RK.UNIVERSITY

INTERNATIONAL CONFERENCE ON **RESEARCH, ENTREPRENEURSHIP, & PEDAGOGY:** INDIA'S CENTURY IN THE MAKING



JAN. 5 & JAN. 6, 2016

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RKUNIVERSITY

INTERNATIONAL CONFERENCE ON **RESEARCH, ENTREPRENEURSHIP, & PEDAGOGY:** INDIA'S CENTURY IN THE MAKING



International Conference on Research, Entrepreneurship, and Pedagogy: India's Century in the Making

ISBN: 978-93-5254-061-7

Publication Year: 2016

Publisher: RK University, Rajkot, Gujarat (India)

Publication Type: Online Conference Proceedings

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ABOUT ICRE 2016

RK University had recently organized its first *International Conference on Research & Entrepreneurship* (ICRE 2016) in the sprawling RKU campus on Jan. 5th & Jan. 6th, 2016. Nobel Laureate Dr. Alan J. Heeger and two experts from the renowned Massachusetts Institute of Technology (Alok R. Singh, Sloan Fellow, MIT; USA and Katherine Taylor, CEO, Khethworks; USA) delivered their pre-recorded video addresses during this conference. TOTO Express Co-Founder, Gary Mao (Former Social Innovation Fellow, Stanford University), William Ruiz (Marketing Manager, PPM Works, Inc., Massachusetts; USA), Dr. Anamik Shah (Vice-Chancellor, Gujarat Vidyapith), Dr. Deepak Kumar (Director, Capri Institute of Manual Therapy), Dr. Nagesh Nanda (Director, Novel Consultants), Dr. Shesheer Kumar (CEO/Founding Director, RAS Lifesciences), and Dr. Suvarn Kulkarni (Associate Professor, Department of Chemistry, IIT Bombay) also shared their insightful thoughts with the ICRE 2016 audience. The conference was attended by several other dignitaries and by 300+ delegates from all over India and abroad. More than 200 research papers/posters were published/exhibited during this conference.

ICRE 2016 focused on the following sub-themes: Current Concepts in Physiotherapy, Management Research for Start-Ups and Family Businesses, Latest Trends in Science, Latest Trends in Pharmacy, Innovative Materials, Methods, and Mechanisms in Engineering, and Innovations in Pedagogy. This International Conference received partnership from the Indian Society for Technical Education (ISTE), the Institution of Engineers, India (IE), and from the local body (RMA) of the All India Management Association (AIMA). Inderscience Publishers (with offices based in Switzerland and UK) will be publishing the extended versions of select conference papers from the field of science, pharmacy, medicine, technology, and management in a special issue of the *International Journal of Business And Globalisation (IJBG). IJBG* is a peer-reviewed journal indexed by Elsevier's "Scopus," arguably the world's largest database of high quality peer-reviewed articles. ICRE 2016 got significant coverage from the mainstream and local media. Expert talks were delivered by Dr. Harihara Prakash, Dr. N. Ramesh, Mr. Nagesh Nanda, Dr. Balaganapathy, Dr. Amit Sharma, Dr. Hitesh Shukla, Dr. Reji Samuel, Dr. Priyanshu Rathod, Dr. Anjali Bhise, and by Dr. Mehul Jadav during the parallel sessions. The ICRE 2016 Committee also gave away awards for the *Best Papers* and *Best Posters* in each category. *Special certificates of recognition* were awarded to several faculty members who won prizes during RKU's *Research Competition* last year. ICRE 2016 Organizer, Dr. Ranjit Goswami (Vice-Chancellor, RK University) delivered a welcome speech and Dr. Nikhil A. Gokhale (ICRE 2016 Coordinator) offered a vote of thanks to everyone on behalf of the ICRE 2016 Committee. The ICRE 2016 Committee thanks everyone who contributed to RKU's First International Conference and looks forward to hosting more such events in the future.

ICRE 2016 COMMITTEE MEMBERS

Dr. Ranjit Goswami (Organizer), Dr. Nikhil A. Gokhale (Coordinator), Dr. Ajit Shukla, Dr. Devang Pandya, Dr. Dharmesh Raval, Prof. Krunal Vaghela, Prof. Mayank Pandya, Prof. Nilesh Kalani, Dr. Priyanshu Rathod, Dr. Amit Lathigara (Academic Communications), Mr. Yash Chawla (Media and PR), and Research Facilitators.

ICRE 2016 ADVISORY COMMITTEE

Dr. Huanchen Wang (National Institutes of Health, USA), Dr. Nithin V. George (IIT Gandhinagar), Dr. Shivani Agarwal (Northwestern University, USA), Dr. Uttama Lahiri (IIT Gandhinagar), Dr. Shivangi Agarwal (Northwestern University, USA), Dr. Vasudha Nair (National Institutes of Health, USA), Dr. C. H. Vithlani (GEC Rajkot), Dr. P. H. Parsania (Saurashtra University, Rajkot), Dr. Hiren H. Joshi (Saurashtra University, Rajkot), Dr. K. R. Desai (Uka Tarsadia University, Surat), Dr. Anamik Shah (VC, Gujarat Vidyapith, Ahmedabad), Dr. Ketan Kotecha (VC, Parul University, Vadodara), Dr. Subhash Mandal (Jadavpur University, Kolkata), Mr. Apoorve Mohan (Northeastern University, USA), and Dr. Nagasamy Venkatesh (JSS College of Pharmacy, Ooty).

KEYNOTE SPEAKERS



Nobel Laureate Dr. Alan J. Heeger delivered a brief pre-recorded video address to the ICRE 2016 attendees on Jan. 5, 2016. Dr. Alan Heeger received the Nobel Prize in Chemistry in the year 2000 for his pioneering research on conductive polymers. He is currently directing research work on semi-conducting polymers and plastic solar cells at the University of California, Santa Barbara (USA). He is a member of the U.S. National Academy of Science and the U.S. National Academy of Engineering.

The Nobel Prize in Chemistry was jointly awarded to Dr. Alan J. Heeger, Dr. Alan G. MacDiarmid, and Dr. Hideki Shirakawa (in the year 2000) for the discovery and development of conductive polymers (http://www.nobelprize.org/nobel_prizes/chemistry/laureates/2000/). Widely recognized for his groundbreaking research in the field of semi-conducting and metallic polymers, Nobel Laureate Dr. Heeger is also the recipient of numerous other prestigious awards. Dr. Heeger has over 900 publications in scientific journals and more than 50 patents to his credit. As a passionate entrepreneur, he founded UNIAX Corporation back in 1990. He is the Co-Founder/Chairman of CBrite Inc. (makers of next generation flat panel displays), Co-founder/Vice-Chairman of Cynvenio (micro-fluidics for cell sorting), and Co-Founder/Vice-Chairman of Cytomx Therapeutics (novel technology for targeted drug delivery). Dr. Alan Heeger has participated in the production of several Broadway plays including *In the Heights*, *West Side Story*, and *Cinderella.*



Ms. Katie Taylor (CEO, Khethworks) earned her Master's degree in Mechanical Engineering from the Massachusetts Institute of Technology (USA) in 2015. She obtained a B.S. in Mechanical Engineering from the California Institute of Technology and a B.A. in Physics from Pomona College. Katie is the co-founder and CEO of Khethworks, a Tata Center spinout company, which builds reliable, solar-powered irrigation systems that enable their customers to farm through all three seasons. She was a Tata Fellow at MIT (USA) between 2013 and 2015. Katie delivered her pre-recorded video address during ICRE 2016.



Mr. Alok R. Singh is a Sloan Fellow at the Massachusetts Institute of Technology (USA). He has 15+ years of global experience in sales, marketing, operations, analytics, and strategic program management in the IT industry. Between 2013 and

2015, Alok was the Regional Sales Director (South Asia) at Dell (listed as one of the Top 60 Fortune 500 companies in 2014). He has been associated with Dell for more than 12 years, in various global and regional leadership roles for businesses across AMERICAS, EMEA, and APJ. Alok specializes in global leadership, business strategy and execution, program management, business intelligence, and business process improvement. During ICRE 2016, Alok e-shared his views on the importance of entrepreneurial initiatives in the area of science and technology.



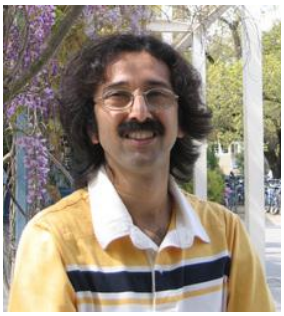
Gary Mao is the Co-Founder of TOTO Express and a former Social Innovation Fellow of Stanford University. Armed with a Bachelor's Degree from the University of California, Berkeley and with a Master's Degree in Business Administration from the Stanford Graduate School of Business, Gary is now creating the right infrastructure to connect rural artists with the emerging markets. At TOTO Express, he has been instrumental in securing partnership with a high-end apparel company, thereby creating a royalty structure for *Gond* artists from remote Indian villages. Gary has also worked as a Business Analyst for McKinsey and Company between 2009 and 2011.



Dr. Anamik Shah (Vice-Chancellor, Gujarat Vidyapith) has been instrumental in creating a Rs. 13.2 crore National Facility for Drug Discovery (Center of Excellence) in the Saurashtra University campus. He has received several grants from government funding agencies and has been successful in establishing multiple industrial collaborations. Dr. Shah has carried out high impact research work in several areas of national and global importance. He has directed various research projects involving reputed research institutes such as the U.S. National Institutes of Health (NIH), the Department of Atomic Energy, and the National Medicinal Plant Board (New Delhi). He has published several well-cited research articles in the area of medicinal chemistry and drug discovery. Dr. Shah has also received an award from the International Scientific Partnership Foundation in Russia.



Mr. Nagesh Nanda has more than 20 years of industrial experience and has held managerial positions in top pharmaceutical companies in India (Torrent, AFDIL) and abroad (Chemo Pharma, Spain). His firm *Novel Consultants* offers services in R&D management, formulation development, IVIVC, QbD, R&D centre establishment, clinical research, acquisition, GMP, GLP &, GCP and in resolving critical regulatory queries related to formulations.



Dr. Suvarn Kulkarni (Associate Professor, IIT Bombay) is a distinguished researcher in the field of organic chemistry and has successfully published research articles in *Nature*, *Nature Protocols*, and many other world-renowned journals. After obtaining his Ph.D. from the University of Pune in the year 2001, Dr. Kulkarni visited Taiwan and the United States to conduct post-doctoral research work at Academia Sinica and at UC Davis. He has been actively involved in publishing and patenting his work in the field of organic synthesis. His current research interests include devising newer ways for the efficient chemical synthesis of complex glycoconjugates implicated in various infectious diseases as well as in cancer, and the development of carbohydrate-based vaccines.



Dr. Shesheer Kumar is the CEO/Founding Director of RAS Lifesciences and an accomplished entrepreneur in the area of biotechnology and molecular diagnostics. Dr. Kumar worked as a scientist at Shantha Biotechnics, Hyderabad, prior to the launch of RAS Life Sciences in March 2008. He has been successful in developing recombinant clones for the production of Human Insulin, Streptokinase, EPO, Interferon, etc. Dr. Kumar has also been actively involved in developing molecular biology kits and has successfully cloned, expressed, and purified HCV proteins. He has published several research articles in peer-reviewed national and international journals.



Dr. Deepak Kumar is a successful healthcare entrepreneur and the Director of the Capri Institute of Manual Therapy. He has obtained special training in the area of manipulative physiotherapy from Australia's Curtin University. He has also been awarded a fellowship for his exemplary service by the Indian Association of Physiotherapists. Dr. Kumar has trained more than 10,000 students during the last 11 years and has successfully treated over 85,000 patients during the last 22 years of his career. He also has 12 inventions (in the area of manual therapy, electrotherapy, and exercise therapy) to his credit. Dr. Kumar's innovative techniques have been duly recognized by Brian Mulligan, the founder of the Mulligan Concept.

EXPERT SPEAKERS (PHYSIOTHERAPY TRACK)



Dr. M. Balaganapathy (PT) completed his Master of Physiotherapy in Orthopaedics and P.G. Diploma in Emergency & Trauma Care and is currently pursuing his PhD from Gujarat University. He is the Principal of the Ashok & Rita Patel Institute of Physiotherapy (affiliated to CHARUSAT University, Anand, Gujarat). He has more than 12 years of professional experience in physiotherapy. He is a member of IAP, ISE (Indian Society of Ergonomics), RTTF, and Handicap International. He has published papers in international journals and conference proceedings and presented papers in various national and international conferences. He was also an entrepreneur a few years ago and his area of expertise in research is primarily focused on low back pain, physiotherapy education, and ergonomics. He was instrumental in introducing the course "Ergonomics" in the undergraduate curriculum of BPT in Charotar University of Science & Technology (the first of its kind in India). He has spoken at various Ergonomics workshops and has worked as an expert for introducing curriculum changes at the undergraduate level. He has also been a jury member for various academic events.



Dr. Mehul Jadav is a Director at the Sai Global Therapy Fitness Solution, Vadodara, Gujarat. He has more than 12 years of experience in the field of physiotherapy. He is a co-opted EC member of the Central Executive Council of the Indian Association of Physiotherapy (IAP). He received the Physio Excellence Award during the 4th INCPT conference at New Delhi and an award for his significant contribution to the field of physiotherapy during the national conference of IAP at Indore. He has presented papers in various national conferences and he has been invited as a resource person by various institutes.



Dr. Reji K. Samuel is a Professor and the Principal of the C. U. Shah Physiotherapy College, Surendranagar, Gujarat. He has more than 14 years of professional experience in physiotherapy, as an academician and clinician. He has organized various camps and has imparted services in the villages located near Surendranagar, with the noble mission to serve the underprivileged rural people. He has been invited as a speaker to various conferences and he is also a member of the UK-based Health & Care Professions Council.



Dr. **R. Harihara Prakash** is a Professor and the Principal of the K. M. Patel Institute of Physiotherapy, Karamsad. He has 18 years of professional experience in clinical as well as academic areas of physiotherapy. He holds a Doctorate in Physical Therapy from the National University of Medical Science, Spain. He was awarded the "RASHTRIYA VIDHYA SARASWATHI PURASKAR AWARD" for his excellent contribution to academics. He is the former Dean, Faculty of Physiotherapy, Baba Farid University of Health Sciences, Punjab. Dr. Prakash serves as an editor and peer reviewer for various international journals. He is also a NAAC panel member. He has obtained certification in the field of neurology, osteopathy, and manual therapy from various countries and has published and presented research papers in various national and international conferences**.** He is an eminent speaker and a renowned academician.

ICRE 2016 Schedule (Jan. 5th & Jan. 6th, 2016)

International Conference on Research & Entrepreneurship: India's Century in the Making

Table 1. Program Schedule for Jan. 5th, 2016.

Sr. No.	Event	Time	Venue
1.	**ICRE 2016 Registration**	8:00 a.m.–9:30 a.m.	School of Engineering Entrance
2.	**Breakfast**	8:00 a.m.–9:30 a.m.	School of Physiotherapy Hall
3.	**ICRE 2016 Inauguration** **1. *Ceremonial Lamp Lighting*** ***(दीप प्रज्वलन;*** Mr. Denish Patel (Exec. VP, RKU), Dr. Ranjit Goswami (VC, RKU/ICRE 2016 Organizer), Mr. Shivlal Ramani (Registrar, RKU), Mr. Gary Mao (Co-Founder, TOTO Express; USA), Dr. Anamik Shah (VC, Gujarat Vidyapith), Mr. Deepak Kumar (Dir., Capri Institute of Manual Therapy), Mr. Nagesh Nanda (Dir., Novel Consultants), and Dr. K. S. Murthy (VC, ITM Voc. Univ., Baroda). **2. Welcome Messages** (Mr. Denish Patel (Exec. VP, RKU)/Mr. Mohit Patel (VP, RKU), Dr. Ranjit Goswami (VC, RKU/ICRE 2016 Organizer), & Dr. Nikhil A. Gokhale, ICRE 2016 Coordinator). **3. Nobel Laureate Dr. Alan J. Heeger's Keynote Video Address** on Research & Entrepreneurship, **Keynote Addresses** on Research/Entrepreneurship (Mr. Gary Mao (Co-Founder, TOTO Express; Social Innovation Fellow, Stanford University; USA), Dr. Anamik Shah (VC, Gujarat Vidyapith), Dr. Deepak Kumar (Dir., Capri Institute of Manual Therapy), Mr. Nagesh Nanda (Dir., Novel Consultants); Pre-Recorded Messages from Mr. Alok R. Singh (MIT, USA) and Ms. Katherine Taylor (CEO, Khethworks; USA).	9:30 a.m.–9:35 a.m. 9:35 a.m.–9:50 a.m. 9:50 a.m.–10:50 a.m.	School of Engineering Seminar Hall
4.	Participants Disperse to Various Seminar Halls for the Parallel Sessions	10:50 a.m.–11:00 a.m.	--
5.	**Parallel Track Sessions/Invited Talks** (Management, Medicine, Pedagogy, Pharmacy, Science, and Technology)	11:00 a.m.–Noon	Various seminar halls across RKU

	Management Track – Mr. Gary Mao ("Entrepreneurship")	11:00 a.m.–Noon	School of Comp. Sci. Seminar Hall
	Medicine Track – Chairperson/Expert: 1. Dr. Deepak Kumar, MPT., PhD., FIAP, (CEO, Capri Institute of Manual Therapy, Delhi). 2. Dr. Harihara Prakash, MPT., Ph.D. (Principal, K. M. Patel Institute of Physiotherapy, Karamsad). Topic: "Evidence-Based Practice In Physiotherapy." 3. Dr. N. Ramesh, MPT, MBA (Assistant Professor, School Of Physiotherapy, RK University). Topic: "Technological Advancement In Physiotherapy." Paper Presentations: 1. Dr. Maitrey Pandya, MPT; Topic: "Neurophysiological Changes in Person with Insulin-Dependent and Non-Insulin-Dependent Diabetes Mellitus." 2. Dr. Shweta Rakholia, MPT; Topic: "Comparison of Effect of Proprioceptive Neuromuscular Facilitation Technique versus Motor Relearning Programme on Improvement of Trunk Control in Hemiparetic Patients." 3. Dr. Hetal Patel, MPT; Topic: "To assess the effect of physiotherapy in modified radical mastectomy patients."	11:30 a.m.–12:30 p.m.	Auditorium, School of Diploma Studies
	Science & Pharmacy Track – Invited Talks Mr. Nagesh Nanda: "Research and Entrepreneurship: My Journey."	11:30 a.m.–1:00 p.m.	School of Management Seminar Hall
	Technology Track – Oral Presentations (EE: 215, 163, 165, 42, 155; EE Session Chairs: Dr. Dineshkumar & Prof. Dhaval Pipalia)	11:00 a.m.–12:30 p.m.	F101 (School of Comp. Sci.)
	Technology Track – Oral Presentations (CV: 132, 272, 271, 58)	11:00 a.m.–12:30 p.m.	F103 (School of Comp. Sci.)
	Technology Track – Oral Presentations (CE/IT: 15, 37, 77, 90)	11:00 a.m.–12:30 p.m.	School of Comp. Sci.
	Technology Track - Poster Presentations (CV: 243, 204; Physics: 25, 80, 96, 138, 209)	1:00 p.m.–5:00 p.m.	School of Comp. Sci.
6.	Lunch Break	Noon–2:00 p.m.	School of Physiotherapy Hall
7.	**Parallel Track Sessions/Invited Talks** (Management, Medicine, Pedagogy, Pharmacy,	1:00 p.m.–3:00 p.m.	Various seminar halls across RKU

	Science, and Technology)		
	Management Track – Oral Presentations ("Entrepreneurship & Innovation") Submission nos. 18, 40, 82, 146, 157, 236, 254, 148, 193, 149, 26, 160, 14, 67, and 89	1:00 p.m.–3:00 p.m.	G2 (School of Comp. Sci.)
	Medicine Track – Chairperson/Expert: 1. Dr. Balaganapathy, MPT (PhD) (Principal, Ashok & Rita Patel institute of Physiotherapy), Anand; Topic: "Role of physiotherapist in public health." 2. Dr. Amit Sharma, MPT (Deputy Director, School of Physiotherapy; RK University); Topic: "Home health care in India: Building an entrepreneur model." Paper Presentations: 1. Dr. Ankur Khant, MPT (PhD); Topic: "Efficacy Of Muscle Energy Technique For Pectoralis Minor In patient With Frozen Shoulder." 2. Dr. Priyanshu Rathod, PT, PhD; Topic: "Computer Based Developmental Paediatric Screening (CBDPS): Evolving Healthcare Entrepreneurship." 3. Dr. Ankur Parekh, MPT; Topic: "Efficacy Of Plyometric Training In Football Players." 4. Dr. Dheli Kadachha, MPT; Topic: "Effects Of Yoga On Balance In Geriatric Population." 5. Dr. N. Ramesh, MPT; Topic: "Effectiveness of Additional Backward Walking Training to Improve Balance in Stroke Patients." 6. Dr. Keny Macwan, (MPT); Topic: "Effect of slow breathing and progressive muscle relaxation technique in persons with essential hypertension - A randomized controlled trial." 7. Dr. Yagna Yadav, (MPT); Topic: "A Study to evaluate the Effectiveness Of Constraint Induced Movement Therapy on affected Upper Extremity functions in Children suffering from Hemiplegic Cerebral Palsy." 8. Dr. Krupa Raithatha, MPT; Topic: "Effectiveness of scapular muscle strengthening in management of lateral epicondylalgia."	1:30 p.m.–3:30 p.m.	Auditorium, School of Diploma Studies
	Science & Pharmacy Track – Invited Talks	2:00 p.m –3:15 p.m.	School of Management Seminar Hall

	Mr. Shesheer Kumar: "Research and Entrepreneurship: My Journey as a Biotechnologist." Dr. Suvarn Kulkarni: "Research in Chemistry: My Journey."		
	Technology Track – Oral Presentations (EE: 22, 184, 191, 69, 151, 13, 19, 154, 232 (EE Session Chairs: Dr. Dineshkumar & Prof. Dhaval Pipalia)	1:00 p.m.–3:15 p.m.	F101 (School of Comp. Sci.)
	Technology Track – Oral Presentations (ME: 98, 139, 105, 21, 36, 213, 140, 173, 24, 186, 56, 212, 104, 23, 198, 158, 102)	1:00 p.m.–3:15 p.m.	F103 (School of Comp. Sci.)
	Technology Track – Oral Presentations (CE & IT: 38, 54, 55, 65, 72, 75, 99, 181; 113, 121, 128, 167, 177, 200, 216, 223)	3:00 p.m.–5:00 p.m.	G1 & F102 (School of Comp. Sci.)
	Technology Track – Poster Presentations: (ME: 176, 119, 190, 156, 16, 180, 197, 282, 201, 10, 84	11:00 a.m. to Noon and 1:00 p.m.–3:00 p.m.	School of Comp. Sci
	Technology Track – Poster Presentations: (CE&IT: 59, 60, 91, 94, 100, 101, 111, 114, 118, 135, 142, 211, 283)	1:00 p.m.–5:00 p.m.	School of Comp. Sci
	Tea & Networking	3:00 p.m.–3:30 p.m.	Across RKU
8.	Local Bus Tour (Limited Rides Available on a First-Come, First-Served Basis.)	3:15 p.m.–6:00 p.m.	*Ramakrishna Ashrama*

ICRE 2016 Program Schedule (Jan. 5th & Jan. 6th, 2016)

International Conference on Research & Entrepreneurship: India's Century in the Making

Table 2. Program Schedule for Jan. 6th, 2016.

Sr. No.	Event	Time	Venue
1.	**ICRE 2016 Late Registration**	7:30 a.m.–8:30 a.m.	School of Engineering Entrance
2.	**Breakfast**	7:30 a.m.–8:30 a.m.	School of Physiotherapy Hall
3.	**Parallel Track Sessions/Invited Talks** (Management, Medicine, Pedagogy, Pharmacy, Science, and Technology)	8:30 a.m.–Noon	Various seminar halls across RKU
	Management Track – Dr. Hitesh Shukla ("Leading and Managing Family Businesses")	8:30 a.m.–9:45 a.m.	School of Comp. Sci. Seminar Hall
	Management Track – Oral Presentations ("Business & Management") Submission nos. 52, 68, 87, 115, 120, 144, 168, 183, 185, 219, 242, 258, 287, 302	10:00 a.m.–Noon	G2 (School of Comp. Sci.)
	Medicine Track - Chairperson/Expert: 1. Dr. Reji K Samuel, MPT (Principal, C.U. Shah College of Physiotherapy, Surendranagar); Topic: "Continuing professional development in physiotherapy." 2. Dr. Priyanshu V. Rathod, MPT, PhD (Director, School of Physiotherapy, RK University); Topic: "Physiotherapist: Health Care Entrepreneur." Paper Presentations: 1. Dr. Nalina Gupta, MPT, (PhD); Topic: "Patients' Expectations: A Necessary Focus Of Care In Physiotherapy." 2. Dr. Rozy Patel, MPT; Topic: "Women's Health In India." 3. Dr. Navjoyt Trivedi, MPT, (PhD); Topic: "Physiotherapy In Disaster Management: Preliminary Investigation." 4. Dr. Vaibhavi Ved, MPT; Topic: "A study to evaluate the efficacy of body's own weight	9:00 a.m.–10:30 a.m.	Auditorium, School of Diploma Studies

	training program on lower limb strength & function in elderly individuals." 5. Mr. Mohindrasingh Varma, (BPT); Topic: "Abdominal muscle recruitment in various degree of bilateral SLR."		
	Medicine Track – Chairperson/Expert: 1. Dr. Anjali Bhise, MPT, (PhD) Principal, Government Physiotherapy College, Ahmadabad; Topic: "Current Concepts in health promotion and fitness." 2. Dr. Mehul Jadav, MPT, (PhD) Director, Sai Global Fitness Academy, Vadodara; Topic: "Recent advances in shoulder rehabilitation". Paper Presentations: 1. Dr. Shanawaz Syed, MPT, (PhD); Topic: "A Validated 11-Item Multi-Component Exercise Program On Dizziness Caused By Benign Paroxysmal Positional Vertigo Individuals: A Pilot Study." 2. Dr. Amit Sharma, MPT; Topic: "Relationship Between Functional Capacity And Functional Impairments Of Upper Extremity In Female Beedi Workers." 3. Dr. Vaitianande, MPT, (PhD); Topic: "A Study to Find the Effectiveness of Mulligan's Mobilization Along With Conservative Exercise in Improving Pain and Functional Activity in Patients with Knee Osteoarthritis .A Randomized Controlled Trail." 4. Dr. Chirag Parmar, (MPT); Topic: "A Study to Isolate Radial Tunnel Syndrome from Lateral Elbow Pain Patients". 5. Dr. Vrunda Paghi, (MPT); Topic: "Using the Pediatric balance scale to determined balance in 5 to 6 year old children."	9:00 a.m–Noon	Auditorium, School of Diploma Studies
	Science & Pharmacy Tracks– Oral Presentations (Submission nos. 9, 30, 133, 152, 172, 182, 252, 253, 49, 92, 126, 214, 229, 257, 279, 281, 194, 206, 222, 230, 248, 300)	8:30 a.m.–11:30a.m.	School of Computer Science First Floor Rooms 102 (Pharmaceutics), 103 (Chemistry), 104 (Miscellaneous).

	Science & Pharmacy Tracks – Poster Presentations (Submission nos. 12, 88, 95, 136, 145, 147, 189, 231, 235, 240, 246, 278, 299, 6, 73, 83, 141, 164, 175, 255, 275, 277, 280, 296, 298, 314A, 47, 48, 169, 174, 178, 187, 205, 226, 244, 250, 263, 267, 269, 270, 288)	11:30am –2:00 p.m.	School of Comp. Sci
	Technology Track – Oral Presentations (EC: 125, 196, 268, 17, 228, 27, 108, 44, 86, 161, 20 (EC Session Chairs: Dr. Divyang D. Vyas & Prof. Nilesh Kalani)	9:00 a.m.–11:30 a.m.	F101 (School of Comp. Sci.)
	Pedagogy Track – Oral Presentations (8, 26, 130, 131)	11:00 a.m.–12:30 p.m.	S201 (School of Comp. Sci.)
4.	Lunch Break	Noon–1:00 p.m.	School of Physiotherapy Hall
5.	**Special Talks** by Mr. Gary Mao (Co-Founder, TOTO Express; Social Innovation Fellow, Stanford University (California); USA) and by Mr. William Ruiz (Founder, Food ME; Marketing Manager, PPM Works Inc. (Massachusetts); USA)	1:00 p.m.–1:30 p.m.	School of Engineering Seminar Hall
6.	Valedictory Speeches (Dr. Ranjit Goswami & RKU School Directors), Vote of Thanks (Dr. Nikhil A. Gokhale), and the Awards Ceremony (Best Paper Awards/Best Poster Awards).	1:30 p.m.–2:15 p.m.	School of Engineering Seminar Hall
7.	Certificate Collection at the Registration Desk	2:15–3:00 p.m.	School of Engineering Entrance

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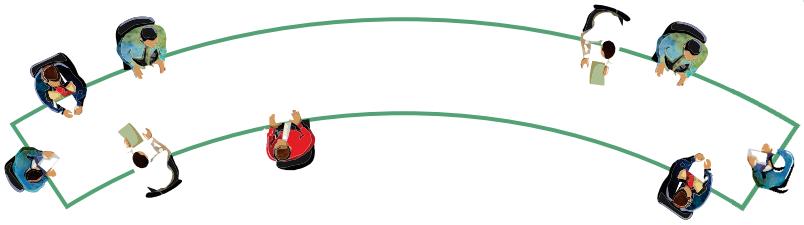
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SECTION NO. 1: MANAGEMENT RESEARCH

The prosperity of any economy largely depends on the amount of success achieved by promoting entrepreneurship, innovation, and the use of newer technologies in businesses. Globalization has brought upon many challenges that can be addressed by promoting entrepreneurship and by supporting start up eco-systems. Small family-owned businesses are initially managed by a few family members. However, as the business grows in size, more and more people start contributing, and many of these contributors/collaborators are not necessarily members of the family. India in general and Gujarat in particular have a rich heritage of small- and medium-sized family businesses, wherein the families and their corresponding businesses have been naturally intertwined for several generations. This section primarily focuses on the various aspects of business management and entrepreneurship.





MANAGEMENT RESEARCH FOR START-UPS AND FAMILY BUSINESSES

ISBN: 978-93-5254-061-7 | www.rkuconference.org | JAN. 5 & JAN. 6, 2016

Proceedings of R K University's First International Conference on Research and Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

(Proceedings available for download at rku.ac.in/icre)

R K University's First International Conference on Research & Entrepreneurship (ICRE 2016)

Exploring of the factors that help sustain family managed businesses in the state of Gujarat

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ABSTRACT

The aim of the study was to identify the factors that help in sustaining the family managed businesses in the state of Gujarat. The methodology used was interpretative case study to explore more about the factors from different perspectives and explore more about the cases through individuals. The findings of the study reveal that values that businesses hold play a demanding role in survival of it as well as the support needed around the crucial times. The implications of the study state that for family managed business the more pivotal crux lies as to when and how the succession planning takes place along with the handling the reins of the business. The limitation of the study was the time duration provided for the study for the exploration of factors.

SUMMARY

The factors of survival of family managed businesses in the state of Gujarat has been explored and explained in the research with the case study methodology.

Keywords:

Family managed business, interpretative case study, factors for survival of family managed businesses.

INTRODUCTION

Family businesses in the country have shown miracles in sustaining themselves and the economy of the country. It has been defined as the business which has the ownership as well as the management by the member(s) of the family. There are variety of factors that have affected and challenged the survival and growth of the business over the years and the research of them has occurred through both, qualitative and quantitative studies.

Survival is said to be a relative term. But according to Darwin (), survival came from evolution and it is a way of describing the mechanism of natural selection. If one is competitive then its survival can continue in successive generations. There are many challenges that one faces while continuing for the survival and there are various factors that create these challenges. Here the aim is to research the factors affecting their survival over the generations through qualitative study.

Case study is a methodology that falls under the category of qualitative research and is referred to as an approach towards addressing issues through the participants' viewpoint. The insights help in the exploration of the inside world and hence it leads to a formation of theory that would rationalize the conclusions obtained.

The factors that could be addressed could help in identifying the survival strategies of the family managed businesses and the challenges that they faced while they managed to survive for such a long duration of time with the help of case study analysis.

Objectives

The objective of the research was to identify the meaning of family managed business from the family managed businesses; to explore the factors that had helped and have been helping in the survival of family managed business and also to identify the sentiments attached with such businesses of the family:

Data Required	Data Source	Data Collection Tool
Meaning of family managed business	Review of Literature; Entrepreneurs	Journals, articles, references, books. Interviews (Case study analysis).
Factors that helped in survival of family managed business	Entrepreneurs; Review of Literature	Case Study analysis: Interview. Journals, articles, references, books
Sentiment of such businesses in their respective fields	Entrepreneurs; Personal experience	Interviews; Personal experience and its reflection

REVIEW OF LITERATURE

Family managed businesses:

Colli*(7)* states that the history of the definition of family managed business came into existence in 1890's. According to traditional economists, family firms are small or medium sized, have slow growth, depend upon flat structure and internal succession patterns and mostlyself-financed.

Family managed business has been a tradition in the country since long. For understanding family managed business there are two concepts at stake- 'family' and 'business.' To know them both the differences in their individual characteristics should be known as per Schwass*(31)*:

Sr. No.	Characteristics of a family	Characteristics of a business
1.	Formed when group of individuals combine themselves not by choice	Formed on the willingness level of individuals forming a group together.
2.	The involvement is for life	The involvement is for a timeline with a job description
3.	The support is for as long as the member needs- financial and moral	The compensation to an employee is for as long as his/her involvement is with the organization
4.	Governed by emotions	Governed by rationality

5.	More inclined towards socialistic approach	More inclined towards capitalist approach

According to McKinsey & Company *(26)* there are five dimensions to a family managed business:

- ✓ **Business and portfolio governance-** The form of ownership is often overlooked. With expansion come the challenges of performance and governance. To achieve success, there has to be an intertwining of business performance and family commitments to carry on.
- ✓ **Family-** Family is a crucial factor for survival of family managed businesses. The business can cease to exist because of many reasons which could include conflicts for money, poor management, etc. Family institutions aid in making continued ownership by nurturing family values and giving the next generation a sense of motivation to contribute to the society.
- ✓ **Ownership-** Maintaining control over the business is what makes the difference for family managed business. Hence, most of them are private held organizations. They restrict the trading of shares.
- ✓ **Wealth management-** These businesses need strong capabilities for managing wealth and with diversifying risk they preserve harmony for management of wealth.
- ✓ **Foundations-** Creating meaningful jobs, promoting values, sharing wealth are important charities that family businesses do. The five dimensions should work in synchronization for the success of family managed businesses.

Some major family managed business has influenced the economy of the country in a deeper way and have managed to survive for a long period of time without having a setback. The literature has defined family business as "A Family-owned business is one that is owned and managed (that is controlled) by one or more family members" as per Hunt and Handler *(20)*.

The following are some of the characteristics noticed in the family- managed business in India according to Bhattarcharyya*(2)*:

1. Family relationship is the most important factor in determining the position in business about a person.
2. The Board of Directors includes family members who are not contributing or are involved in the business.
3. Few castes' have been very successful and thus are synonym with family-owned businesses in India The Aggarwals and Guptas in the North, the Chettiars in the South, the Parsees, Gujarati Jains and Banias, Muslim Khojas and Memons in the West, and Marwaris in the East. Of these, the Marwaris have been the most successful.
4. Every caste has a dominant culture which is to be followed in the business by all the members.
5. Those members of the family and/or extended family and relatives who have a very strong sense of loyalty to the family are translated to be loyal towards business too.
6. The single minded dedication of the CEO and the family ensures that family-owned business survives through the toughest times.
7. Sons and male members are more likely to hold higher positions and succeed the CEO. Role of women is that of facilitator to the male members and as the mother figure to the family and employees.

The Indian Perspective for family managed businesses:

According to the survey held by PwC in 2013-14*(27)*, the Indian perspective on family managed businesses with certain facts and figures are as follows:

- 78% of the Indian family enterprises support various community initiatives
- 78% of the Indian family enterprises go out of their way to help and retain their staff in bad conditions
- 80% of the Indian family enterprises support employment in areas of their operation
- 90% of the Indian family enterprises are confident of achieving their predicted growth

Family managed businesses have long been a part of India and hence their unique challenges for survival and growth are:

- Diverse opinions of family members for business, issues, management etc.
- Succession planning for the next generation
- Difficulties the younger generation may face in proving themselves to the former generation

- Managing the differed views between older and newer generation
- External staff (hiring) that may prove to be an advancement for business as well as the individuals
- Capital requirements for business

Factors for the survival of family managed businesses:

The literature points out that the following factors may be the reasons that affect the survival and growth of family businesses-

1. ***Succession planning****(42, 35, 36, 21, 27, 18, 33, 24, 16,* 6*,* 5*,* 8*, 25)*
2. ***Environmental factors****(41)*
3. ***Political factors****(41)*
4. ***Relation of the family****(35, 23, 21,33,*6*)*
5. ***Control of the business****(23, 21, 18, 33,* 5*)*

Research Methodology and case study research:

According to Rajashekhar*(30)* Research methodology is a structured way of solving the issues that arise in the research.

Stake *(34)* states that Case studies, in which the researcher explores in depth a program, an event, an activity, a process, or one or more individuals. The case(s) are bounded by time and activity, and researchers collect detailed information using a variety of data collection procedures over a sustained period of time.

Key Characteristics of Case Studies according to Benbasat*(*1*)*:

- Phenomenon is examined in a natural setting.
- Data are collected by multiple means.
- One or few entities (person, group, or organization) are examined.
- The complexity of the unit is studied intensively.
- Case studies are more suitable for the exploration, classification and hypothesis development stages of the knowledge building process; the investigator should have receptive attitude towards exploration.
- No experimental controls or manipulation are involved.
- The investigator may not specify the set of independent and dependent variables in advance.
- The results derived depend heavily on the integrative powers of the investigator.
- Change in site selection and data collection methods could take place as the investigator develops new hypotheses.
- Case research is useful in the study of "why" and "how" questions because these deal with operational links to be traced over time rather than with frequency or incidence.
- The focus is on contemporary events.

According to Creswell *(10)* there are three variations in case studies: single instrument case study, collective case study and intrinsic case study. In single instrument case study the researcher focuses on an issue or concern and then selects one bounded case to illustrate the issue. In collective case study, the one issue or concern is again selected but the inquirer selects multiple case studies to illustrate the issue and in the intrinsic case study the focus remains on the case itself because it represents unusual or unique situations.

The approach towards the case study is more crucial in the research. The following table gives the details about the same:

Approach	**Characteristics**	**Criticisms**	**Key references**
Critical	Involves questioning one's own assumptions taking into account the wider	It can possibly neglect other factors by focusing	*(19,* 3*, 12, 13)*

	political and social environment. Interprets the limiting conditions in relation to power and control that are thought to influence behavior.	only on power relationships and may give the researcher a position that is too privileged.	*(*4*)*
Interpretative	Involves understanding meanings/contexts and processes as perceived from different perspectives, trying to understand individual and shared social meanings. Focus is on theory building.	Often difficult to explain unintended consequences and for neglecting surrounding historical contexts	*(34, 12)*
Positivist	Involves establishing which variables one wishes to study in advance and seeing whether they fit in with the findings. Focus is often on testing and refining theory on the basis of case study findings.	It does not take into account the role of the researcher in influencing findings.	*(37, 38, 40, 32)*

Validity of the study:

One of the criteria for judging the quality of research designs is by establishing the domain to which a study's finding can be generalized, and thereby addressing the issue of external validity. According to Yin *(***Error! Unknown switch argument.***)*, external validity problem has been a major barrier in doing case studies, particularly for single-case study and "critics typically state that single case offer a poor basis for generalizing" (p.36).

MATERIAL AND METHOD

The research study started with exploring the existing family businesses in the area as per convenient sampling. Based on the interviews, the comparison between the existing mentioned factors as per literature and the interviews that explored factors was made.

The objective of the research being exploration, hence, the semi-structured interview format was adopted to have a better apprehension of the process of the family managed businesses. The questions included from the meaning of family managed businesses to the interviewees to the sentiments attached towards the business. The assistance of local language was used for the convenience of interaction of the interview process.The interview was designed in two phases: the first was in the format of interviewing the entrepreneurs currently having the control of the business and the second was the next generation who would take over the business in the near future.

The sample includes the entrepreneurs currently holding the reins of family business as well as the next generation working in the business. Convenient and snowball sampling were employed to collect the data for the research.As it is a case study the successful family managed businesses in the vicinity were the target of the study to help in better understanding of the survival of businesses over the period of time. Thus, 10 such cases that have survived over second and/or third generation and/or fourth generation have been studied.

RESULTS

Description of the sample:

The sample that have been a part of the study is the first, second and third generation entrepreneurs. Their business has been functioning for more than 10 plus years now. Out of the total sample size, the first generation was 30% of the population, the second were 40% and the third were 20% and the fourth were 10%. The sample profile has been created for a better clarification (See table 1). The respondents were introduced with the purpose of research as basic information and the anonymity was guaranteed and whatever information was provided from their end was welcomed.

Data gathering:

The study was conducted in interviewee's work setting and the data was gathered with the help of tools such as recorder and jotting of major points during the interview process and no attempt was made to influence or to control the process. For the follow up, the flexibility in the area of setting was made.The process was about interacting with the current members of the business about the family managed business- meaning and the factors for its survival and threats.

The recorded interaction, the transcribed interviews, the written key words from the interaction, observation towards the participant and the change in views of the two or three working generations were observed were the tools for the research.

Data analysis:

The data analysis was made by the method of controlled comparison. The factors affecting a survival of family managed business given by the literature were compared to the factors mentioned by the participants and the similar factors were decided to be the findings of the study. The following were the steps taken to analyze the given data:

The first step in the analysis was to identify the meaning of family managed business as given by different participants. Hence all the definitions were compared and the common conclusions from it were drawn on defining the meaning. The variations were seen in defining family business according but the platform of treating it as an important and individual identity was derived. Hence, with all these common threads it was decided to draft a common definition of family managed business as a conclusion to this paper.

Defining of the term family managed business may or may not provide justification to the feelings attached to it from the participants but attempting to define it within a parameter was the aim in data analysis. The next step was to enlist the factors given by the participants that have helped them in the survival of the business for over the years. The factors that affected them were listed and compared to the ones as mentioned in literature and a list of the factors helping them for survival have been listed in the findings for the study. Various factors have an impact on the survival and everyday functioning of the business and hence only the frequent occurring factors have been mentioned. For the analysis purpose, the meaning of defining the family managed business as well as exploring the factors that succor in its survival have not varied.

The observation into the participants and data analysis as well shed a light into a new observation stating that new generation are more acquiescent to joining a family business rather than settling on a regular employment and are more accommodating to the norms of the business.

DISCUSSION

The main aim of the paper was to explore the factors that have helped in family businesses to sustain over the period of time since its inception. The interaction with the interviewshas lead towards discovering certain factorswhich have been mentioned in the literature along with their practical application. With the cases that were taken into

consideration, they are from different parts of the state and their businesses have been functioning for over 20+ years now. In the current scenario, some businesses have had their fourth generation involved and hence the sustainability seemed to be incredible.

While studying these cases it shed light on certain concepts which have been discussed here. The factors that have been a major issue for the survival of the family managed businesses as well as the ones that have helped cross the ocean have been in sync with the factors mentioned in the literature (Table 2). The classification of the factors has also been done in order to have a detailed description of them. The first and foremost factor that is observed is the *succession planning* which have been a curse and a boon. With the scale of leadership styles ranging from dormant decision makers to self-governance, succession planning has been an issue. Businesses that have planned it as soon as the next generation enters in the business have taken the position of dormant decision makers and businesses in which the former generation is still on hold of everyday functioning faces major issues with decisions related to business are active leaders. They are the know-it-alls and have hold over petty as well as major decisions related to business. There is third category as well which are at the self-governance level and have reached the self-satisfaction level as per Maslow's hierarchy of needs.

The second most observed factor that affects in making or breaking is the customer relationship with the business as well as the owners of the business. The founders of the business which are the current owners as well as not the current owners have stated the same fact that the business is a sum of its owners; risk-taking but majorly customers and the relationships with them. It has helped businesses to grow and achieve profuse revenues.

The third highest factor in rating that helped in sustaining of the business is the goodwill that the owners and/or founders have established since the existence of business but over the period of time. There is a classification for goodwill as well: business made i.e. the goodwill that the business has to offer in terms of products and owner made i.e. the owner of the business has made with his/her name associated with the business.

The fourth factor is the support from the family that is available through the thick and thin of the business as well as the owner(s) and/or founder(s). The support which could be financial and/or emotional is the one that sometimes helps to make it through the darkest period(s).

Other than these, the factors that help are diversification of either the product or the business; favorable environment and reassuring personnel.Diversification of product has two things: one, expansion of product basket either due to technological changes or change in trend and diversification into another segment which is diversification of business. This might help the chief business during the unfortunate situation(s) and provide better opportunities for expansion as well. The next factor that helped in survival includes favorable environment. It includes: support from the government i.e. political support and the availability of resources i.e. which are crucial for business scenario and last is the personnel related to the business. This factor is considered as a boon to business if the staff and labor are reliable and supportive.

CONCLUSION

Qualitative studies help in exploring of subject matter and are a research method that helps in investigating to obtain deeper knowledge. The procedure that has been followed here, the collection of data and its analysis have been helpful in exploring of factors required for the study.

The objective of defining family managed business from the internal sources and also exploring the factors behind their survival have been stated. In conclusion, the nature of the business might have been different but the factors affecting them were to be similar as mentioned. *Succession planning* has been the prima facie factor affecting the family managed businesses all around. The interpretative case study here had provided an opportunity in exploring the other factors that helped in survival of the business and assurance of growth if the factors can be handled.

The limitations of the study that can be stated here is that the in depth view about all the factors that have impacted businesses in their sustainability couldn't be reviewed and more number of cases in different areas of industry should have been explored in order to identify other factors as well. Nonetheless, the factors explored here have been validated to know about what affects businesses and what helps in sustainment of it. It is hoped that certain limitations of this study will provide another platform for this subject matter to be explored.

TABLES

Table 1: Of the sample data used for the study, the following is the sample demographics:

No.	Gender	Designation	Age	Education	Field of work	City/Town of business
1	Male	First generation	65	Nil	Retailing of tyres	Gandhidham
2	Male	First generation	60	Bcom	Transport	Gandhidham
3	Male	Second generation	26	BCA	FMCG	Surat
4	Male	Fourth generation	26	MBA	Petroleum	Ahmedabad
5	Female	Third generation	25	MBA	Textile	Ahmedabad
6	Male	Third generation	25	MBA	Metal Industry	Jamnagar
7	Male	Second generation	41	BCom	Transport	Mithiruhar
8	Male	Second generation	25	BCom	Optical showrooms	Ahmedabad
9	Male	First generation	64	12^{th}	Provisional store	Porbander
10	Female	Second generation	24	MBA	Retailing of Tyres	Gandhidham

Table 2: Factors that have posed as issues while survival of businesses:

General Factors	Classification of factors that helped in survival	Classification of factors that pose issues or problems
A. Succession planning	• Timely handovers	• Control of business with former generation
B. Customer relationship	• Old customers and repetition • New customers	• Losing on old customers for newer ones
C. Goodwill	• On the basis of business ethics • On the basis of owners	-
D. Support from the family	• Financial aid • Lasting relationships	• No financial aid • Insubstantial relationships
E. Presentation of new opportunities i.e. diversification	• Product diversification • Expansion of product basket	• Industry growth but no change in business per se
F. Competition in the market		• High competition
G. Favorable environment	• Support from environment i.e. availability of resources	• Political thwart
H. Personnel- Labor and staff	• Low turnover due to relationships	• High turnover • Less reliability

ACKNOWLEDGEMENT

I am grateful to my doctoral studies guide Dr. RaashidSaiyed, R K University (Personnel, Staff and Technical help), my colleagues and organization (IISFT), corporate networks and my family.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

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Venture Capital Financing: Performance Evaluation of Aavishkaar India Micro Venture Capital Fund

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ABSTRACT

Venture Capital is a crucial source of finance, which is employed in newly start- up businesses, businesses which are of high growth potential and risky projects. Venture Capital is an Equity investment, so the company on return expects high rate of return. Aavishkaar India Micro Venture Capital Fund was launched in 2001. Itespecially focuses on financing Agricultural sector, Health, Water and Sanitation, Education, Energy, Technology and Micro finance. The company finances in the rural areas. The purpose of this study was to know the pattern of investments made by the company in various sectors. So keeping in mind this the researcher has collected 5 years (2010-11 to 2014-15) annual Impact report of the company. The present study analyses the performance of the company with the help of secondary data. The company's investments in multiple sectors were analyzed with the help of descriptive Statistics and one way ANOVA.

SUMMARY

The research study analyzes the performance of Venture Capital Financing of Aavishkaar Venture Capital Fundduring the year 2010-11 to 2014-15.

Keywords: Venture Capital Financing, Performance Evaluation, Aavishkaar Venture Capital Fund, ANOVA

INTRODUCTION

Venture Capital Financing is a very crucial source of finance as compare to other traditional sources. It is generally invested in high growth firms, businesses having potential, which are risky. Aavishkaar India Micro Venture Capital Fund invests at early stage in underserved businesses. Aavishkaar India Micro Venture Capital Fund (AIMVCF) (here after Company) was established in the year 2001 with the main aim to serve underserved regions. It especially contributes to the rural areas. The company finances venture capital fund in multiple sectors like Agriculture and dairy, Education, Energy, Health and Sanitation, Technology, handicrafts, livelihood. Company has also started contributing to Microfinance sector recently. The company has raised four funds namely Aavishkaar I, Aavishkaar II, Aavishkaar Good well I and Aavishkaar Good Well II. The company invests in above mentioned sectors through these funds. The purpose of this research study was to know the performance of the Venture Capital Fund, pattern of investment made in multiple sectors and the contribution made in various sectors through venture capital financing.

LITERATURE REVIEW

1. *M.P. Shiva Kumar,* "**Evaluation of Entrepreneurs on Indian Venture Capital Firms-A Study",** has analysed the data through correlation and regression collected from TheFunded.com an online community of CEOs and Entrepreneurs. He analysed the perception of entrepreneurs regarding selection of venture capital financing. Results found that the age of venture capital firm has no significant relationship. So venture capital firm needs to grow efficiently should keep commitments towards entrepreneurs and should serve them at their best.
2. *Devlyn Tedesco*, **"An Overview of the Venture Capital Process for Entrepreneurs" (2013)** has explained advantages and disadvantages of venture capital financing. He had also explained the Due Diligence. Due diligence means the thorough survey of the investee company before actual investment by the venture fund.
3. *Hans Landström*, "**Research on institutional venture capital: the past, the present, the future"** has reviewed the research papers on venture capital. He had also analyses the growth of venture capital research during the 1990s and 2000s. he studied the venture capital exits, why, how and when venture capitalists exit.
4. *ThillaiRajan A. and Ashish Deshmukh,* **"Venture capital and private equity in India: an analysis of investments and exits"** He analysed total of 1912 VCPE transactions involving 1503 firms during the years 2004 – 08. He studied that whether venture capitalists should invest at an early stage of the incorporation of the investee company or at a later stage. The paper also focused on exit pattern followed by the venture capitalists. It concluded that venture capitalists should increase the time interval between successive funding rounds.
5. *Jagongo Ambrose*, **"Venture capital (VC): The all important MSMEs financing Strategy under neglect in Kenya" (2012)** A sample of 60 SMEs from Thika Municipal council was interviewed to get their perception and attitude towards utilizing venture capital as an alternative source of financing. The study indicated that 90% of the respondents lacked information about existence and operations of venture capitalists.
6. *Srinivas K T*, **"A study on venture capitalists' funding in different sector of karnataka" (2013)** The author had collected thedata of 20 venture capital firms and had been analysed for the period of 15 years. Both primary and secondary data have been used. The descriptive and ANOVA test were used. The study concluded that the venture capitalists have invested more in service sectors in the state of Karnataka. They should also focus in other sectors.
7. Jim Valrio," **The performance of Canadian Firms that received Venture Capital Financing" (2013)** examined the performance of Canadian Firms that received Venture Capital Fund. The study also examined sector wise & Region wise performance of firms. The study concluded that the Venture Capital backed firms performed better in cumulative revenue growth over the

period. Cumulative revenue growth was higher by 53% as compared to 29%. The Venture Capital backed firms demonstrated superior performance in sales growth, employee growth and asset growth as compare to non- Venture Capital backed firms.

8. Xiaoquing Eleanor Xu, **"A Comparative Study of Venture Capital performance in the US and Europe" (2004)** Examined the quarterly data from 1993 to 2003. The study compared the return and risk performance of Venture Capital Fund in US and Europe. Venture Capital returns were 3.273% and .0765% above Capital Asset Pricing Model risk adjusted return.
9. Tom S. Lindstrom, **"Venture Capital Performance Determinants and Differences between Europe and Northern America" (2006)** the study compared the Europe and American Venture Capital Environments. The 'Profitability" was identified as main determinants. The study concluded that the corporate affiliated Venture Funds seem to yield significantly higher returns compared to independent Venture Capitalists.

OBJECTIVES OF THE STUDY

This study has been selected keeping in view the following objectives.

1. To know the investment pattern of the Aavishkaar India Micro Venture Capital Fund during the period of study.
2. To examine the sector wise investments made by the Aavishkaar India Micro Venture Capital Fund during the period of study.
3. To analyze total investments made by the Aavishkaar India Micro Venture Capital Fund during the period of study.

RESEARCH METHODOLOGY

Data Collection: The present study was mainly based on secondary data. The researcher has used the published data (Annual Impact Reports- 2010-11 to 2014-15) available on official website of the sample unit. Moreover other relevant information related to Venture Capital was also used for the study. The study was also supported with articles published in journals. The data were in million US dollar, the researcher has converted the figures in Indian rupees taking year wise average exchange rates. (2010-11 47.774, 2011-12 49.124, 2012-13 55.911, 2013-14 60.936 and 2014-15 63.469)

Period of the study: The period of the present research study was 5 years begin from 2010-11 to 2014-15.

Hypothesis: H_o: There is no significant difference in investment pattern of sample unit during the period of study.

H_1: There is no significant difference in investment pattern of sample unit during the period of study.

Tools and Techniques: Descriptive statistics have been used to analyze individual sectors. It is also represented by charts. One way ANOVA has been used to investigate the difference in investment pattern of the sample unit during the period of study.

RESULTS AND ANALYSIS

Aavishkaar India Micro Venture Capital Fund (The Company) invests in below mentioned multiple sectors. The results, discussion and analysis of the each sector are as below.

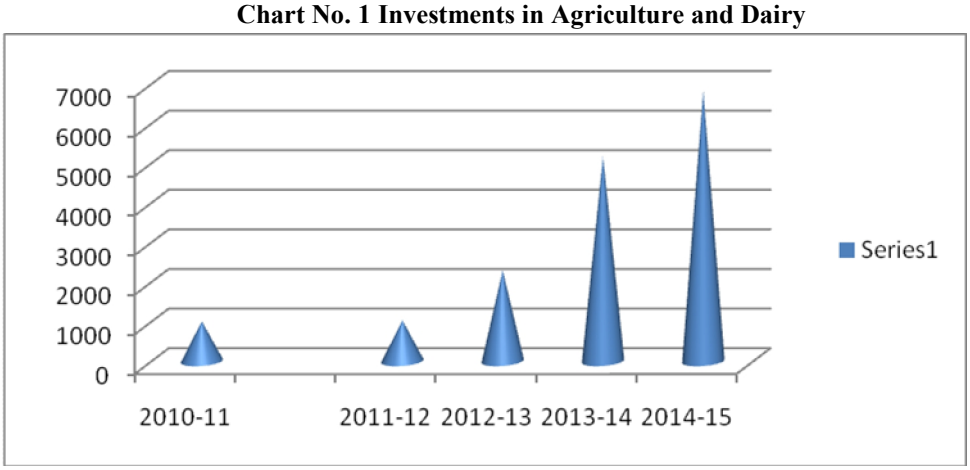
1. **Agriculture and Dairy**

Table No. 1 Investments in Agriculture and Dairy & Descriptive Statistics

1. Sr. No.	2. Years	3. Investments (Rs. In lacs)	4. Mean	5. 3264.766
6. 1	7. 2010-11 8.	9. 1003.25	10. Standard Error	11. 1169.884
12. 2	13. 2011-12	14. 1031.60	15. Median	16. 2292.35
17. 3	18. 2012-13	19. 2292.35	20. Standard Deviation	21. 2615.941
22. 4	23. 2013-14	24. 5167.37	25. Sample Variance	26. 6843149.334
27. 5	28. 2014-15	29. 6829.26	30. Confidence Level (95.0%)	31. 3248.12

Source: Compiled from secondary data

Chart No. 1 Investments in Agriculture and Dairy



The above table and chart reveals that the investment in agriculture and dairy during the period of the study is increasing. The SD (standard Deviation) is 2615.941 and standard Error is 1169.884. If we compare the standard error, it would be decreases as the investment increases and would increase the variance.

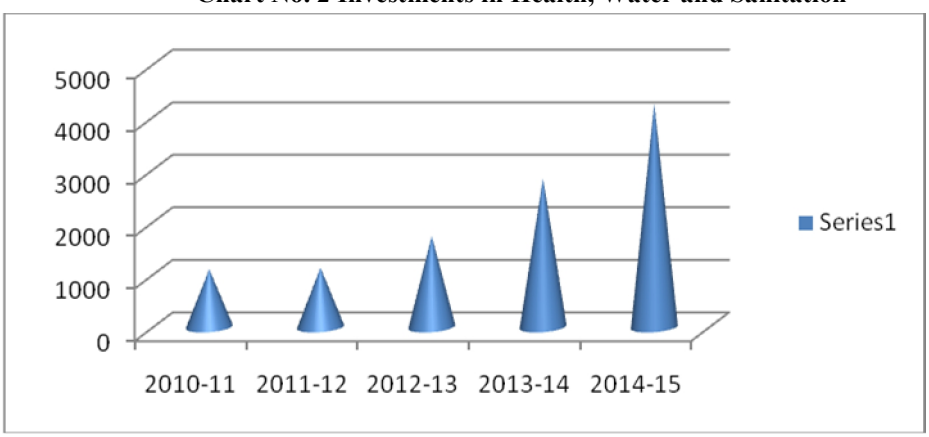
2. Health, Water and Sanitation

Table No. 2 Investments in Health, Water and Sanitation & Descriptive Statistics

32. Sr. No.	33. Years	34. Investments (Rs. In lacs)	35. Mean	36. 2210.832
37. 1	38. 2010-11	39. 1098.80	40. Standard Error	41. 600.62
42. 2	43. 2011-12	44. 1129.85	45. Median	46. 1733.24
47. 3	48. 2012-13	49. 1733.24	50. Standard Deviation	51. 1343.034
52. 4	53. 2013-14	54. 2833.51	55. Sample Variance	56. 1803742.089
57. 5	58. 2014-15	59. 4258.76	60. Confidence Level(95.0%)	61. 1667.59

Source: Compiled from secondary data

Chart No. 2 Investments in Health, Water and Sanitation



The above table and chart reveals that the investment in health, water and sanitation during the period of the study is increasing. The SD (standard Deviation) is 1343.034 and standard Error is 600.62If we compare the standard error, it would be decreases as the investment increases and would increase the variance.

62. Sr. No.	63. Years	64. Investments (Rs. In lacs)	65. Mean	66. 1371.604
67. 1	68. 2010-11	69. 264.19	70. Standard Error	71. 418.949

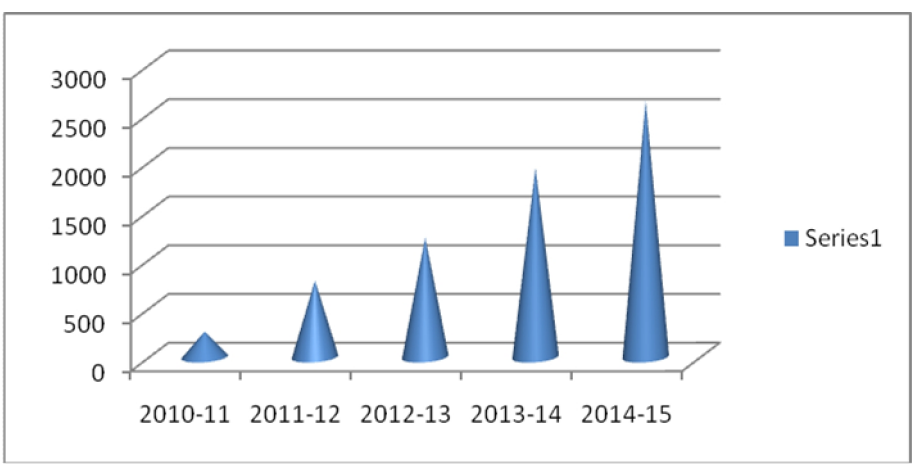
72. 2	**73. 2011-12**	**74. 785.98**	**75. Median**	**76. 1230.04**
77. 3	**78. 2012-13**	**79. 1230.04**	**80. Standard Deviation**	**81. 936.799**
82. 4	**83. 2013-14**	**84. 1943.85**	**85. Sample Variance**	**86. 877592.439**
87. 5	**88. 2014-15**	**89. 2633.96**	**90. Confidence Level (95.0%)**	**91. 1163.189**

3. Education

Table No. 3 Investments in Education & Descriptive Statistics

Source: Compiled from secondary data

Chart No. 3 Investments in Education



The above table and chart reveals that the investment in education during the period of the study is increasing. The SD (standard Deviation) is 936.799 and standard Error is 418.949 If we compare the standard error, it would be decreases as the investment increases and would increase the variance.

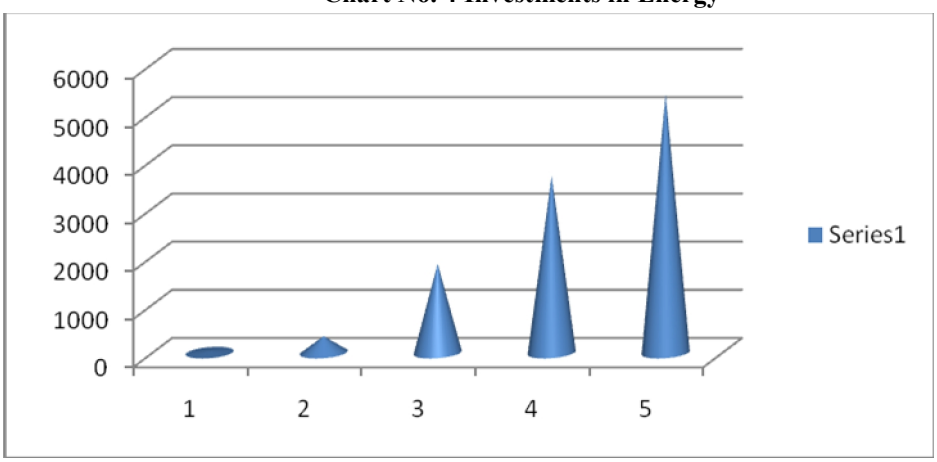
4. Energy

Table No. 4 Investments in Energy & Descriptive Statistics

92. Sr. No.	93. Years	94. Investments (Rs. In lacs)	95. Mean	96. 2255.384
97. 1	98. 2010-11	99. 37.74	100. Standard Error	101. 1012.202
102. 2	103. 2011-12	104. 343.86	105. Median	106. 1845.06
107. 3	108. 2012-13	109. 1845.06	110. Standard Deviation	111. 2263.353
112. 4	113. 2013-14	114. 3674.44	115. Sample Variance	116. 5122768.864
117. 5	118. 2014-15	119. 5375.82	120. Confidence Level (95.0%)	121. 2810.324

Source: Compiled from secondary data

Chart No. 4 Investments in Energy



The above table and chart reveals that the investment in energy during the period of the study is increasing. The SD (standard Deviation) is 2263.353 and standard Error is 1012.202 If we compare the standard error, it would be decreases as the investment increases and would increase the variance.

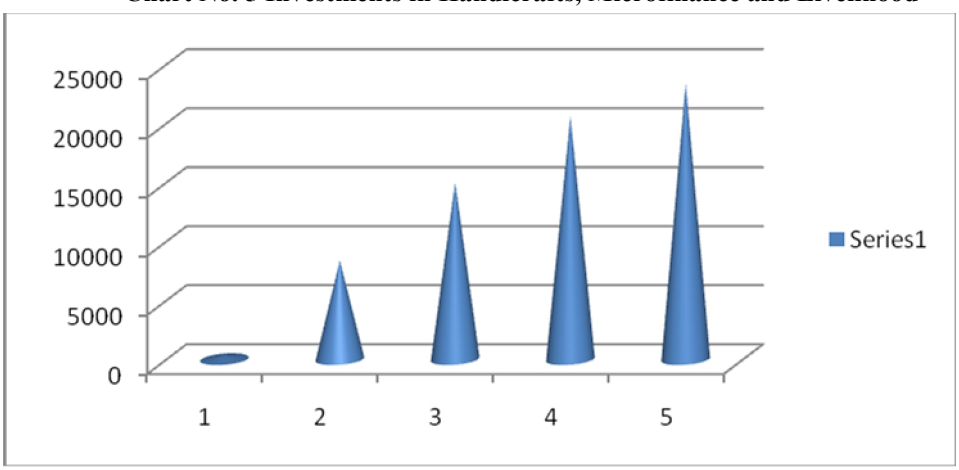
5. Handicrafts, Microfinance and Livelihood

Table No. 5 Investments in Handicrafts, Microfinance and Livelihood & Descriptive Statistics

122. Sr. No.	123. Years	124. Investments 125. (Rs. In lacs)	126. Mean	127. 13527.1
128. 1	129. 2010-11	130. 334.41	131. Standard Error	132. 4182.361
133. 2	134. 2011-12	135. 8351.07	136. Median	137. 14984.14
138. 3	139. 2012-13	140. 14984.14	141. Standard Deviation	142. 9352.043
143. 4	144. 2013-14	145. 20590.26	146. Sample Variance	147. 87460724.23
148. 5	149. 2014-15	150. 23375.62	151. Confidence Level (95.0%)	152. 11612.096

Source: Compiled from secondary data

Chart No. 5 Investments in Handicrafts, Microfinance and Livelihood



The above table and chart reveals that the investment in Handicrafts, Microfinance and Livelihood during the period of the study is increasing. The SD (standard Deviation) is 9352.043 and standard Error is 4182.361 If we compare the standard error, it would be decreases as the investment increases and would increase the variance.

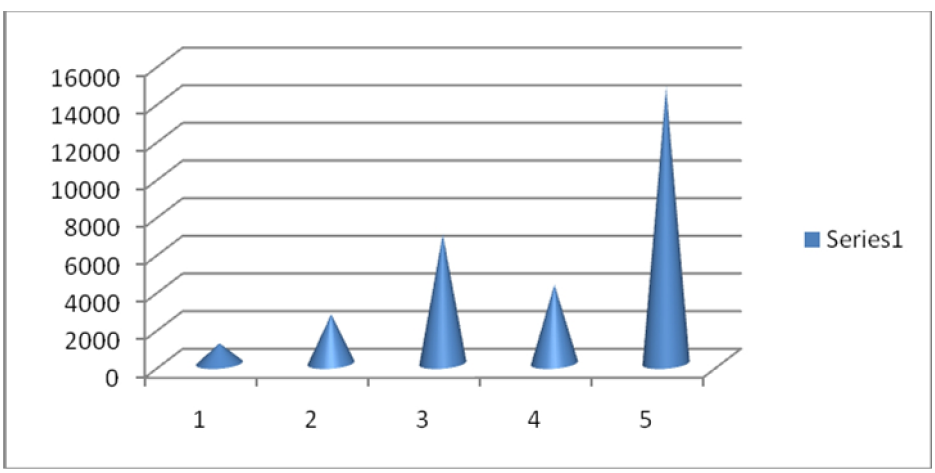
6. Technology for Development

Table No. 6 Investments in Technology & Descriptive Statistics

153. Sr. No.	154. Years	155. Investments 156. (Rs. In lacs)	157. Mean	158. 5884.114
159. 1	160. 2010-11	161. 1060.58	162. Standard Error	163. 2403.067
164. 2	165. 2011-12	166. 2603.57	167. Median	168. 4186.3
169. 3	170. 2012-13	171. 6877.05	172. Standard Deviation	173. 5373.421
174. 4	175. 2013-14	176. 4186.30	177. Sample Variance	178. 28873662.32
179. 5	180. 2014-15	181. 14693.07	182. Confidence Level(95.0%)	183. 6671.984

Source: Compiled from secondary data

Chart No. 6 Investments in Technology



The above table and chart reveals that the investment in Technology during the period of the study is fluctuating. In the year 2013-14 as compare to the year 2012-13 it decreases. The SD (standard Deviation) is 5373.421 and standard Error is 2403.067 If we compare the standard error, it would be decreases as the investment increases and would increase the variance.

7. Total Investments- Sector Wise

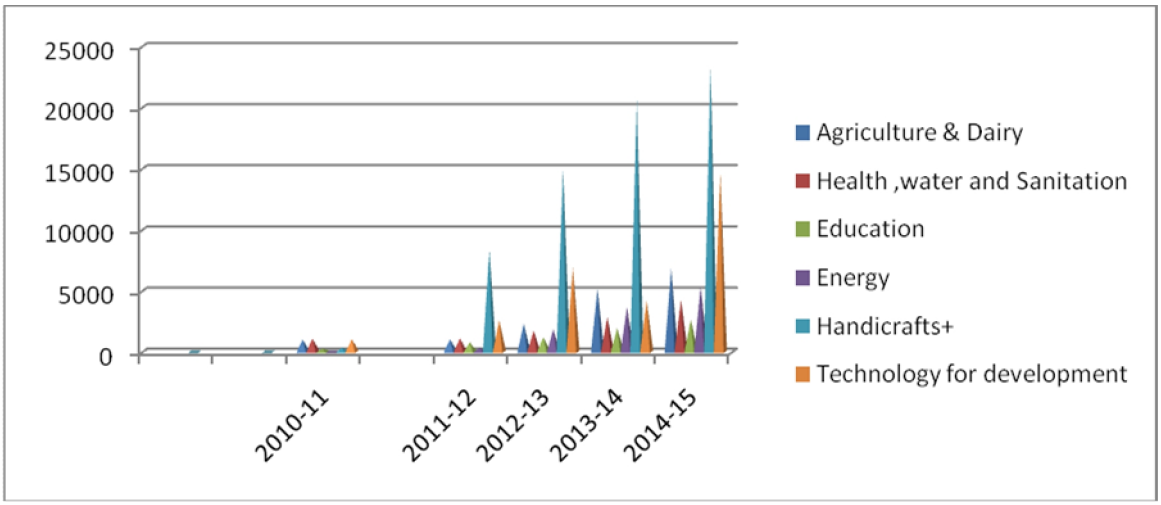
Table No. 7 Sector wise Investments

Rs. in lacs

184. Sr. No.	185. Years	186. Agriculture & Dairy	187. Health ,water and Sanitation	188. Education	189. Energy	190. Handicrafts + 191. Microfinance+ 192. Livelihoods	193. Technology for development
194. 1	195. 2010-11 196.	197. 1003.25	198. 1098.80	199. 264.19	200. 37.74	201. 334.41	202. 1060.58
203. 2	204. 2011-12	205. 1031.60	206. 1129.85	207. 785.98	208. 343.86	209. 8351.07	210. 2603.57
211. 3	212. 2012-13	213. 2292.35	214. 1733.24	215. 1230.04	216. 1845.06	217. 14984.14	218. 6877.05
219. 4	220. 2013-14	221. 5167.37	222. 2833.51	223. 1943.85	224. 3674.44	225. 20590.26	226. 4186.30
227. 5	228. 2014-15	229. 6829.26	230. 4258.76	231. 2633.96	232. 5375.82	233. 23375.62	234. 14693.07

Source: Compiled from secondary data

Chart No. 7 Sector wise Investment



The above table and chart reveals that there is a maximum investment in Handicrafts and Microfinance sector as compare to other sectors during the period of the study. In the year 2010-11 there is a balanced investment in each sector. Year by year the company has focused maximum in Handicrafts & Microfinance and Technology sectors.

***Testing Hypothesis*,**

Ho: There is no significant difference in investment pattern of sample unit during the period of study.

Table No.8 One Way ANOVA of Sector wise Investments

Source of Variation	*SS*	*df*	*MS*	*F*	*F crit.*
Between Groups	523068149.6	5	104613629.9	4.792135623	2.620654147
Within Groups	523926557.1	24	21830273.21		
Total	1046994707	29			

Fc > Ft 4.79 > 2.62

Hence Null hypothesis is rejected at 5 % level of significance. Alternate hypothesis fails to reject, it means that there is significant difference in investment pattern of sample unit during the period of study.

LIMITATIONS OF THE STUDY

Following are the limitations of the study.

1. The study was based on secondary data therefore its findings depend entirely on the accuracy of the data.
2. The study was restricted to only one venture capital firm.

CONCLUSION

Aavishkaar India Micro Venture Capital Fund is a venture capital financing company. The company has been focusing on rural area. The performance of the company was consistent during the period of the study. Even results and analysis also reveals that the company has the maximum investment in the

handicrafts and microfinance sector as compare to other sectors during the period of the study. The analysis (ANOVA) shows that the company has a significant difference in investment pattern among the various sectors during the period of the study. The company has very less investment in energy sector as compare to other sectors. The company should maintain uniformity in invest pattern. The company should also focus on other sectors to achieve balanced regional development in all the sectors of the economy.

ACKNOWLEDGEMENT

The author would like to gratefully acknowledge the Aavishkaar Micro Venture Capital Fundfor this research. Without such data the research work would not be possible. The author would also like to acknowledge the support offamily members for this research.

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Trans-Generational Entrepreneurship: A Challenge or An Opportunity for Family Businesses

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ABSTRACT

Though Family business has its own advantages like strong business insight-speedy decision making-strong values-agility, managing and transferring business to family members has its own unique challenges. Younger generation may face difficultiesto prove themselves to the former generation becomes acute challenge for family leaded business. This research focus on mainly two research problems: 1.) How first generations look forward to transfer their business to next generation? 2.) What are the challenges and opportunities 2nd generations experience to take up business responsibilities from first generation? Selected reviews of literature, surveyed reports of Indian families were selected as sources along with few1st and 2nd generations from different family business had been chosen and interviewed at their convenience. Analysis of data revealed various challenges and opportunities both the generations experience which reflect upon good and bad faces of trans-generational entrepreneurship.

SUMMARY

Researcher focused on entrepreneurs' responses when interviewed and asked to share their opinions about difficulties they faces or faced in transferring business and derived various opportunities and challenges both generations find in trans-generational entrepreneurship mode.

Keywords: Trans-generational, Entrepreneurship, challenges, opportunities, family business

INTRODUCTION

Nurturing entrepreneurial skills and putting trust on family members over generations to sustain competitive advantage in family business becomes quest in incumbents of family business. Since financial crisis, more people have come to realize the importance of family leaded business for stability and resilience.Where Family companies contribute two-thirds of India's GDP (KPMG, 2013) with increasing size of business to ensure hold in competitive market, family business needs family ownership

with assurance to save, extend established business value. Generation, credibility and communication are mainly three gaps which needs to be filled up by family business in order to ensure succession planning in family business. The transition from one generation to the next is a crucial problem, which can construct or destruct the business, according to the PwC report. In the UK, history of family business had begun from 1541AD by john brooke as he had started business of supplying uniform to soldiers of army and then after that business transferred to generations. In India, before independence 'swadeshi' movement indirectly encouraged family business with the intent to make people self-reliant. Tata Group started by Jamshedji Tata in 1968 is the oldest and the most trustworthy Indian business group in the world. Most Business families can't survive beyond 3rd generations. There are few companies run by Birla, Ambani, and Bajaj split after third or second generations. Those family firms management and ownership spread across generations need to review their ways of doing business to reduce risk of losing their entrepreneurial capacity. As per the Ernest &Young report in global survey of students, 22.7% students express their intention to join business. So, the low proportion of Younger's intention to join business urges family firms to change their approaches to attract them towards family business. The problem of succession transferring leadership and ownership to the next generation of family members has captivated most of the research interest. Many companies like Du-Pont and Foard are considered as examples where the succession problem deeply affected the business and the family. All the challenges of family business extend the quest to resechers that how families in business survive and grab competitive advantage generation wide and challenges they find during transformation.

PROBLEM STATEMENTS

- How first generations look forward to transfer their business to next generation?
- What are the challenges and opportunities 2nd generations experience to take up business responsibilities from first generation?

LITERATURE REVIEWS: GENERATION CONCERN AND FAMILY BUSINESS

Trans-Generational Entrepreneurship is the business activities to develop and grow business by transferring resources, capabilities and mind-sets across generation to transfer ownership and management.

Mohammad Jasim Uddin contributed by addressing linkage between resources available and identifying their links with how family get sustainable competitive advantages through it if penetrated properly. Proper transfer of tacit knowledge about firm values, behavioural norms generation wide becomes a bridge between each generation to build relationship and trust amongst. Transfer of that knowledge is only possible if stability prevails in family business. Involvement and organization socialization plays vital role in that. (Uddain, 2011)[i]

Martínez, Galván, Palacios analyze the factors that influence knowledge transfer both intra and intergenerational in family firms and concluded that knowledge is best transferred when family members value the following factors like trust between family members, commitment to the family business, intergenerational relationships, psychological ownership of the family business, successor's aspects and training, predecessor involvement in the successor training, Organizational culture. (Martínez, Galván, Palacios, 2013)[ii]

Randel S. Carlock has said in his working paper on Why Planning is needed for family business derived some instincts which helps to understand the needs when one think to survive family business. Parallel planning process helps firms to become professionally emotional. To ensure value creation an maintain

family influence, proactive planning and decision making is deadly needed that helps to govern family business generation wide.[iii]

W. Gibb Dyer, Jr.Wendy Handler explored that instead of ignoring the connection between the entrepreneurship and the family, importance of family involvement in shaping career of family members through supporting at the time of new start-ups, offering employment in new venture, sharing early experiences are much required to make succession planning smooth.[iv]

K. Ramachandran sated in his study of Indian family Business:Their Survival beyond Three Generationsthat most of the family businesses go for unrelated diversification to cope with poor communication structure, regulatory constraints and inefficient judiciary (Khanna and Palepu 1997). Business grown over third generation get government protection as one of the most expected benefit from family business. (Kim, Kandemir and Cavusgil 2004).[v]

RESEARCH OBJECTIVES

- To know the challenges experienced by early generation to transfer business to late generation
- To know younger generations challenges to get hold in existing family business
- To Study trans-generational challenges and opportunities in family business

MATERIALS AND METHODS

Research is focused to explore challenges and opportunities faced by family business with the focus of Trans - generational entrepreneurship through research base views and selected small and medium size family businesses. Early and young generations of family had been selected as respondents and their dialogues and words had been collected through interviews.

I have conducted 6 semi-structured personal interviews of respondents from Rajkot (Gujarat, India) city who are founders or family members of family business. Below are the profiles of the respondents.

Table 1. Profiles of respondents:

Respondents	Age (yrs)	Occupation	Gender	Generation
1	45	Product head	Male	1st
2	65	founder	Male	1st
3	50	founder	Male	1st
4	21	Pursuing graduation	Male	2nd
5	23	Pursuing post-graduation	Male	2nd
6	36	General Manger	Male	2nd

I have prepared below questions for these interviews and involved respondents in discussion.
Questions to 1st Generation

1.) Are you a founder member of family business?
2.) What is your status in family business?

3.) How many members are involved in business?
4.) To whom they can accommodate in family business in future?
5.) Do you have confidence in next generations of your family?
6.) What you are doing to share knowledge, challenges and complexities of business to next coming generations?
7.) What do you think about scope of family values and its understanding in family business?

Question to 2nd Generation

1.) Do you know about your family business?
2.) Are you working for family business?
3.) Do you like to join/have you already joined?
4.) Have you heard any words from your family members which shows low confidence in you?
5.) What you are doing to increase confidence of them for you?
6.) Why you like to join your family business?
7.) Why you do not like to join your family business?

CHALLENGES FACED BY EARLY GENERATION TO TRANSFER BUSINESS TO LATE GENERATION

A 25 years old large firm mainly in submersible pump business is being handled by many family members including 2nd generations. During interview one of the member handling pump as managing director stated in his response, "Youngers in our family are doing well as they have related education and understanding of our family business values we have inculcated since long back. They remain more accountable than outsiders", when asked share their opinion about challenges experienced by them while transferring business responsibilities to next generation.

A father who had handover responsibilities to their sons responded in interview, "In early stage I had fear as they did not have sufficient market knowledge but full trust on their knowledge about technology and creativeness. Today they are doing well".

One of the large NRI family, which had a huge succession planning challenge said in their interview that though our daughters are talented and well qualified, we were not being considered them in our business succession as it was clear earlier that only sons could be considered.[vi]

According to PwC's Family Business Survey 2012-13, 33% of the Indian family businesses interviewed planned to pass on the Ownership but not the Management of the company to the next generation. Reason came behind is the uncertainty regarding skills and aptitude of young generations.[vii]

YOUNGER GENERATIONS' CHALLENGES TO GET HOLD IN EXISTING FAMILY BUSINESS

From the same family as in submersible pump interviewed, one of the younger who is going to join business in near future responded in interview, "I am definitely going to join my family business as there is ample growth opportunities in it and I will get already established market which takes long time that I don't want to spend as my father and uncles have vested in their life. They are happy to give me management responsibility of any of the area in which I am interested"

A young studying in management institute having family business of manufacturing brass parts being handled by his father and uncle responded in interview, "I want to jump into some new business as there

is no any work to offer me and I may not get the importance as new comer by my family members or employees".

PwC surveyed more than 200 next generation family members who are likely to take over their family businesses. The study showed that 88% of the next generation say they have to work harder than others in the firm to prove themselves to both colleagues and customers and 59% said gaining the respect of co-workers is one of their biggest challenges. 22% of the next generation are concerned about working with family members and understanding the family dynamic.[viii]

RESULTS AND DISCUSSION

What is evident from the studied literature and interviewed business families in this research that, there is no one way to approach trans-generational entrepreneurship. What is apparent through reviewed papers is transfer of tacit knowledge by valuing trust commitment and intergenerational interrelationship, predecessor involvement in successor training and proactive planning is deadly needed to transfer family business generation wide. Challenges related to insufficient market knowledge gender discrimination, uncertainty about skills and aptitude of the younger generations stressed old generations. Wherein interest, innovativeness, early experiences in youngers are considered opportunities. Convince family members to get involved in other business, realize them the capacity of handling running business or to prove their area of interests are the main challenges felt by young generations at a time of taking decisions to join or not their family business. Trust and early experiences of business transferred from family become opportunities for them when they ready to join family business along with established business and ready costumers.

CONCLUSION

Few of interviewed responses provide a deep understanding that both the generations early and late have interest to get competitive advantage and growth of their business while offering tag as family business. Though both the generations have their own different expectations, they see opportunities in either handover or takeover management or ownership. The key findings reinforce first generations to come out from gender bias, believe their interest and provide youngers an early opportunity to learn market and business before offering ownership or management. Another important suggestions to young generation before giving thought to enter into family business check their own interest and capability with proving capacity which shows imbibed family values.

ACKNOWLEDGEMENT

I would like to express my sincere gratitude from the bottom of my heart to Founder and co-founder of Falcon pump Ltd., Owner of KK Hotel, Owner of Modern super Market and youngers of all those businesses for sharing their opinions and experiences. Also my sincere thanks to Dr Hitesh Shukla, Professor, Department of Management, Saurashtra University towards value addition of this study.

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'ROLE OF MOTIVATIONS AND CHALLENGES TO BECOME AN ENTREPRENEUR: A STUDY AMONG BHAVNAGAR UNIVERSITY STUDENTS'

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ABSTRACT

The person, who undertakes the risk of new enterprise, is called as entrepreneur. That is the person who undertakes the risk of new enterprise, is called as entrepreneur. The entrepreneur is the one who crates ideas, bring together all the four factors of production and produces marketable goods and services. Entrepreneurs are the economic agents who bring economic development by the way of entrepreneurial activities. To become an entrepreneur is not an easy task. Bhavnagar University offers entrepreneurship subject at graduate and post graduate level. This study examines the understanding of entrepreneurship, motivations as well as challenges faced by 123 Bhavnagar University undergraduate students to become an entrepreneur. This study reveals that motivational factors like 'high profits', 'no boss' attract them to start a new business. While raising capital for start ups, assembling a business team, dealing with competition are the main challenges faced by them

SUMMARY

This study examines the understanding of entrepreneurship, motivations as well as challenges faced by undergraduate students to become an entrepreneur.
Keywords: Challenges, Entrepreneur, Motivational factor, new business, Undergraduate students

INTRODUCTION

The term entrepreneur is derived from the French work 'Entrependure'. It means to undertake. That is the person who undertakes the risk of new enterprise, is called as entrepreneur. The entrepreneur is the one who identifies the business idea; bring together all the four factors of production (Land, Labour, Capital and Organization) and produces marketable goods and services. Entrepreneurs are the economic agents who bring economic development by the way of entrepreneurial activities. ***'Entrepreneurs are born, not created'. The entrepreneurs are different from managers and common man. They set an objective to establish a business unit and try to fulfil the objective by applying his skills and knowledge.***

To establish the business organization and to achieve the business objectives is not one man show. It requires skills, knowledge, Technical know-how and vision for long term planning. To be a successful entrepreneur, one must have some qualities like building an organization, managing the resources etc. Thus, to be an entrepreneur, a person must have various skills.

Entrepreneurship is an act of being an entrepreneur. In other words the entrepreneurs pass through various processes of actions to become an entrepreneur. These processes include;

1. The process of Idea generation
2. The practices to Acquire financial and human resources
3. To construct or acquire business premises
4. To take various decisions etc…

Thus, entrepreneurs undertake various activities to develop and manage a business unit. All these processes of actions are called as 'Entrepreneurship'.

Entrepreneur is the one who provides forth factor of productions that is 'Enterprise'. With the help of forth factor of production, the entrepreneur assembles, co-ordinates and manages the other factors namely land, labor and capital.

Entrepreneurship is the act of being an entrepreneur, which can be defined as 'one who undertakes innovations, finance and business acumen in an effort to transform innovations into economic good'. Entrepreneur is the person who generates new ideas and put it into action by way of starting a new business. However, in recent years, the work of entrepreneur is not limited to only generation of ideas and starting a new business but it extends to social and political sides also. Entrepreneur meaning does not limited to only starting a new business but it also includes decisions regarding restructuring like mergers, acquisitions, amalgamation, spin –offs, split-offs etc..

MOTIVATIONAL FACTOR:

'Motivation' is the reason that why person inspired to do that activity. Without motivational factor nobody wants to involve their personal, financial sources into that

activity. There are various motivational factors that why a person want to start a new business. Proper motivation is needed when a person is going to start a new venture. It is a psychology of human being that if he/she get something from the activity then only they think over to start a new activity. Motivation plays an important role for entrepreneurs. While starting a new business entrepreneur invest his/her financial and personal resources in the form of capital and new idea and as a reward they get internal as well as external reward from business. If there are no such rewards then no one wants to start a new business. Motivational factors are not same for everyone. It different from person to person but some motivational factors like high profit, no risk etc are common when a person starts new business.

Motivational factors are categorized into two: Internal gain and External gain. Internal gain includes excited for new venture, ready for new challenges, social status, public recognition, want to be free from corporate culture etc. while external gain includes increase in income, increase in personal income, increase in wealth etc.. In addition to these some motivational factors are they want to be their own boss, voluntary retirement, security of family members, new opportunities in management etc.

CHALLENGES OR OBSTCLES OF SUSTAINING IN A NEW VENTURE:

Entrepreneurs have to face many challenges on the way to success. When entrepreneurs think of starting a new venture, he/she visualize motivational factors in setting up a new business but they have to also face the dark side of new business i.e. obstacles. Various studies had been conducted on challenges faced by new entrepreneur from all the studies it was found that the main challenge is of procurement of finance i.e. rising of capital. Young and Welsch (1993) identified that entrepreneurs face several obstacles, such as lack of financial assistance, lack of information on various aspects of business, excessive taxation, and high rate of inflation. In addition to these challenges common challenges faced by entrepreneurs are poor organization set up, lack of confidence, negative attitude, poor customer response, frequently changing technology, frequently changing customer preferences, lack of coordination in organization etc..

MATERIALS AND METHODS

OBJECTIVES OF THE STUDY:

1. To get an idea about the student's perception towards entrepreneurship
2. To identify the factors motivates to start a new business.
3. To identify the challenges/obstacles faced by university students while starting a new business.

HYPOTHESIS OF THE STUDY:

H1: There is no significant difference between Male and Female students for their intention to become an entrepreneur.

H2: There is no significant difference between Male and Female students regarding Motivational factors to become an entrepreneur.

H3: There is no significant difference between Male and Female students regarding Challenges faced while starting a new business.

RESEARCH METHODOLOGY:

The research methodology used in this study is given below:

Selection of Sample:

The students selected for the sample were those in final year of Bachelor of Business Administration. A total of 200 set of questionnaires were distributed to the students of institutions running Bachelor of Business Administration programme, namely Swami Sahajanand College of Commerce and Management (SSCCM), The KPES. Out of that 123 students returned filled up questionnaire i.e response rate is 61.5%.

Data collection Tool:

For the study data collected using primary source. A questionnaire was developed to collect data.

Data analysis:

Primary data collected through questionnaire was analyzed using statistical techniques and statistical software SPSS.

RESULTS AND DISCUSSION

Table 1 shows the gender profile of the respondents. It can be seen from the table that 72.36% respondents were female as compared to male respondents 27.64%. This shows majority female respondents.

Table 2 and figure: 2 shows that 120 respondents (97.56%) age between 19-23 years while only 2.44% student's age between 23-25 years.

Above table reveals that 34.15% respondents were elder in the family, which consists of 8 male students and 34 female students. 7.32% respondents were single child in the family, they were 5 male and 4 female.

Place of residence of most of the students were rural (64.23%).

Table 3 shows that most of the respondent's specialization of study was marketing management (39.02%).

Respondents reported that 30 male students and 51 female students had working experience. Out of them total 69 students (85.19%) had working experience of less than 12 months. Analysis also shows that father's occupation is employed in 39.84% cases, and self employed in 34.96% cases. 64.23% respondent's mother's occupation was unemployed. 59.35% respondents says that their parents didn't influence on their career selection.

Respondents were asked to rate their intention to become an entrepreneurs on a five point scale. Table 4 shows that the respondent had strong intention (M=3.86, S=0.804) to become an entrepreneur. There were main two reasons "career goal is to become an entrepreneur" (M=4.14, S=0.653) and "want no boss" (M=4.27, S=0.660).

Table 5 shows that external gain is the main motivational factor with mean 4.18 and standard deviation 0.518. External gain includes increase in income opportunity and increase in wealth. While mean for motivational factor- Independent and new opportunities in management were same 4.14. Independent motivational factor includes – they want to be their own boss, want self employment, personal security and voluntary retirement with the mean 4.22, 4.30, 4.26, and 4.27 with standard deviation 0.573, 0.460, 0.610, 0.676 respectively.

New opportunities in management include development of new ideas, acceptance of new technologies with mean 4.13 and 4.11 respectively.

Factor analysis was also applied by categorizing challenges into three heads: External factors, Personal factors and internal factors (related to finance and production). Table 6 shows results.

T-TEST

T test was conducted to prove hypothesis. To compare the entrepreneurial intention, motivational factors and obstacles and challenges for males and females independent t test was conducted.

H1: There is no significant difference between male and female students for intention to become an entrepreneur.

Above hypothesis was rejected as $p<0.05$. From the calculations given below it can be analyzed that male student's intention to become an entrepreneur was higher than that of female students.

H2: There is no significant difference between Male and Female students regarding motivational factors to become an entrepreneur.

Above hypothesis was rejected as $p<0.05$. From the calculations given below it can be analyzed that male students are highly motivated to become an entrepreneur than that of female students.

H3: There is no significant difference between Male and Female students regarding Challenges faced while starting a new business.

Above hypothesis was rejected. Male students were found to be higher in facing challenges while starting a new business as compared to female students.

FINDINGS:

The objective of this paper is to get an idea about university undergraduate student's perception towards starting a new business, to identify the factor which motivates them to start a new business and the challenges faced by them while starting a new business. From the analysis it was found that student's had a positive perception towards starting a new business and they were highly motivated to start a new business. Mainly male students were found to be highly motivated as compared to female students. They are highly motivated to start a new business due to internal reward, external reward, and independent.

CONCLUSION

This study provides a platform for both university and government to cultivate, develop and train a more entrepreneurially- oriented and proactive university students able to launch business ventures upon graduation. It is strongly recommended that university must have to offer entrepreneurship development as a specialization subject at undergraduate level. This can be helpful to the students to understand stages of starting up a new business because without understanding these stages it is difficult to start a new venture.

FIGURES

Fig:1 Gender Profile of the Respondents

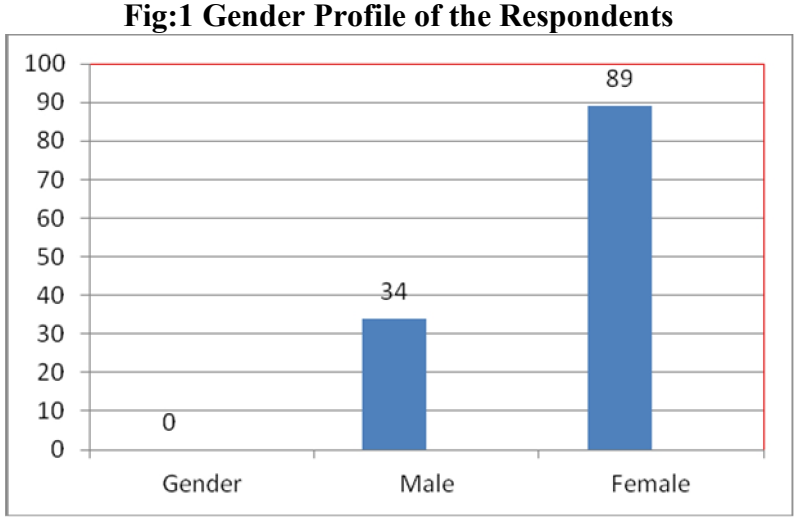
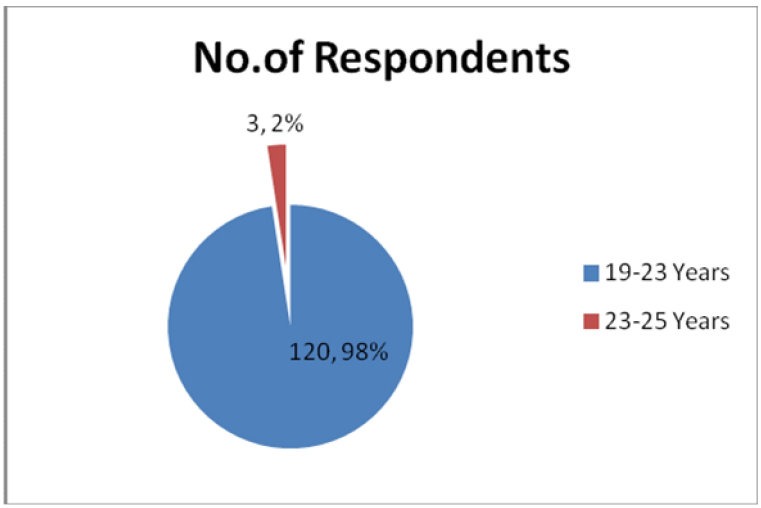


Fig 2: Age Profile of the Respondents



TABLES

Table 1: Gender Profile of the Respondents

Gender	No.of Respondents
Male	34
Female	89

Table 2: Age Profile of the Respondents

Age	**No.of Respondents**	**Percentage (%)**
19-23 Years	120	97.56
23-25 Years	3	2.44
Total	**123**	**100**

Table 3: Other Profile of the Respondents

Other profile	**No.of Respondents**		**Total**	**Percentage (%)**
	Male	**Female**		
Order of Birth in Family				
Single child	5	**4**	**9**	**7.32**
Second child	9	**22**	**31**	**25.20**
Elder	8	**34**	**42**	**34.15**
Younger	9	**12**	**21**	**17.07**

None of the above	3	**17**	**20**	**16.26**
Place of Residence				
Rural	16	**63**	**79**	**64.23**
Urban	17	**23**	**40**	**32.52**
Metro city	1	**3**	**04**	**3.25**
Specialization of study				
Marketing management	17	**31**	**48**	**39.02**
Human resource management	12	**28**	**40**	**32.52**
Financial management	5	**30**	**35**	**28.46**
Working experience				
Yes	30	**51**	**81**	**65.85**
No	4	**38**	**42**	**34.15**
Working period (for 81 respondents)				
Less than 12 months	23	**46**	**69**	**85.19**
1-3 year	7	**5**	**12**	**14.81**
Place of Work (for 81 respondents)				
Govt.Sector	2	**3**	**5**	**6.17**
Private Sector	4	**32**	**36**	**44.44**
Own Business	23	**9**	**32**	**39.51**
Relative Business	1	**7**	**8**	**9.88**
Father's Occupation				
Employed	10	**39**	**49**	**39.84**
Self employed	22	**21**	**43**	**34.96**
Unemployed	00	**2**	**2**	**1.63**
Retired	02	**24**	**26**	**21.14**
Other	00	**3**	**3**	**2.44**
Mother's occupation				
Employed	10	**11**	**21**	**17.07**
Self employed	02	**16**	**18**	**14.63**
Unemployed	20	**59**	**79**	**64.23**
Retired	01	**00**	**1**	**0.81**
Other	01	**03**	**4**	**3.25**
Parent's influence on career selection				
Yes	20	**53**	**73**	**59.35**
No	14	**39**	**53**	**40.65**

'Table 4: Mean of Student's intention to become an entrepreneur after graduation'

Student's intention	Mean	Std.dev.
- **Want to become businessman**	**3.73**	**0.786**
- **Career goal is to become an**	**4.14**	**0.653**

entrepreneur		
- **Inspired from family business**	**3.60**	**0.965**
- **Decided to create a partnership firm in the future**	**3.91**	**0.768**
- **Want no boss**	**4.27**	**0.660**
- **Will work hard to be an entrepreneur**	**3.83**	**0.912**
- **Has financial support to start a new business**	**3.59**	**0.970**
- **Think to start new business in near future**	**3.93**	**0.770**
- **Want to start new business other than family business**	**3.71**	**0.756**
AVERAGE	**3.86**	**0.804**

'Table 5: Mean and Standard deviation of motivational factors'

MOTIVATIONAL FACTORS	MEAN	STD.DEV
Internal gain	**4.02**	**0.421**
External gain	**4.18**	**0.518**
Independent	**4.14**	**0.425**
Security of family members	**4.05**	**0.466**
New opportunities in management	**4.14**	**0.437**

'Table 6: Mean of Challenges faced'

Challenges faced	Mean	Std. Deviation
Eternal factors:		
Inflation rate	**3.48**	**0.804**
Poor labor supply	**3.67**	**0.785**
Strict government laws	**3.92**	**0.743**
Expensive labor	**3.73**	**0.669**
High tax rates	**3.73**	**0.752**
Lack of government support	**3.60**	**0.690**
Tight competition	**3.73**	**0.691**
	3.69	**0.523**
Personal factors		
Over stress	**3.68**	**0.740**
Negative attitude	**3.50**	**0.878**
Lack of Managerial skill	**3.50**	**0.828**
Lack of coordination	**3.57**	**0.889**
More risk	**3.64**	**0.833**
	3.58	**0.651**

Internal Factors (related to production and finance)		
High production expenses	**3.73**	**0.843**
Require high working capital/investment	**3.66**	**0.812**
Poor financial support	**3.80**	**0.860**
Lack of material suppliers	**3.66**	**0.770**
	3.72	**0.667**

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

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RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

Investors' Awareness and Financial Risk Tolerance towards Stock Market: An Empirical Study in Rajkot

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ABSTRACT

This study attempts to analyze the factors influencing the perceived risk towards stock market. The paper empirically examines whether demographic and market awareness elements could be the differentiate factors for financial investors in terms of perceived risk and risk taking behavior. A survey of investors belongs to Rajkot city was conducted through a structured close-ended questionnaire including a variety of demographic factors. The stock market awareness and financial risk tolerance factors significantly predict the attitude towards investing in the stock market. In India, the stock market investment instruments are considered as the risk by the investors. Still the investors are willing to invest in the stock market through the help of news collected from the TV, newspaper, seminars, and workshops. The study was carried out in order to find the awareness level of retail investors and perceived risks associated with investing in stock market. The study also highlights the ways by which investors are minimizing the risk. Finally, the paper analyses the factors influencing the stock selection by a retail investor. The research work is confined to the retail investors of Rajkot city only. The further research can be carried out at the state level to get the larger picture of the study. The same research can also be carried out for the mutual fund market, derivative market or commodity market.

SUMMARY

The study reveals the low risk tolerance and high market awareness sample with respect to the stock market.

Keywords: Investors; Risk tolerance; Investors' awareness; Stock market

INTRODUCTION

The presence of diversified financial service sectors provides investors with a wide range of opportunities to invest. The investor's decision to invest in the stock market is significantly predisposed by the variety of benefits each investor seeks from it. In general, the investment schemes of one investor are different from that of another. The behavior of an individual investor to make the investment decision is multifaceted and be subject to a number of demographic and behavioral factors. The diversified researches have been carried out across the globe in order to analyze the behavior of retail investors. The extensive review of literature also available that seeks to elucidate the investor's awareness, financial risk tolerance, perceived risk, patterns of investment, and factors affecting the stock selection (Grable & Lytton, 1999; Grable, 19997; and Grable and Joo, 2004). The study has considered the financial risk tolerance and financial risk behavior is an important factor for the investment decision. The study of M Kannadhasan (2015) confirms that the demographic factors do play a role in differentiating and classifying retail investors and they motivate practitioners to continue to use them in the future as well. Gable (1999) argued that the financial risk tolerance is a fundamental issue underlying a number of financial decisions. This is the reason why researchers are giving more importance to understand the relationship between individual financial risk tolerances.

REVIEW OF LITERATURE:

Cohn et. al. (1975) investigated the effect of changes in income on the proportion of risky portfolio. They found that the amount of investors' contribution to risky stocks decreases with increased with the increase in wealth. William et. al. (1990) has done a discriminant analysis to segment the investors based on investment behavior and demographic characteristics. The study reveals the light and heavy investors as well as active and passive investor. The descriptive analysis carried out by Gupta (1993) reveals that there is a substantial swing towards mutual fund products while there is a moderate move towards stocks and debt instruments and a shift away from traditional financial assets. A report of NCAER (2000) studied that the safety and liquidity were the primary consideration for an investor while choosing the stocks. Rajarajan (2002) found the strong association between the risk tolerance capacity and the demographic profile of the retail investors. Kiran and Roa (2004) classified the investors based on the psychographic and demographic characteristics. The study reveals that the risk tolerance capacity of the investors was strongly dependent on the psychographic and demographic variables. Al-Ajmi (2008) found that the investors those approaching towards the retirement age show decline in risk tolerance capacity. The descriptive study of Gupta and Jain (2008) shows the investors' preference for the selection of various financial instruments. The study reveals that the household investors less preferred the mutual fund due to the lower returns, management fees and entry/exit load. The study also exposes that the household investor is likely to invest in the stock market as compared to other financial instruments. Parashar (2010) examined the effect of various personality traits on investment decision by the investors. The study finds the significant association between demographic characteristics such as education, occupation, age, gender etc. and the investment decision.

The study of Lehave& Sloan (2008) shows the investors recognition and stock return. The study finds that the changes in investor recognition have a positive correlation with the contemporary stock return while the returns of future stock were negatively correlated to the investors recognition change. Besides it, they found that there is a positive correlation between corporate investment & financing activities and investor recognition change. The study of Barneaet. al. (2010) reveals that the environment of the family plays an important role on the young investors. However, it last for the short period only and vanishes, as they become experience. They have also analyzed over the genetic factors. Their results show that even the twins nurtured in different atmospheres, they display the similar investment behavior. Van et. al. (2003)

examinedthe stock selection strategies in emerging market. The study shows the significant effect of value, momentum, and earning revisions over stock selection in contrast to selection based on size and liquidity. There was no significant evidence that supports the risk factors as the selection strategy. Wang & Hanna (1997) has studied the effect of age on risk tolerance. He had measured the risk tolerance by the total wealth and risky assets factors. The finds that if other factors remain constant, the risk tolerance behavior increases with the increase in age.

OBJECTIVES:

- To classify the investors in terms of financial risk tolerance towards stock market
- To classify the investors in terms of the investor's awareness about the stock market
- To examine the factors influencing the stock selection by the retail investors

RESEARCH METHODOLOGY:

Sampling and Data Collection

It is a single cross-sectional survey design. The structured questionnaire has been designed to collect the data from retail investors from Rajkot City. The study was carried out in October and November 2015 with 50 respondents of diverse experience and demographic factors. The respondents were selected conveniently to fill up the questionnaires.

Data Analysis Tools

The cluster analysis technique has been used to classify the respondents into different risk tolerance categories. The independent sample t-test has been employed to identify the significant differences among various demographic factors.

RESULTS AND DISCUSSION

The following table shows the frequency of independent variables. It can be observed from the table 1 that most of the (83 %) male members are taking the interest in the stock market activities. In Rajkot, only 27 % investors are regularly trades on the stock market. In addition, most of the investors (48 %) are from the private organization.

The Chronbach's alpha has been calculated for each factor i.e. financial risk tolerance 0.68; self-awareness 0.81 and Stock Selection 0.83. It indicates the scale selected for the study is reliable for further analysis (see table 2).

To classify the investors in different groups, the K-means cluster analysis has been done. The analysis has produced two clusters (Table 3). The cluster has been categorized as the high-risk tolerance and low-risk tolerance investors. The F-values of the cluster analysis were significant at 1 % level of significance. The cluster analysis classified 44 % investors as the high-risk tolerance while 56 % investors as the low risk tolerance.

After classifying the categories for financial risk tolerance, the investors were classified based on the market awareness (Table 4). The cluster of the both categories has been categorized the investors with high market awareness and low market awareness. In each case, the F-values of the cluster analysis were significant at 1 % level of significance. The stock market awareness classification shows that the 62 % of the investors are highly aware of the stock market while 38 % investors are low aware of the stock market.

Table 5 shows the investors' stock selection criteria. The table reveals that in about 50 percent of cases, price, price earnings ratio, and earning per share remain the significant factors for the stock selection. While, investors relatively least influence (14.3 % + 28.6 % case) by the dividend paid history for the stock selection. The table also reveals that the investors are indifferent (26 %) in the case of 52 weeks

high/low price of the stocks. The overall results of the table show the factors mentioned in the table makes the significant influence while selecting a stock.

CONCLUSION

The object of this paper was to classify the investors into the categories of high-risk tolerance and low risk tolerance towards stock market. From the research, it can be concluded that the majority investors of Rajkot city seem low risk tolerance. On the contrary, the categories of investors' awareness reveal high stock market awareness. The more than 62 percent of respondents belong to the categories of high market awareness. It shows that the investors follow the newspaper, TV, seminars, workshop etc. to update their information about the stock market. The investors of Rajkot are irregular in term of trading on the stock market. The third objective of research i.e. factors influencing stock market selection decision, reveals the significance of price of the stock, earning per share, and price earnings ratio while selecting a stock. The sample size of the research is very small as far as the population of investors in the Rajkot city is concerned. The further research can be carried out with the large sample and varied demographic variables.

TABLES

Table 1. Independent variables

Independent Variables		**Frequency**	**Percentage**
Gender	Male	64	83.1
	Female	13	16.9
Education	Post-Graduation	53	68.8
	Graduation	24	31.2
Occupation	Private Organization	48	62.3
	Self-Employed	15	19.5
	Professional Practices	14	18.2
Frequency of Trading	Irregularly	41	53.2
	Weekly/Monthly	15	19.5
	Regularly	21	27.3

Table 2. Chronbach's alpha

Sr. No.	**Factors**	**No. of Items**	**Chronbach's alpha**
1	**Financial Risk Tolerance**	**5**	**0.68**
2	**Self-Awareness**	**6**	**0.81**
3	**Stock Selection**	**6**	**0.83**

Table 3. Cluster analysis of financial risk tolerance

Final Cluster Centers				
	Cluster		F-Value	P-value
	1 High Risk Tolerance	2 Low Risk Tolerance		
[Investing is too difficult to understand]	3	2	17.762	0.000
[I am more comfortable to put my money in a bank than in the stock market]	4	2	30.155	0.000

[When I think of the word "risk," the term "loss" comes to mind immediately.]			3	2	34.239	0.000
[Making money in stocks is the matter of luck]			3	2	25.621	0.000
[In terms of investment, safety is more important than returns]			4	3	43.922	0.000
Number of Cases in each Cluster						
Cluster	1	(44 %) 34.000				
	2	(56 %) 43.000				
Valid		77.000				

Table 4. Cluster analysis of Investors' Awareness

Final Cluster Centers				
	Cluster			
	1 High Market Awareness	2 Low Market Awareness	F-Value	P-Value
[I have a sufficient knowledge about stock market activities]	4	2	26.872	0.000
[I regularly follow the stock market news from financial news papers]	4	2	86.300	0.000
[I regularly follow the stock market news from TV channels]	4	2	126.414	0.000
[I regularly follow the stock market news from websites]	4	2	73.770	0.000
[I update my financial knowledge by attending relevant seminars, conferences, and workshops]	3	2	7.522	0.008
[I usually acknowledge the public information while trading]	4	2	13.677	0.000

Number of Cases in each Cluster		
Cluster	1	(62 %) 48.000
	2	(38 %) 29.000
Valid		77.000

Table 5. Factors influencing stock selection (In percentage)

Response	**Price**	**EPS**	**P/E**	**Dividend**	**Book Value**	**52 Weeks High/Low**
Least Influence	11.7	9.1	10.4	14.3	14.3	7.8
Influence	24.7	23.4	26.0	28.6	20.8	26.0
Neutral	15.6	19.5	16.9	23.4	24.7	26.0
Significantly Influence	29.9	24.7	32.5	18.2	23.4	22.1
Most Significantly Influence	18.2	23.4	14.3	15.6	16.9	18.2

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Experiencing Organizational Politics in Businesses and Institutes: Incidences & Interpretation in Indian Context

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ABSTRACT

As a working definition (derived from various dictionary meanings), human nature is a "behavioral, temperamental, emotional and mental tendencies that are assumed to be shared by all human beings." [9]'

Work place politics is the power used by the individual for their own benefit, without regards to their effect on organizational goal as well as on their own personal and professional life. When this occurs, the best decisions are not always made.

The study will do a phenomenological exploration of incidences of organizational politics experienced, 6 people to delineate the meanings created by them and juxtapose them against available frameworks of human nature to infer the additional dimensions of human nature.The study will thus contribute towards greater understanding of the gestalt of human nature and proposes to achieve this through phenomenological exploration of incidences of organizational politics

SUMMARY

As part of the proposed project work, an attempt will be made to study how the organization politics is interpreted by an individual , how their personal and organizational goal suffers.

Keywords: Keyword ' Human Nature', 'Organizational politics', 'Office Politics', 'Human Behavior', 'Organization Behavior'

INTRODUCTION

Human nature defines the identity of every individual. Every human being exhibits a characteristic set of behavioral patterns that largely depend on the individual'snature and personality.Individuals differ from each other largely because of the way they process their thoughts. An individual's culture, background, religion, education, and environment all have a great impact on his/her general attitude and social behavior. However, there are certain distinguishing behavioral characteristics that are independent of the individual's culture and environment. Recent research in genetics, evolutionary biology, and cultural anthropology suggest a complex interaction between genetically inherited factors ("nature") and socially acquired factors ("nurture"). The basic driving forces shared with other primates include food, the need for procreation, security, play, and social status [1].

When an individual inappropriately uses his/her authority at the workplacefor personal gains, without taking into account its adverse effects on the business goals, it amounts to workplacepolitics, which not only ruins the individual's professional life, but also ruins his/her family life in the long run.Such a negative attitude (e.g., refraining from information sharing, non-cooperation, etc.) is not ideal for the growth of the individual or business. Virtually all human resource decisions, right from employee promotion to new recruitment can be adversely affected by workplacepolitics and hidden agendas. When this occurs, the decision-making capacity of the concerned authorities in the impacted organizationgets adversely affected. The more we knowabout organizational politics, the easier it is to determine why or how workplacepolitics enters the human resource decision-making process.

It is a known fact that employees often competewith each other in order to achieve certain common goals(or to get rewarded), but sometimes their personal interestsdo not line up with the organization's short-term and long-term objectives. Organizational politics is extremely destructive for any organization and is also a major source of work-related stress. Workplacepolitics includes the use of subversive

methods to promote personal agenda.It not only jeopardizes the organization's goals, but also proves detrimental to the progress of the concerned employees. Individuals involved in workplacepolitics inappropriately blame others for their own mistakes, threaten their co-workers, and also resort to information filtering/distortion, non-cooperation, reprisal, dishonesty, and obstructionism.Often, political behaviour and manoeuvring within an organization is caused by uncertainty (e.g., unclear objectives, poorly defined decisions, extreme competition,rapid change, unstructured organization,inappropriate hiring, lack of qualification, lack of transparency, etc.) [2].

Gandz and Murray[3] noted that most people agree with the statements "successful executives must be great politicians" and "the existence of workplace politics is common in most organizations." Robert Aunger & Valerie Curtis*Biology and Philosophy*[11] argues that in biological science evolutionary natural kinds are the adaption which can be identified by their history of selection human brains."Neta C. Crawford[4]" in her article "Human nature and world politics" argue that their theories of world politics are rooted in specific assumptions about human nature, that they do not trust others and decision makers always seeks to promote their self-interest. Organization politics, Job attitudes and work outcomes in this article "EranVigoda"[5] argue on the understanding of employees' reaction to organizational politics the relationship between perception of organizational politics , job attitudes and several other work outcomes was examined. Drory(1993)[7] founded that perception of politics were negatively related to job satisfaction and organizational commitment and the damaging effect is more on lower side that on higher side because the employees on the lower side those who do not have influence suffer more and this also leads to the frustration and negative work environment."The Relation of organizational politics and support to work behaviors attitude and stress" "Russell Crapanzano , John C Howes , Alicia A Grandey and Paul Toth" [6]he tested on two sample that was part time and full time employees , in their research their major finding was withdrawal behavior , turnover , job satisfaction and commitment and stress, variables , tension , fatigue, burnout they found both was on the same level in part time as well as full time employees."BolanleOgungbamila" in "Perception of organizational politics and job related negative emotions as predictors of workplace among employees of distressed bank" [7] he extended his literature on workplace incivility by investigating the extent to which perception of organizational politics and job related negative emotions predicted workplace incivility. He focused on how the workplace politics takes place because of an employee reaction to uncertainty , unfavorable politics manipulation and negative workplace takes place due to this and how the management should react and should effectively manage employees negative emotions and perception of organizational politics. "Joseph M Goodman, W Randy , Charles M Carson" [8] they argue on the interaction of perceived accountability on the politics perception – job stress relationship their findings indicated on how the individual who have higher responsibility perceives more stress than the employees on the lower responsibilities.Prof. Roselyne W Gakure&Dr,

George Orwa[17] argue in their article "The impact of organizational politics on the effectiveness of development in the Kenya civil service" on how Organizational Politics affects effectiveness of management development in the civil service of Kenya their findings agreed with the findings by Bacharach(2005) that politics is essential skill in manager who wish to get things done and with Kim (2004) that politics and power are critical factor in Organizational Process. Valle &Perrewe (2000) argued that management development should include political skills because organization politics can build or destroy careers. The art of how to get them on your side is crucial at any rank and has human resource management implications (Vigoda, 2003) the study concluded that managers in civil service are aware of the political environment they operate in but did not think it had much impact on their work which is surprising considering that Organizational Politics is natural phenomenon in any organization.Nathanael S Campbell,Sara Jansen Perry,Carl P MaertzJr,David G Allen,Rodger W Griffeth,08/07/2014 , All you need is … resources: The effects of justice and support on burnout and turnover[15] in their study They propose and test a comprehensive model of burnout, as influenced by justice and support, and as it impacts the turnover process. Theoretical contributions and implications in the areas of justice, burnout, and turnover are discussed.David A. Buchanan, You Stab My Back, I'll Stab Yours: Management Experience and Perceptions of Organization Political Behaviour[16]In exploring their experience and perceptions of organization politics. Political behaviour appeared to be common. Most managers viewed political behaviour as ethical and necessary, and aspects of organizational effectiveness, change, resourcing and reputation were attributed to political tactics. EranVigoda-Gadot, Leadership style, 0rganizational,politics, and employees' performance. An empirical examination of two competing models.[12]They studied the perceptions of politics among public sector employees as a possible mediator between the supervisor's leadership style and formal and informal aspects of employees' performance.EranVigoda-Gadot and YinnonDryzin-Amit, Organizational politics, leadership and performance in modern public worksites: A theoretical framework[13]in this article the author focuses on how, much attention has been given to theories of leadership in organizations. Leadership is considered to have a major influence on the performance of organizations,managers and employees. Early theories tried to define effective leadership styles (democratic or autocratic, socially oriented or target oriented etc.).Bronston T. Mayes[1] and Robert W. Allen[2],Toward A Definition of Organizational Politics[14]In this article the authors examine political behaviour in organizations and attempt to define the concept of organizational politics. They note that certain criteria have to be met in order for behaviour to be considered political such as intention and a desire to influence outcomes.

MATERIALS AND METHODS

Objectives are as follows:

1. To identify significant incidences of organizational politics as experienced by employees.
2. To understand the meanings of such incidences.

Methodology

The proposed study is exploratory in nature, keeping in mind the purpose and objectives of the study, the following multi method qualitative study data collection methods and sampling techniques have been chosen,

Data Required	Data Source	Data Collection
Significant incidences of organizational politics	Employee	Phenomenological Interview
Meanings of such incidence	Employee	Phenomenological Interview

Sampling unit : Employees

Sample size : 6 Individuals

Sampling procedure: Convenience

RESULTS AND DISCUSSION

Dimensions of Human Nature as emerging from the field of Organizational politics will be studied.

The researcher has asked some questions on phenomena related to organization politicsso that researcher can best analyze how the organization politics has been interpreted by the employee. However it is very difficult to draw conclusion from one single interview but what researcher have found in this interview is that there was communication gap in the organization, poor reporting structure, employees were placed on wrong position, Improper workload distribution, Un-defined roles and responsibility, Lack of information from both the side i.e. management and employee, Rigidity towards changes and fear of losing power. Transcript of the interview is mentioned below in the appendix. For brief highlights of data more samples need to be collected then only researcher would be able to conclude proper conclusion. Hence this research is in process.

CONCLUSION

Phenomenology, most simply stated, is the study of conscious phenomena: that is, an analysis of the way in which things or experiences show themselves. The term "phenomena" is derived from the Greek verb, which means to show oneself or to appear.

Phenomenology can also be described as a qualitative research technique where we can analyse the meaning of human experience, it is the search for essences that cannot be revealed by ordinary

experiences. The point of phenomenology is to get straight to the pure and unencumbered vision of what an experience essentially is.

According to the Internet Encyclopedia of philosophy Phenomenology refers to the way of doing philosophy that is more or less closely related to the corresponding movement phenomenology utilizes a distinctive method to study the structural features of experience and of the things as experiences.

In intentional analysis phenomenology has to place before its own eyes as instances certain pure conscious events, to bring these to complete clearness, and within this zone of clearness to subject them to analysis and the apprehension of their essence, to follow up the essential connections that can be clearly understood, to grasp what is momentarily perceived in faithful conceptual expressions, of which the meaning is prescribed purely by the objective perceived or in some way transparently understood.

Thus, intentionality refers to the total meaning of the object, which is always more than what is given in the perception of a single profile or perspective. Intentionality is the direction and internal shape of experience or consciousness.

Thereare three fundamental components in a phenomenological research design:

1. Determiningthelimitsofwhatandwhoistobeinvestigated.
2. Collection of data.
3. Phenomenological analysisofthedata.

Phenomenological researcher tend to choose the interview due to their interest in the meaning of phenomenon as it is lived by the other subject and collecting data solely from oneself would be more of a philosophical endeavour.

However the phenomenological researcher are interested in the subjectivity of other persons and this it seems logical that we would get a description of such subjectivity and collecting description from other is also an attempt at a discovery of human scientific meaning of a particular phenomenon.

The idea is to identify the influences likely to be most important, analyze how people are apt to respond to them, and revise them if necessary so that they create the right kinds working environment at office and in home, and will be able to enjoy their work .The researcher thus will investigated the influence of perception of organizational politics on employee's.

Since the topic is about Exploring "Human Nature as reflected in meanings given to incidences of organizational politics" and it relates to exploring human nature, qualitative research method is the most useful method hence I have opted for this method, also the topic require me to enquire into meanings

given to incidences of the organizational politics, hence Phenomenology is the most appropriate methodology.

The study would thus contribute towards greater understanding of the gestalt of human nature and proposes to achieve this through phenomenological exploration of incidences of organizational politics

ACKNOWLEDGEMENT

I would like to show my great gratitude to my guide Dr.RaashidSaiyed for his constant support and his valuable guidance. Sincere thanks to Dr. Nikhil Gokhale(Dean Doctoral Research RK University) for always guiding and motivating me to do something innovative.

One person who actually inspired me to write this paper is Dr.RanjitGoswami (Vice Chancellor, RK University). I would like to thank him for his one sentence conversation which motivated me to write.

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APPENDIX

Transcript of the 1st Interview

Interviewer: Researcher

Interviewee: Asst. store Manager, Store Manager 1 year, 2 Years Component store 13 year Front office manager. Total 16 years.

Interview Setting: Interview conducted in the house of researcher in Rajkot the Interview was conducted on 15/12/15 @22.44 pm

(Start of Interview)

Interviewer: Please describe one incidence of your life of organizational politics which has impacted your life a lot.

Interviewee: In 2013 when I was working as front office manager I was handling whole communication department of my organization, that time only one new person was appointment he wanted the same position which I was handling, he went to the GM and said that the communication department is the part of EDP department and we want that department under us. He told this directly to GM of the company

and said same thing the GM told me, that you handover your department to him(new joined person). I was handling a big network and I was reporting to Director. Hence I told him that I am handling this department as per the instruction of our Director if he says I will handover. They went to the director miscommunicated the things that I am refusing to handover the department. Director called me and said go to the GM and handover the department I was performing very well in that department and management told me that my performance is not good in communication department hence I have to quit that department.

Interviewer : How many years you worked with communication department?

Interviewee: 13 years

Interviewer: In the same company?

Interviewee: Yes, whole department was under me. In 2013 I was transferred in consumable stores as Asst.Manager consumables.

Interviewer: You had knowledge of consumable?

Interviewee: I went earlier for 6-7 times to support them if anybody in on leave as per the instruction of Director so I was having only basic knowledge of that department i.e. in 2000 but that time we were not using any computerized system we doing only paper work but this time when I was transferred there was huge gap whole system was changed and computerized and there was very less paperwork. Hence I took 6 months to learn that system.

Interviewer: Did that director called you to for the clarification?

Interviewee: No, actually I am very much emotionally attached with the Director for me whatever he says it is final; he called me and said that from tomorrow I am transferring you to consumable store. I said ok, and till today I have not asked him anything neither he has asked me any clarification.

Interviewer : How you learnt the things in new department?

Interviewee: That department people helped me out.

Interviewer: But you went on the designation of Asst.Manager?

Interviewee: That designation was because I was working with that company since last 13 years. But since I have to get acquainted to new system I have learned from my assistants.

Interviewer: How did you manage to do that?

Interviewee: I used to keep watching my assistants and this is how I learnt and gained the knowledge now I know complete system.

Interviewer: Are you still working with the same department i.e. consumable

Interviewee: No, now I am transferred to component store. 11 Nov 2014 I was again transferred because of manpower shortage. And they wanted somebody who is good in operating computers and can manage people; so I was transferred.

Interviewer: But you were not having knowledge of that department (component) too.

Interviewee: I am now dealing with vendors and handling staff.

Interviewer: Now you have been transferred from component to consumable so there would be vacancy in consumable?

Interviewee: No manpower was already in excess in that department**.**

Interviewer: Why HR has not checked this that there is excess manpower?

Interviewee: No earlier one duty to each staff was assigned now they have transferred my duties to existing staff.

Interviewer: Now you have knowledge of three departments i.e. communication, consumables, component

Interviewee: No now I have again started handling communication. Now officially director has made committee of 4 members because since 2 years we have not negotiated with any company since I was transferred. He called me regarding this, I told him that you only told me that I have better person than you in communication department. I have handed over all the charges and also explained him the functioning of the department, rest he should have taken care of. Now if he is not thinking of negotiation in communication department than it is his fault. We have one consultant in the company they work for audit he called me once for negotiation in 2014 and have made one proposal changed plan and saved 40000/- and forwarded the proposal to the management but management didn't gave any response the consultant he took 2-3 times follow up then he stopped taking follow up also.

Interviewer: No but the department which was taken from you and handed over to that person he didn't do anything?

Interviewee: He was just following the GM and EDP manager he was not using his brain and I was working independently hence I was able to think on this. But then director called me and said why I have not done this, I replied you took away this department from me and transferred me in component so how can I interfere in other department, he replied no you have to look all this also. He said since last year there is no negotiation, I said who told you this I have given proposal last year only this is the wrong document you are holding. He said I have not received anything.

Interviewer: Why the proposal which you have prepared was not given to the management?

Interviewee: No actually they decided to have meeting before giving to management with GM but that meeting itself didn't happened and the project failed.

Interviewer: That person is an employee or a consultant?

Interviewee: Consultant with team of 15 they do process audit.

Interviewer: But that person was not from the communication department right?

Interviewee : No he is CA

Interviewer: How he can work for communication?

Interviewee: He took my help and did, but even though director has refused I have done those efforts, hence when he asked him I told him that I have done it but the managers have not put in front of you. However I have already negotiated with the companies for reduction of the prices but since you took the rights from me to work in that department I could not process it, I was waiting for the approval. He asked me how much loses we made because of this I answered 480000/- loss. He said now what, I said you tell..

Interviewer: Now how you will do?

Interviewee: I will handle that from my existing department i.e. component. Now I am handling both department.

Interviewer: So now you are handling both the department how?

Interviewee: Now I have committee of 4 members which includes the advisor of the company the CA and the documentation part will be done by the person who is sitting in my place.

Interviewer: Please explain how the advisor, CA, and you three people are going to handle communication department whereas earlier it was handled by you alone?

Interviewee: But since company told to take 3 more, so I took.

Interviewee: Ya we also want some responsible person to answer the parties so we kept them.

Interviewer: Now since you are handing 2 department do you think you component department must be suffering?

Interviewee: No

Interviewer: How?

Interviewee: Because I have command on software and stay back complete m work and then leave.

Interviewer: You have worked in 3 departments' communication, component, and consumables what do you think you would have performed better in which department?

Interviewee: Communication

Interviewer: One year you learnt consumable, that was loss to company then again you were transferred to component that means it was total loss of 2 years and 480000/- , so now that director has finally handed over that department to you or it is still pending.

Interviewee: No only negotiation part is given to me.

Interviewer: What do you think all this what ever happened with you what could be the reason behind that?

Interviewee: Ego, they did not like my hold on the department, hierarchy

Interviewer: The person who took charges from you, which department he was handling earlier

Interviewee: EDP they convince in this way that since EDP is communication centre we will keep communication department under EDP.

Interviewer: What do you think that what exactly that EDP head wanted from you?

Interviewee: He just came and said give me all the network you are handling

Interviewer: But why suddenly he did this?

Interviewee: He may have thought that changed which he wanted to do will not be possible if I stay.

Interviewer: But what changes he was demanding?

Interviewee: I do not know, because when he came to take charges he himself was new.

Interviewee: He was good in networking and related to director, one day he came and told that you have to hand over your department to me.

Interviewer: Who told him to do this?

Interviewee: The GM

Interviewer: You didn't asked him why?

Interviewee: He didn't tell anything he just said this should come in my department and I want it.

Interviewer: Have you asked him how the communication department comes in EDP?

Interviewee: Yes I asked, he said all the communication will be computerized and you don't keep any computerized record hence hand over this department to me.

Interviewee: I refused by saying that I reporting to Director.

Interviewer: But what he wanted to computerized?

Interviewee: No Idea, Just wanted to show.

Interviewer: still I have not understood how he wanted to link communication with EDP?

Interviewee: No he just wanted some load in his department and to justify that he said he has to link both department and he wants this department. He wanted to check the bills, but see if the management is not having any problem and the user is using as per the limit then why should we check the detail bills, if check bills if management says.

But Director instead of calling me for this he called the GM and said that see these bill are so high and you don't see, now what idea GM will have on this, he came and instructed me I gave him consolidated data. He started shouting me that why I don't keep track of value added service I told him that see users should be instructed earlier on this now since you have not instructed them anything you cannot take action. I said you tell I will do. He took various type of report from me. Then GM discussed with EDP head , EDP head told I will handle this department ask communication person to give me.

Interviewer: So he just wanted the department,

Interviewee: Ya he came to me earlier also asking for department but I refused and then he did all this.

Interviewer: Management have not given thought on this that how an EDP person can handle communication department?

Interviewee: No

Interviewer: They didn't asked him also that why he want communication department?

Interviewee: No because he presented in this way that communication department should be linked with EDP department. So that misuse can be stopped and we will also be able to get quick service.

Till today whenever management have some problem their first call comes to me only. Because I have a very good relation with the suppliers and if any problems come I have that power to get my work done through the heads also. This is because I have maintained good relationship with them.

Interviewer: Management never called you for clarification?

Interviewee: No the very next day another director called me and instructed me that since the other director has given this decision I cannot change his decision but you keep working for this department from back.

Interviewer: when your department was shifted they have not thought of that it is communication department and entry in the computer will not be sufficient?

Interviewee: He told me to support that department but he deleted all the correspondence

Interviewer: EDP people have not restored it?

Interviewee: it was done by them only and not only this they convinced management so well that I have setting with supplier. Just because that supplier listens me and quick action is taken by them.

Interviewer: But that was because of your relation?

Interviewee: Ya but who will make them understand see all this depend on the way you convince your supplier and how you maintain relations with them

Interviewer: I am still not finding and reason why they removed you from your department.

Interviewee: I even told that I will handle both the department it was just a politics. Because I was handling good PR and also communication and I was good with customers.

Interviewer: But it was good for company?

Interviewee: Ya and I was handling PR , Vehicle , Hotels , Communication but they convinced management that I have setting in all the things.

Interviewer: When you knew that these people are doing this miscommunication, then why you have not gone to director for clarification.

Interviewee: yes I knew everything, but why should I give clarification when he should have asked this, he didn't asked me then why should I give clarification. I was not wrong I would have given clarification if I would have done wrong. I was just performing my duty. If you ask police not to fire criminals and if he escapes then you say why you have not fired him then this will definitely not work. They were saying that I was leaking information.

Interviewer: Who gave this information to him?

Interviewee: There are always many.

Interviewer: You never thought that you should meet him and clarify all these?

Interviewee: No if you are attached emotionally to someone you can't, I have been working with that company when I was very small since 1999. I was directly reporting to him , he was also having lot of faith on me so I thought if he is thinking wrong for me then it is his problem. Even if he says you sit on gate I will do that.

Interviewer: Till now he has not realised this?

Interviewee: No and I am not going to do that. I regularly meet him for some other work but he has never asked this. Because if I do so he will feel I might have done this.

Interviewer: What do you think that if in this whole process if organization structure, reporting structure would have been good then this would have not happened?

Interviewee: yes they would have save 480000/- which would have covered my salary.

Interviewer: you were reporting to him then why this problem occurred?

Interviewee: He heard bad thing and concluded.

Interviewer: He has heard that don't you think he would have checked this information once?

Interviewee: He would have done this, but he did not.

Interviewer: This is 2 years old incidence, what was your metal situation that time?

Interviewee: I was transferred to new department I was learning they were also guiding me, I did mistakes also for which I had to listen from my boss, I was taking it positively but my mental situation was that if I get good opportunity I will leave the company. I gave many interviewees I was even selected but I could not join because my father was suffering from cancer and I was getting medical help from ESIC. And if I would have left that then new company would have taken long time to start this and my father was on last stage. I was not having time.

Interviewer: I was thinking that you worked for so many years with that company and department and one day you are transferred. It's like changing house and mentally you get disturbed with that.

Interviewee: I was disturbed that is way per day I use to call you thrice for job. Even in less salary why I was doing that because this has affected my image In front of management and even I was thinking I have worked with loyalty then how these people can think in this way but because of my father I was stuck with that company. That one year was very difficult for me to work there. One fine day you are transferred to some other department without any intimation and you also do not know new role and responsibility I was not knowing what to do.

Interviewer: How you use to manage this stress level because at home your father was also not well?

Interviewee: No I never carry this at home.

Interviewee: No I keep personal and professional separate

Interviewer: Then why you use to shout?

Interviewee: I was still not able to shout more because my father use to say keep office tension in office. Then where should I go.

Interviewer: Even if we say that we keep personal and professional life separate still that is always there in back of mind.

Interviewee: I have learned from all senior people like you, because you people have lot of tension but still you all manage your home problems also very well, hence I use to think same and managed. Because if you have tension at home then it is not the problem of your family then why should they suffer?

Interviewer: But you use to shout?

Interviewee: that was my nature.

Interviewee : I use to share all the tension with my mom, she use to council me. She always motivated me to work hard. But still I was not able to share everything with my mom because she was also in my father's tension.

Interviewer: You never share with you wife?

Interviewee: No, because if wife is working and educated and have working experience in same industries then sharing the problem can help a person because you get the solution as your partner is from the same area, but if not then share does not help you because she is totally unaware of that.

Interviewer: Now in this whole process what do you think that company should have to avoid such stress and problem because you have lot of experience and you must have experienced lot of organizational politics incidences

Interviewee: Right person should be placed in right place and should take care of his department and staff then only employee can work and perform, this will also reduce rejection and increase productivity.

Interviewer: How should management work for as an employee what do you think?

Interviewee: Managers should report directly to them, and if there is problem then they should find the solution first not the action and still if it is not possible then take action. Now what is happening nobody analyses the problem that why and how it happened who was responsible they directly take action. Nobody even give a thought that if the person responsible has taken some action out of the way then why he has done that.

Interviewer: Do the manager meet regularly?

Interviewee: Daily

Interviewer: No I am not talking about daily I am talking about monthly review meeting.

Interviewee: No

Interviewer: Then how management come to know about the performance.

Interviewee: No they have given charges to manager they gather for daily meeting (all department head with GM & Project head) they discuss their daily issue.

Interviewer: But that meeting is for routine problem?

Interviewee: I went to director and asked this question what is the meaning of plant head, he said you tell I said all maintenance follow up , production follow up you are taking then what is the use of plant head. He still said I have not understood then I asked him what is the meaning of chairmen he said you tell , I explained your work is to get the work done from plant head**.**

Interviewer: I think reporting structure is not proper in that company.

Interviewee : Exactly if this is proper then everything can be managed. Suppose for example if I would have been handling communication department and if director or anybody ask for any issue in the mobile then I have immediate answer if not I even have the capability to get the work done. But if he ask the same question to some other person of different department he will not be able to answer what he will do that since director level person is asking this question he will just try to answer that question.

Interviewer: That means there is not assigned responsibility and author assigned and poor organization structure.

Interviewee: No one more thing if any old employee is holding one position then he/she in any situation don't leave that position because if suppose you are not educated and you have only experience then you can only perform in the area of your experience but if you are qualified then you can perform.Right person is not in right department for eg: our GM QC has been given the designation of Head HR now how will he justify the position. Besides this managerial skills are also essential.

Interviewer: How does and QC person can become HR?

Interviewee: I have still not understood.

Interviewer: How he manages to do that

Interviewee: Only report is done to him for problem he ask the solution from them and work.

Interviewer: Now still I have not understood that a QC manager has been assigned job of HR manager also how he is able to justify both the role , because both the role is very difficult.

Interviewee: No only in case of problem team approaches him

Interviewer: How then the HR department must not be working, Management do not understand this?

Interviewee: No

Interviewer: who gave me this position

Interviewee: Management

Interviewer: Management do not think this should be rectified

Interviewee: who will do that …..but since the good HR is not available in company employees are also suffering there is nobody to help the employee which is really bad.

Interviewer: But you said Asst.Manager is their

Interviewee: He don't know anything.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

(Proceedings available for download at rku.ac.in/icre)

RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

Analysis of the Social & Economic impact of Self Help Group in Junagadh City

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ABSTRACT

The SHG Bank linkage programme plays an important role for the development of the rural poor in India. Self Help Group is a small team of poor people, which majorly contains group of all women or all men, which is formed voluntary by the members from the same social and economic background. These groups promote small amount of savings among its members and such savings are accumulated and deposited in a bank in the name of SHG as collective fund. Here an attempt is made is to analyze the impact of Self-Help-Groups (SHGs) on social and economic development of the respondents of Junagadh. The Primary data were collected from field survey with the help of filled in questionnaire and direct interview method. The sample size is selected 50 Self Help Groups with 5 members (50*5=250) have been selected randomly.

SUMMARY

Majority of the women are having primary education as the highest qualification and they got various types of social advantages like as Increased Communication ability, increased

confidence level, Respect from the society, Respect from the family members and Enhanced Household consumption of products.

Keywords: Self Help Group (SHG), Financial Inclusion, Socio-economic development

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INTRODUCTION

Self Help Group is a small union of poor people generally including 10 to 20 members, which is formed voluntarily by the members from the same social and economic background. They can be neighbors, relatives etc. The aim of the groups is to promote small savings among its members and such savings are accumulated and deposited in a bank in the name of SHG as collective fund. This collective fund is accumulated by contribution of pre determined and agreed small amount of savings on regular basis (generally monthly) by each member of the group. The accumulated fund is then provided to the needy members as a loan on nominal interest basis. After six months of formation, if their functioning is found to be satisfactory they become eligible to avail various government schemes. These groups get revolving fund from the government which is again utilized to provide such fund as a loan to needy members, which they repay in the installments with subsidized interest rates.

The concept of SHGcame from the Gramin Bank of Bangladesh, which was found by the economist, Prof. Mohammed Yunus of Chittagong University in the Year 1975. It aimed to provide micro-finance to 87 rural women. Micro-finance has been established as one of the most powerful instrument to eliminate povertyIn Bangladesh. The SHGs in India were formed by Mysore Resettlement and Development Agency (MYRADA). It was an NGO and in 1985 due to breakdown of the large cooperatives organized by it. By 1986–87, there were nearly 300 SHGs in its projects. MYRADA then approached NABARD for an action research project on self-help groups which funded the research.

NABARD, in association with the Reserve Bank of India (RBI), Commercial Banks and NGOs, launched a pilot project in 1991–92. It was establishedto create abridge between SHGs with banks.The journey of the Self Help Group – Bank Linkage Programme which started from linking a pilot project of 500 SHGs of rural poor two decades ago, is now boasting the world's largest microfinance initiatives with over 7.4 million SHGs. These SHGs represents 97 million rural households, which are directly becoming part of this great movement.

Functions of SHGs:

SHGs promote:

Savings habit of the members.

Internal lending to the needy members at nominal interest rate.

Engaging the members towards economic activities which help them to raise their economic background as well as provide income generating opportunities.

Regular meetings and discussion of problems of the members and solving them.

Highlights of the SHG-Bank Linkage Programme 2013-14:

Total SHGs registered are now 74.30 lakh amounting to SHG savings with banks Rs. 9897.42 crores as on March 31, 2014

With over 84% of these being all women groups, the poor rural women in India now controls a financial business with turnover of nearly Rs. 1,00,000 crore (deposits + credit)

The extent of savings by the group members still has lower average of over Rs. 13,300 per SHG as on 31.3.2014.

After presuming that 70% of savings would have been utilized into internal lending, the gross savings by all members of SHGs works out to approximately Rs. 44000 only or less than Rs. 3500 per member.

Review of Literature:

Rahul Sarania (February 2015) attempted to examine the effectiveness of SHGs in economic empowerment of women in Assam. It revealed that majority of the respondent's income, employment days and amount of savings increased in the post-SHG situation as compared to pre-SHG situation. Thus the study concluded that SHGs have been playing a vital role in the empowerment of rural women in the study area. Dr.A.Sundaram (2012) examined the impact of Self-help Group in Socio-economic development of India. He gave number of suggestions in his study for promotion of Financial Inclusion, designing of NRLM (National Rural Livelihood Mission), and small and medium enterprises which would lead to the overall development of SHGs in India. SazzadParwez (2014) examined and assessed the role of SHGs in rural development in Jharkhand. He made suggestions for effective and efficient implementation of financial inclusion through SHGs covering socio-economic aspects of Jharkhand state. Dr. Sushil Kumar Mehta (2011) suggest that SHG – Bank Linkage Programme has shown

remarkable improvement in the access to financial services for the rural poor and has sizeable positive impact on the socioeconomic conditions of the members and ultimately contributed towards the reduction of poverty of SHG members and their households.

Objectives:

The present study covers the following aspects relating to the Self Help Groups of Junagadh city

- To study the socio-economic background of respondents.
- To study the impact of SHGs on women in the Junagadh.
- To study the benefits received by the respondent through SHG.
- To examine the Pre SHGs & Post SHGs income status of SHG members

Research Methodology:

The present study is descriptive in nature covering selected SHGs of Junagadh city. The selection of SHGs has been done by using convenience sampling technique. A sample of 50 SHGs consisting of 250 members has been taken for the present study. To judge the impact of SHG, the respondents have been asked questions on various aspects before and after joining SHG.

Data Analysis:

For the analysis & interpretation of data, statistical methods like CHI Square and other tools like Frequency distribution and cross tabulation has been made through Statistical Package for Social Science 22 Software. Frequency Distribution is used to obtain counts of number of responses. Cross Tabulation is used to Study the relationship between different variables and Chi-Square is used to test the association of the variables which are presented in a cross tabulation. Paired T- Test is used to examine the impact on level of income of the respondents before SHGs and after joining SHGs.

Variables used in the Study:

The socio-economic profile is studied in terms of five variables: the age of SHG members, education, marital status, caste and period of joining of the SHG members. In order to find the impact of SHG on members' economic empowerment the main variable considered is annual income and amount of savings.

Results & Findings:

Socio-Economic Background of the respondents:

62% of the respondents were of age 30 to 45 years while 30 to 45 years while 26% of them belong to age group of less than 30 years. Only 12% of the respondents were of more than 45 years of age. 82% of the respondents were having maximum primary education. 89% of the respondents were married 60% belonged to OBC category. 40% of the respondents joined SHG since 2012 while 58% of them joined post 2012. 54% of the respondents availed loan facility from the SHG.

When asked to respondents about what helped them to join SHG, 46% of them responded that NGO & Government Employees promoted them to join SHG, while 26% joined due to family members and friends. This shows the efforts by the government to promote Financial Inclusion of the respondents.

Table 1Socio Economic Benefits after joining SHG

	Frequency	Percent	Cumulative Percent
Increased Communication ability	1	.4	.4
Increased confidence level	7	2.8	3.2
Increased Respect from the Society	36	14.4	17.6
Increased Respect from the family members	44	17.6	35.2
Enhanced Household Consumption of Products	162	64.8	100.0
Total	250	100.0	

Table 2 Opinion of the Respondents about their SHG

Problems faced after joining SHG	Strongly disagree	Disagree	Neutral	Agree	Strongly agree	Remarks
Lack of training programmes	20	183	36	11	0	81.2% Respondents are satisfied with training programmes

Formation of group	42	165	40	3	0	82.8% Respondents did not face any problem in group formation
Higher interest rates for loans	164	54	23	9	0	87.2% Respondents do not agree that interest on loan is higher. This encourages their attraction for taking loan through SHG.
Improper utilization of funds	175	61	13	1	0	94.4% Respondents feel that the fund accumulated in the SHG is utilized in proper manner.
Inequality in issuing loan	193	46	11	0	0	None of the respondents felt inequality in loan issuance. All get equal chance to access credit facility in the group.
Misunderstanding among group members	222	23	5	0	0	None of the respondents faced any kind of misunderstanding among the group members which shows unity in the group.

From the above data, we can say that joining SHG not only gives satisfaction to the respondents, but it also encourages social inclusion and financial inclusion of the members.

Table 3 Cross tabulation

Education * Reasons for Joining SHGs

	Reasons for Joining SHGs					
	To meet Household Expenditures	To promote saving	To raise status in Society	To Promote income Generating activities	To get loan	Total
Uneducated	31	33	3	1	1	69

Primary	55	62	6	8	4	135
Secondary	8	27	0	2	0	37
Higher Secondary	2	1	0	1	0	4
Collage	1	3	1	0	0	5
Total	97	126	10	12	5	250

From the above table we can conclude that 89.2% of the respondents joined SHG to meet household expenditure and to promote savings. This implies that the respondents have gradually developed savings habits, which helps them to raise their economic background and standard of living.

Chi-square tests:

Chi-square test is conducted to check whether there is any association between education level of the respondents and reason for joining a SHG.

H_0: There is no association between education and various reasons to joining SHG.

H_1: There is association between education and various reasons to joining SHG.

Table 4 Chi-Square Tests

	Value	df	Asymp. Sig. (2 sided)
Pearson Chi-Square	21.386[a]	16	.164
N	250		

Above table suggests that there is no association between education & various reasons for joining SHGs. Hence it can be concluded that majority of the respondents being educationally backward are attracted to join SHG.

Paired t test:

Paired t test is conducted to check the impact of SHG on income level before and after joining SHG.

$H_{0:}$ There is no significant difference between the mean values of Level of income before joining SHG & Level of income after joining SHG.

H_1: There is a significant difference between the mean values of Level of income before joining SHG & Level of income after joining SHG

Paired t tests:

TABLE 5 - Paired Sample t Test

	Paired Differences					t	Df	Sig. (2-tailed)
	Mean	Standard Deviation	Standard Error Mean	95% Confidence Interval				
Pair 1 Income Level before Joining SHGs - Income Level after Joining SHGs	-.868	.861	.054	-.975	-.761	-15.938	249	.000

The impact of SHG on income generation of members is tested statistically by paired t-test. It can be derived from the above output that the t value is significant at 5 per cent significance level. It implies that the level of income of the respondents has significantly improved after they joined the SHG which shows the economic impact.

Conclusion:

Above findings suggests that SHG movement has significantly played a vital role in enhancing the Socio-economic development of the members. This contributes to the development of the social upliftment of the group members and also towards the development of financial inclusion of the members.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

(Proceedings available for download at rku.ac.in/icre)

RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

A Comparative Study of Satisfaction of Management Educators of Gujarat State

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ABSTRACT

Higher education grows rapidly in last few decades in India. Management education also emerged as an important discipline and due to these the need of efficient and effective management educators improves drastically. To attain and retain efficient management educators becomes a big challenge for all the management institutes. The researcher has decide to measure satisfaction level of management educators working in private universities and affiliated colleges to state universities of Gujarat. The researcher has chosen management educators from private universities and affiliated colleges to state universities for the study purpose. He has used self administered questionnaire for the study. He used convenience sampling method having response from 185 respondents for the study. The outcomes of research shows the satisfaction level of management educators working in private universities and affiliated colleges to state universities in Gujarat.

SUMMARY

This research was carried out with an aim of doing a comparative study of satisfaction of management educators of Gujarat State.

Keywords: Higher Education , Management Educators,Satisfaction, Private Universities, Affiliated Colleges to State universities.

INTRODUCTION

Job satisfaction has been defined as **"the extent to which people like or dislike their jobs."** (Spector, 1997, p.2). This definition suggests that job satisfaction is a general or global view of employees about their jobs. [1] Job satisfaction states that are the employees enjoying their jobs? Performing it well, and get fairreward for their job. A numerous researches have been carried out for the satisfaction of educators all over the world. In many researches it was found that **"Satisfied employees can bring the success for the organization".** So, every universities/Institutes want to find that if their educators are happy or not? Job satisfaction is a tool to measure if their educators are happy or not. Every university/institute wants that their educators be a part of the growth and success of their university/institute.By keeping these requirements in mind the researcher had decided to do a specific study for the management educators to measure their job satisfaction level. Due to these reason the researcher decided to compare job satisfaction of management educators of Gujarat State.

REVIEW OF LITERATURE

A number of researches have been carried out with an aim of measuring private universities and public universities in Gujarat. The researchers have done some literature review on the dimension of satisfaction level of educators working in private universities and public universities.

According to Bakhshi, A., Sharma, S., Kumar, K., & Sharma, A. (2008) did study at University of Jammu and he has revealed that the level of job satisfaction of faculty member working at private college and government colleges are quite different. Government college faculty members have higher job satisfaction then private college faculty members. They also found that there is not any major dissimilarity in life satisfaction scores of government college faculty members and private college faculty members. They got results that there exist a positive correlation between life satisfaction and job contentment.[2]

According to M I Rehman, R Parveen (2008) on job satisfaction a study among public and private sector teachers of Bangladesh the major characteristics where dissatisfaction of theFaculty members was identified in the areas of pay, promotion and recognition from authority for the good job and performance reviews. They have found that, there exists a clear dissimilarity in the compensation of faculty members of public and private universities. The compensation differences widely found even within the faculty members of private universities. Private university employees are found less satisfied then public university teachers in case of equity of payment. There are some issues were private university teachers were dissatisfied are like payment for exam duties. [3]

According to Tsigilis, N., Zachopoulou, E., & Grammatikopoulos, V. (2006) on their study of job satisfaction and burnout among Greek early educators: a comparison between public and private sector employees have mentioned that faculty members were least contended from the pay they are getting and most contented from their nature of their work and their supervisor . It was found that private sector's faculty members were more contended then their peers from the public sector about their immediate supervisor and salary structure. [4]

According to Khalid, S., Irshad, M. Z., & Mahmood, B. (2012) on their study of job satisfaction among academic staff: a comparative analysis between public and private sector universities of Punjab, Pakistan Salary difference does exists between private and public universities in Punjab, Pakistan. Educators in private sector universities were found more contented with salary, promotional opportunities and job

security while public sector educators were satisfied with Co-worker's behaviour and job security. even if, there exist an importance difference in the satisfaction level of both private and public sector university in the overall job satisfaction they derive from the various parameters like salary, behaviour their relationship with their co-workers, promotional opportunity, supervision and Job security.[5]

According to M I Rehman (2008) on his study on Job Satisfaction among Public and Private College Teachers of Dhaka City faculty members are very sensitive regarding payment they are getting from their work. He also found that there is a clear difference in the pay structure of private and public colleges. The management of public and private college's managements should revise the pay structure for increasing the job satisfaction of management teachers. There are also some factors which leads to faculty member's dissatisfaction were fair promotion procedures and opportunity of research work and in private and public colleges. On other reason of faculty member's dissatisfaction were similar norms policies and rules regarding promotion of the faculty members and they can resolve these problems by having fair promotion policies at public universities. [6]

RESEARCH OBJECTIVE

To compare job satisfaction level of management educators (MBA program) of private universities and affiliated colleges to state universities in Gujarat State.

RESEARCH METHODOLOGY

The researcher has begun the study with an aim of identifying constructs and variables of the study. He has carried out pilot study, review of literature and expert's opinion for the said purpose. He has chosen sample size of 82 respondents for the pilot study. He has identified 6 constructs named as class room teaching and student quality, institutional support, salary, team spirit, freedom research and security, management of institutes and 17 independent variables for the study that are mention in below table.

CONSTRUCTS, INDEPENDENT VARIABLES OR PARAMETERS USED FOR THE STUDY

CONSTRUCT	CONSTRUCT NAME	INDEPENDENT RESEARCH VARIABLES/PARAMETERS
CONSTRUCT 1	CLASS ROOM TEACHING AND STUDENT QUALITY	• Class room Interaction • Student interaction, student IQ, and student curiosity and student eagerness to learn • Contribution in helping my students in achieving their personal and professional goals • Freedom to conduct lecture in my own style, own delivery method and course choice
CONSTRUCT 2	INSTITUTIONAL SUPPORT	• Training and faculty development initiatives • Infrastructure and technological facilities
CONSTRUCT 3	SALARY	• Salary with reference to my knowledge, skill and experience

CONSTRUCT 4	**FREEDOM RESEARCH AND SECURITY**	• Job Security • Research grant/ research leave and motivation for research • Independence of work
CONSTRUCT 5	**MANAGEMENT OF INSTITUTES**	• Performance appraisal and performance feedback system • Recognition for extra work • Objectives and clearly defined HR policies • Participation in decision making • Management style/ management/ philosophy/ vision/mission/ strategy at top management • Organization culture
CONSTRUCT 6	**TEAM SPRIT**	• Team spirit of management teachers

After that the researcher used self administered questionnaire and convenience sampling method for the final study. He chooses a sample size of 185 management educators working in MBA in colleges of private universities and affiliated colleges to state universities in Gujarat state.

RESULTS AND DISCUSSION

According to Figure.1.1

For understanding satisfaction of management educators with regard to classroom teaching and student's quality respondents were surveyed to assess their level of satisfaction and contentment from classroom interaction.

As shown in Figure 1.1 above average satisfaction score of the management teacher's at private universities is 6.37; average satisfaction score of the management teacher's of affiliated colleges is 6.14. The overall average satisfaction score of all surveyed teacher's taken together is 6.22.

The average score of the two groups are relatively closer to each other but average score of private universities teacher's is on relatively higher side.

According to Figure.1.2

For understanding satisfaction of management educators with regard to classroom teaching and student's quality respondents were surveyed to assess their level of satisfaction and contentment from Student interaction, student IQ, and student curiosity and student eagerness to learn.

As shown in chart 1.2 above average satisfaction score of the management teacher's at private universities is 4.86; average satisfaction score of the management teacher's of affiliated colleges is 4.48. The overall average satisfaction score of all surveyed teachers taken together is 4.61.

The average score of the two groups are relatively closer to each other but average score of private universities teacher's is on relatively higher side.

According to Figure.1.3

For understanding satisfaction of management educators with regard to classroom teaching and student's quality respondents were surveyed to assess their level of satisfaction and contentment from Contribution in helping my students in achieving their personal and professional goals.

As shown in chart 1.3 above average satisfaction score of the management teacher's at private universities is 6.17; average satisfaction score of the management teacher's of affiliated colleges is 5.98. The overall average satisfaction score of all surveyed teacher's taken together is 6.05.

The average score of the two groups are relatively closer to each other but average score of private universities is on relatively higher side.

According to Figure.1.4

For understanding satisfaction of management educators with regard to classroom teaching and student's quality respondents were surveyed to assess their level of satisfaction and contentment from freedom to conduct lecture in my own style, own delivery method and course choice.

As shown in chart 1.4 above average satisfaction score of the management teacher's at affiliated colleges is 5.943; average satisfaction score of the management teacher's of private universities is 5.921. The overall average satisfaction score of all surveyed teacher's taken together is 5.935.

The average score of the two groups are relatively closer to each other but average score of affiliated college's teachers is on relatively higher side.

According to Figure.1.5

For understanding satisfaction of management educators with regard to classroom teaching and student's quality respondents were surveyed to assess their level of satisfaction and contentment.

As shown in chart 1.5 above average satisfaction scores of the management teacher's at private universities is 6.37, 4.86, 6.17 , 5.921; average satisfaction score of the management teacher's of affiliated colleges is 6.14, 4.48, 5.98, 5.943. The overall average satisfaction score of all surveyed teacher's taken together is 6.22, 4.61, 6.05, and 5.935.

The average score of the two groups are relatively closer to each other but average scores of private universities is on relatively higher side is 6.37, 4.86, and 6.17 while average score of affiliated colleges is on relatively higher side is 5.935.

According to Figure.1.6

For understanding satisfaction of management educators with regard toInstitutional Support respondents were surveyed to assess their level of satisfaction and contentment from training and faculty development initiatives.

As shown in chart 1.6 above average satisfaction score of the management teacher's at private universities is 5.65; average satisfaction score of the management teacher's of affiliated colleges is 5.02. The overall average satisfaction score of all surveyed teacher's taken together is 5.23.

The average score of the two groups are relatively closer to each other but average score of private universities teacher's is on relatively higher side.

According to Figure.1.7

For understanding satisfaction of management educators with regard toInstitutional Support respondents were surveyed to assess their level of satisfaction and contentment from infrastructure and technological facilities.

As shown in chart 1.6 above average satisfaction score of the management teacher's at private universities is 6.00; average satisfaction score of the management teacher's of affiliated colleges is 5.74. The overall average satisfaction score of all surveyed teacher's taken together is 5.83.

The average score of the two groups are relatively closer to each other but average score of private universities teacher's is on relatively higher side.

According to Figure.1.8

For understanding satisfaction of management educators with regard to Institutional Support respondents were surveyed to assess their level of satisfaction and contentment.

As shown in chart 1.8 above average satisfaction scores of the management teacher's at private universities is 5.65, 6.00; average satisfaction score of the management teacher's of affiliated colleges is 5.02,5.74. The overall average satisfaction score of all surveyed teacher's taken together is 5.23, 5.83.

The average score of the two groups are relatively closer to each other but average scores of private universities is on relatively higher side is 5.23, 5.83.

According to Figure.1.9

For understanding satisfaction of management educators with regard tosalary at Institutes/ Universities respondents were surveyed to assess their level of satisfaction and contentment from salary with reference to my knowledge, skills and abilities.

As shown in chart 1.9 above average satisfaction score of the management teacher's at private universities is 4.90; average satisfaction score of the management teacher's of affiliated colleges is 4.39. The overall average satisfaction score of all surveyed teacher's taken together is 4.56.

The average score of the two groups are relatively closer to each other but average score of private universities teacher's is on relatively higher side.

According to Figure.1.10

For understanding satisfaction of management educators with regard toFreedom, Research and Security respondents were surveyed to assess their level of satisfaction and contentment from job security.

As shown in chart 1.10 above average satisfaction score of the management teacher's at private universities is 4.78; average satisfaction score of the management teacher's of affiliated colleges is 5.03. The overall average satisfaction score of all surveyed teacher's taken together is 4.95.

The average score of the two groups are relatively closer to each other but average score of affiliated college's teacher's is on relatively higher side.

According to Figure.1.11

For understanding satisfaction of management educators with regard toFreedom, Research and Security respondents were surveyed to assess their level of satisfaction and contentment from job security.

As shown in chart 1.11 above average satisfaction score of the management teacher's at private universities is 5.05; average satisfaction score of the management teacher's of affiliated colleges is 4.31. The overall average satisfaction score of all surveyed teacher's taken together is 4.56.

The average score of the two groups are relatively closer to each other but average score of private universities teacher's is on relatively higher side.

According to Figure.1.12

For understanding satisfaction of management educators with regard toFreedom, Research and Security respondents were surveyed to assess their level of satisfaction and contentment from Independence of work.

As shown in chart 1.12 above average satisfaction score of the management teacher's at private universities is 5.54; average satisfaction score of the management teacher's of affiliated colleges is 5.69. The overall average satisfaction score of all surveyed teacher's taken together is 5.64.

The average score of the two groups are relatively closer to each other but average score of affiliated college's teacher's is on relatively higher side.

According to Figure.1.13

For understanding satisfaction of management educators with regard toFreedom, Research and Security respondents were surveyed to assess their level of satisfaction and contentment.

As shown in chart 1.13 above average satisfaction score of the management teacher's at private universities is 4.78,5.05 and 5.54; average satisfaction score of the management teacher's of affiliated colleges is 5.03,4.31,5.69. The overall average satisfaction score of all surveyed teacher's taken together is 4.95, 4.56, and 5.64.

The average score of the two groups are relatively closer to each other but average score of private universities teacher's 5.05 is on relatively higher side while average score of affiliated college's teacher's 5.03 and 5.69 is on higher side.

According to Figure.1.14

For understanding satisfaction of management educators with regard toManagement of Institutesrespondents were surveyed to assess their level of satisfaction and contentment from Performance appraisal and performance feedback system.

As shown in chart 1.14 above average satisfaction score of the management teacher's at private universities is 4.54; average satisfaction score of the management teacher's of affiliated colleges is 4.60. The overall average satisfaction score of all surveyed teacher's taken together is 4.58.

The average score of the two groups are relatively closer to each other but average score of affiliated college's teacher's is on relatively higher side.

According to Figure.1.15

For understanding satisfaction of management educators with regard toManagement of Institutesrespondents were surveyed to assess their level of satisfaction and contentment from Recognition for extra work.

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As shown in chart 1.15 above average satisfaction score of the management teacher's at private universities is 4.78; average satisfaction score of the management teacher's of affiliated colleges is 4.73. The overall average satisfaction score of all surveyed teacher's taken together is 4.75.

The average score of the two groups are relatively closer to each other but average score of private universities teacher's is on relatively higher side.

According to Figure.1.16

For understanding satisfaction of management educators with regard toManagement of Institutesrespondents were surveyed to assess their level of satisfaction and contentment from objectives and clearly defined HR policies.

As shown in chart 1.16 above average satisfaction score of the management teacher's at private universities is 4.65; average satisfaction score of the management teacher's of affiliated colleges is 4.29. The overall average satisfaction score of all surveyed teacher's taken together is 4.41.

The average score of the two groups are relatively closer to each other but average score of private universities teacher's is on relatively higher side.

According to Figure.1.17

For understanding satisfaction of management educators with regard toManagement of Institutesrespondents were surveyed to assess their level of satisfaction and contentment from objectives and clearly defined HR policies.

As shown in chart 1.17 above average satisfaction score of the management teacher's at private universities is 5.03; average satisfaction score of the management teacher's of affiliated colleges is 4.87. The overall average satisfaction score of all surveyed teacher's taken together is 4.92.

The average score of the two groups are relatively closer to each other but average score of private universities teacher's is on relatively higher side.

According to Figure.1.18

For understanding satisfaction of management educators with regard toManagement of Institutesrespondents were surveyed to assess their level of satisfaction and contentment from Management style/ management/ philosophy/ vision/mission/ strategy at top management.

As shown in chart 1.18 above average satisfaction score of the management teacher's at private universities is 5.32; average satisfaction score of the management teacher's of affiliated colleges is 4.79. The overall average satisfaction score of all surveyed teacher's taken together is 4.97.

The average score of the two groups are relatively closer to each other but average score of private universities teacher's is on relatively higher side.

According to Figure.1.19

For understanding satisfaction of management educators with regard toManagement of Institutesrespondents were surveyed to assess their level of satisfaction and contentment from Management style/ management/ philosophy/ vision/mission/ strategy at top management.

As shown in chart 1.19 above average satisfaction score of the management teacher's at private universities is 5.24; average satisfaction score of the management teacher's of affiliated colleges is 5.21. The overall average satisfaction score of all surveyed teacher's taken together is 5.22.

The average score of the two groups are relatively closer to each other but average score of private universities teacher's is on relatively higher side.

According to Figure.1.20

For understanding satisfaction of management educators with regard toManagement of Institutesrespondents were surveyed to assess their level of satisfaction and contentment.

As shown in chart 1.20 above average satisfaction score of the management teacher's at private universities is 4.54,4.78,4.65,5.03,5.32,5.24; average satisfaction score of the management teacher's of affiliated colleges is 4.60,4.73,4.29,4.87,4.79,5.21. The overall average satisfaction score of all surveyed teacher's taken together is 4.58, 4.75, 4.41, 4.92, 4.97, and 5.22.

The average score of the two groups are relatively closer to each other but average score of private universities teacher's 4.78, 4.65, 5.03, 5.32, 5.24 is on relatively higher side while average score of affiliated college's teacher's 4.60 is on higher side.

According to Figure.1.21

For understanding satisfaction of management educators with regard toTeam Spiritrespondents were surveyed to assess their level of satisfaction and contentment.

As shown in chart 1.19 above average satisfaction score of the management teacher's at private universities is 5.29; average satisfaction score of the management teacher's of affiliated colleges is 5.56. The overall average satisfaction score of all surveyed teacher's taken together is 5.46.

The average score of the two groups are relatively closer to each other but average score of affiliated college's teacher's is on relatively higher side.

According to Figure.1.22

For understanding satisfaction of management educators with regard toTeam Spiritrespondents were surveyed to assess their level of satisfaction and contentment.

As shown in chart 1.22 above average satisfaction score of the management teacher's at private universities is 5.30; average satisfaction score of the management teacher's of affiliated colleges is 5.10. The overall average satisfaction score of all surveyed teacher's taken together is 5.17.

The average score of the two groups are relatively closer to each other but average score of private universities teacher's is on relatively higher side.

CONCLUSION

The findings of study reveals that the overall satisfaction level of two groups of management educators are relatively closer to each other but the satisfaction level of the management educators of private universities are relatively on higher side in comparison of colleges affiliated to state universities. Male management teachers are more satisfied then female management educators working at private universities and colleges affiliated to state universities. There is no significant difference found in the satisfaction level of management teachers having PhD. degree and not having PhD. degree, the level of satisfaction of both the groups are relatively closer to each other this indicate that higher education does not make any difference in the satisfaction level of management educators. Satisfaction level of the management teachers of different age group is relatively very close to each other this indicates that age does not matter for the job satisfaction of management teachers. The research indicate that higher the level of experience, higher the level of satisfaction from all the constructs used in the study.

SUGGESTIONS

The management educators of private universities and affiliated colleges to state university should adopt interactive methods for teaching rather than traditional lecture method to have personal satisfaction and contentment from classroom interaction. They should generate student eagerness and students curiosity while teaching by innovative methods of teaching. The private universities and affiliated colleges to state universities need to arrange some standard entry level selection procedure to assess student quality and student quality, student innovativeness, student curiosity, student eagerness to learn, student IQ can make a significant difference in satisfaction level of management educators. Management educators should also realize that they are responsible for achieving personnel and professional goals of their students so they should help their students in achieving their personal and professional goals.Management educators also need to have autonomy to conduct lecture in their own style, own delivery method and course choice for having more satisfaction especially at colleges affiliated to state universities.

The management of private universities and affiliated colleges to state universities should arrange maximum number of training programme for their student development faculty development programme (FDP) for their management teacher's development and management development programmes(MDP) for their management development by inviting well-known trainers, coaches and mentors at their universities/institutes. They should arrange modern infrastructure and latest technologies to facilitate student learning at their university/institute.

The management of private universities and affiliated colleges to state universities should have very well structured compensation structure. Their salary increments, incentives, fringe benefits and promotion all are linked up with performance. They should have appropriate performance appraisal system linked up with knowledge, skills and abilities of management educators.

The management educator should fell job security at their university institutes for having satisfaction and reducing stress from the job. They should have freedom for research work because research leads to innovativeness and success of universities and institutes. The management of the private universities and affiliated colleges to state universities should develop research environment at their university/institutes. They should give research leave and research grant to their management teachers and motivate them for research work. The independence of work is also one of the important factors for management educators to boost up their performance and have satisfaction from their job.

The private universities and affiliated colleges to state universities should have well defined performance appraisal and performance feedback system to assess the performance of management teachers. They also need to appreciate their management teacher's for their extra work. The managements of institutes should

have clear objectives and well defined HR policies which lead to satisfaction of management teachers. They also need to take view of their management teacher's in decision making activities prevailing at their universities/institutes to have sense of participative culture at their university/institute. Management also need to be very clear about management style/management philosophy/vision/mission/strategy at top management which leads to satisfaction and growth and development at their institute/university. They should have good organization culture because organization culture is vital for satisfaction of management teachers and success of the universities/institutes.

The management educators should believe in team work and cooperation because it is essential for achieving goals and objectives of the university/institute and lead them to growth and development of their universities/institutes. Every university/institutes should support teamwork and supportive environment at their institute /university.

FIGURES

Figure. 1.1

Construct 1 - class room teaching and students quality
Q.1 - Class Room Interaction

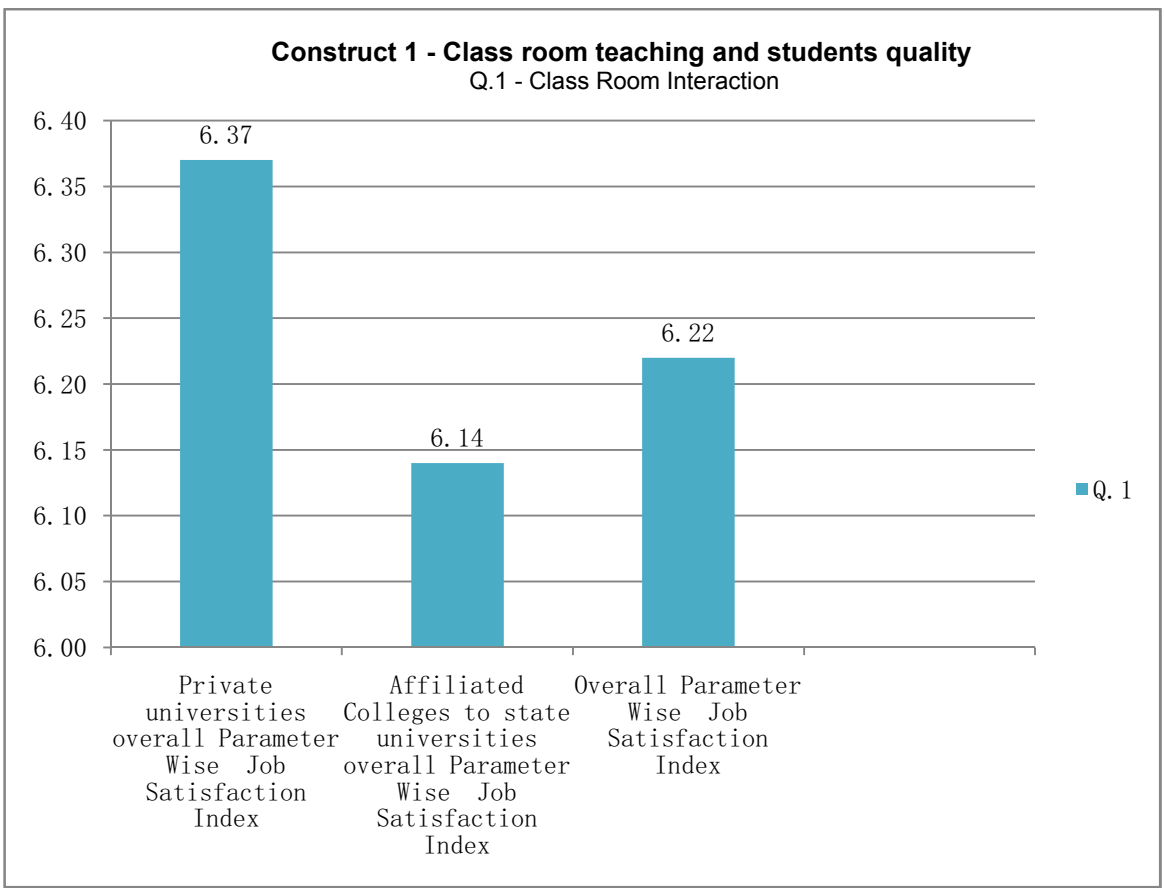


Figure. 1.2

Construct 1Class room teaching and students quality

Q.6 - Student interaction, student IQ, and student curiosity and student eagerness to learn

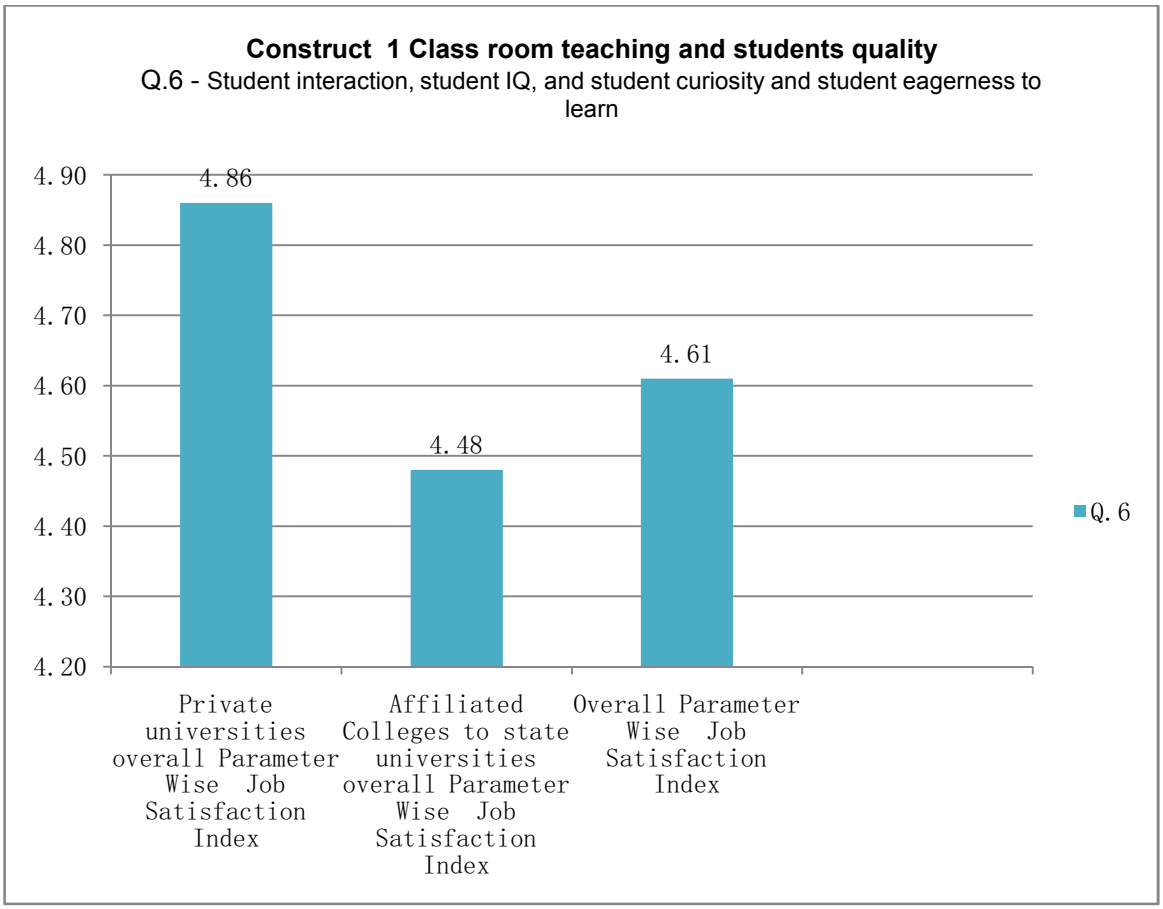


Figure 1.3

Construct 1 Class room teaching and students quality
Q.12 - Contribution in helping my students in achieving their personal and professional goals

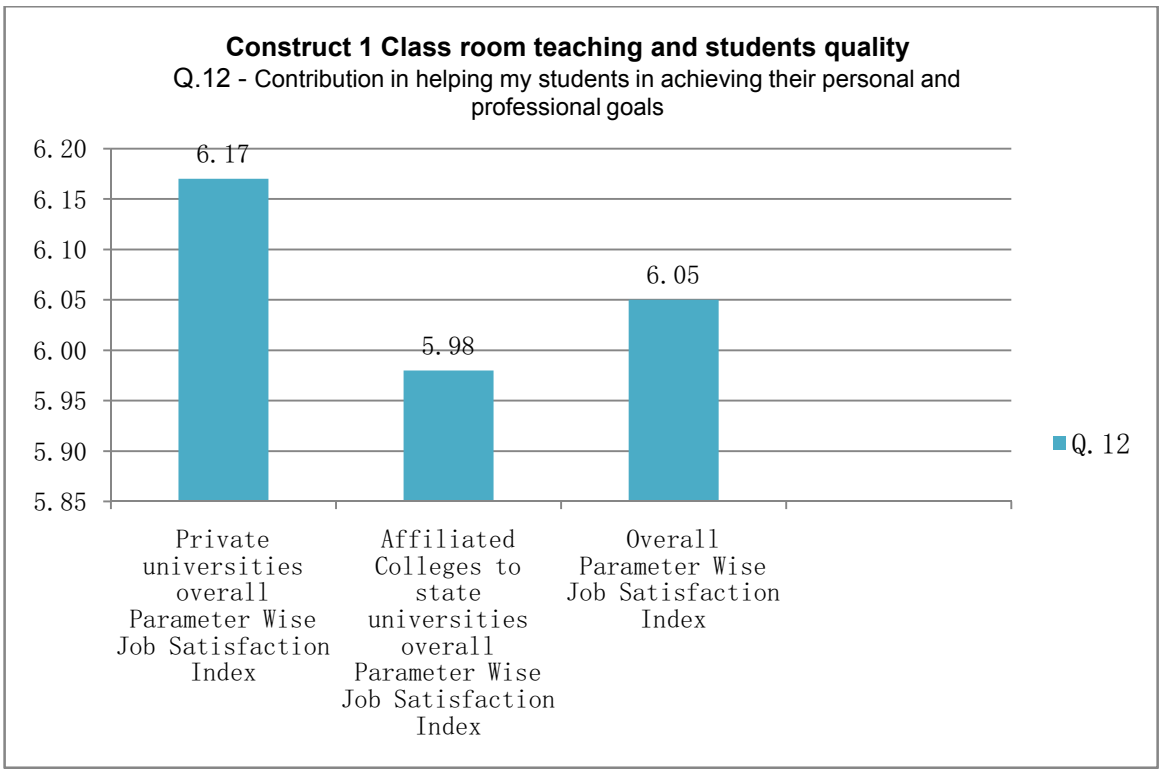


Figure 1.4

Construct 1 Class room teaching and students quality
Q.16 Freedom to conduct lecture in my own style, own delivery method and course choice

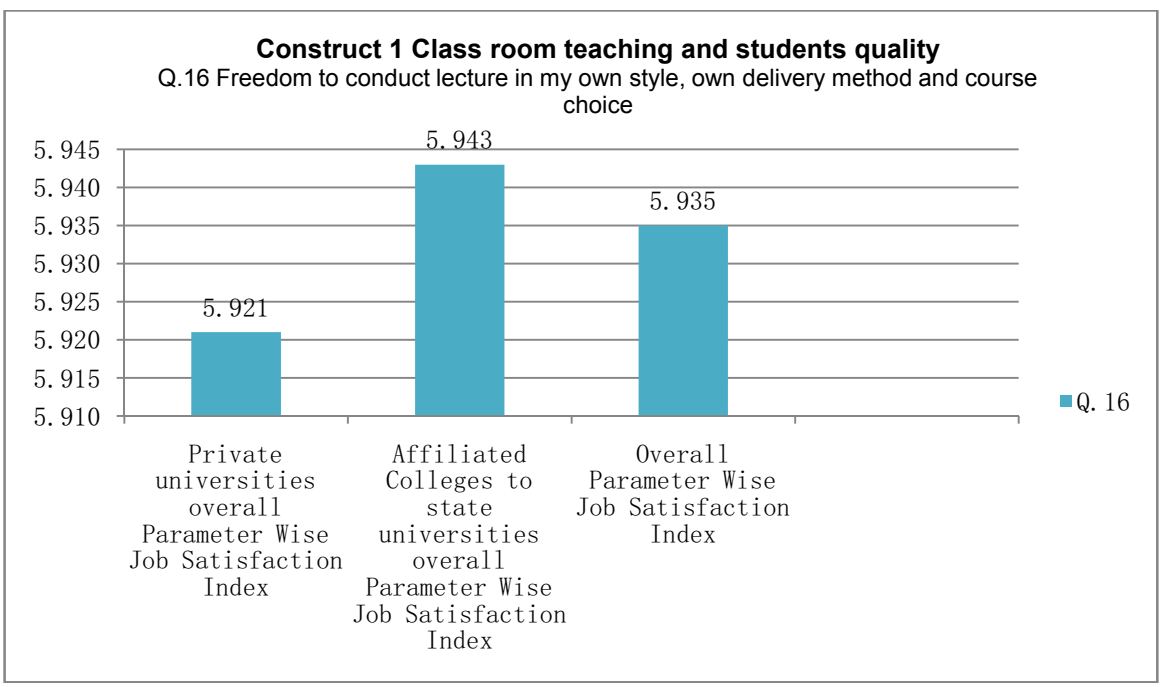
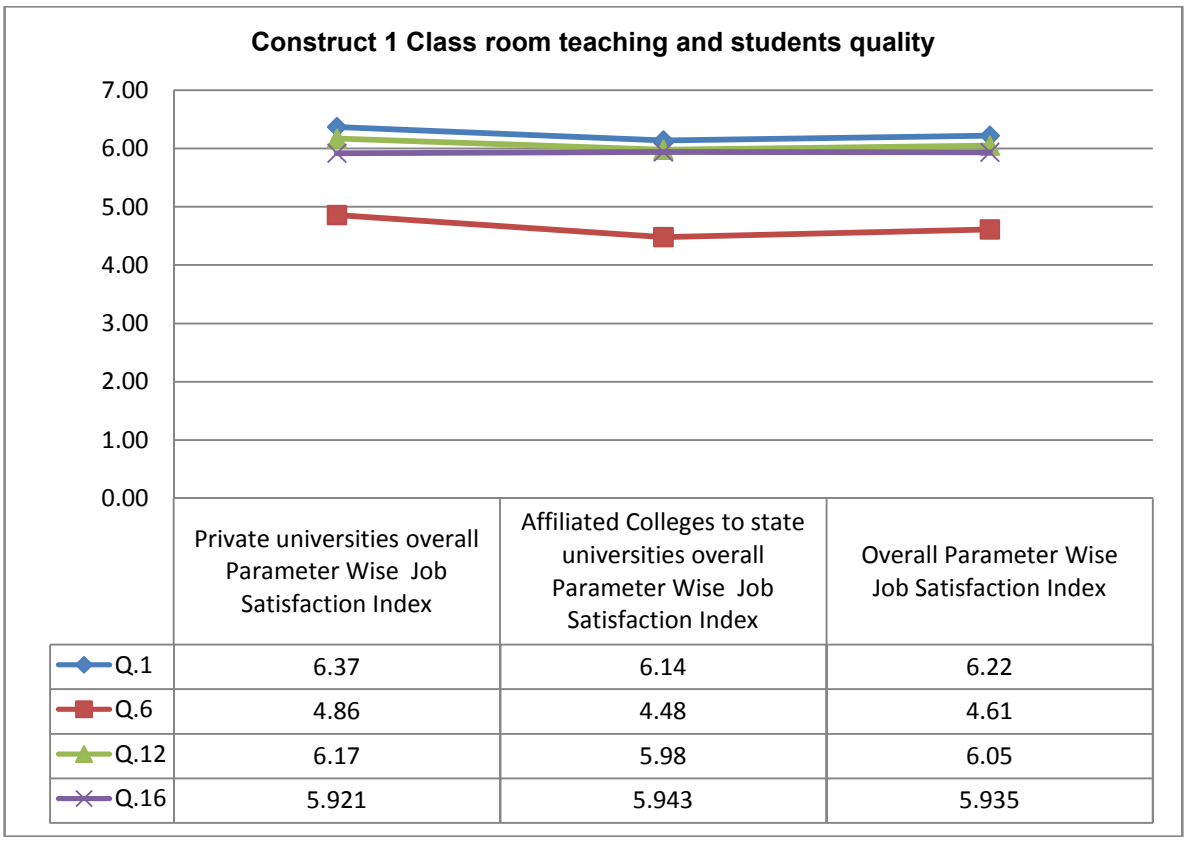


Figure. 1.5

Construct 1 Class room teaching and students quality



	Private universities overall Parameter Wise Job Satisfaction Index	Affiliated Colleges to state universities overall Parameter Wise Job Satisfaction Index	Overall Parameter Wise Job Satisfaction Index
Q.1	6.37	6.14	6.22
Q.6	4.86	4.48	4.61
Q.12	6.17	5.98	6.05
Q.16	5.921	5.943	5.935

Figure. 1.6

Construct 2 InstitutionalSupports
Q-2 Training and faculty development initiatives

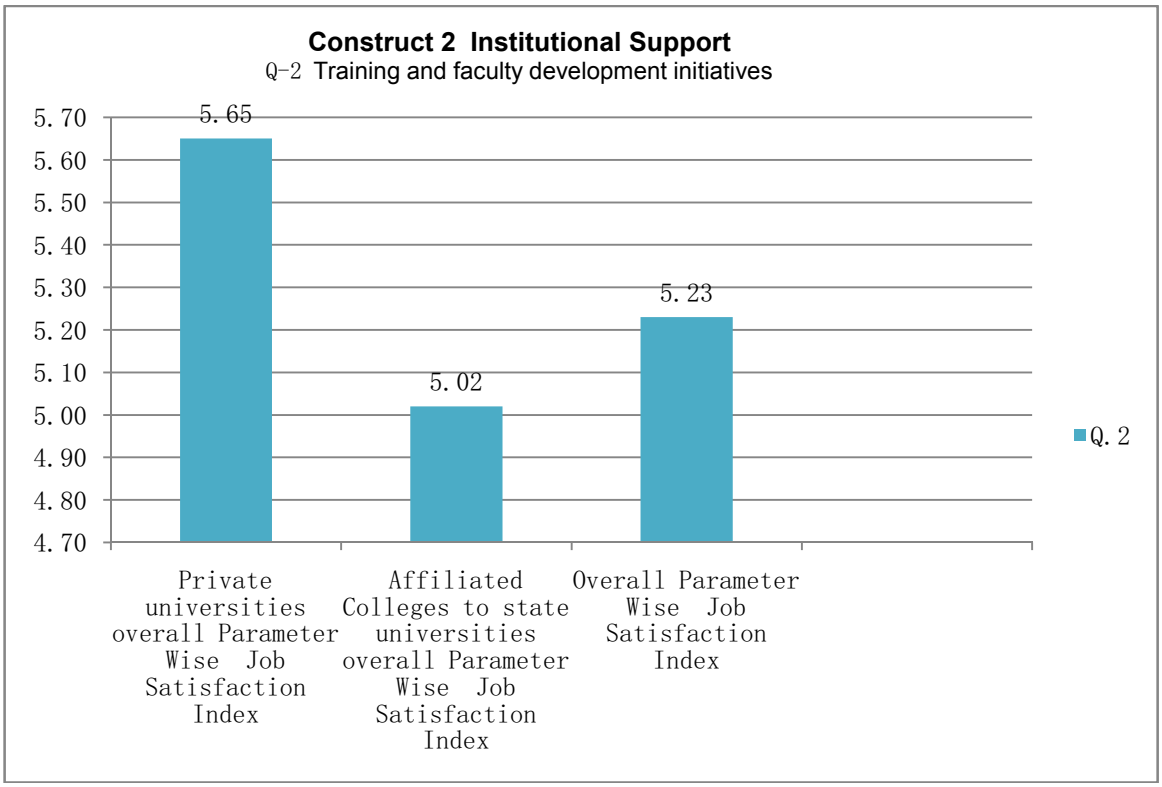


Figure. 1.7

Construct 2 Institutional Supports
Q.5 Infrastructure and technological facilities

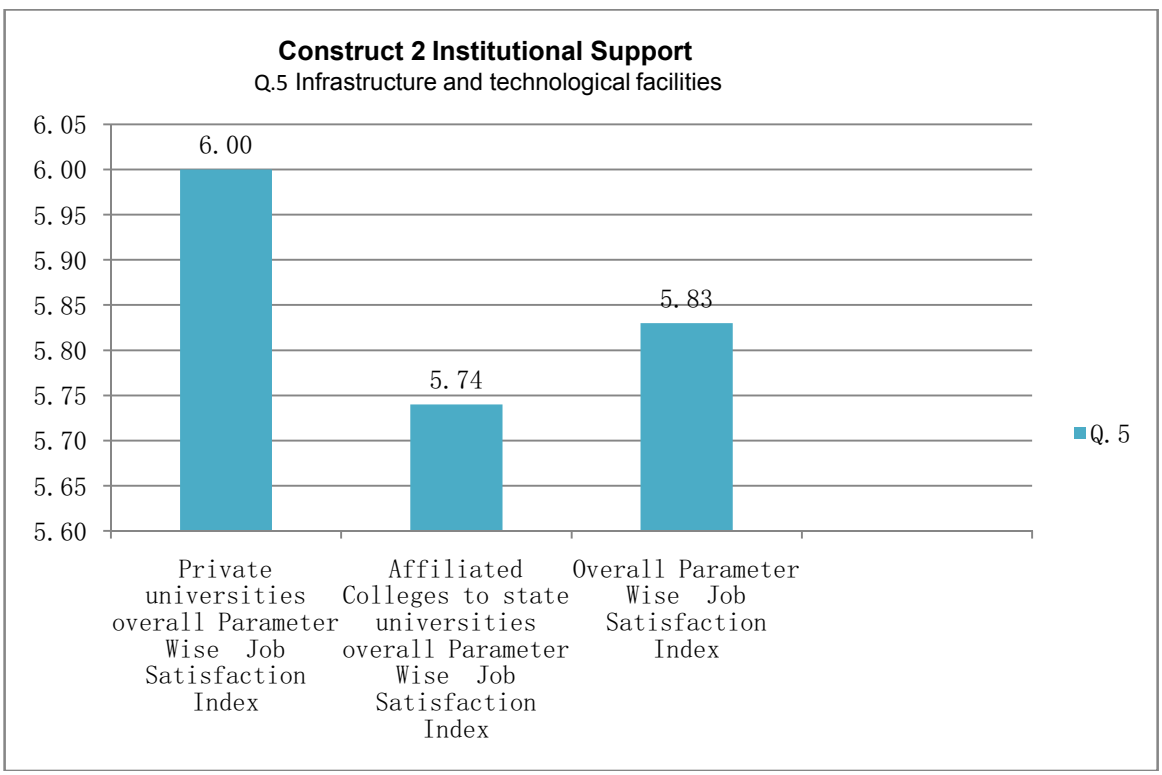
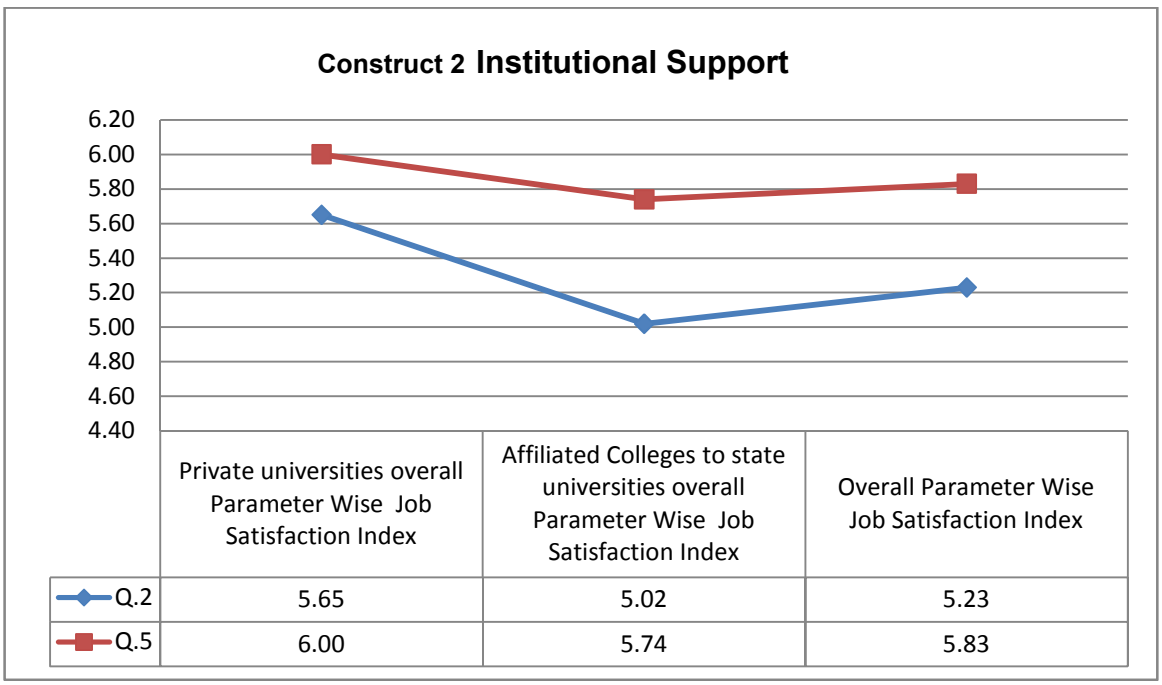


Figure 1.8

Construct 2 Institutional Support



	Private universities overall Parameter Wise Job Satisfaction Index	Affiliated Colleges to state universities overall Parameter Wise Job Satisfaction Index	Overall Parameter Wise Job Satisfaction Index
Q.2	5.65	5.02	5.23
Q.5	6.00	5.74	5.83

Figure. 1.9

Construct 3 Salary
Q.9 Salary with reference to my knowledge,skills and Abilities

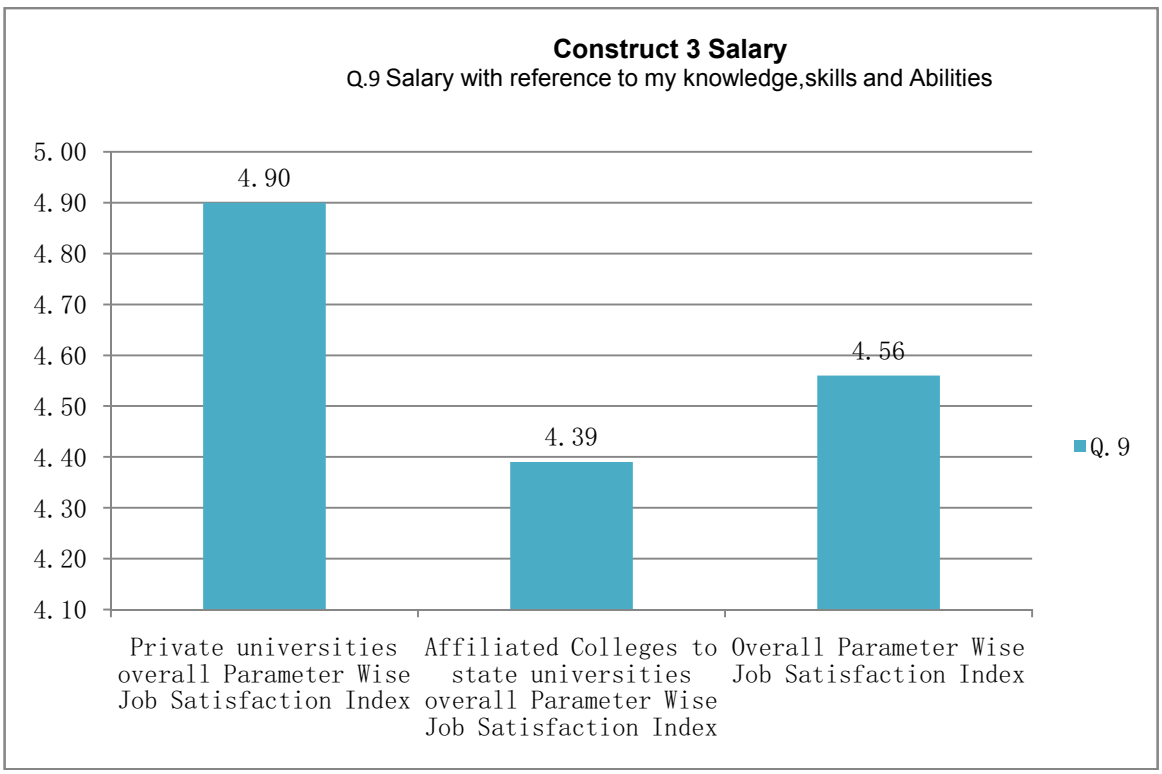


Figure. 1.10

Construct - 4 Freedom, Research and Security
Q.13 Job Security

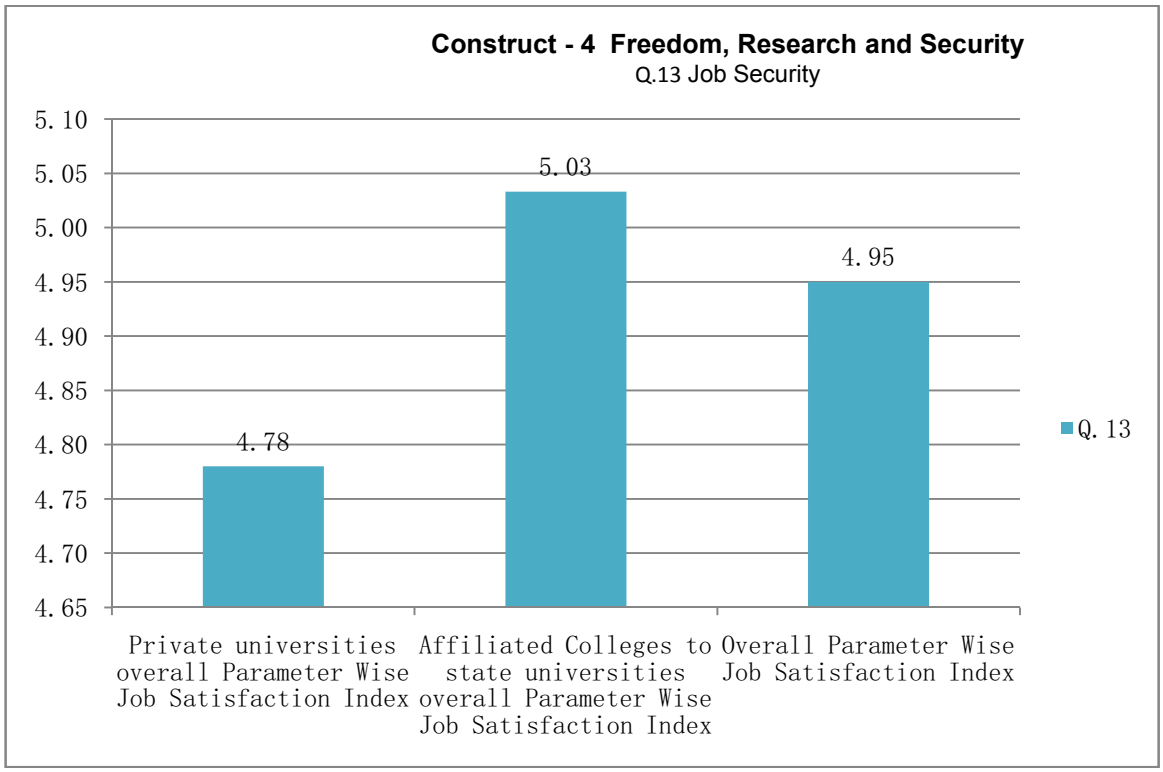


Figure. 1.11

Construct 4 Freedoms, Research and Security
Q.15 Research grants/ research leave and motivation for research

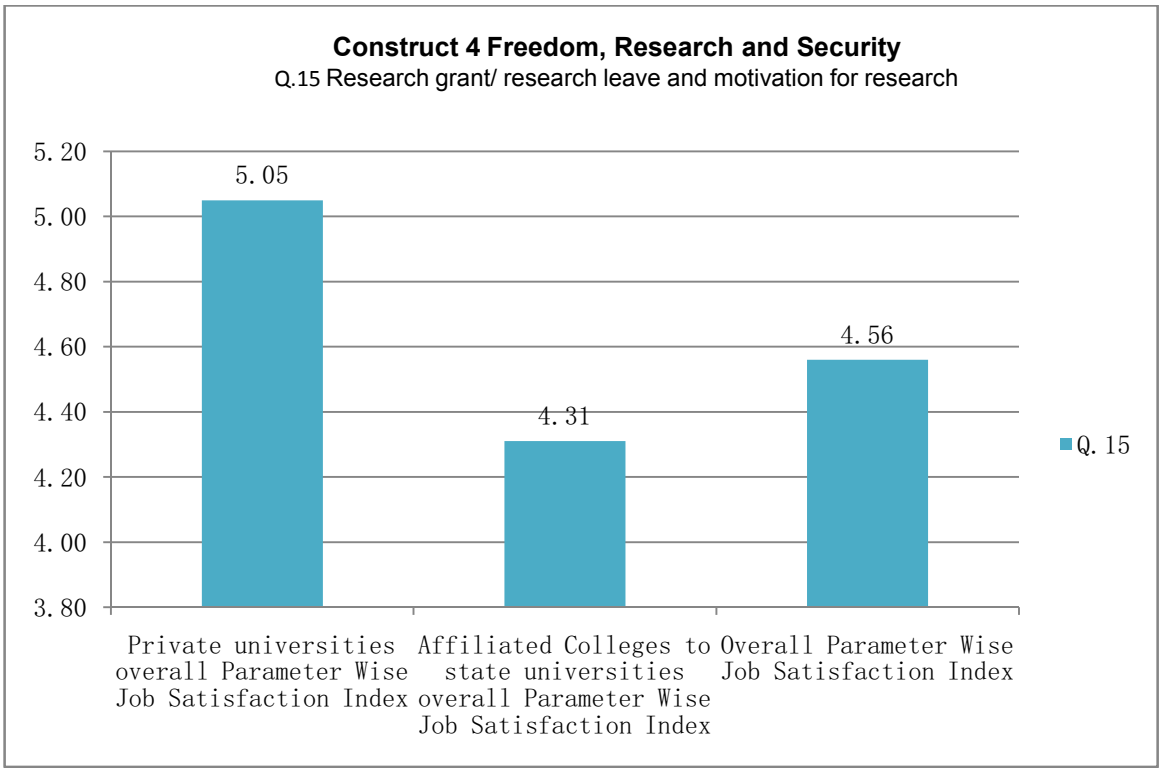


Figure. 1.12

Construct 4 Freedom, Research and Security
Q.17 Independence of work

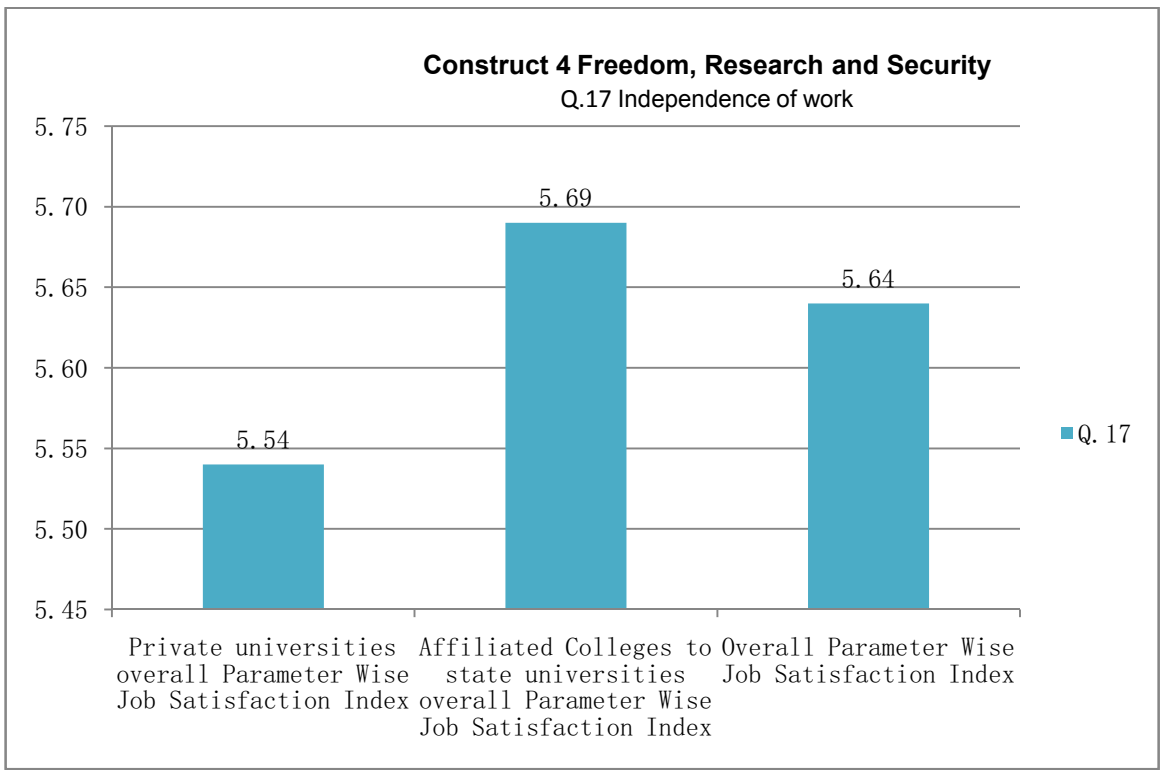
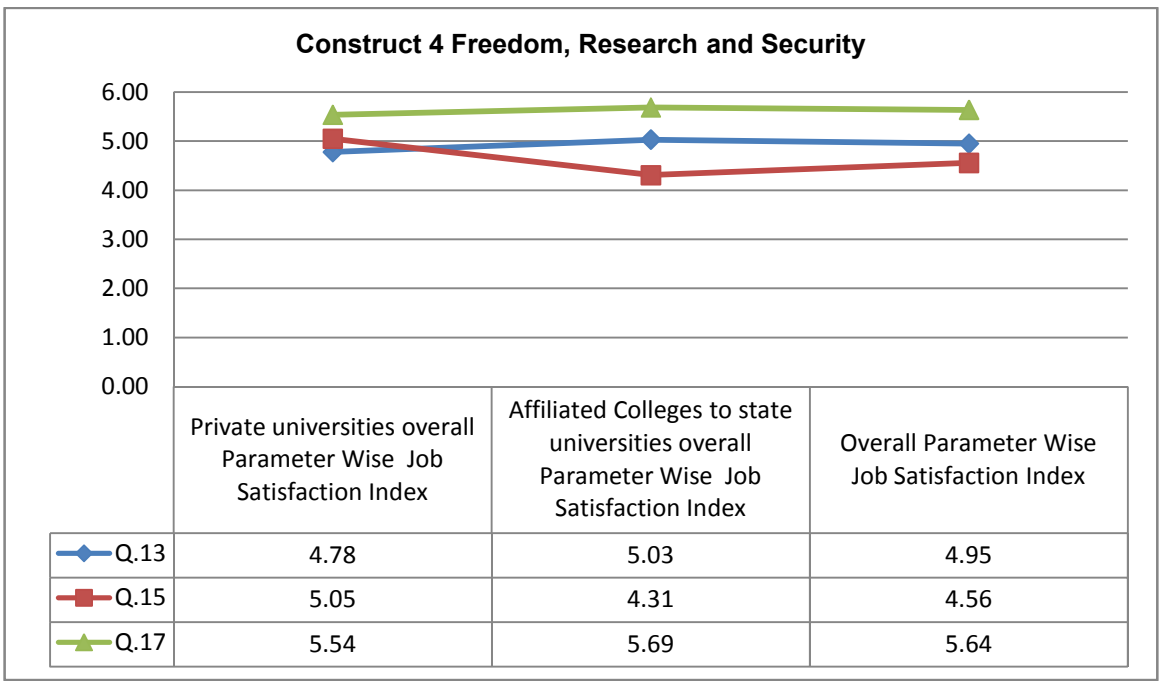


Figure 1.13

Construct 4 Freedom, Research and Security



	Private universities overall Parameter Wise Job Satisfaction Index	Affiliated Colleges to state universities overall Parameter Wise Job Satisfaction Index	Overall Parameter Wise Job Satisfaction Index
Q.13	4.78	5.03	4.95
Q.15	5.05	4.31	4.56
Q.17	5.54	5.69	5.64

Figure1.14

Construct 5 Management ofInstitutes
Q.3 Performance appraisal and performance feedback system

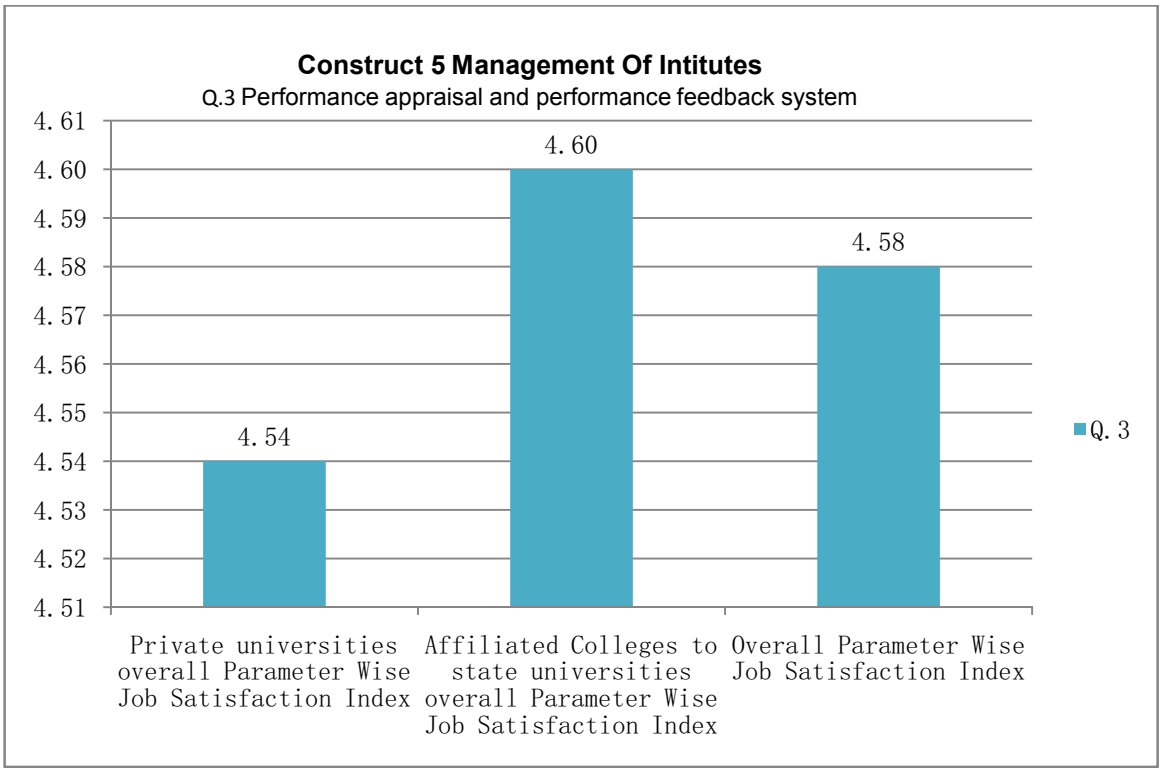


Figure 1.15

Construct 5 Management Of Institutes
Q.7 Recognition for extra work

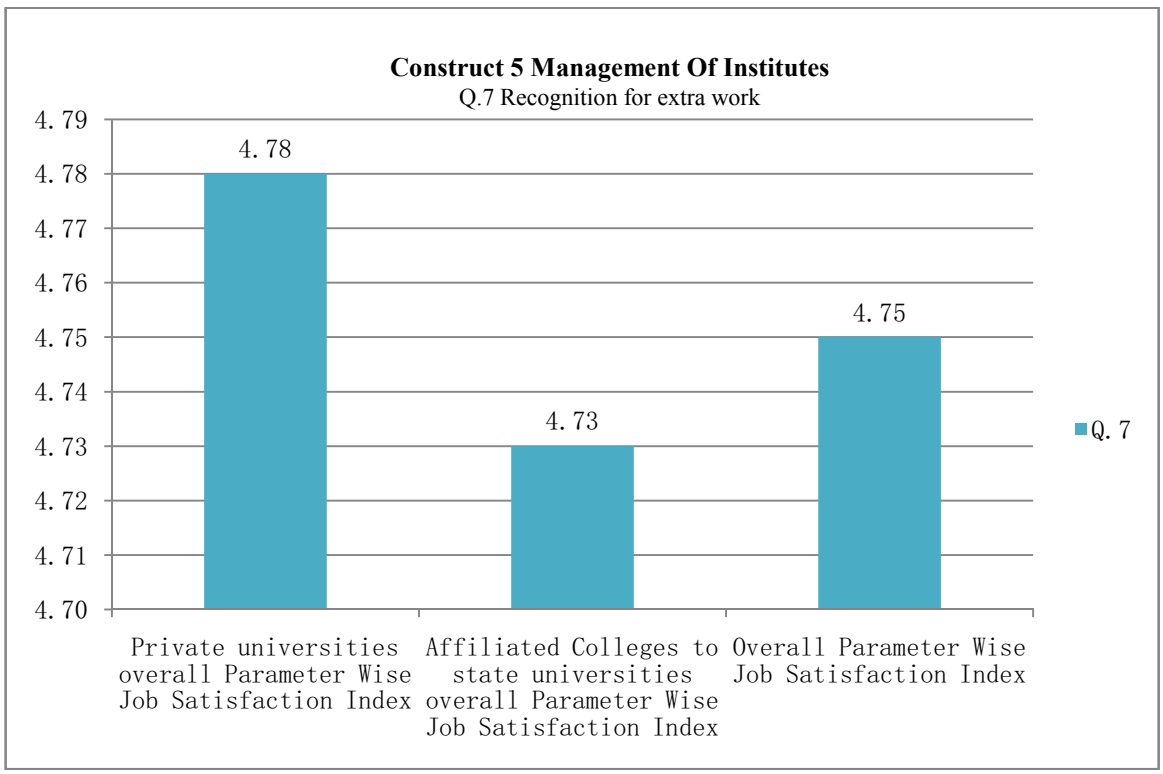


Figure 1.16

Construct 5 Management of Institutes
Q.8 Objectives and clearly defined HR policies

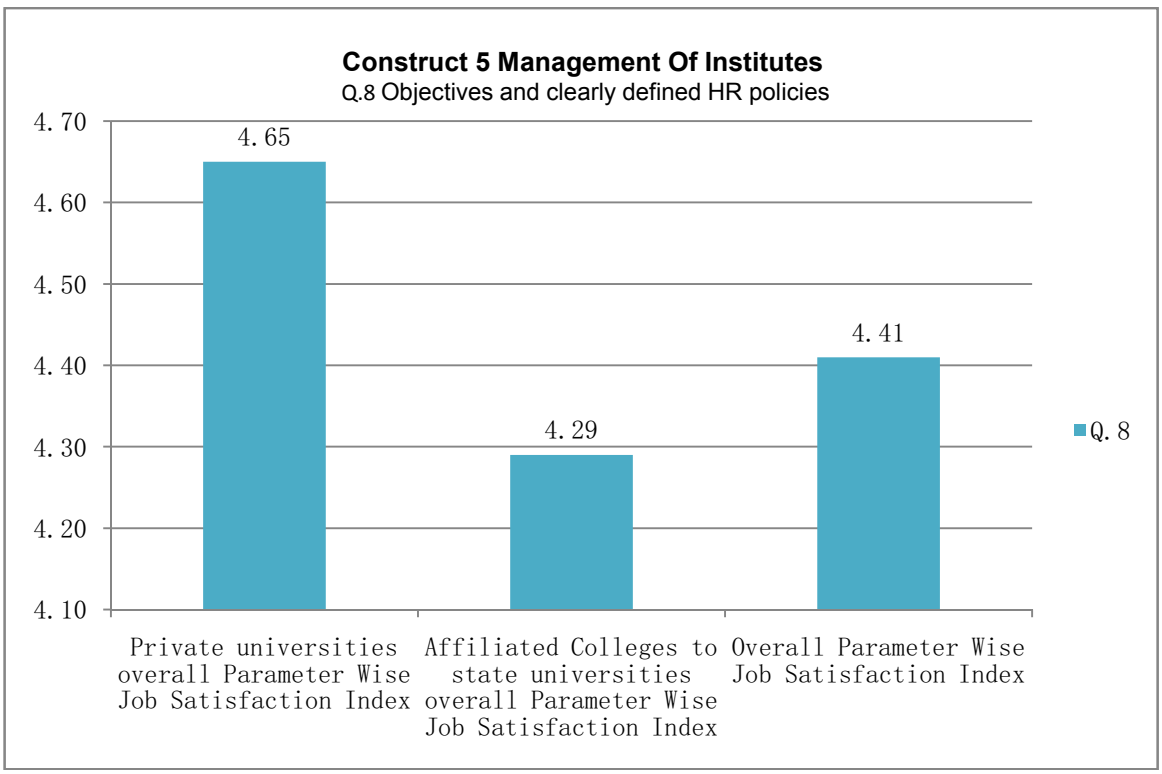


Figure 1.17

Construct 5 Management of Institutes
Q.10 Participation in decision making

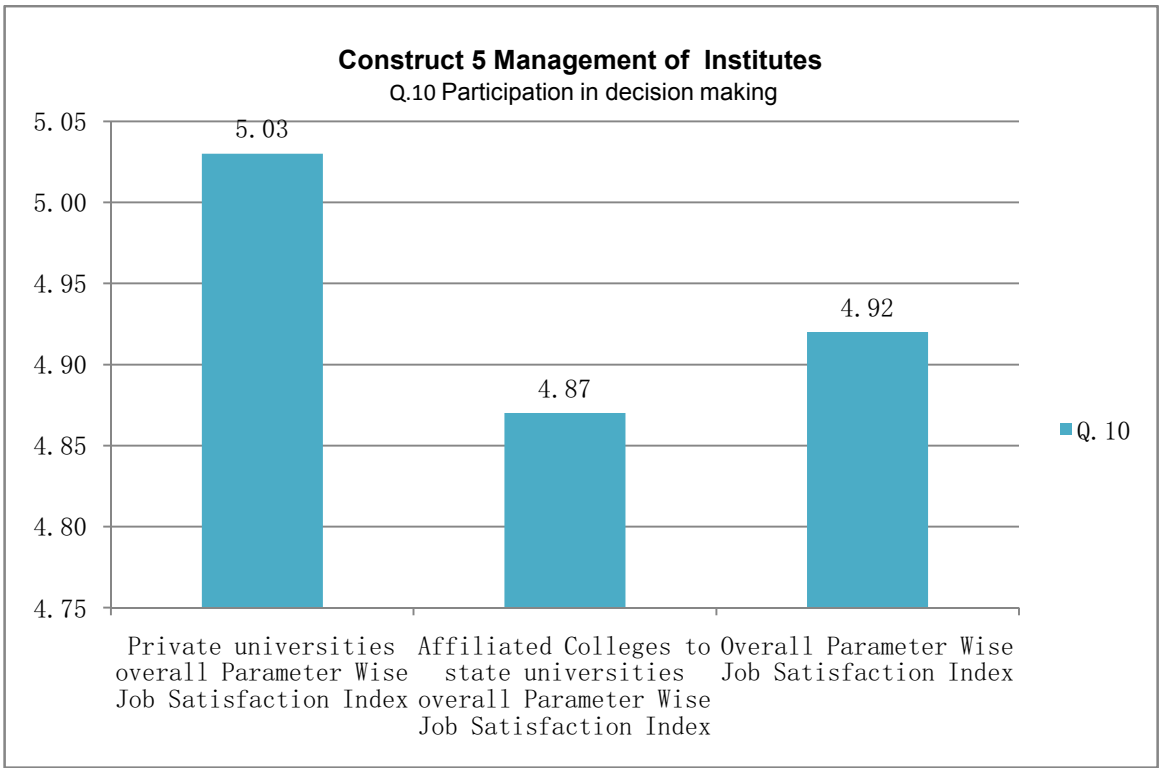


Figure 1.18

Construct 5 Management Of Institutes
Q.11 Management style/ management/ philosophy/ vision/mission/ strategy at top management

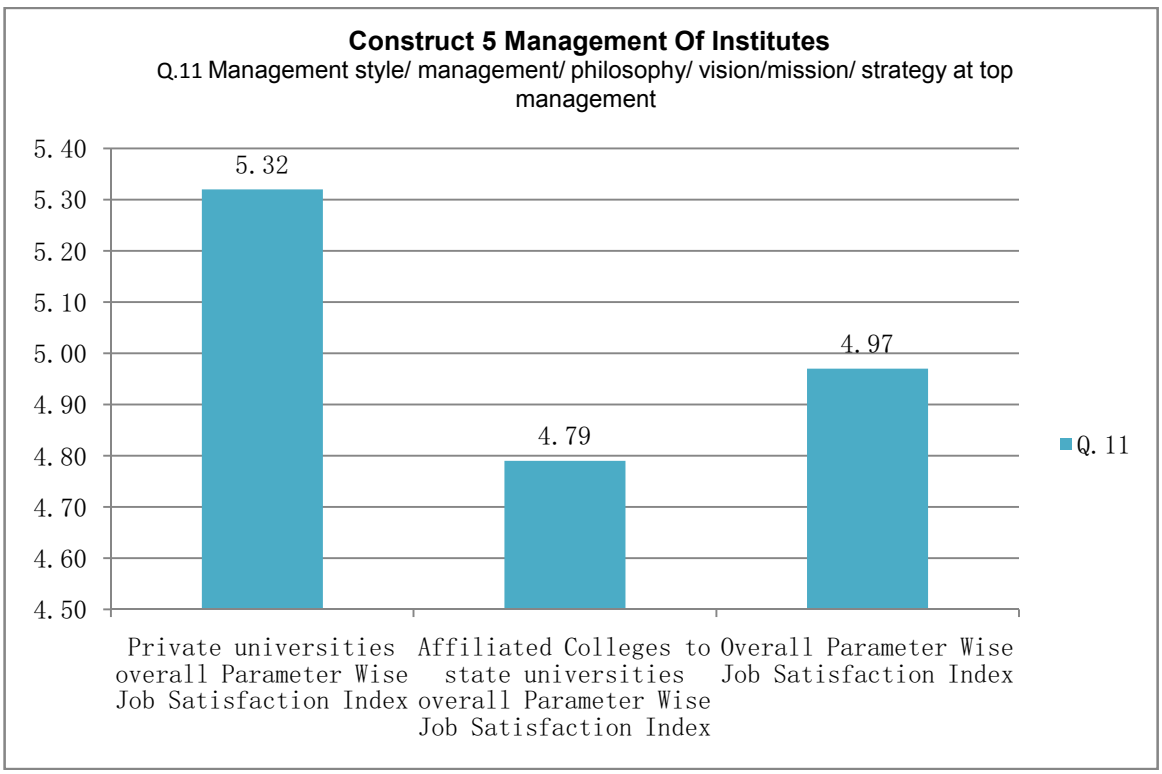


Figure 1.19

Construct 5 Management Of Institutes
Q.14 Organization culture

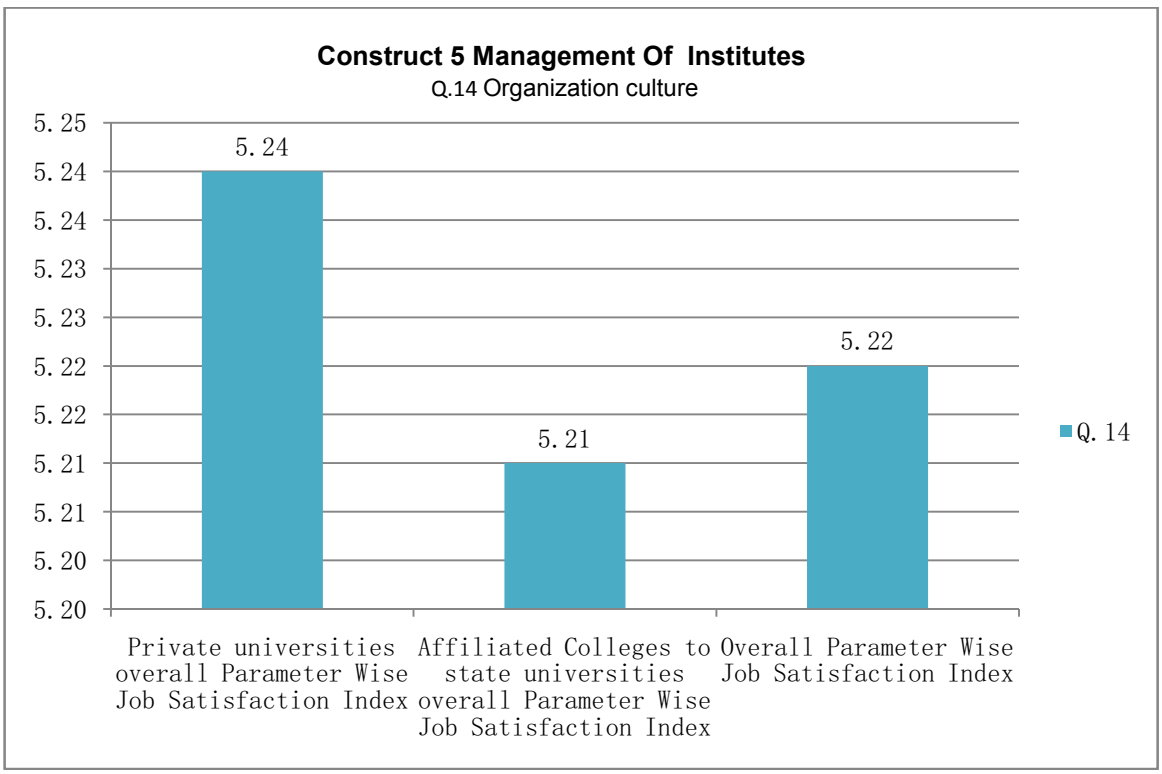
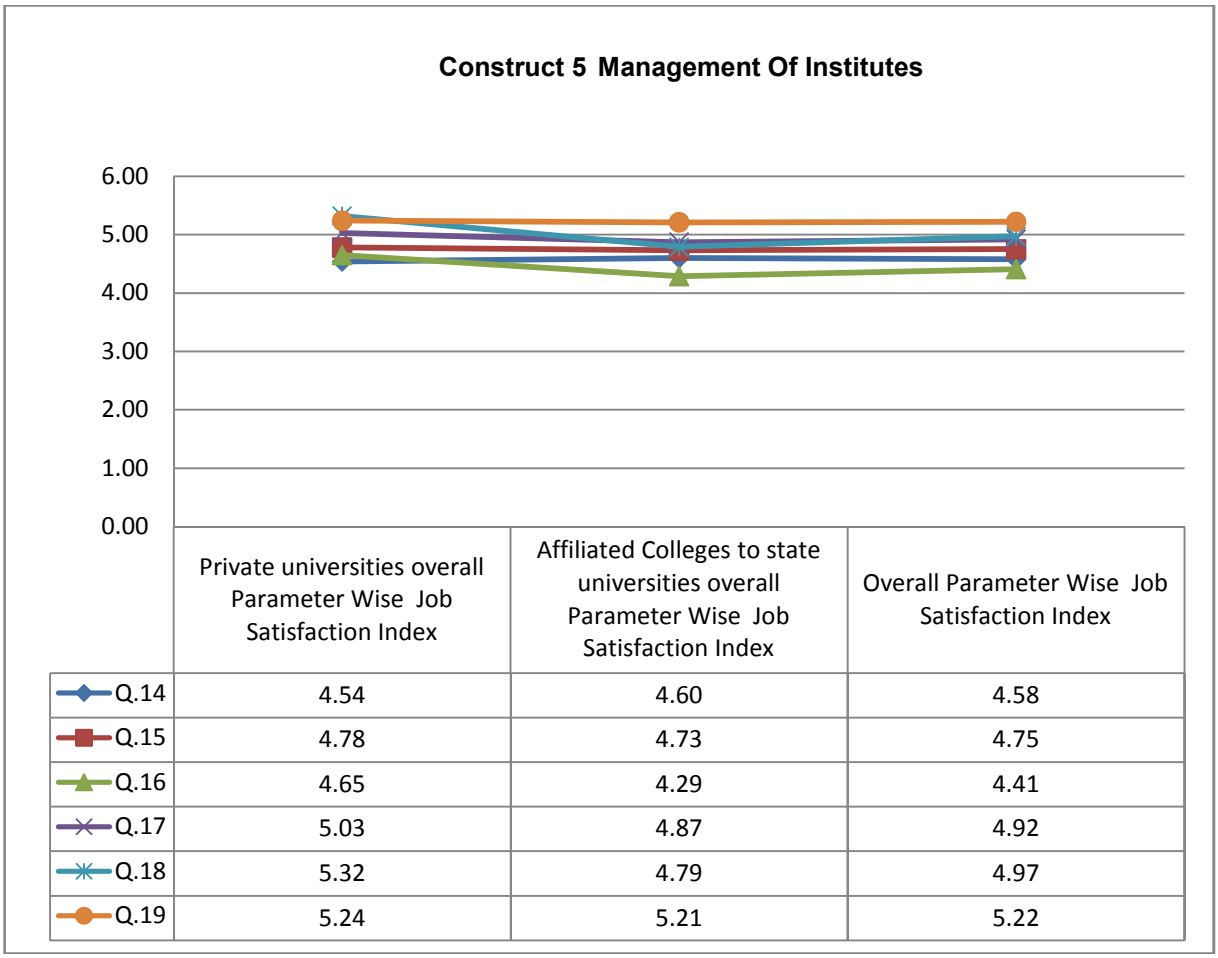


Figure 1.20

Construct 5 Management Of Institutes



	Private universities overall Parameter Wise Job Satisfaction Index	Affiliated Colleges to state universities overall Parameter Wise Job Satisfaction Index	Overall Parameter Wise Job Satisfaction Index
Q.14	4.54	4.60	4.58
Q.15	4.78	4.73	4.75
Q.16	4.65	4.29	4.41
Q.17	5.03	4.87	4.92
Q.18	5.32	4.79	4.97
Q.19	5.24	5.21	5.22

Figure 1.21

Construct 6 Team Spirit
Q.4 Team spirit of faculty members

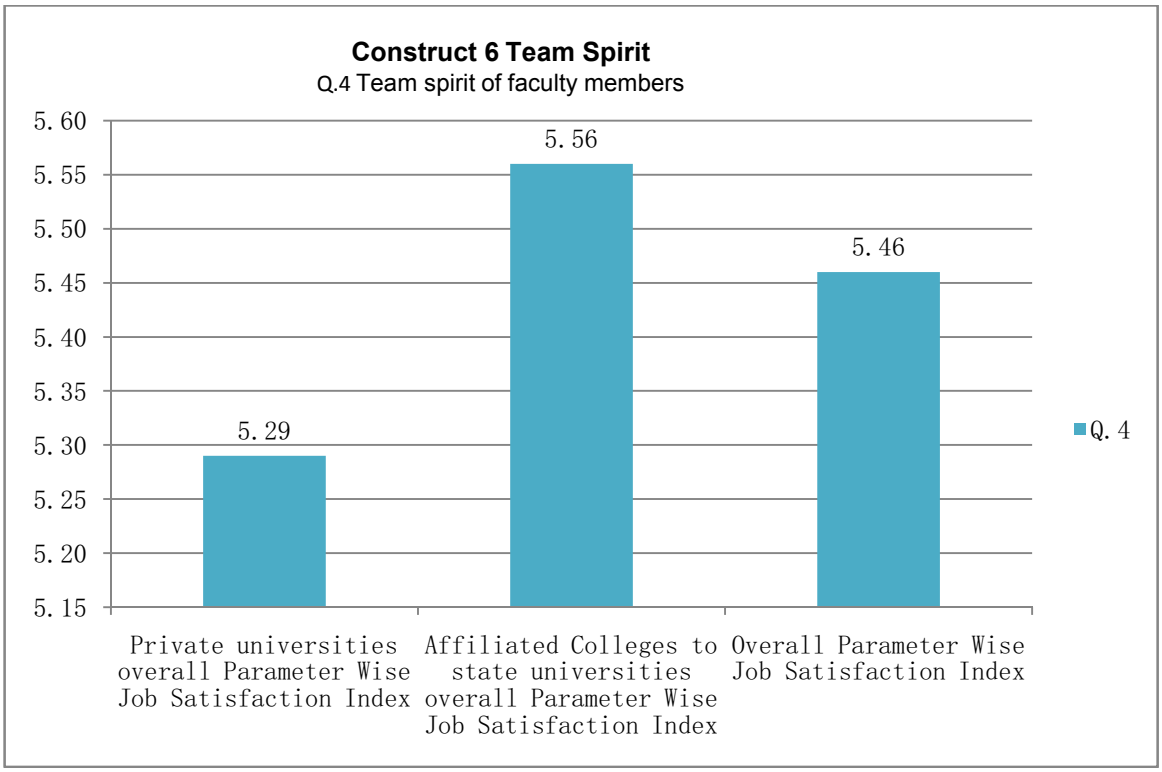
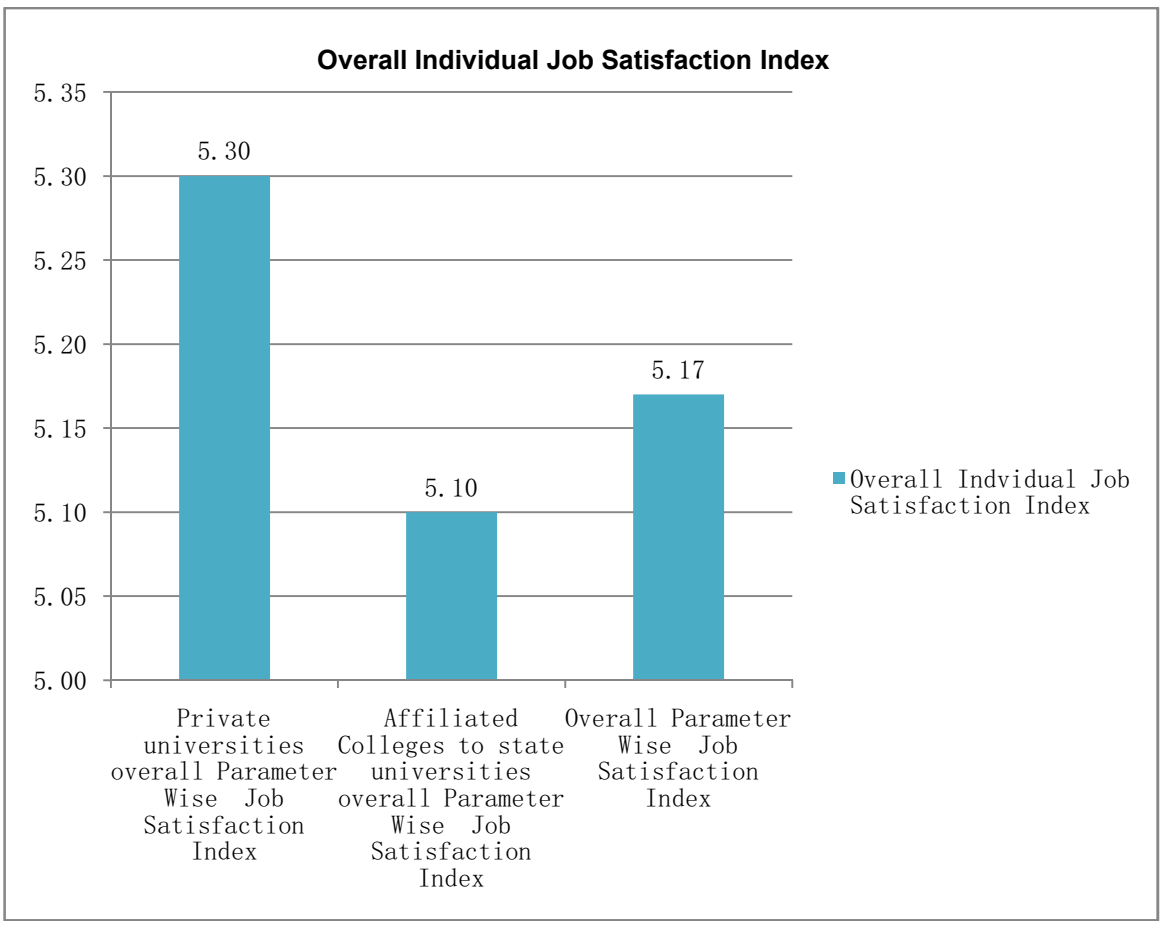


Figure 1.22

Overall Individual Job Satisfaction Index



TABLES

TABLE 1.1
GENDER WISE JOB SATISFACTION SCORE

GENDER	FREQUENCY (MALE, FEMALE)	JOB SATISFACTION SCORE
Male	99	5.29
Female	86	5.03
Total(Overall)	185	5.17

TABLE 1.2
QUALIFICATIONS WISE JOB SATISFACTION SCORE

QUALIFICATIONS	FREQUENCY (PH.D., NON.PHD.)	JOB SATISFACTION SCORE
PhD.	45	5.21
Non PhD.	140	5.16
Total(overall)	185	5.17

TABLE 1.3
AGE WISE JOB SATISFACTION SCORE

AGE	FREQUENCY(21-30,31-40,41-50 & ABOVE)	JOB SATISFACTION SCORE
21-30	99	5.23
31-40	63	5.08
41-50 & Above	21	5.09
Missing Age	02	5.76
Total(overall)	185	5.17

TABLE 1.4
Experience wise job satisfaction Score

EXPERIENCE	FREQUENCY(0-5,6-10,11-15 AND 16 & ABOVE)	JOB SATISFACTION SCORE
0-5	95	5.12
6-10	65	4.97
11-15	14	5.77
16 & Above	11	5.53
Total(overall)	185	5.17

ACKNOWLEDGEMENT

We owe a deep gratitude to professorsworking at various private universities and colleges affiliated to state universities in Gujarat for supporting in our research work.

For completing the present study, we got assistance, valuable advice and suggestions from many of teachers, well wishers, colleagues, officials and experts, who took keen interest through the work and inspired us.

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ABSTRACT

The Blue Ocean Strategy is most discussed in the last decade (2005-2015). The authors came up with an expanded edition of in the year 2015. It covers two new topics, challenges faced by industries, while applying Blue Ocean Strategy and how to overcome it. Zivame.com India's first online lingerie store, where women can discuss, share their views and emotions in it. The study carried out is to check whether zivame.com is sailing in the Blue Ocean and creating any value innovation in the unorganized lingerie market of India. The study covers analysis of two things about zivame.com i.e. growth in customer and investment. The study will provide a clear picture about zivame.com value innovation and creating a leap in value for buyers and companies. The outcome indicates that there are significant relationships between zivame.com growths by sailing in uncontested market space known as Blue Ocean Strategy.

SUMMARY

An entrepreneurial initiative towards educating women to select correct lingerie product, add a new dimension in the unorganized market of lingerie selling in India.

Keywords: Blue Ocean Strategy, Red Ocean Strategy, Value Innovation, E-commerce, Uncontested Market Space, Entrepreneurship

INTRODUCTION

Making India Awesome[a], the latest book by India's prominent writer Chetan Bhagat, highlighted a special portion rights of women. The concern, on which he emphasized in the book referred to women's rights and gave new dimension and understanding towards it. India is a male dominated country, where in the new age of development bringing equality is vital. (Beaman, L., Duflo, E., 2012). Richa Kar, founder of zivame.com employee turn entrepreneur taken the first steps towards in the month of August 2011. She has started India's first online lingerie store. The first step taken by her was actually in uncontested market space and till the date, no one had thought of that as well. But the enterprising initiative taken by Richa Kar was appreciable and bring valuable change in the business practices of intimate apparel, which is treated by men in the retail marketplace.

A conversation about lingerie is taboo in India, here no one talks about the intimate wears amenably. An under-served category brings an opportunity for Richa Kar. After completing MBA from NMIMS and Engineering Degree from BITS Pilani, Richa Joined SAP and working on a project for the big conglomerate called Limited Brands, which owns Vitoria's Secret. While doing research for the brand, Richa came to understand that most of the clientele come for this product online and that's eureka moment for Richa Kar. Intimate apparel is a merchandise category, which is not considered as a prospective business and neglected product in Indian Woman wardrobe (Nair, 2015). She started doing tons of market research, visit department stores, study the brands and its impacts and discuss with the customer as well, she got to the conclusion that, this is an untouched market where value innovation in business achieve an enormous difference. The market research about India Lingerie category identified four things, first, women in India doesn't have an idea which lingerie will be consider as a right fitting for her, second, in retail market all the size with specific design not available, third lingerie retail business mostly manage by male salesmen which create social discomfort for women while shopping for it, and fourth one there is no consultancy about product only push sales about a product which gives high margin to sellers, even women doesn't know which product is correct choice for her. She identified that 8 out of 10 women select wrong size lingerie, which indicates that lingerie business boundaries need to be redefined. (YS, T., 2012).

[a] A Non-Fiction book written by Chetan Bhagat, 2015

She had an idea and want to move forward on the idea, in the month of February 2011 meet Mr. Kapil Karekar and discuss idea also, both were interested in working further on the idea. Then the journey begins and finally in August 2011 launch the business under the name *"Actoserba Active Wholesale Pvt Ltd"* with a brand name called *"ZIVAME.COM"*. Ziva in Hebrew means *"radiant"* and added *"me",* so it becomes *"radiance in me"* and these maxims about premium with comfort (Tewari, S., 2014), India's first online lingerie e-commerce store to break the boundaries and to overcome the limitation of retail market (Singh, S., 2014). The idea clicks through e-commerce has two reasons, *first*according to IMRB International[b] research group in India, internet access were available to 240 million consumers (2011), and India ranked third after the US and China, which may take over by India at the end of this year (Pandya, K., 2015) and *second* reason was to address the gap in retail market lingerie consult and availability of size and maintain privacy, because this category needs privacy (Goveas, J., 2014).

The first order was from Indore worth of `7000, which boost Richa's motivation to continue the business with a higher goal and for Zivame biggest achievement was an order worth of `45000 by one customer. In a four year of span Zivame have enormous set of experience, i.e. in the August 2011 number of visitors 6000 increased to 2.50 Lakh visitors at the end of the year, (Kola, V., 2012), growing 400% annually(YS, T.,2013), 5 million visitors out of which 1 million unique customers (YS, T.,2012), started India's First lingerie consultant and fitting room named as "Zia" and "Zoe" where consultant offers guidelines for correct size and shape, product for different occasion and mood, 80% of buyers are first-time buyers in first three years of starting of operation, started India's first lingerie discussion blog "*followmycurves*"[c] to discuss issues, concerns and what's new in the lingerie world (YS, T.,2011). Some noteworthy growth numbers of zivame.com are 40% of repeat buyers, 60% have never thought of purchasing lingerie online, 20% have never purchased online lingerie, the orders coming from tier-2 and tier-3 cities, increasing year by 15 % in the first year and went up to 30% the second year and increasing year-on-year, every month adding 20000 new customers and 80% of customers are women (Tilley, N., & Reddy, N., 2013), launch of America's prominent brand "Wonderbra" on zivame.com and during 5 days pre-booking time 90000 customers visited the website and 100% merchandise that was set aside was sold out (Martin, R., 2012). The customer is King, but building a relationship with the customer is what trending now (Ghosh, A., 2015,).

[b]IMRB International formerly known as Indian Market Research Bureau
[c]http://www.followmycurves.com/ – Indian lingerie blog, blog about lingerie and fit advice for the Indian woman.

A company currently valued $100 million received funding from Kalaari Capital (Series A, B and C), IDG Ventures India (Series A)[d], Ronnie Screwvala & Unilazer Ventures (Series B)[e], IDG Ventures and Zodius Capital (Series C)[f] and highest ever funding from Khazanah Nasional Berhad (Series C) $40 million (Rodionova, Z., 2015). In Series A and B funding round zivame.com respectively receive fund worth of $ 3 million and $ 6 million (Chakraborty, S., 2015). The business model of zivamehas some uniqueness to offer to its customer as well as to investors. First, customers can ask, share and discusstheir issues, concern and feedback with 24x7 women customer relations team" (Agarwal, S., 2015), offer all the size in national and international brand which were previously not available in India, bra size calculator, delivering in 20000 pin codes, quality product with latest design, launched India's first most exclusive lingerie app, selling more than one bra every minute, 5000 styles in 100 sizes and 50 brands, mobile fitting lounge, custom made bras (Prabhan, P., 2012), introducing new category i.e. Shapewear, Fashion wear, Leisurewear, Sportswear, Swimwear, and Plus Size, Bridal lingerie, Everyday wear, Proprietary content to guide regarding product size, Fitting, and selection to give ease of selection (Nair, R., 2013). Second, Investors identified lingerie is niche branded category (Ghosh, D., & J, A., 2013), where other e-commerce, fighting for space competitive edge of e-commerce Zivame became most loved brand in India lingerie market and growth rate is 300% (Chadha, S.,2013), a private label strategy[g] which helps design own pricing strategy (Ghosh, D., & Chaudhary, D., 2014), investor engagement and involvement, less capital intensive, direct dealing with brands, Just-in-time replenishment model (Brain, M., 2011), continuous market research and customer engagement and involvement, segment scalability market potentiality, consultative approach to modern retailing, estimated market valuation for lingerie in India was 3 Billion of which 1% was online last year and projected to increase 5 Billion in next 5 year (Krishnamurthy, K.,2015), broken customer experience, increasing customer loyalty towards brands zivame, 60% of revenues comes from in-house products (Mehta, J., 2015).

[d]Series A funding means the first round of financing undergone for a new business venture after seed capital. (Source http://www.investopedia.com/)

[e] Series B funding means the second round of financing for a business by private equity investors or venture capitalists. (Source http://www.investopedia.com/)

[f] Series C funding means a venture capital firms goes for this round of funding when the company has proved its mettle and success in market. (Source http://www.startupfreak.com/)

[g]Private Label Strategy - Private-label products or services are typically those manufactured or provided by one company for offer under another company's brand. (Source - https://en.wikipedia.org/wiki/Private_label)

Some remarkable steps taken by zivame.com as a part of corporate social responsibility were "Gift a Bra" program for underprivileged women with the support from NGO Goonj[h], awareness campaign against wrong bra size (Zakaria, N., 2015), with the support from NGO Milaap[i] arrange special screening and awareness camp in rural areas and advise women how to select correct lingerie (Gore, D., 2014). Future plan of zivame.com is to open 100 Fitting rooms to consult the size and help the customer select right fitted product online (Prabhakar, M., 2015).

BLUE OCEAN STRATEGY

A business or nonprofit organization or leaders of the government never want to drive in a market where the level of competition squeezes their investment and a market became Red Ocean day by day, where every companytries to invest for survival, not for sustainable growth. But the situation became worse when companies are not able to distinguish their position in the market on the basis of creating value for their stakeholders. How can you address this challenge? , a question raised by Blue Ocean strategist distinguish Professor from INSEAD W. Chan Kim and Renee Mauborgne.

To address this challenge professors came with some innovative solutions in the year 2005 with a concept called "Blue Ocean Strategy" which now became a part of Business terminology, the author released an updated edition in March 2015 by addressing challenges faced by Industries while implementing Blue Ocean Strategy and also design new tools, lessons, and framework to overcome the hurdles of implementation.

Blue ocean strategy is a concept focused on two aspects, first to find uncontested market space and value innovation. In a competitive business world, creating buyers' value is not enough, companies should find certain moves which create sustainable business for a longer period of time. Blue Ocean Strategy design with the same objective. Blue Ocean Strategy is a systemized and sequential process to break the traditional boundaries of competitive strategy by identifying value innovation and simultaneous pursuit of low cost and differentiation. (Kim & Renee Mauborgne, 2005)

[h]Goonj is a non-governmental organisation based in Delhi, India which undertakes disaster relief, humanitarian aid and community development

[i]Milaap is non-governmental organization the Association of People with Disability (APD) is a non-governmental organization working in the field of disability for the last fifty six years.

LITERATURE REVIEW

In the first edition, the author designed the concept, and prepare framework and tools for implementation by studying 30 diversified industries for the period of 1880 to 2000, they studied 150 strategic moves in these industries and come up with a concept called blue ocean strategy. The first edition studied about what way industries are able to sustain by applying the concept called uncontested market space and value innovation. Whatever the data collected is on the basis of past data and records. What about current business, how they can apply Blue Ocean Strategy, this was the first challenge faced by the author while implementing, they help 30 plus companies, government, and nonprofit organizations (Kim & Renee Mauborgne, 2005).

During the journey of implementing most of the organization facing three challenges, which required be answered. The challenges are: How do companies align all their efforts towards achieving blue ocean strategy? What steps organization can take while blue ocean turning red? How can companies avoid red ocean traps while pursuing Blue Ocean? (Kim, W. C., & Mauborgne, R. 2015). These questions addressed in a new edition with appropriate tools, framework, and lessons. Creating and capturing Blue Ocean is required to achieve a sustainable business strategy for high performance.

The three strategy propositions, i.e. Value propositions, profit propositions, and people propositions. The conceptually designed with an objective to make companies high performance and achieve sustainable growth. Value propositions explained utility buyers receive minus price pay for it, profit propositions explained revenue generates an organization from an offering minus the cost to produce and deliver it, people propositions the positive motivations and incentives put in place for the people needed to support and implement the strategy. The alignment between these propositions is must create sustainable growth because, Blue Ocean Strategy not only create differentiation and low cost simultaneously, but it also creates value for their stakeholder also. Failure of Tata Nano is the best example which doesn't meet the three strategy propositions and failed to address sustainable growth in spite of initial success. Next issue discusses in updated edition was how to renew Blue Ocean, it is not easy in a dynamic business. First thing authors highlighted where it is not onetime, but it can be continued by identifying new Blue Ocean waves. To create new waves of Blue Ocean companies need to study the barriers known as alignment barrier, cognitive or organizational barrier, brand barrier, economic and legal barrier and then need to identify at what level the barrier required change business level or corporate level. Through these process business can overcome the limitation of the imitative market condition.

A third issue addressed was Red Ocean traps, Red Ocean traps occurred due to perspective and people's mental model for traditional theories, these happen due to pre-existing knowledge and backgrounds. There are ten red ocean traps which consider as traps i.e. Blue Ocean Strategy is customer oriented, need to diversify from your core business, about new technologies, first to market, differentiation is same as Blue Ocean, focus on low prices too, same as innovation, niche marketing strategy, competition is bad, synonymous with creative disruption or destruction. By putting these theories in expanded editions, authors want to achieve the ultimate goals of implementing Blue Ocean Strategy into practice.

The Research Methodology

To discuss whether there is a significant relation between zivame.com strategy and Blue Ocean strategy, in this section different tools and procedures elucidated. To evaluate whether zivame.com is Blue Ocean or not following suggested tools use for study. The study carried out is analytical and empirical in nature, to study the significance of Blue Ocean Strategy at Zivame.com. To study data for analysis collected for the time period of August 2011 (started) to September 2015 (4 Years). The present study covers the analysis of Zivame's different strategy used to start a business and different techniques used to earn profit plus the highest number of customers in the shortest period of time by 400%. The study will provide detailed analysis how zivame's value innovation helps the organization to be sailed in Blue Ocean. It will also describe what steps taken by zivame, help organization to be a strong player in the business of online lingerie store. It provides a base to analyze the impact of Blue Ocean Strategy in terms of identifying uncontested market space and how zivame achieved the highest growth rate and sustainable position in the unorganized lingerie business in India.

Objective of the Study

- To study whether zivame.com India's first online lingerie store is Blue Ocean or not.
- To study whether sailing in Blue Ocean deliver sustainable growth to the zivame.com.
- To find the relationship between value innovation adopted by zivame.com and its impact on the growth rate of business.

To study the relation, the data from various authentic sources like ZIVAME's website, articles published in reputed newspaper, magazine, journals and websites for the above mentioned period i.e. August 2011 (started) to September 2015. To study the Blue Ocean Strategy suggested tools and framework used are

Strategy Canvas Model, Four Action Framework, Three Characteristics of Good Strategy, Three Tiers of Noncustomers, Six Paths Framework and Value Innovation *(see Figure 1)*.

RESULTS AND DISCUSSION

This section explained the different tools and framework designed by the INSEAD Professor from INSEAD W. Chan Kim and Renee Mauborgne. All the framework used with an intention to use the study strategy and steps taken by zivame.com to sail in Blue Ocean.

Strategy Canvas Model

The Strategy Canvas model is both a diagnostic and action framework to study the current state of the market and help organizations to find space to convert current practices into Blue Ocean. Figure 2 represents a graphical form of presentation of zivame.com, how zivame.com sailing in Blue Ocean and creating uncontested market space in Indian lingerie business, bring lingerie online is the most challenging things, but technology disruptions make it happen. The horizontal axis of the graph represents in what factors industry competes and how the zivame.com is creating an uncontested market space in unorganized lingerie market space.

Zivame.com business model became sustainable after having appropriate market research by founder Richa Kar, who identifies certain key factors on which current business is not focusing or ignoring. The factors which identified during market research were male salesmen, privacy, availability of size, low ease of selection, wrong size selection, price, after sales service. By studying these factors, she came up with an innovative solution and brings the concept selling lingerie online all over the India. E-commerce is on the pace of growth in India that create an opportunity for zivame.com. Zivame.com eliminated the concept of male salesmen, help women to find the correct size of lingerie, customer education, wide range and availability of all the size, adding international brands, and ease of selection, easy return, building a relationship through interactive CRM, fast delivery, and quality is a must. These all notion became factors of sustainability for zivame.com and due to this number of customer increase from 6500 to 250000 in just the first 6 months of business.

Four Action Framework

The Four Action Framework tools focus on creating buyers value by breaking the tradeoffs between differentiation and low cost strategy. The four question series helps companies to understand the level of

competitiveness and design new value curve for uncontested market space i.e. Which factors need to eliminate, reduce, raise and create?

The ERRC framework *(see Figure 3)* of zivame.com, *eliminated* medium of selling – male salesman and taboos for lingerie product, *reduced* abrupt selling, and immediate payment, *raised* quality of product by private label strategy, effective pricing, 24 x 7 interactive CRM where women customer care consult, guide and express the importance of perfect selection of the product, maintain privacy by discreet packaging and 15 day product return policy with no question ask concept and *created* a platform where all the size of product which is not available in normal market, availability of international brand, product selection according age group, body size, preference, specific requirement for body fitness, bra-size calculator to select exact size product, customer education and feedback and most important ease of buying as per the convenience and time.

Three Characteristics of Good Strategy

The Blue curve in figure 3 represents zivame.com focusing on alternatives which current market is ignoring while selling products. To validate whether zivame.com is compelling towards Blue Ocean or not, three concepts should validate first these factors i.e. Focus, Divergent and Compelling tagline of zivame.com. The zivame are *focusing* on ease of buying and maintaining privacy while shopping and delivering product, the focus is on women first and feeling safe, the *divergent point* of zivame is selling all the size with a number of varieties according to size, income, body, fitness and feelings and *compelling tagline* are consult for perfect selection and educate more for understanding requirement of body fitness first *(see Figure 4)*.

Three Tiers of Noncustomers

The universe of noncustomers represents enormous Blue Ocean opportunities. To convert this latent demand of *noncustomers* into real customers, companies need to understand first noncustomers and pace of opportunity available to contest in uncontested market space. To understand zivame.com noncustomers' customer *(see Figure 5)*. There three tiers of noncustomers are *First Tier* - Soon to be noncustomers, *Second Tier* – Refusing noncustomers, *Third Tier* – Unexplored noncustomers. To understand in detail *soon to be noncustomers* in lingerie business is in search for international brand and selections, *refusing noncustomers* looking for ease of buying and privacy, *unexplored noncustomers* searching for the precise fitting and availability of all the size. The third tier of noncustomers creates a

blue ocean curve for zivame.com where the market was failing to deliver expected requirement and became an uncontested market for zivame.com.

Six Paths Framework

A Blue Ocean theory based on the foundation of creating uncontested market space and reconstruction of market boundaries. Six Paths framework is a systematic way to study the industry boundaries and it help companies to take a decision based on space available to redefine market boundaries. The six basic approaches to creating a Blue Ocean are: Look across – alternative industries, a strategic group within industries, the chain of buyers, complementary product and service offerings, functional or emotional appeal to buyers, across time.

The zivame.com reconstructed market boundaries of the industries by looking at following perspectives which created uncontested market space are, lingerie business divided primarily in India is the three alternatives local retail market, malls and online marketplace through e-commerce. The strategic group within industries are housewives, working women, and young girls. The demography of chain of buyers is the internet user, working women, educated housewives and young girls. The complementary service offerings in the local retail market, malls, physical availability, immediate purchase, and exchange. The emotional things which expected by most of the buyers are consultation, availability of all the sizes, ease of selection, privacy, and discreet packaging. What makes these things possible is through expansion of internet facility all over the India, which across the times brings ease of buying, availability of internet facility and purchase through a mobile application *(see Figure 6).* Zivame creates value for buyers, which create three things for customers, i.e. ease of buying, customer education and privacy that delivers satisfaction to their customers.

Value Innovation

A leap in value for both buyers and companies known as value innovation. The leap in value can be achieved by creating a sequence of exceptional utility, strategic pricing and target costing based on win-win games for buyers and companies, delivering the product or service with low cost and differentiation simultaneously. The strategic moves designed in value innovation leads to rapid cost advantages for both buyer and companies.

Zivame.com created a leap in value by creating online lingerie e-commerce store where the customerhas ease of buying, customer education, bra-size calculator, and consult for the exact size, interactive CRM

and availability of size. These factors converted noncustomers into customers for zivame.com because it created value for them. The ease of buying, the addition of international product, customer education and consultation and availability of all the size reduce the gap between noncustomers and companies because it created a leap in value for them *(see Figure 7)*. The online platform brings both low cost and differentiation both simultaneously, which became unique value innovation in selling lingerie in the unorganized lingerie market of India.

CONCLUSION

The research work done to find whether zivame.com adopted Blue Ocean Strategy or not, have some key findings as follows:

- The growth rate in number customers purchased online lingerie were increased by 400% from August 2011 and annually increase by 300% due to ease of buying, privacy, customer education and consultation.
- The investment made by a different venture capitalist increase of 3 million dollars to 40 million dollars by September 2015, which shows how potential is a business and sailing in uncontested market space brings new segmentation in India's unorganized lingerie market *(see Figure 8)*.
- There is a significant relationship between adopting Blue Ocean Strategy and growth rate of business.

LIMITATION OF STUDY

The research work with utmost diligence and taken due care to cover up the aspects related to study the concept, lack of adequate time and resources forced the author to consider limited variable for study. The limitations of the study are:

- The financial data sheets are not available to check the profitability in details.
- The period of study is limited August 2011 to September 2015 (4 Years) because zivame.com started in 2011.
- To study the relationship the tools used which is suggested by authors of Blue Ocean Strategy, other tools may apply to check the relevance.
- There may be other factors have impacted on the growth rate of business which could not be identified due to the limitation of tools used for the study.

FIGURES

Step 1 Strategy Canvas Model

- Visual cue describing the strategic landscape of business tomorrow.

Step 2 Four Action Framework

- Structure that encourages planners to reduce the cost structure of the industry and increase buyer value by asking four questions about their business.

Step 3 Three Tiers of Noncustomers

- Noncustomers can be defined in three tiers "Soon to Be", "Refusing" and "Unexplpored"

Step 4 Six Path Framework

- Structure that provides planners a variety of "paths" to "value innovation."

Step 5 Value Innovation

- A cornerstone of Blue Ocean Strategy, making competition irrelevant and by creating leap in value for buyer and a company.

Fig. 1 Research Process - Blue Ocean Strategy – zivame.com

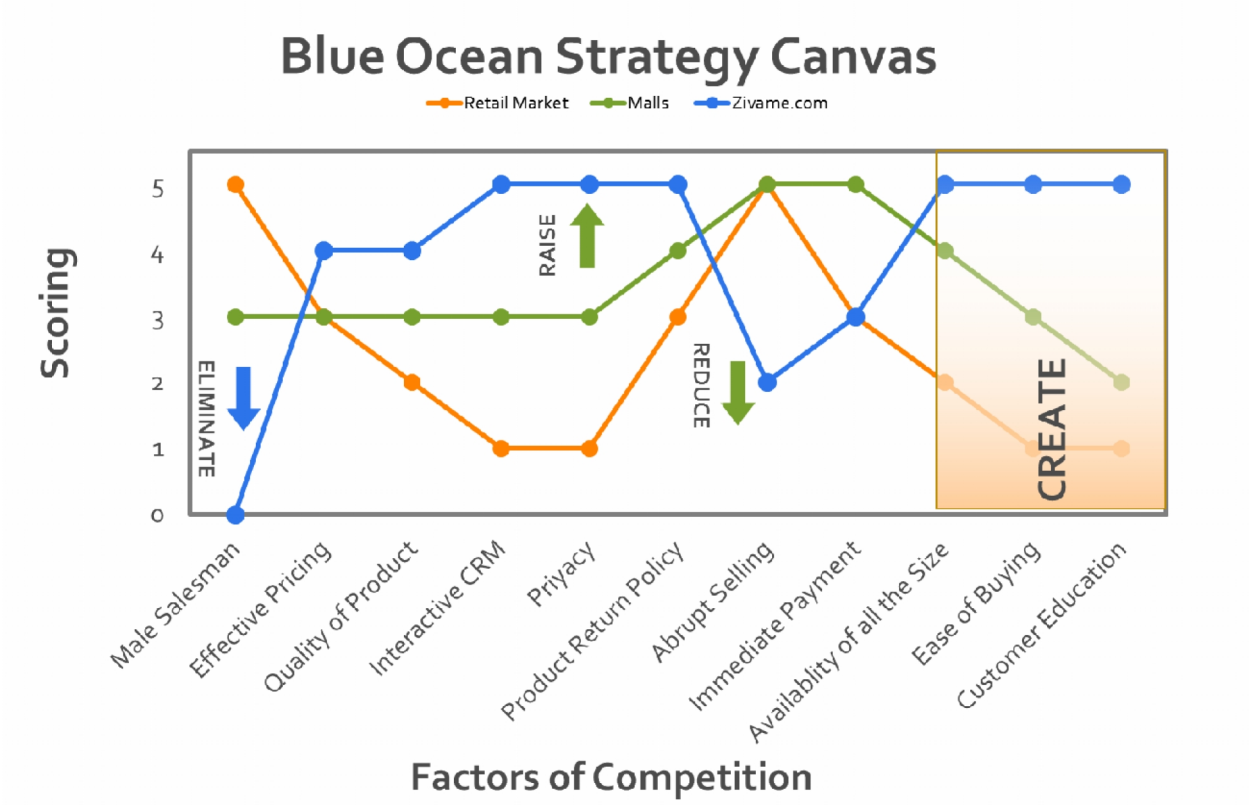


Fig.1 Blue Ocean Strategy Canvas – zivame.com

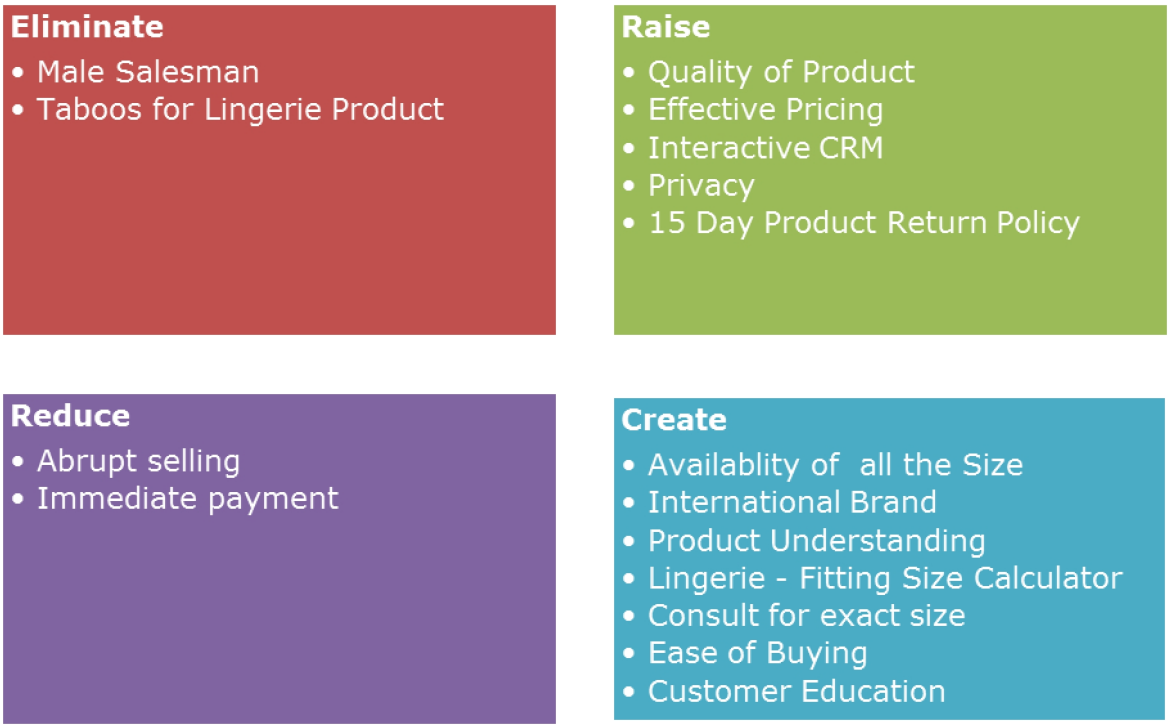


Fig.3 ERRC Framework – zivame.com

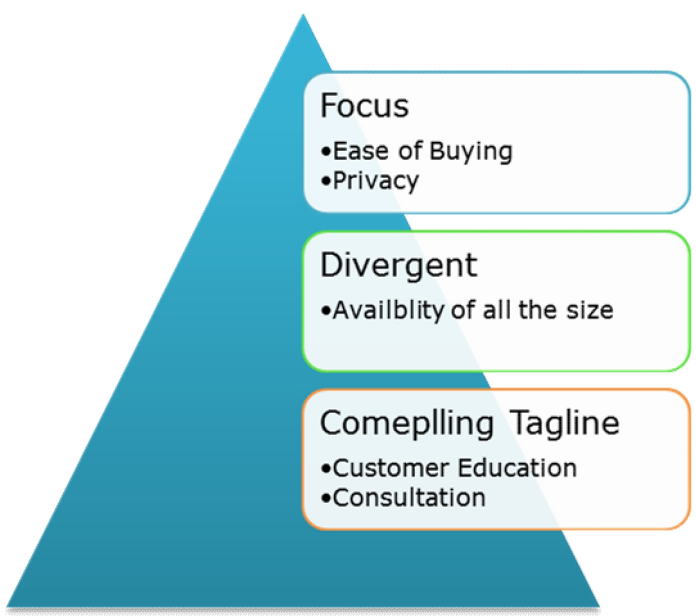


Fig.2 Three Characteristics of Good Strategy – zivame.com

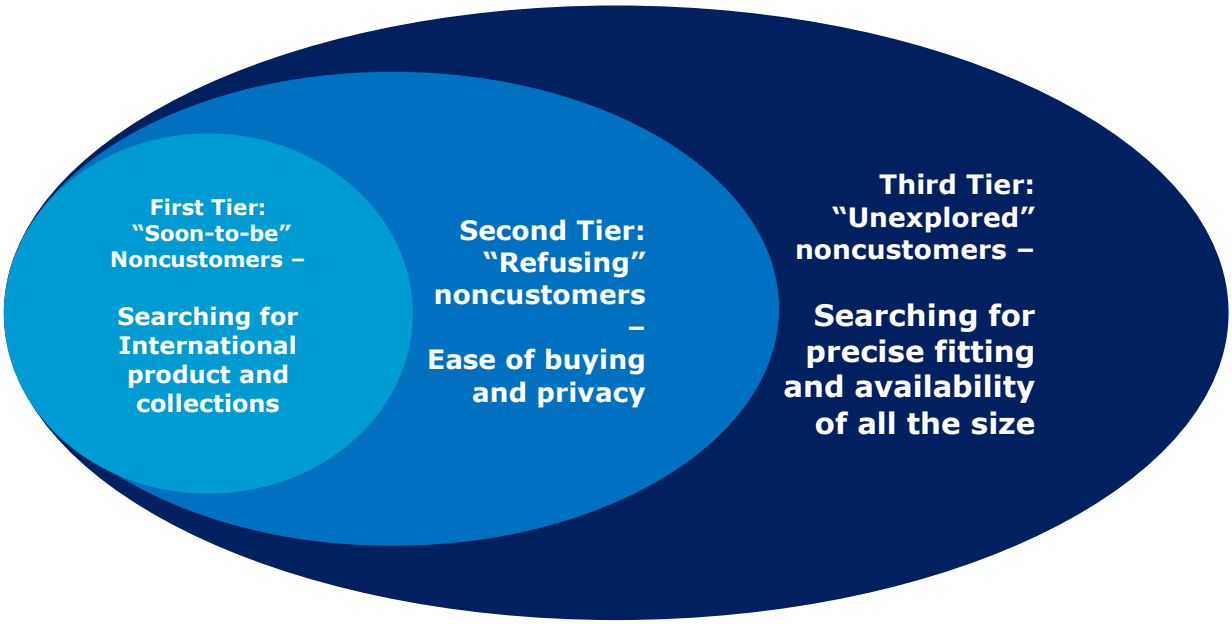


Fig. 4 Three Tiers of Noncustomers – zivame.com



Fig. 5 Six Path Framework – zivame.com

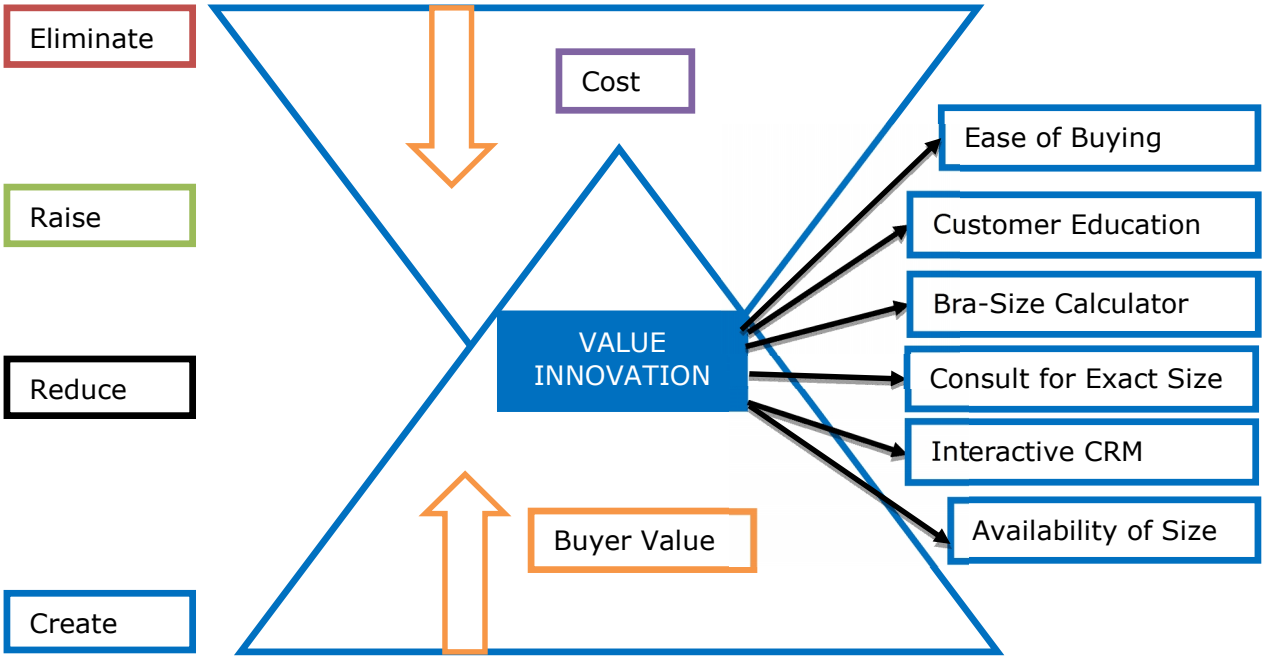


Fig. 7 Value Innovation – zivame.com

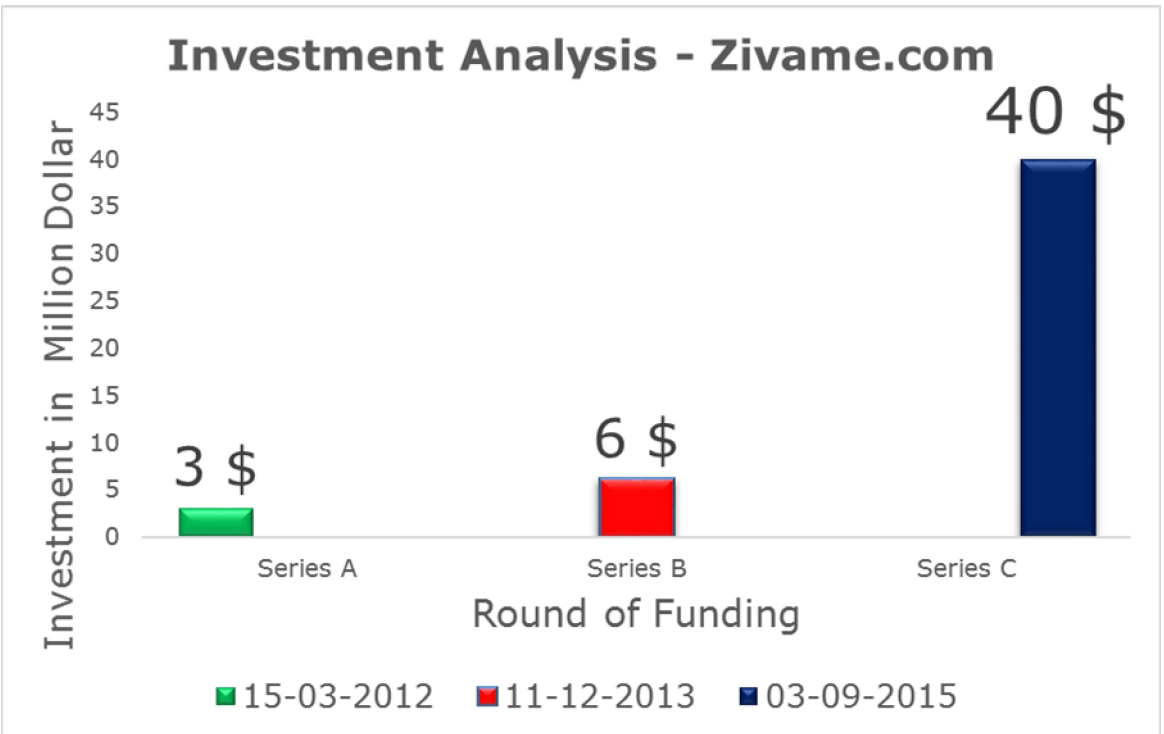


Fig. 6 Investment Analysis – zivame.com source – www.zivame.com

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(Proceedings available for download at rku.ac.in/icre)

RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

Liquidity Analysis of Sun Pharmaceutical Company Limited

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ABSTRACT

Working capital management plays very important role in any organization.It is needed to payment of day to day activities and short term obligations. It is very important to maintain optimum level of working capital because shortage of it and excess amount of fund both become harmful to organization. Here, Sun pharmaceutical company limited selected for working capital analysis. Data was collected from websites and annual reports of ten years andanalyzed with the help of Ratios and statistical techniques. It can be concluded from the data that company having good working capital position till 2012-13. But from last two years it shows decreasing trendand became negative in last year of study period it may be because of good credit availability from suppliers.

SUMMARY

The study shows working capital management and liquidity position of the Sun Pharmaceutical Ltd.

Keywords: Liquidity, Current Assets, Current Liabilities, Current Ratio, Liquied Ratio

INTRODUCTION

Working capital management plays a very important role to run any business. It is needed to run day to day activities as well as pay off short term obligations. It can be said lifeblood of any firm.It is very important for any company or organization to manage working capital at an optimul level. Because shortage of working capital may become dangerous for company it can disturb production continuity as well as leads closer to the company. Besides excess amount of funds invested into current assets affect profitability of the firm.

LIQUIDITY ANALYSIS

Liquidity analysis refers to proportion of liquid assets with firm's liabilities. It is very important to manager to maintain optimum level of liquidity. It shows how firm meet its liabilities in a within time with a minimum cost. Here, liquidity position of the Sun Pharmaceutical Limited analysed with the help of financial ratios.

ABOUT THE COMPANY

Globally, The Indian pharmaceutical industry has become the third largest producer in the world and is poised to grow into an industry of $20 billion in 2015 from the current turnover of $12 billion. [1]Mumbai, Hyderabad and Ahmedabad are the major pharmaceutical hubs of India. Sun Pharmaceutical company having highest rank in market capitalisation with 2,17,636crores.Sun Pharmaceuticals was established by Mr.DilipShanghvi in 1983 in Vapi with five products to treat psychiatry ailments. Cardiology products were introduced in 1987 followed by gastroenterology products in 1989. Today it is the largest chronic prescription company in India and a market leader in psychiatry, neurology, cardiology, orthopedics, ophthalmology, gastroenterology and nephrology. After the acquisition of Ranbaxy in 2014 it become the largest pharma company in India, the largest Indian pharma company in the US, and the 5th largest specialist generic company globally.[2]

MATERIALS AND METHODS

REVIEW OF LITERATURE

Shin and Soenen, (1998): Studied that there was a great effect of working capital management on creating value of shareholders. The working capital had effect on profitability as well as liquidity. The statistical method correlation and regression analysis used to study relationship between the length of Net Trading Cycle profitability and a risk adjusted stock return. The finding shown negative relationship between the firm's net trading cycle and profitability.

Ghosh and Maji, (2003): studied in this paper the efficiency of the working capital management of the cement industry for ten years. Indices used for measuring working capital management, performance, utilization and overall performance. The results shown the selected cement industry did not perform well during the study period.

Eljelly, (2004): this study examined the relationship between profitability and liquidity, for this statistical technique like correlation and regression were used. Current ratio and cash gap (cash conversation cycle) also calculated to drawn conclusion on the basis of samples selected from the joint stock companies of Saudi Arabia. Results revealed negative relationship between the profitability and liquidity. Major variation found among the companies while measuring the liquidity.

Doutora, (2013): analyse the effect of working capital on firm's profitability with reference to Portugal. For analysed collected samples non-linear and panel data methodology with the help of applying fixed effects model with robust standard errors used. Result found concave relationship between working capital and profitability. Selected firm's used optimum level of working capital.

Dr. PushplataChouksey and and Miss RakhiHotwani, (2013): examined the liquidity position of Bajaj Auto Ltd for a ten years period with the help of ratio analysis, standard deviation and C.V.. Study concluded that the selected firm not maintained enough liquidity to pay of short term liabilities.

Srinivas K. T., (2013): studied the working capital management through ratio analysis with reference to Karnataka Power Corporation Limited. Secondary data collected for five years to examine working capital management of it. Results shown there were company maintained good liquid position for payment of liabilities. Company was trying to increase production and profit ratio.

OBJECTIVES OF THE STUDY

- To analyse the working capital management of the selected company
- To analyse the liquidity position of the selected company

PERIOD OF THE STUDY

Here, for analyse the working capital management andliquidity position of the Sun Pharmaceutical Ltd study covers ten year's period from 2005-06 to 2014-15.

COLLECTION OF DATA

Secondary data is used for analyse the working capital management and liquidity position of the selected company. For this data is collected from various websites and annual reports for ten years i.e. 2005-06 to 2014-15.

DATAANALYSIS

Collected data analysewith the help of Ratio analysis as well as statistical techniquesto study working capital management and liquidity position of the selected firm. Current assets, Current liabilities and Cash & balances analysed with the help of various components of working capital and conclusion drawn from this.

RESULTS AND DISCUSSION

Working Capital Management of Sun Pharmaceutical Ltd

Working capital works as a lifeblood for a company if firm wants to keep continue its production activity it is needed to maintain working capital properly. Working capital may be defined in a two ways current assets or Current assets minus Current liabilities. Current assets includes inventories, sundry debtors, cash and balances, loan and advances etc which converted into cash easily and production activity being continue. While current liabilities includes sundry creditors, unclaimed dividend warrants, short term credit, tax provisions etc. Funds invested into current assets get converted into cash within a short time. The below Table shows the Current Assets and Current Liabilities of Sun Pharmaceutical Ltd

Liquidity Ratios of Sun Pharmaceutical Limited:

The above table – 2 shows the liquidity parameters to measure the liquidity position of the Sun Pharmaceutical Company Limited for ten years.

Current Ratio

Current ratio shows working capital position of the firm. It is also known as a working capital ratio. It shows excess amount of current assets than current liabilities. It is used to judge firm's short term capacity to pay off its obligation. Current Ratio =

Current assets/current liabilities. Higher ratio shows good position of the company to pay off its obligation in time.

Traditionally current ratio of any production firm is 2:1 considered good except banking sector. It's also depends on nature of business. Table – 3 shows the fluctuation trend in current ratio of sun pharmaceutical ltd. It is varied -0.41 to 6.82 times during study period. It shown from the table that in 2005-06 current ratio is 6.82 times which increases in 2006-07 with 1.49 times. Then after in 2007-08 it decrease 3.20 times. From 2010-11 current ratios shown decreasing trend till 2013-14. In last year 2014-15 current ratio reveled negative with -0.41 times.

Table – 3 shows Current ratio with an average of 3.17 for ten years with 2.10 and 66.24% Std. Deviation and C.V. respectively. Standard deviation shows high difference between the current ratios of study period. There was a big difference between the ratios of 2005-06 to 2014-15. In lasttwoyear company have been merge with Ranbaxy and became India's first rank pharmaceutical company. Its increases companies creditability in suppliers so, investment in current assets became law.

Liquid Ratio

Liquid Ratio refers the liquid position of the firm. It is also known as a quick ratio or acid test ratio. Liquid Ratio = liquid assets / liquid liabilities. Here, a liquid asset is equal to short term liabilities minus inventories. This ratio shows firms ability to pay off short term liabilities from liquid assets. Mostly the liquid ratio 1:1 shows ideal position of the firm to pay off short term liabilities from the liquid assets.

From the above Table – 3 it can be observed that liquid ratio shown fluctuating trend during the study period. In 2005-06 current ratio was 5.86 times which decreased continuously for a two years and reached at 1.67 times in 2007-08. Afterwards ratio shown steady position for two years with minor difference but in 2010-11 it increases with 2.40 times. In last two years of study period it shown with 0 .17 and -0.41 respectively in 2013-14 and 2014-15.

Average ratio of the above company shown 2.23 which shows that firm have a ability to pay off its short term liabilities within stipulated time but excess amount of cash invested in liquid assets refers loss of optional income. The standard deviation and C.V. 1.91 times 86.08% respectively shown high variation between the liquid ratios of the firm during the study period.

Cash Ratio

Cash and bank is a highly liquid asset from current assets. This ratio shows amount of cash in proportion to current liabilities. Cash ratio = Cash and Bank/Current liabilities. Traditionally 0.5 consider ideal ratio. This ratio measures cash availability to pay off obligations.

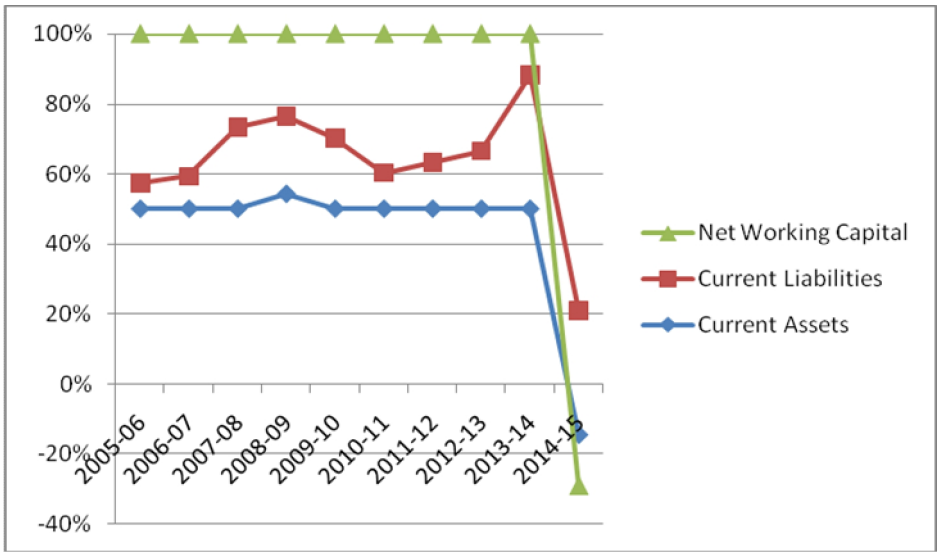
From above table-3 it showsthat cash ratio of the selected firm has been in a very low position during the study period. It revealed firm's good credit in a market and suppliers. The table-3 shows average ratio with0.04, standard deviation and C.V.0.03 and 66.88% respectively. It can be concluded that company adopted low cash maintenance policy. The C.V. revealed huge difference between cash maintenance policy of the selected firm.

CONCLUSION

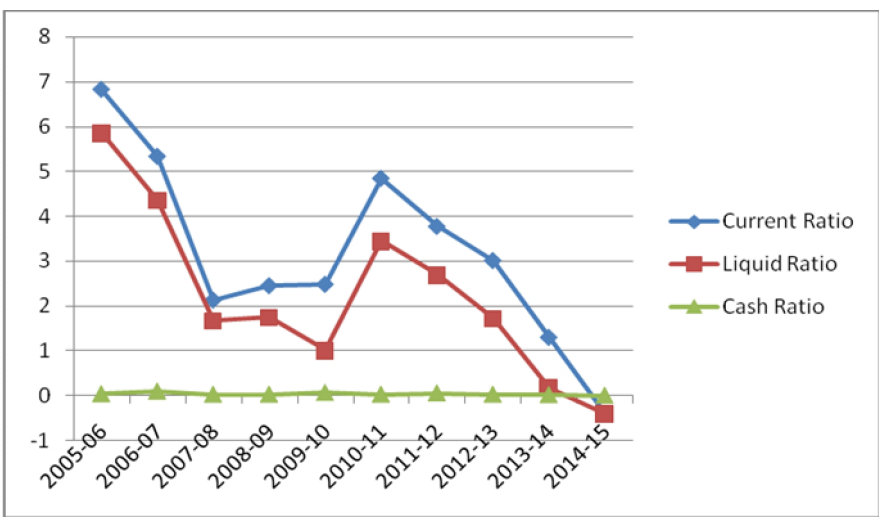
Liquidity plays a very important role to run any business. It is needed to run day to day activities as well as pay off short term obligations. It can be say it is lifeblood of any firm. Liquidity management is very important for any company. That's why in this study the liquidity position of the Sun pharmaceutical limited anlysed with the help of very famous liquidity ratios. For this three ratios calculated with the help of financial data of the company. The result discussed that sun pharmaceutical company having average good current ratio and liquid ratio to pay off firms short term obligations within time. Average Cash ratio is not good as to pay off short term obligation. It may be the reason of company's good credit availability from suppliers.

FIGURES

"**Fig. 1**Current assets and Current liabilities"



"**Fig.-2**Liquidity Ratios of Sun Pharmaceutical Ltd"



TABLES

"**Table 1.**Current asset and Current liabilities of Sun Pharmaceutical Ltd"

(Rs. in crore)

Years	Current Assets	Current Liabilities	Net Working Capital
2005-06	1,864.15	273.30	1,590.85

2006-07	1,838.95	345.23	1,493.72
2007-08	1,803.10	845.73	957.37
2008-09	1,705.22	696.34	742.11
2009-10	963.11	388.45	574.66
2010-11	2,139.60	442.44	1,697.16
2011-12	2,254.47	598.67	1,655.80
2012-13	2,030.64	674.84	1,355.80
2013-14	1,053.53	807.89	245.64
2014-15	-1,942.62	4,746.95	-6,689.57
Average	1,371.02	981.98	362.35
Std. Deviation	1175.33	1337.40	2,528.01
C. V. %	85.73	136.19	697.67

Source: Annual Reports of the Company

"Table **2.**Current assets, Current liabilities, Liquid Assets Cash & BankBalance of Sun Pharmaceutical Ltd."

(Rs. in crore)

Years	Current Assets	Current Liabilities	Liquid Assets	Cash & bank Balance
2005-06	1,864.15	273.30	1,600.74	11.26
2006-07	1,838.95	345.23	1,505.57	35.69
2007-08	1,803.10	845.73	1,413.47	23.29
2008-09	1,705.22	696.34	1,218.48	20.17
2009-10	963.11	388.45	392.97	26.11
2010-11	2,139.60	442.44	1,521.40	11.26
2011-12	2,254.47	598.67	1,614.40	35.69
2012-13	2,030.64	674.84	1,161.88	23.29
2013-14	1,053.53	807.89	135.15	20.17
2014-15	-1,942.62	4,746.95	-1,942.62	26.11

Source: Annual Reports of the Company

"**Table 3.**Liquidity Ratios of Sun Pharmaceutical Ltd"

(Rs. in crore)

Years	Current Ratio	Liquid Ratio	Cash Ratio
2005-06	6.82	5.86	0.041

2006-07	5.33	4.36	0.10
2007-08	2.13	1.67	0.03
2008-09	2.45	1.75	0.03
2009-10	2.48	1.01	0.07
2010-11	4.84	3.44	0.03
2011-12	3.77	2.69	0.06
2012-13	3.01	1.72	0.03
2013-14	1.30	0.17	0.02
2014-15	-0.41	-0.41	0.005
Average	3.17	2.23	0.04
Std. Deviation	2.10	1.91	0.03
C.V.%	66.24	86.08	66.88

Source: Annual Reports of the Company

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

(Proceedings available for download at rku.ac.in/icre)

RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

A Study on Firm Performance of IT-ITES Organization: Financial and Non-Financial Perspective

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ABSTRACT

Organizations make most of their investments in Capital and Human Capital, for HR Personals or Managing Heads of the organization, it becomes important to know whether HR Practices like Staffing, Training, Performance Appraisal, Benefits give those advantages like ROI, Productivity, Reduced Turnover etc. However, organizations always want a way to diagnose their HR investment and its return, and to explore whether Human Resource Practices links their Firm Performance. This empirical study with primary data collected from HR Heads/Managing Heads of selected IT-ITES

organizations of Vadodara and Ahmedabad in central Gujarat. The researchers are able to study performances in terms of financial and non financial. ANOVA reveals that there exist a relationship among employees looking for better jobs outside, Return on Investment gets affected by investing in HRM Practices, and Financial Performance of organization gets affected by HRM Practices and Organization size.

SUMMARY

The researchers have studied financial and non financial performances. The present study indicates that majority of the statements related to firms' performance are having relationship with size of the firm. This means that overall firm performance depends on size of the firm. Those organizations which invest better in HRM practices to achieve better firm performance are also related to size of the firm.

Keywords: ***Firm Performance, Employee Performance, Organization Performance, Financial Performance***

INTRODUCTION

The requirement for answerability in terms of investment-return aspect among all business functions has been increasing day in and day out. Many researches demonstrate HR decisions and its importance in an increasingly and uncertain interconnected world. The key job of Organization Heads, HR Heads, and Managing Heads of the organization is to articulate the logical links between progressive HR practices and performance of firm. Investing in human resource practices should be as methodical as investing in other vital resource, based on rational frameworks and purposeful on optimization, not simply on falling expenses or mimicking best practices of other benchmarked organization.

As per the research of Hay Group the companies listed in Fortune as world's most admired or to be worked with companies invest in human resource and foresee as the assets to be developed and not the cost to simply be cut. As per it, 3 out of list of 64 most admired companies like UPS, Disney, McDonald's, and Marriott International were successful enough to manage their people during the Great Recession, compared to competitor players. These companies hardly laid off their resources say for eg 10% as compared to 30% of their competitors and with that they were on the top to brand themselves as one of the best employers and not just the marketers to their clients. This is possible because they consider their people as assets, not expenses. Conceivably the most significant message from the 2010 World's Most Admired companies is that they did not begin their enlightened human capital philosophies when the recession hit; they'd been practicing them for years. It becomes late after recession starts. "Champions know what their most valuable asset is, and they give it the investment it deserves—through good times and Bad".

Business privileged and employees routinely are expected to have the basics that explain how decisions about funds and customers connect to organization success. Even non-finance people are expected to understand philosophy of cash flow and return on investment; similarly even non marketing people are expected to understand principles of market segmentation and product life cycle, so they are expected to know how human resource management systems can enhance organization success to decisions about their own talent.

MATERIALS AND METHODS

FIRM PERFORMANCE DEFINED

As defined by (Becker and Huselid, 1998) (2), (Horngren et al., 2000) (9) although there are various stakeholders in an organization, the chief goal of any organization is

higher financials and wealth generation for stakeholders. As per (Curtis et al, 1995) (4) financial performance of an organization depends on operational performance. The operational performance of an organization is a function of people, process and technology and as per (Huselid, 1995) (10) and (Barney and Wright, 1997) (1) for effective interactions of people with technology and process, the people in the organization have to be competent enough, with the required knowledge, skill and abilities. The perception of (Brooks, 1987) (3) and (Wynekoop and Walz, 2000) (18) is that the competence among individual is an important factor for operational effectiveness for providing timely quality products and services. At the same time an array of authors like (Latham and Wexley, 1981) (14) (Snell and Dean, 1992) (16); (Lado and Wilson, 1994) (13) (Terpestra and Rozell, 1993) (17) (Koch and McGrath, 1996) (12) (Pfeffer, 1998) (15) viewed HRM practices like staffing, training, work environment and performance appraisal may enhance the competence of employees for higher performance.

The ultimate goal of a business is higher financials and maximisation of wealth for stake holders as per (Horngren, C.T., Foster, G., and Datar, S.M., 2000) (9) and (Becker & Huselid, 1998) (2). Nonetheless, attaining the organization's goals depends upon the extent to which its organizational performance is reached that is what (Katou & Budhwar, 2007) (11) believed. (Delaney & Huselid, 1996) (5), (Dyer & Reeves, 1995),(6) (Guest, 2001) (7), (Katou & Budhwar, 2007) (11) were of opinion that organizational performance is generally indicated by effectiveness , efficiency , customer and employee satisfaction, innovation, quality of products or services, and ability to maintain an exclusive HR pool.

IMPORTANCE OF HRM PRACTICES-FIRM PERFORMANCE

The importance of these topic will be apparent when senior HR heads, managing heads of your organization will be able to pose some heart wrenching HRM practices – performance issues like---"I can see that there exists correlation between human resource practices and sales revenue across various branches, but does it mean that if we invest more on providing best HR practices our sales will raise?, or another issue like "on any given day, about 5 percent of employees remain absent. Even though my present employees are in a state to cover for absent employees and at the end the work gets done. So the question remains as should we make efforts to reduce the absent rate? Which would benefit our organization or the commitment level of present employees is high to do work on behalf of others?, still another serious one issue like "if our turnover rate is higher than that of our competitors, what our HR should institute to bring it at par, because it is quite possible employee turnover directly affect cost and benefit of the organization?, still one more to go like" there are various low-priced health-care and insurance programs available in the market to offer to our employees which may reduce our cost, but does it shows an exit to talented employees or the doors for skilled employees gets closed forever?, more on " does there exist any sound relationship between personal-professional life conflict and organization productivity, if so than how to know the links to tract the benefits of providing family friendly programs to organization?, further " are great performers that different from routine performers?, if so how to handle high performers and how to motivate low performers and that too by not exceeding cost", still on " is it that worth to invest heavily on fresh bloods to improve their work, if yes, then whether it will save future time and money both in terms of staffing?

BACKGROUND OF THE STUDY

In order to proceed with the above mentioned study, the researchers had undergone the empirical study of IT-ITES organizations of Vadodara city, and found that Human

Resource Planning, Recruitment and Selection, Staffing, Training and Development, Compensation, Pay and Benefits, Performance Appraisal, Work-Life Balance or Family-Friendly workplace etc are widely practiced HRM practices. Proceeding towards next step was to identify the performance indicators of the IT-ITES organizations. After detailed literature review on performance indicators or firm performance, the researchers limited his reach up to two widely categorized performances i.e. Financial Performance and Non-Financial Performance. Detailing further to the extent of categorizing Financial Performance into Return on Investment, Market Share and Financial Profit and Non Financial Performance in to Employee Performance like Commitment, Absenteeism, Turnover and Organizational Performance like Productivity, Quality and Service.

OBJECTIVES

The purpose of this research is to study financial and non-financial performance of the selected IT-ITES organizations and its relationship with size of the organization.

RESEARCH METHODOLOGY

The current study is a descriptive study with an objective to study financial and non-financial performance of the selected IT-ITES organizations and its relationship with size of the organization. For this primary data is collected through personal survey method. The data is collected through a structured questionnaire sent to senior HR heads / Managing heads of about 50 IT-ITES organizations through email, out of which we received 36 samples. . The sampling technique taken on for the survey is non-probability convenience sampling technique.

Definition: Size of the organization (1) Small size is defined as 5 to 149 employees (2) Medium is defined as 150 to 300 employees and (3) large is defined as more than 300 employees.

DATA ANALYSIS

For the current study Analysis of Variance is carried out with an aim to know the alliance between size of the organization and performance of the firm. For the same the subsequent hypotheses were framed showed in Table 1.

Table 2 indicates ANOVA for size of the firm and employees leaving the job or looking for better opportunities outside. Return on Investment gets affected by investing in HRM practices and size of the organization. Financial performance of the organization gets affected by investing in HRM practices and size of the organization As seen in table 2 the significance values of 3 statements are statistically significant i.e. are less than 0.05. Hence for all these assertions the null hypothesis can't be accepted. This demonstrates that for these statements there is a link between employees looking for better opportunities outside, ROI gets affected by investing in HRM practices, financial performance gets affected by investing in HRM practices and size of the organization.

RESULTS AND DISCUSSION

The empirical research study on *a Study on Firm Performance of IT-ITES organization: Financial and Non-Financial perspective* indicates that (1) There exist a relationship between size of the organization and its employees actively looking for better jobs outside. This means that in smaller organizations employees look for opportunities in other organizations for growth, which is lesser found in large organizations where employees are getting enough in-house opportunities. (2) There exist a relationship between size of the organization and the effect on ROI by investing in HRM practices. This means that those organizations that are relatively large in size can afford investing in HRM practices and by that can leverage in return on investment. (3) There exist a relationship between size of the organization and its Financial Performance getting affected by HRM practices. This means that organization which are relatively large in size, can achieve better financial performance by sound HRM practices.

Thus the organizations has to focus on Financial Performance in terms of Return on Investment, Market Share and Profit and Non Financial Performance like Employee Performance in terms of Commitment, Turnover and Absenteeism and Organizational Performance like Product Quality and Service in order to achieve overall Firm Performance.

LIMITATIONS

The present study is restricted to the companies in the State of Gujarat particularly Vadodara and Ahmedabad. Also only one sector has been included for analysis viz: IT-ITES. The analysis is limited to 36 companies. The researchers wish to deal with these restrictions in their future work.

CONCLUSION

The current study may be of help to IT-ITES organizations. The organizations get an idea that Firm Performance in terms of financial and non financial has relationship size of the firm. Those organizations which are able to invest more in hiring employees, invest more in providing best of the HR practices to the resources can get better advantages in terms of Employees Performance, Organization Performance and Financial Performance.

TABLES

Table: 1 Hypothesis

Hypothesis	Variable	Significance Value (From Table 2)

H0: There is no relationship between size of the organization and its employees actively look for better jobs outside.	Employees actively look for better jobs outside	**.003**
H1: There is a relationship between size of the organization and its employees actively look for better jobs outside.		
H0: There is no relationship between size of the organization and the effect on ROI by investing in HRM practices	Return on Investment gets affected by investing in HRM Practices	**.031**
H1: There is a relationship between size of the organization and the effect on ROI by investing in HRM practices		
H0: There is no relationship between size of the organization and its Financial Performance getting affected by HRM practices	Financial Performance of your organization gets affected by HRM Practices	**.009**
H1: There is a relationship between size of the organization and its Financial Performance getting affected by HRM practices		

Table 2: ANOVA of ROI, Financial Performance and HRM Practices Investment and Size of the Organization

		Sum of Squares	**df**	**Mean Square**	**F**	**Sig.**
	Total	14.171	34			

Employees actively look for better jobs outside	Between Groups	7.590	2	3.795	7.104	**.003****
	Within Groups	17.095	32	.534		
	Total	24.686	34			
Do you think that Return on Investment gets affected by investing in HRM Practices	Between Groups	4.804	2	2.402	3.866	**.031****
	Within Groups	19.882	32	.621		
	Total	24.686	34			
Do you think that Financial Performance of your organization gets affected by HRM Practices	Between Groups	7.790	2	3.895	5.541	**.009****
	Within Groups	22.495	32	.703		
	Total	30.286	34			

ACKNOWLEDGEMENT

I would like to express my sincere gratitude from the bottom of my heart to Dr Kunjal Sinha, Asso. Professor, C K Shah Vijapurwala Institute of Management, Vadodara for her continuous and significance guidance towards the study. Also my sincere thanks to Dr Kerav Pandya, Asso. Professor C K Shah Vijapurwala Institute of Management towards value addition of this study.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

(Proceedings available for download at rku.ac.in/icre)

RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

A Study on Antecedents and Structure of Brand Loyalty: Developing the Conceptual Model

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ABSTRACT

This paper is focused on the most intelligible and extensively pertinent antecedents of brand loyalty. For the same the authors have reviewed books, research journals and thesis available on internet. The study explores the existing literature on brand loyalty and brings out four major variables which are accountable for structure of brand loyalty which are like brand equity, brand association, brand trust and customer satisfaction. By undergoing detailed study into these four antecedents and their relationship with brand loyalty the authors feel that it will definitely affect consumers to change their attitudinal or behavioural loyalty which helps them to be brand loyal. The paper holds significant implications for academicians interested in dynamics of brand loyalty as well as the marketers of who are interested in building brand loyal customers.

SUMMARY

The study is aims to study the antecedents of brand loyalty.
K*eywords:* ***Brand equity, Brand Association, Brant Trust, Customer Satisfaction and Brand Loyalty***

1. Introduction

Brand loyalty is considered as one of the preferred subject for market researchers, winning brands are one of the most important ways for a company to achieve economical advantage and superior performance. In the present scenario the main workings for the brand is sustenance, sustainability and sustained advantage to keep hold of its customers which will make them brand loyal. One the most significant challenges for the brand managers are to understand the relationship between loyalty and its antecedents so that they can incorporate marketing strategies accordingly. This research article is emphasis on four major antecedents like brand equity, brand association, brand trust and customer satisfaction and their relationship with brand loyalty. The research paper is useful for developing the antecedents of brand loyalty.

2. Antecedents of Brand Loyalty: Conceptual Foundation

- **Brand Equity**

Brand equity something which is adding values of the products and services. Brand equity is perceived by (1, 21) as mixture of all the ingredients in creating brand, in addition to the brand name which contributes towards the product. It is being (29) continuously argue that strong brand equity increase the likelihood of brand choice and is also considered a tool for attaining aggressive advantage. Further Keller K.L. (22) defines brand equity from the customers' perspective as the difference in the mind of consumers about the brand can affect marketing of the brand. Brand equity is a condition through which consumers can recall their positive strong and exclusive

ssociation with the brand.Based on the above definition brand equity is conceptualised as it is brands' value in consumer's mind.

- **Brand Association**

Brand associations are the link between consumers' brand memory and brand meaning towards the brand (16). Brand association is the initial belief about the brand which comes in customer's mind and makes them brand loyal. Aaker (1), identify variables affecting brand associations such as; customer benefits, product attributes intangibles, celebrity/person, price, product usage, lifestyle/personality user/customer, product class, country of origin and competitors. In the similar study he describes product-related characteristics like product price, packaging and functional are those factors which influence brand association.

- **Brand Trust**

The term brand trust is established through long lasting relationship with the customers, and in the long run it could result in to brand loyalty. Marketers want to maximize profit and market share through loyal customers(8),the brand loyalty can be build through brand trust, which is defined as consumers' confidence and their expectations towards the brand (9). Many disciplines such as economics, psychology, sociology management and marketing have stated giving attention to the term brand trust. (9), but still the study of brand trust needs extensive literature on branding (8).

- **Customer Satisfaction**

Consumer Satisfaction is a key variable affecting consumer behaviour (10). It is widely accepted by marketers as an important factor for increasing market share. The term customer satisfaction is widely used as a benchmark by the marketers for evaluating company's performance (6). Furthermore, it is believed that a satisfied customer can exhibit loyalty behaviour in terms of repetitive buying behaviour and positive word of mouth publicity (32-6-30).

3. Brand Loyalty: Conceptual Foundation

Recently, Brand loyalty has received much attention of market researchers. But still the variables which determine customers' loyalty towards a brand are yet not clearly stated. (3, 26). Brand Loyalty is considered as an important construct for making a consumer psychology for purchasing a particular brand. (17-18-7).

Loyalty can be defined as behavioural response; (11) it can be further expressed as consumers' biased behavioural response expressed over time towards some specific brands. Brand loyalty helps marketers to estimate the customers' repeat purchase buying behaviour which leads to greater market share (19). Brand loyalty is considered as customers' assurance towards product or service and brand which will make them brand loyal. In more recent loyalty studies on brand loyalty, it is explained that brand loyalty can be considered as one of the strategic importance for the market researchers. Further, it is noted that loyal customers are easy to attract which may reduce marketing expenses. Ultimately, brand loyalty brings high rates of return on market expenditure and could increase market share. (14) In some recent study brand loyalty defined as attitudinal and emotional attachments of the consumer's towards their favourite brand (15). Emotional and attitudinal attachment of the customers towards particular brand can be considered as one of the significant construct of brand loyalty. Further, behavioural loyalty considered as another important construct of brand loyalty. It is to be said that a consumer who buy similar brand repeatedly are showing their behavioural loyalty towards particular brand (28).The attitudinal loyalty definition implies that loyalty is a mindset of the customers about the product or brand.If the consumer like the particular manufacturer, its products or brands, their attitude influence to buy from it, rather than its competitors. In strictly economic terms, the customer loyalty towards attitudinal is the consumer who is intentionally willing to pay a premium for particular brand than its competitor brand, even if the competitor offers nearly comparative or even lesser price

4. Relationship between Brand Loyalty and Its Antecedents

- **Brand Equity and Brand Loyalty**

The first significant antecedent of brand loyalty is brand equity known as one of the most powerful factors for both behavioural and attitudinal brand loyalty (32). Brand equity can be defined as value of a product/service in the mind of customer which is linked with brand's name, brand image, brand symbol etc. (2). He identify variables such as brand equity, name awareness, brand association, perceived quality and brand loyalty and other proprietary brand assets such as patents and channel relationships. According to brand equity model given by Aakar it helps marketers to evaluate brand equity over various markets and products. Majorly variables such as brand awareness, perceived quality, brand association and brand loyalty have direct association with brand equity (2).

Further in the similar study by Kumaravel and Kandasamy (23) conducted study on brand equity of hypermarket store in India and they empirically evaluated five variables affecting brand equitywhich are; Brand Awareness, Brand Loyalty, Perceived Quality, Brand Association and Brand Image, perceived quality was an important indicator affecting brand equity.

Thus, the conceptual understanding shows that there is a positive association between brand equity and brand loyalty and it plays a vital role while forming a structure of brand loyalty.

- **Brand Association and Brand Loyalty**

Another predecessor of brand loyalty is brand associations. To have brand loyal customer marketers must implement those strategies through which they can establish customers' association with brand and would be one of the influencing variable affecting brand loyalty. Variables such as product category, packaging, price and functional attribute are the important sources of brand association which influence

brand loyalty. (2) Al-Abdallaha and Abo-Rummanb (12) recently conducted study on customers association with the brand with an objective to study effects of brand associations on customers' brand loyalty, and they concluded that brand association of all independent variables like product attributes, customer benefits, lifestyle/personality, price, product class, Use/Application, User/Customer, and Celebrity/Spokesperson are directly influenced on customer loyalty. The above conceptual understanding shows that there is a relationship between brand association and brand loyalty.

- **Brand Trust and Brand Loyalty**

Since many years brand trust is measured as one of the most significant antecedents affecting brand loyalty, (4-5-24) and other variables such as customers' satisfaction, perceive quality of products or services and positive or negative attitude have positively effect on brand trust. (13, 27). Brand trust is conceptualised, as a feeling consumers' that brand will meet their expectation (10). The customer relationship can be developed by enhancing the trust of the consumers, which will create positive impact towards brand loyalty. Thus, brand trust is vital for building and maintaining long-term relationships and brand loyalty (31).

- **Customer Satisfaction and Brand Loyalty**

Customer satisfaction since many years considered as one of the vital factor affecting brand loyalty. There is the link between customer satisfaction and brand loyalty which can be formed through customers' attitude and behaviour. It is also considered as one of the most important construct of brand loyalty (20,11). Customer satisfaction is a long term relationship between customers and their preferred brand which shows customers' commitment and loyalty towards the brand (25).

5. Model Development - Antecedents of Brand Loyalty

The conceptual model tested in this paper contains antecedents and structure of brand loyalty demonstrated theoretically based on the researches done in this area. The model examines the antecedents and structure of brand loyalty. The brand loyalty model given

below will be useful model to which helps managers to build and sustain their brands. Firstly, it indicates the brand equity; a contributing variable affecting brand loyalty. Secondly, brand association; customer will be brand loyal only when he/she is associated with brand. Thirdly, brand trust; at the time of repeat purchase customers always calculate perceived risk and quality of the product and their trust on respective brands. Lastly, customer satisfaction; while developing their brand loyalty plan, managers' needs to understand type of association they want their brand to build with their customers and it is possible when consumers meet their expectations. Thus, all the major contributing antecedents are those variables which are affecting to either behavioural or attitudinal loyalty or both together. For all these four antecedents a detailed literature review is performed to find the contributing variables. These contributing variables are also considered for developing the model on antecedents of brand loyalty is mentioned below in the figure 1.

6. Managerial Implications

Based on the conceptual understanding of above model given in this study, decision maker/s have to pay attention on the strategies and measures which primarily help to build brand loyalty, such as brand image, qualitative products, fair price, differentiated and unique product attributes, and meeting customers' expectations, guarantee and warrantee to reduce customers' perceived risk.

The conceptual model on antecedents of brand loyalty proves that brand equity, brand association, brand trust and customer satisfaction will definitely affect consumers to change their attitudinal or behavioural loyalty which helps them to be brand loyal. Thus, this model encourages managers to adopt a strategic and balanced approach which will lead them towards building brand loyalty.

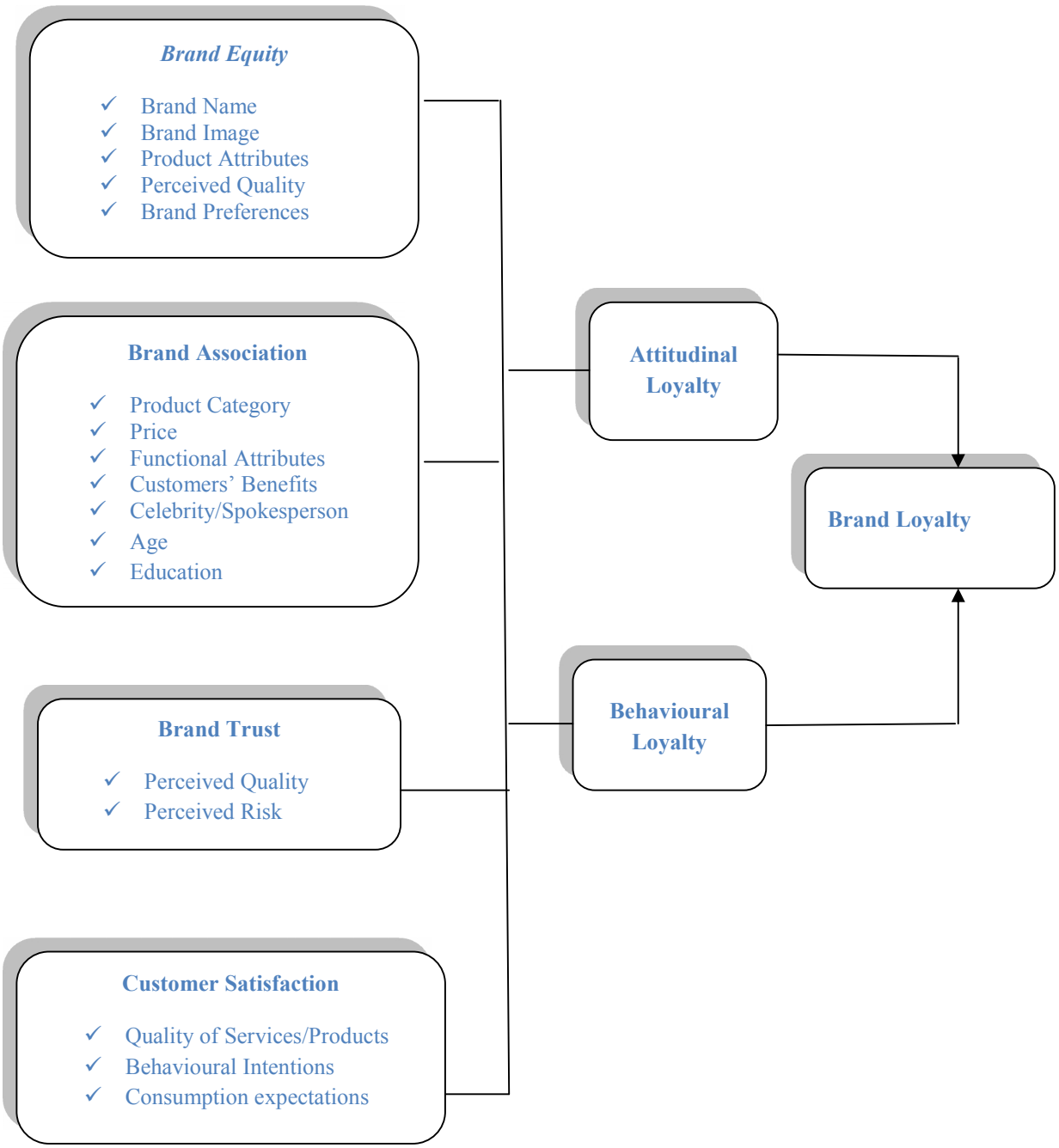
7. Proposed Research Methodology

The author hasextended the same study as part of doctoral program and will conduct empirical research. The author will adopt descriptive research methodwith an objective to study antecedents and structure of brand loyalty which in turn helps managers to adopt different strategies through which they can build brand loyal customers.For this the author will collect primary data by using personal survey method. The data will be collected through a structured questionnaire with a five point likert scale ranging from strongly disagree to strongly agree for all the statements related to brand loyalty and its antecedents. The author will adopt non-probability convenience sampling technique for collecting the samples size of 500 working women which from cities like; Vadodara, Ahmedabad, Surat, Rajkot and Bhavnagar of Gujarat State, India.

8. Conclusion

The authors have investigated antecedents and structure of the brand loyalty. As brand loyalty is found to be combination of several components, all the antecedents considered for the study should be given equal importance. The result of the study shows that Brand satisfaction, Brand equity and Brand trust and Brand association has higher impact with brand loyalty. By giving noteworthy importance towards the antecedents of brand loyalty, several competitive advantages can be gained through brand loyal customer.

Figure: 1 Developed Model on Antecedents of Brand Loyalty



9. Acknowledgment

I owe my profound gratitude to Dr Kunjal Sinha, Associate Professor, C K Shah Vijapurwala Institute of Management, Vadodara for her significant contribution and towards present study. I am extremely grateful to her for providing such a nice support.

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"Movies close to hearts"- A Qualitative study of young audience of Bollywood movies

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ABSTRACT

Movie is an experiential product. Some of the movies are closed to viewers' heart. It gives experience to audience which is interesting, repeated, remembered and valued for long time. Young audience is potential target audience of Bollywood, so it is useful to know their 'Loved Movies' and reasons for loving the movies. Purpose of this qualitative study is to find out various reasons for some Bollywood movies being loved by young audience. Findings of the paper suggest that story/message of Bollywood movies and the psychological traits and behavior of character/s played in the movies, are the main reasons of loving particular Bollywood movies by young audience along with other contents include acting, flow, music, songs, dance, direction, locations etc. The paper will help movie makers to understand the young audience of Bollywood movies. The paper also reflects on Bollywood movies' impact on personal life of young audience.

SUMMARY

A qualitative study to find out various reasons for some Bollywood movies being loved by young audience.

Keywords: Bollywood movies, young audience, qualitative study.

INTRODUCTION

Movies are considered as one of the preferred entertainment avenues in India. Every year more than 1000 movies releases in India by Mumbai film industry, called Bollywood.
Movie is an experiential product. It gives different experiences to different individuals. Audiences get experience while watching and post watching the movies. Some of the movies are loved by audiences which give experiences to audiences which are interesting, repeated, remembered and valued for long time.
Movie watching is considered as hedonic consumption. Elizabeth C. Hirschman (1982) and Morris B. Holbrook (1982)[(1)] have defined "hedonic consumption as those facets of consumer behavior that relate to the multisensory, fantasy and emotive aspects of product usage experience."
Viewers may share more positive information regarding the movies they love and it may influence other viewers' movie watching behavior. Elizabeth Cooper-Martin (1992)[(2)]found that experiential information such as subjects rated previews and friends' comments are more valuable for audience than non experiential information of movie such as magazine advertisements, radio advertisements.
Theatre is also an experiential product. Ben Walmsley (2013)[(3)] have explored the impact that theatre can have on its audiences, both immediately and over time. He found that regular attendees of theatres who seek a deeper engagement often became emotional and for them it is difficult to imagine a life without theatre.
Enjoyment from movies can be different for different viewers due to their psychological traits. Gilmore, Linda A. and Campbell, Marilyn A. (2008)[(4)] found that children enjoyed certain scary experiences like theme park ride, scary movies and books. They found children who are more fearful according to their parents, did not enjoy such scary activities as their less fearful peers.
Young audience is potential target audience of Bollywood, so it is useful to know movies they love and reasons for loving these movies. Purpose of the paper is to find out "why does young audience love some Bollywood movies?" objective of the paper is to delineate main content of Bollywood movies that that are loved by young audience and to find out reasons for some Bollywood movies being loved by young audience. Possible outcome of the paper will be the reasons for some Bollywood movies being loved by audience and main content of such movies and it gives greater understanding of hedonic consumption of young audience of Bollywood movies.

MATERIALS AND METHODS

I have used qualitative approach for the study. As mentioned by John W. Cresswell (2013)[(5)] that the approach is appropriate when detailed understanding is needed and when we want to empower our respondents to share their stories and hear their voices.

I have conducted 6 semi-structured personal interviews of respondents from Rajkot (Gujarat, India) city who have watched Bollywood movies. See Table 1. for Profiles of respondents. I have selected respondents from the age of 19 years -27 years as 'young audience' of Bollywood movies.

I have prepared below questions for these interviews and involved respondents in discussion. I have recorded audio of these interviews.

"Tell me something about yourself/ the journey of your life so far."
"What are your hobbies?" "Do you watch Bollywood movies?"
"How often do you watch Bollywood movies?"

“Can you name few movies which you like and enjoyed most?”
“Can you name few movies which you really love; I mean you feel you get/got emotionally attached with it?”
“What makes you emotional with this particular movie/s?”/ "Why do you love this movie/s?"
"What are the content of the movie which were the reasons of your attachment towards these movie/s?” “Can you tell me something more about the movie/s and your experience while and after watching it?”
“Do these movies impact your life?” "How?”

RESULTS AND DISCUSSION

See Table 2 for the self reported profile of respondents, list of their liked movies and list of loved movies of respondents. Here 'Liked Movies' includes list of movies which viewer likes in his/her life so far and 'Loved Movies' includes list of movies which viewer love and feel emotionally attached with it.

Reasons for loving particular movie:

Very surprising that most the respondents answered that story/message of the movies and the psychological traits and behavior of character/s played in the movies, are the main reasons of loving particular movies along with other contents include acting, flow, music, songs, dance, direction, locations etc.

Respondents love particular movies because they feel the story or the character's psychological traits and behavior are similar o their life situation and their belief system.

A respondent love ‘Mohabbatein’ as it gives him feeling of satisfaction of living the life which he has not lived and want to live it again.

He said, “While I was in college, I never thought of having love with any girl. But after my college days over and I started my business, I realized that I have missed my college days. So while watching ‘Mohabbatein’ I am living life which I have missed and want to live again.”

A respondent love movies like ‘A Wednesday’, ‘Baby’, ‘Nayak: The Real Hero’ and ‘Sarfarosh’ because it gives him satisfaction that someone (the character) is fighting with wrong doers and help our nation which the respondent is not able to do for the country. He also anticipates the future that if someone will fight against wrong doers then our country will live better life.

An unmarried respondent said she loved ‘Mohabbatein’ as it makes her to dream her future life. She also loved ‘Phir Hera Pheri’ as it is having situations which she experience in routine life, dialogues which she speaks in routine life and a location which is very familiar to her.

Respondents who love ‘3 Idiots’ said that the message of the movie and the similar life situation are the main reason of loving the movie. It reminded their college days and the message of the movie match their perspective towards career.

A respondent love ‘Baghban’ because he is observing similar situation in his family and believe every one need to respect their parents and give them good life ahead.

A respondent love ‘Anand’ because he feels that character of lead actor is similar to him. The respondent believes in helping others, love ‘BajrangiBhaijaan’ because the lead actor is helping a small girl to reach her home in the movie.

'Loved Movie' impact on real life:

'Loved Movies' have huge impact on audience life. After watching ‘3 Idiot’, one respondent decided to pursue business career of his choice rather than following traditional path of doing job after completion of his graduation. And he has started his own business. One respondent said he developed ‘never give up’

approach in his life and attempted difficult challenges after watching movies like ‘Tango Charlie’ and ‘Lakshya’.

CONCLUSION

Findings of the research paper suggest that story/message of Bollywood movies and the psychological traits and behavior of character/s played in the movies, are the main reasons of loving particular Bollywood movies by young audience along with other contents include acting, flow, music, songs, dance, direction, locations etc. It also suggests that young audience love Bollywood movies because it helps them to live unfulfilled desires and dream of future life. It also suggests that Bollywood movies have huge impact on young audience’s life. The paper may help movie makers to understand young audience of Bollywood movies.

TABLES

Table 1. Profiles of respondents:

Respondents	Age (years)	Occupation	Gender	Education	Marital Status
1	21	Student	Female	Pursuing Post Graduation	Unmarried
2	19	Student	Male	Pursuing Graduation	Unmarried
3	23	Self employed	Male	Graduate	Unmarried
4	23	Self employed	Male	Graduate	Unmarried
5	22	Student	Male	Pursuing Post Graduation	Unmarried
6	27	Employed	Male	Post Graduate	Married

Table 2. Self reported profile, list of ‘Liked Movies’ and list of ‘Loved Movies' of respondents.

Respondents	Age (years)	Gender	Occupation	Self reported profile	'Liked' Movies'	'Loved' Movies'
1	21	Female	Student	short temper, extrovert, Social	Herapheri (new), PhirHerapheri, AwaraPaagalDeewana, DilwaleDulhania Le Jayenge, KabhiKhushiKabhieGham, Hum ApkeHaiKaun, Hum SaathSaathHai,Golmal(new), Golmal 3, Veer-Zaara, Mohabbatein	Mohabbatein, Phir Hera Pheri

2	19	Male	Student	Want to be different, successful in student career, adventures, want to explore existing concepts, love tracking, travelling.	Tango Charlie, Lakshya, LOC Kargil, Goal, Iqbal, Badmaash Company, Student Of The Year, Lakshya	3 idiots, Tango Charlie, Lakshya, Student Of The Year
3	23	Male	Self employed	independent person, love to do what I want, struggling for career development, want to be self made, helping nature, extrovert,	3 Idiots, Dhoom&Dhoom 2, Mohabbatein, Lagaan, AjabPremkiGhazabKahani	3 Idiots, Dhoom, Dhoom 2, Mohabbatein, AjabPremkiGhazabKahani
4	23	Male	Self employed	introvert, stay away from crowds, scholar in studies in childhood, calm nature, never get angry	Anand, ChupkeChupke (old), Chhotisibaat, Rang De Basanti, BajrangiBhaijaan, Drishyam	Anand, BajrangiBhaijaan
5	22	Male	Student	happy going life, never get confused, naughty, extrovert,	3 Idiots, Baghban, TaareZameen Par, Chak de India, Lagaan, YehJawaaniHaiDeewani, Luck, Lakshya, Guzaarish	Baghban, Luck
6	27	Male	Employed	Scholar in student life, attachment with science, extrovert, Social	A Wednesday, Sarfarosh, Nayak: The Real Hero, Gangaajal, shagird(new), Singham, Tango Charlie, Zameen, Gabbar is Back, Baby, Holiday, Phantom	A Wednesday, Baby, Nayak: The Real Hero, Sarfarosh, Gangaajal

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

(Proceedings available for download at rku.ac.in/icre)

RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

Offer for Sell – An Event Study

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ABSTRACT

Ministry of Finance and Securities and Exchange Board of India – SEBI has made it mandatory for allpubliccompanies tohaveatleast25%and PSUstohaveatleast10%publicshareholding byJune2013. Corporate are employing various methods to dilute their shareholding with majority of them going with Offer for Sale of shares – OFS route because of its easy process and fast processing. In this study we have observed the market reaction to Offer for Sale using Event Study Methodology. Event Study is powerful tool that can help researchers assess the financial changes in corporate policy. Various non-parametric tests are applied to the observed Abnormal Returns and statistical significance has been derived. All test results indicates significant negative returns before OFS, on the day of OFS and after OFS is over. This clearly indicates not even semi strong market efficiency but weak form of market efficiency.

SUMMARY

Event Study using non parametric tests performed for Offer for Sell of shares.

Keywords: Offer for Sale of Shares (OFS), Event Study, Non- Parametric test, Event window

1. INTRODUCTION

1.1 Indian Equity Market

The Indian equity market has its roots older than 150 years. We can categorize it as an emerging market. Bombay Stock Exchange (BSE) and National Stock Exchange (NSE) are the two major stock exchanges

in India where BSE being the oldest one and NSE being the one of the most technologically advanced stock exchanges. Sensex and Nifty are very famous market barometers – indices, of BSE and NSE respectively. Online trade based systems are implemented on both these exchanges and securities are settled in dematerialized form. Securities and Exchange Board of India (SEBI) is the regulatory authority for capital markets in India and regulates stock exchanges, stock brokers, sub brokers, mutual funds, merchant bankers, depositories etc.

1.2 Efficient Market Hypothesis

A theory which tries to explain the functioning of capital markets is popularly known as Efficient Market Hypothesis – EMH. According to EMH share prices observe Random Walk and that can not be predicted if the market is efficient. It advocates that the share process reflects all the information available to general public quickly. So today's prices incorporate all the information available today [1].

Fama (1970) [2] further classified the market in to three levels of market efficiency: (1) Weak-form – It says that excess returns cannot be earned *in the long run* by using investment strategies based on historical share prices or other historical data. Today's price of a security incorporates all the information which is publicly available. (2) Semi-strong-form – it says that the security prices reacts very fast to the newly available public information. This happens in unbiased way i.e. no one is able to trade on particular information and can earn profit. That means that fundamental analysis or technical analysis cannot be useful in earning the excess returns. This means that firm specific information like earning announcement, dividend, rights etc. or industry / economy specific announcements like budget, monetarily policy, interest rates, country ratings, WPI announcement etc cannot trigger the security process to change. It can be possible only if there exist some novel factor which is not anticipated by mass – a surprise factor available in the information. (3) Strong-form efficiency - it says that security prices incorporates all the information available publicly and in private. There is no scope t earn excess return. In such markets persons who have some internal information available can gain till the time the information is not made publicly available. Regulator has employed various measures to curb this and exercised stringent norms for insider trading.

2. EVENT STUDY

Event study helps in gauging the impact of any economic, non economic, regulatory etc. event on the prices of the security. As it considers share process for calculation of excess return, unbiased information is accessed.

2.1 History of the Event Study

Event study has a long history as old as from 1933, when the first event study was published by Dolly

(1933). Then remarkable studies were done by Ball and Brown (1968), and Fama, Fisher, Jensen and Roll (1969). They proposed a formal even study methodology. It is still in vogue with little changes in the methodology. The main changes which have happened are daily security returns in place of monthly returns and more sophistication has been introduced in the statistical methods employed to verify significance of abnormal returns [4].

Campbell, Lo and MacKinlay (1997)[4] have also stated that in the years since those pioneering studies, several modifications of the basic methodology have been suggested, and two main changes in the methodology have taken place. First, the use of daily rather than monthly security return data has become relevant. Second, the methods used to estimate abnormal returns and calibrate their statistical significance have become more sophisticated [4]. Kothari and Warner (2007) major financial journals. This clearly indicated the importance of event study in the field of research.

2.2 Steps of Event Study

Entire process of event study can be portrayed as in the following steps [4]. (1) Clearly articulate the event that needed to be studied. Along with that also identify the period for which we are interested to examine the effect of the event. Such a specified period is known as event window or event period. (2) The event may have effect on various companies. So decide the factors for selecting the companies to be observed for the study. The effect or the impact of the event can be measured by determining abnormal returns (AR). (3) Finalize on method for calculating normal returns and also the additional – abnormal returns. The AR will be nothing but the difference between the actual returns and the expected returns as calculated above during the event window. Normal return or expected return is calculated using he parameters which are in turn calculated during the estimation window. This estimation window is the period prior to the event window and should be long enough to judge the estimation parameters reasonably. (5) Now need to test the ARs and define the null hypothesis. Also devise the manner in which ARs of all the firms will be aggregated. (6) Come up with the finding and the possible reason for the same. (7) Explain for the findings and conclude.

2.3 Event Study Test Statistics

As stated earlier it is very important to test the AR that we have calculated. Various statistical methods and more and more sophistication is being introduced in these tests from time to time.

2.3.1 Parametric Test Statistics

Various parametric tests like an ordinary *t*-statistic, Patell's t-statistics [6], Boehmer, Musumeci and Poulsen test (BMP) (1991) [7] etc are widely used for judging the significance of ARs. Almost all parametric tests require the daily returns data to be normal. But as per Fama (1976), actual daily stock returns are far away from normal in distribution. This is also applicable to daily excess returns. depart from normality and also the same stands true for daily excess returns as per Brown and Warner (1985). Due to this limitation, parametric test are not so reliable for accessing significance of ARs.

2.3.2 Non - Parametric Test Statistics

As opposed to parametric tests, non parametric test do not have any requirement for distribution of daily returns as well as daily abnormal returns. These test are more powerful than their counterpart, parametric tests. The rank test of Corrado and Zivney (1992) is designed to standardize returns and resulted in to a more robust against event induced volatility. Apart from this various Sign tests are useful and proved to be more powerful.

ARs are cumulated over a certain period of time if there exists uncertainty over the happening of the event. This is known as Cumulated ARs – CAR. SIGN – COWAN test is designed for testing CARs by Cowan (1992). It computes the ratio of positive ARs around the event period and the same for the period unaffected by the event. Table 1 shows a summary of non – parametric tests.

Table I. Comparison of non parametric test statics

Name	Key Reference	Antecedent	Strength	Weakness
Corrado Rank Test [C Rank]	Corrado and Zivney (1989)			Loses power for larger CAR [-10, 10]
Corrado-Zivney Rank Test [CZ Rank]	Corrado and Zivney (1992)	C Rank	Cross sectional variance adjustment is applied to the data	
Corrado-Zivney Sign Test [CZ Sign]	Corrado and Zivney (1992)		Accounts for skewness in security returns	

3. DATA AND METHODOLOGY

3.1 Data

The dates of Offer for Sell of shares have been taken from www.nseindia.com and www.bseindia.com the official web sites of India's leading stock exchanges. Total companies opted for Offer for Sell of shares is

more than 70 till date combining both the exchanges. Out of this we have selected 36 companies on the following selection criteria:

- Total OFS value is more than Rs.1000 Million. OFS value is a product of quantity of OFS shares allocated and the settlement price.
- Price data availability for the period of estimation period.
- Substantial part of promoters' holding is being offered for OFS.

The list of sample stocks is given in Appendix 1. These stocks represent diverse sectors of the economy. We have considered daily closing prices while for the market return, CNX-500 is considered.

3.2 Methodology

Event study measures the impact of Offer for sell of shares of the sample companies. We have kept the event window of 31 days, i.e., -15 – 0 – 15 means starting from 15 days earlier to event date, event date and then 15 days after the event day. Estimation window has been selected as 250 days which ends just before the start of event window. This is almost a calendar year.

Actual return of each sample stock is calculated as under during event window as well as during estimation window.

$$r_{it} = \left(p_{it} - p_{it-1}\right) / p_{it-1} \quad \ldots\ldots\ldots\

$$R_{i,t} = \alpha_i + \beta R_{mt} + \varepsilon_{i,t} \quad \text{.....(3)}$$

Where,

$R_{i,t}$ = Return on stock i on day t

α_i = Intercept

β_i = Beta of the stock i

$R_{m,t}$ = Market return of CNX-500 on day t

$\varepsilon_{i,t}$ = Residual error term which is assumed to satisfy the usual assumptions of a linear regression model

We have used these estimated coefficients of the market model, α_i and β_i, and calculated the expected return during the event window. AR is nothing but the difference between actual return and expected return which is given by:

$$A\,R_{i,t} = R_{i,t} - \alpha_i - \beta_i R_{m,t} \quad \text{.....(4)}$$

Here we will start with the assumption that the event has no effect on the prices and so AR = 0 [H_0:$E(AR_{it}) = 0$]. For that we need to aggregate the ARs of individual companies. Such an Aggregation should happen in the time series dimension as well as in cross-sectional dimension. Here we assume that there is no correlation between two different abnormal stock returns. It is also assumed that there no clustering i. e. the event being studied has also the same date for the sample stocks. This way, the cross-sectional average of the abnormal returns is calculated as:

$$\overline{AR_t} = \frac{1}{N}\sum_{i=1}^{N} AR_{it} \quad \text{.....(5)}$$

where N indicates the number of firms included in the study.

If in the event period it is thought that the abnormal performance lasts not one day but a few days starting from the event date until the concerned date, and then Cumulative Abnormal Returns (CAR) should be calculated. CAR is found by addition of the CAR values of the previous days to *it AR* value on t day.

$$CAR_t = CAR_{t-1} + AR_t \quad \text{.....(6)}$$

The average of CAR value is calculated as;

$$\overline{CAR} = \frac{1}{N}\sum_{i=1}^{N} CAR_i \quad \ldots\ldots\ldots\

$$S_{sar} = \sqrt{\frac{1}{N-1}\sum_{i=1}^{N}\left(SAR_{i0} - \overline{SAR}\right)^2} \quad \ldots\ldots\ldots\

$$s_{\overline{K}} = \sqrt{\frac{1}{m}\sum_{t=1}^{m}\frac{N_t}{N}\left(\overline{K} - \frac{1}{2}\right)^2} \quad \ldots\ldots\ldots\

As stated earlier we have selected 36 companies for this study. Table 2 shows the OFS date, intercept α, slope β, Cumulative abnormal return and Standard deviation of these companies.The value of α and β are calculated using estimation window data. We have used the Market Model to estimate the returns.

Table 2. α, β, CAR and STD Deviation of Sample Companies

Company Name	OFS Date	α	β	CAR	STD Deviation
L&T Finance Holdings Limited	06/11/14	-0.001	1.2371	-0.49%	0.02072
L&T Finance Holdings Limited	03/14/14	0.000	1.3091	-16.69%	0.01904
Gillette India Limited	11/13/13	0.000	0.3552	-8.22%	0.01577
Oberoi Realty Limited	09/26/13	-0.001	1.0698	-5.31%	0.02265
National Fertilizers Limited	07/31/13	-0.004	1.2571	-29.43%	0.02053
Hindustan Copper Limited	07/03/13	-0.004	0.8624	-27.36%	0.02899
MMTC Limited	06/13/13	-0.005	0.7722	-86.18%	0.02818
Essar Ports Limited	05/30/13	0.000	0.5866	-13.19%	0.01793
Jet Airways (India) Limited	05/30/13	0.002	1.4969	-16.15%	0.03294
Sun TV Network Limited	05/29/13	0.001	1.2827	-16.68%	0.02343
Jaypee Infratech Limited	05/29/13	-0.001	1.3158	-23.56%	0.02259
AstraZeneca Pharma India Limited	05/28/13	-0.003	0.8589	7.75%	0.02803
Puravankara Projects Limited	05/24/13	0.001	1.1274	-15.44%	0.02907
JSW Energy Limited	05/22/13	0.001	1.9253	-21.30%	0.01993
Oracle Fin. Ser Software Limited	05/22/13	0.000	0.6740	4.70%	0.01486
NALCO	03/15/13	-0.001	1.0241	-19.65%	0.01675
RCF	03/08/13	-0.002	1.1877	-14.76%	0.01764
Mahindra Holiday	03/07/13	0.000	0.7994	-5.12%	0.01642
Adani Enterprise	04/03/13	-0.002	1.9783	3.19%	0.02487
NTPC	02/07/13	-0.001	0.7113	4.36%	0.01173
OIL Ind	02/01/13	0.000	0.3907	8.74%	0.01300
Adani Enterprise Ltd	12/21/12	-0.002	1.8379	15.55%	0.02862
Reliance Power Ltd	12/19/12	-0.001	1.9864	-7.33%	0.02022
Honeywell Auto Ltd	12/14/12	0.001	0.5091	1.12%	0.02815
NMDC	12/12/12	-0.001	1.1947	-11.15%	0.01845
Hind Copper	11/23/12	0.001	0.9786	-57.96%	0.03047
DB Corp Ltd	11/09/12	0.000	0.2880	-0.75%	0.01615
Adani Power Ltd	10/08/12	-0.003	1.4899	16.92%	0.02426
Sical Logistics	09/26/12	0.000	0.0956	-2.67%	0.01411
Muthoot Capital Service	08/01/12	0.000	0.5575	-3.73%	0.02161
Uttam Sugar Mills Ltd	07/23/12	-0.001	0.8752	22.25%	0.02841
J P Power Company Ltd	06/29/12	0.000	1.3287	-13.57%	0.01854
D B Corp Ltd	05/10/12	-0.001	0.3401	0.03%	0.01521
Wipro Ltd	03/14/12	0.000	0.9317	3.35%	0.01399
Oil and Natural Gas Corp Ltd	03/01/12	0.000	0.6105	-4.91%	0.01624
Blue Dart Ltd	11/23/12	0.000	0.3714	13.58%	0.01657

Company wise cumulative returns CAR_i are depicted in Table 3. It shows summation of all the abnormal returns in the event window. Further it shows company wise abnormal returns for Pre Event window i.e. from t- 15 day to t-1 day, as on event day i.e. t = 0 day and for post event window i.e. from day t+1 to t + 15.

Table 3. CAR_i on Pre Event, Event Day and Post Event

Company Name	Value			Sign				
	Pre Event	Event Day	Post Event	Pre Event + Ve	Pre Event -Ve	Event Day + / - Ve	Post Event + Ve	Post Event -Ve
L&T Finance Holding Ltd	5.40%	-4.09%	-1.80%	9	6	-Ve	9	6
L&T Finance Holding Ltd	1.19%	-6.35%	-11.54%	8	7	-Ve	6	9
Gillette India Limited	-4.49%	5.03%	-8.76%	6	9	+ Ve	6	9
Oberoi Realty Limited	-4.49%	3.46%	-4.28%	8	7	+ Ve	4	11
National Fertilizers Ltd	-14.74%	2.94%	-17.63%	5	10	+ Ve	5	10
Hindustan Copper Ltd	-12.58%	-1.09%	-13.69%	8	7	-Ve	3	12
MMTC Limited	-3.56%	-8.59%	-74.03%	6	9	-Ve	0	15
Essar Ports Limited	-10.71%	1.23%	-3.71%	5	10	+ Ve	8	7
Jet Airways (India) Ltd	-13.48%	-2.02%	-0.65%	5	10	-Ve	8	7
Sun TV Network Limited	-8.78%	4.37%	-12.27%	6	9	+ Ve	7	8
Jaypee Infratech Limited	4.58%	-9.21%	-18.92%	6	9	-Ve	6	9
AstraZeneca Pharma I Ltd	2.66%	-17.00%	22.09%	8	7	-Ve	5	10
Puravankara Project Ltd	-16.03%	-2.69%	3.28%	3	12	-Ve	10	5
JSW Energy Limited	-7.47%	-3.04%	-10.79%	6	9	-Ve	6	9
Oracle Fin. Ser Soft Ltd	-9.49%	1.20%	12.99%	6	9	+ Ve	10	5
NALCO	-1.39%	-7.76%	-10.51%	8	7	-Ve	5	10
RCF	-9.61%	2.16%	-7.31%	5	10	+ Ve	4	11
Mahindra Holiday	-4.80%	-0.58%	0.26%	5	10	-Ve	7	8
Adani Enterprise	2.68%	0.82%	-0.30%	10	5	+ Ve	7	8
NTPC	-0.42%	-2.02%	6.80%	7	8	-Ve	9	6
OIL Ind	9.13%	-2.54%	2.16%	10	5	-Ve	8	7
Adani Enterprise Ltd	15.60%	3.22%	-3.27%	10	5	+ Ve	7	8
Reliance Power Ltd	-2.22%	-1.48%	-3.62%	7	8	-Ve	4	11
Honeywell Auto Ltd	-8.19%	-0.68%	9.99%	4	11	-Ve	7	8
NMDC	-11.81%	-3.03%	3.69%	5	10	-Ve	9	6
Hind Copper	5.39%	-20.11%	-43.24%	6	9	-Ve	1	14
DB Corp Ltd	-1.70%	-3.22%	4.16%	7	8	-Ve	10	5
Adani Power Ltd	18.95%	-0.30%	-1.72%	9	6	-Ve	8	7
Sical Logistics	-3.23%	0.11%	0.45%	6	9	+ Ve	7	8
Muthoot Capital Service	-1.55%	1.66%	-3.85%	6	9	+ Ve	6	9
Uttam Sugar Mills Ltd	25.22%	0.46%	-3.43%	9	6	+ Ve	5	10
J P Power Company Ltd	-10.12%	-2.35%	-1.11%	3	12	-Ve	7	8
D B Corp Ltd	-1.00%	-0.46%	1.49%	7	8	-Ve	8	7
Wipro Ltd	-0.41%	-1.70%	5.47%	9	6	-Ve	10	5
Oil & Natural Gas Corp L	1.36%	-1.25%	-5.01%	6	9	-Ve	7	8
Blue Dart Ltd	17.68%	-3.45%	-0.65%	7	8	-Ve	5	10

4.2 Statistical Significance of Average Abnormal Returns

In the study, we have considered the event window of 31 days consisting of $t = -15$ to +15 relative to the event day t_0. Event date is the date of Offer for sell of shares.

Hypothesis:

The null hypothesis (H_{0a}) is set forth as the market being semi-efficient; as such the Offer for Sell of Shares does not impact the return. In other words, Average Abnormal Returns (AAR) for any day falling within the event window is not different from 'zero' statistically.

Similarly the alternative hypothesis (H_{1a}) is set forth as, the Offer for Sell of Shares does impact the returns. In other words, Average Abnormal Returns (AAR) for any day falling within the event window is different from 'zero' and significant statistically.

4.2.1 Non Parametric Test

4.2.1.1

C Rank Test: As per the table 4 we can see that t- values for most of the event days are negative but no any value is statistically significant at 5% or 1%.

4.2.1.2

CZ Rank Test: Table shows that for t-8, t-3 and t+6 the t- values obtained are statistically significant at 5% level and for t+1 it is even significant at 1% level. We can clearly see that from the results that CZ Rank test has taken in to account for cross sectional variance.

4.2.1.3

CZ Sign test:

Table 4 shows that for t-2, t= 0 and for t+3, the results of CZ sign test are statistically significant even at 1% level. As the sign test is not taking absolute values into account we have found only three instances of significant abnormal returns.

To sum up most of the non-parametric tests also indicates that there is significant negative abnormal return during Offer for Sell of shares event. These tests rejects our hypothesis H_{0a} and according to H_{1a} we can say that Offer for sell of Shares has a significant effect on Average Abnormal Returns of shares and this is negative in nature.

Table 4: Stock Market Reaction to Offer for Sell

Day	AAR (%)	CAAR (%)	Non Parametric Test ARt		
			C Rank	CZ Rank	CZ Sign
-15	-0.32%	-0.32%	-0.0105	-0.06280	0.00000
-14	0.12%	-0.20%	0.00271	0.00000	0.00000
-13	-0.22%	-0.42%	-0.1631	-0.86303	0.00000

-12	-0.13%	-0.55%	-0.0460	-0.25931	0.00000
-11	-0.48%	-1.04%	-0.3288	-1.73010	0.00000
-10	0.55%	-0.49%	-0.0209	-0.12560	0.00000
-9	0.57%	0.08%	0.23357	1.20945	0.00000
-8	-0.80%	-0.72%	-0.4458	-2.3358[b]	0.00000
-7	0.20%	-0.52%	0.21885	1.15273	0.00000
-6	0.10%	-0.41%	-0.0708	-0.38492	0.00000
-5	0.03%	-0.38%	0.14332	0.74552	0.00000
-4	-0.25%	-0.63%	0.04764	0.24716	0.00000
-3	-0.91%	-1.54%	-0.4729	-2.4878[b]	0.00000
-2	-0.01%	-1.56%	0.07398	0.38694	20.9720[c]
-1	0.10%	-1.46%	-0.3001	-1.58019	0.00000
0	-2.18%	-3.63%	-0.5411	-1.89825	-20.972[c]
1	-0.88%	-4.52%	-0.6186	-3.2515[c]	0.00000
2	-1.07%	-5.59%	-0.3525	-1.85571	0.00000
3	-0.04%	-5.63%	0.11775	0.62397	20.9720[c]
4	-0.85%	-6.48%	-0.3339	-1.75036	0.00000
5	-0.07%	-6.55%	0.14099	0.73540	0.00000
6	-0.83%	-7.39%	-0.4462	-2.3541[b]	0.00000
7	-0.27%	-7.66%	0.07282	0.38289	0.00000
8	-0.26%	-7.92%	-0.0124	-0.06888	0.00000
9	0.18%	-7.74%	0.03060	0.16004	0.00000
10	0.47%	-7.28%	0.28044	1.46876	0.00000
11	-0.64%	-7.92%	-0.1859	-0.97850	0.00000
12	-0.16%	-8.08%	-0.0290	-0.16207	0.00000
13	-0.04%	-8.12%	0.10458	0.54496	0.00000
14	-0.59%	-8.71%	-0.1452	-0.76173	0.00000
15	-0.18%	-8.89%	-0.0465	-0.24108	0.00000

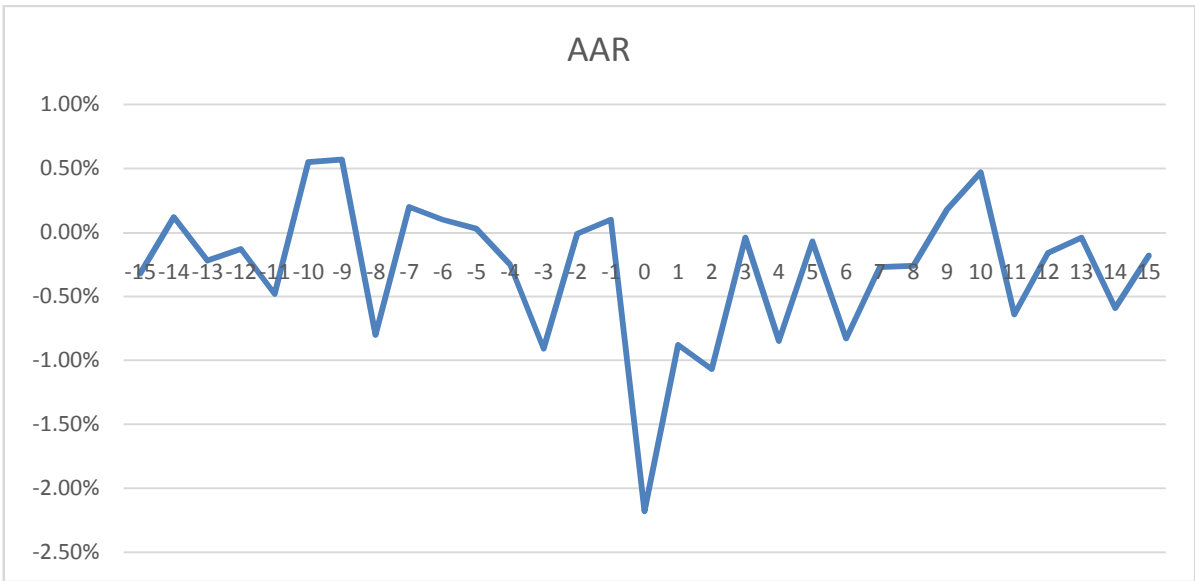


Figure 1. Average Abnormal Returns in Event Window

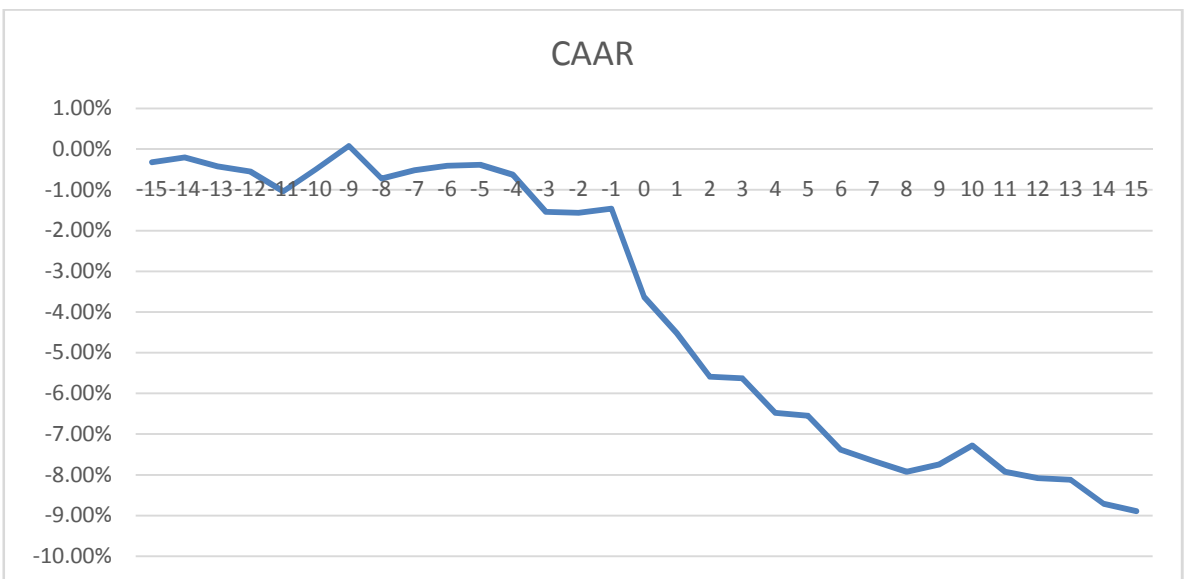


Figure 2. Cumulative Abnormal Returns in Event Window

4.3 Statistical Significance of Cumulative Average Abnormal Returns

In the study, we have considered the event window of 31 days consisting of t = –15 to +15 relative to the event day t_0. Event date is the date of Offer for sell of shares. To study the effect of the event in cluster of more than one day, we have selected various time intervals from event window like t-15 to t+15, t-1 to

t+1 etc and corresponding cumulative abnormal return are CAR(-15,15) and CAR(-1,1) correspondingly etc. We have selected CAR(-15,6) and CAR(-5,-1) as pre event CARs to access any information leakage before the event. Similarly, CAR(1,5) and CAR(6,15) post event window has been selected to access any delay price adjustment after OFS day.

Hypothesis:

The null hypothesis (H_{0b}) is set forth as the market being semi-efficient; as such the Offer for Sell of Shares does not impact the return. In other words, Cumulative Average Abnormal Returns (CAR) for any time period falling within the event window is not different from 'zero' statistically.

Similarly the alternative hypothesis (H_{1b}) is set forth as, the Offer for Sell of Shares does impact the returns. In other words, Cumulative Average Abnormal Returns (CAR) for any time period falling within the event window is different from 'zero' and significant statistically.

Table 5. CAAR for Offer for Sell of Shares

CAR Interval	CAR	Non Parametric Test
		Z Cowan Sign Test
CAR(-1,1)	-2.96%	-2.5552610[b]
CAR(-3,3)	-5.59%	-2.5552610[b]
CAR(-5,5)	-6.14%	-1.8866946
CAR(-10,10)	-6.24%	-0.8838449
CAR(-15,15)	-8.89%	-1.5524113
Pre Event Window		
CAR(-15,6)	-0.41%	-1.2181281
CAR(-5,1)	-1.04%	-0.2152784
Post Event Window		
CAR(1,5)	-2.92%	-1.21812809
CAR(6,15)	-2.34%	-0.88384486

As per table 5 we can see that all the CARs are statistically significant for all the intervals. This rejects the H_{0b} and as per H1b, Cumulative abnormal returns are not zero and they are statistically significant for different time intervals in event window. We can see that Post event CARs are more significant than the pre event CARs. This indicates a clear view of inefficient market condition. Market takes a long time to adjust for the OFS event.

The probable reason for the same may be as the OFS route is altogether a new route for selling shares by companies; it has not been still clearly understood by the market. People are taking it as a negative sign..

CONCLUSION

In this study we are interested to study the market reaction to Offer for Sale using Event Study Methodology. Event Study is powerful tools that can help researchers assess the financial changes in corporate policy. It helps in gauging the unexpected price movement due to any unanticipated event and there by jugging importance of the event.

We presented the data collected for sample companies chosen for the study. Average Abnormal returns (AAR) and Cumulative Abnormal Returns (CAR) are observed in the event window of -15 to 15 days before and after OFS day. Various Non-parametric tests are applied to the observed Abnormal Returns and statistical significance has been derived. Almost all test results indicates significant negative returns before OFS, on the day of OFS as well as after OFS is over. This clearly indicates not even semi strong market efficiency but weak form of market efficiency.

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Appendix 1 List of Sample Companies

Company Name	OFS Date	No of Shares offered	Floor price	Allotted quantity after settlement	Allotment price	Value in CR
L&T Finance Holdings Limited	06/11/14	27,585,744	74.00	27,585,744	76.00	209.65
L&T Finance Holdings Limited	03/14/14	83,258,633	70.00	83,258,633	71.50	595.30
Gillette India Limited	11/13/13	2,857,744	1,650.00	2,857,744	1,801.00	514.68
Oberoi Realty Limited	09/26/13	11,441,069	158.00	11,441,069	167.50	191.64
National Fertilizers Limited	07/31/13	37,479,940	27.00	37,479,940	27.00	101.20
Hindustan Copper Limited	07/03/13	37,119,152	70.00	37,119,152	70.00	259.83
MMTC Limited	06/13/13	93,312,000	60.00	93,312,000	60.00	559.87
Essar Ports Limited	05/30/13	22,671,161	77.00	20,404,045	77.00	157.11
Jet Airways (India) Limited	05/30/13	4,317,697	510.00	2,986,022	515.17	153.83
Sun TV Network Limited	05/29/13	7,881,700	403.00	7,881,700	420.10	331.11
Jaypee Infratech Limited	05/29/13	120,000,000	35.00	120,000,000	35.00	420.00
AstraZeneca Pharma India Limited	05/28/13	3,749,950	490.00	3,749,950	620.00	232.50
Puravankara Projects Limited	05/24/13	14,135,576	81.00	14,135,576	81.00	114.50
JSW Energy Limited	05/22/13	28,230,000	61.50	28,230,000	61.60	173.90
Oracle Fin. Ser Software Limited	05/22/13	4,430,501	2,275.00	4,430,501	2,420.00	1072.18
NALCO	03/15/13	128,861,925	40.00	128,861,925	40.00	515.45
RCF	03/08/13	68,961,012	45.00	68,961,012	45.00	310.32
Mahindra Holiday	03/07/13	3,400,000	270.00	3,400,000	273.10	92.85
Adani Enterprise	03/04/13	8,500,000	221.00	8,500,000	222.66	189.26
NTPC	02/07/13	783,262,880	145.00	783,262,880	145.55	11400.39
OIL Ind	02/01/13	60,113,157	510.00	60,113,157	520.00	3125.88
Adani Enterprise Ltd	12/21/12	23,000,000	282.00	23,000,000	273.45	628.94
Reliance Power Ltd	12/19/12	152,051,807	93.00	152,051,807	95.53	1452.55
Honeywell Auto Ltd	12/14/12	551,333	2,150.00	551,333	2,341.72	129.11
NMDC	12/12/12	396,471,600	147.00	396,471,600	148.50	5887.60
Hind Copper	11/23/12	88,723,300	155.00	41,765,184	156.56	653.88
DB Corp Ltd	11/09/12	11,927,000	205.00	11,927,000	205.00	244.50
Adani Power Ltd	10/08/12	35,577,597	54.20	35,577,597	54.35	193.36

Appendix 1 List of Sample Companies

Company Name	OFS Date	No of Shares offered	Floor price	Allotted quantity after settlement	Allotment price	Value in CR
Sical Logistics	09/26/12	167,518	68.00	167,518	68.00	1.14
Muthoot Capital Service	08/01/12	309,165	90.00	309,165	90.00	2.78
Uttam Sugar Mills Ltd	07/23/12	790,401	24.00	790,401	24.00	1.90
J P Power Company Ltd	06/29/12	29,055,382	33.50	29,055,382	33.53	97.42
D B Corp Ltd	05/10/12	9,000,000	196.00	9,000,000	196.00	176.40
Wipro Ltd	03/14/12	35,000,000	418.00	24,751,254	425.40	1052.92
ONGC	03/01/12	427,774,504	290.00	420,416,170	366.60	15412.46
Blue Dart Ltd	11/23/12	1,431,937	1,720.00	1,431,937	1,823.11	261.06

Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

(Proceedings available for download at rku.ac.in/icre)

RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

A comparative study on change of scale of online population, mobile internet users and social media users in India.

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ABSTRACT

In last decade technology has played a vital role in growth story on India. PC literacy, number of internet users both in rural and urban India, mobile subscriptions, usage of internet through smartphones and social media are key drivers in evolution of new age mass media. In the current paper an attempt has been made to identify the trends of growth of above mentioned variables over the years and comparative study have been carried out to have clear picture on its penetration level and growth rates.

SUMMARY

Comparative study of growth of online population, mobile users, mobile internet users and social media users

Keywords: Online population, internet users, PC literates, social media users, mobile internet users, social media population

INTRODUCTION

Over a past decade, we observed a wave of rapid change in Indian market. Digitization and easy internet access are the key drivers behind this fast paced advancement of Indian growth story. Further the use of mobile has intensified the access of internet both in rural and urban India. The pace of change will continues as digital channels are constantly growing both in size and numbers. According to latest report

by IMRB International and the Internet & Mobile Association of India (IAMAI) which states that online population has rose to 243 billion in India as on March 2014(*1*). The number of people with access to internet continues to rise, but it is still 20 % of the country (*1*). Moreover in last five years social media has evolved from handful community to major media to connect with people across the globe and which helps people to create their identity on web. As per the latest report of IAMAI total social media users in India have grown to 102 million by March 2014(*1, 2*). As per the report published by Mckinsey global institute, internet users in India is third largest in the world in 2012 and its growth rate in coming years will be very critical for economic and overall development of the country(*4*). Growth of Internet, mobile and social media has change the entire landscape and is part of a routine life of majority of people. In current study an attempt have been made to explore the growth of Indian population, online population, mobile subscriptions and social media users.

MATERIALS AND METHODS

2.1. RESEARCH OBJECTIVES

- To identify the trend and pattern of growth of PC literates, online population, mobile internet users and social media users of India over the years.
- To perform comparative analysis of online population and social media users with total Indian population.
- To calculate the penetration level of internet, mobile subscriptions and social media over the years.
- To carry out comparative analysis of internet growth in urban vs. rural India.

2.2. SAMPLE DESIGN

Sampling Technique: The study has been carried out on the basis of secondary data collected from varied government websites, association websites and published reports. Thus, the technique of sampling used for the study is “Convenience Sampling”.

Sample Size: Data like PC literates, total claimed internet users in India, total mobile subscriptions, total mobile internet users and social media users are taken as main variables for the study and to carry out comparative analysis. Further categories like metros, non-metros, tier II, tier III, towns and others are not covered in this study.

2.3. DATA COLLECTION

Various government websites like TRAI, association websites, internet world population, association of telecom industry, published reports of Internet & Mobile Association of India (IAMAI), I-cube, Nielsen research and others have been used to collect data of the variables selected for the study.

2.4. TIME PERIOD OF THE STUDY

The study has been conducted for the period of 2007 to 2014.

2.5. TOOLS USED FOR ANALYSIS

Here we have used different types of charts and graphs like histogram and line chart for graphical representation. For the calculation of growth rate and penetration level simple methods of percentage and indexing have been used to identify the values.

2.6. LIMITATIONS OF THE STUDY:

The study is based on secondary data collection from different reports published by government agencies, associations and published reports on websites. The limitation of the secondary data if any will also influence the study.

The size of the sample and period of the study are among other limitations. Maybe a large sample would be desirable and period taken for the research is 7 years (2007-14).

RESULTS AND DISCUSSION

This section of study embodies the calculation and analysis of selected variables taken for the study

As one can see from table 1 and figure 1, that over a period of seven years i.e. from March 2007 to March 2014 number of pc literates in India were increased by almost 6.6 times. But total number of claimed internet user are 60.91 % of total PC literates. Moreover total number of active internet users are just 79% of total claimed internet users.On further analysing claimed internet users and active internet users (depicted in figure 2 & 3) according to rural and urban areas. We found that online population in urban areas increased by 10 times over a period of last seven years whereas rural online population has increased by 15 times over same period of time. 86 % of claimed internet users are active in urban areas as compared to 67 % of active internet users in rural area. So we can say that in rural area number of claimed internet users are increasing but many of them remain inactive.

Again, if we see table 4 and on analysing the growth of mobile subscribers in India and number of users accessing internet from mobile phones, we found that over the period of five years i.e. March 2009 to March 2014 number of mobile subscriptions has increased by 2.30 times. Whereas percentage of people using internet from mobile phones have increased from merely 2.55 % in March 2009 to 17.14 % in March 2014, which further likely to increase exponentially mainly because of huge demand of smartphones and easy availability of internet at low cost.One of the major usage of internet by users either through PC or mobile phones is to access social media. If we analyse the growth of social media users (figure 5), we can figure out that number of social media users has increased by 17 times from period of 7 years i.e. March 2007 to June 2014.

As we can see from table 6 and figure 6, that over the period of last 7 years India's population has increased by 1.13 times while pc literates were increased by 6.45 times. Further if we look about claimed internet users than overall it has been increased by 5.93 times while mobile internet users were increased by 73.8 times over the period of study. Social media population has also increased by 16. 2 times which is almost 3 time more than growth of online population and 14 time more than India's population growth over the period of study i.e. from March 2007 to March 2014.

As we can see in table 7 and on further analysing the growth rates of India's population, online population in India, mobile internet users and social media users from March 2007 to March 2014, we can see that average growth rate of mobile internet user is 89 % which is very high followed by social media users which is 50 %. Average growth rate of online population is 29 % whereas growth rate of pc literates is 33 % over the period of study. India's population is growing at the rate of 1.8 % over the same period.

If we analyse the penetration level of pc literates, online population, mobile subscribers, mobile internet users and social media population in India (table 8 & figure 8), we can see that penetration level of pc literates has been increased from 5.47 to 31 % from March 2007 to March 2014. As compared to pc literates, penetration level of online population is very low which still 19 % as on March 2014. Penetration level of mobile subscriptions is 70 % which is fantastic, but mobile internet users are yet to achieve that penetration level which is just 12 % as on March 2014. Penetration of social media is 8 % which is good in terms of growth; if we compare with the figure of penetration level with March 2007 which is just 0.5 %.

HYPOTHESIS TESTING 1

H0: There is no significant difference between growth rates of India's population, internet population and social media population

H1: There is significant difference between growth rates of India's population, internet population and social media population

As we can see from table 9, F cal = 22.97 > F crit = 3.55. Hence we will reject our H0 and our test is significant. Therefore we can say that there is significant difference between growth rates of India's population, internet population and social media population.

HYPOTHESIS TESTING 2

H0: There is no significant difference between growth rate of online population and social media population

H1: There is a significant difference between growth rate of online population and social media population

As we can see from table 10, t cal = -2.42 < t crit. =-2.36. Hence we will reject our H0 and our test is significant. Therefore we can say that there is a significant difference between growth rate of online population and social media population

KEY FINDINGS

- India's online population has reached to 243 million as on March 2014. Among total online internet users 65 % are from urban areas and 35 % from rural areas.
- As on March 2014, total number of mobile users has reached to 904.5 million, among them 17% are accessing internet from their mobile phones.
- Average growth rate of online population from March 2007 to March 2014 is 29%, while that of mobile users and social media users over the same period is 89% and 50 % respectively.
- As on March 2014, penetration level of internet users has reached to 19% while that mobile users and mobile internet users has reached to 70% and 12% respectively. Penetration of social media users in India has reached to 8%

CONCLUSION

On the basis of above study we can say that penetration rate of internet, mobile internet users and social media users in India are increasing at exponential rate. Currently India holds third position worldwide in terms of online population. With current growth rate of online population and rapid transition in place, we are going to see even more intense change in next five years. Constant innovation in technology, improvement in wired and wireless infrastructure, growing demand of smartphones, easy access of internet at cheaper rates are few of the drivers which fuelled the growth of online and social media population. We are going to observe significant impact of this fast growth changes of online and social media on several industries and our daily life in times to come.

FIGURES

FIGURE 1-ONLINE POPULATION OF INDIA (IN MILLIONS)

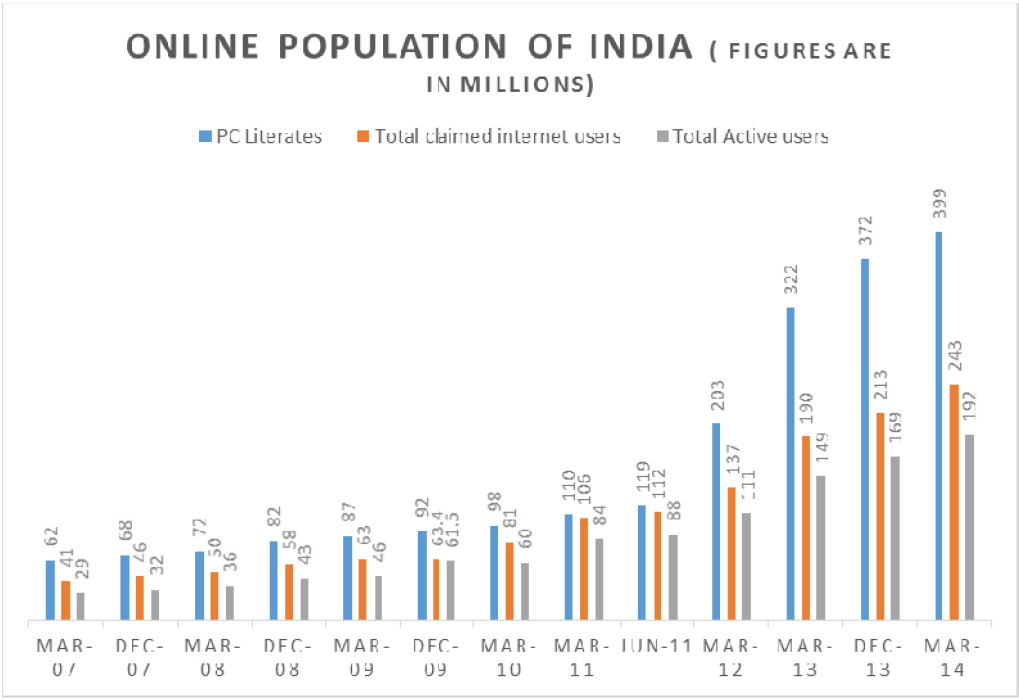


FIGURE 2 – URBAN ONLINE POPULATION OF INDIA (IN MILLIONS)

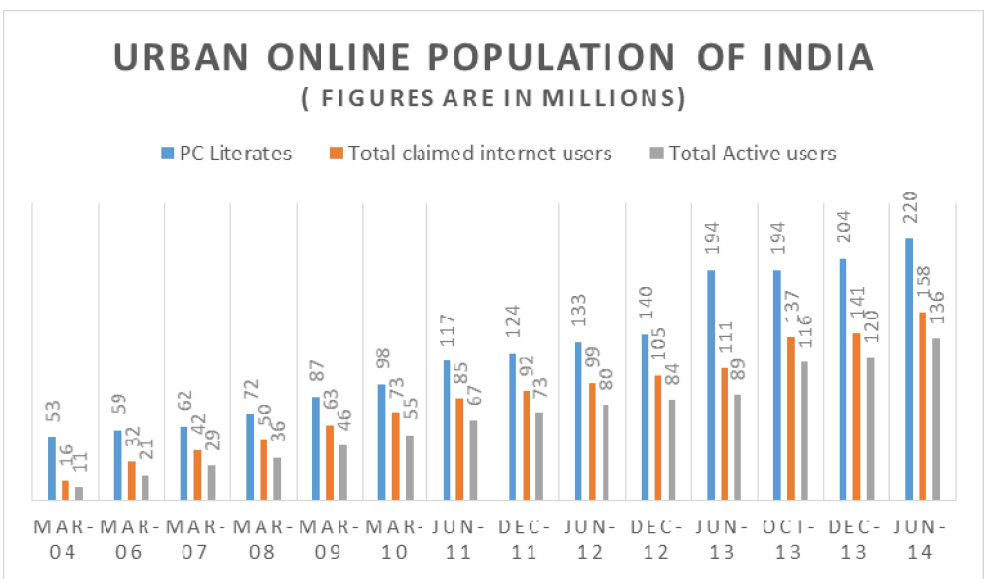


FIGURE 3 - RURAL ONLINE POPULATION OF INDIA (IN MILLIONS)

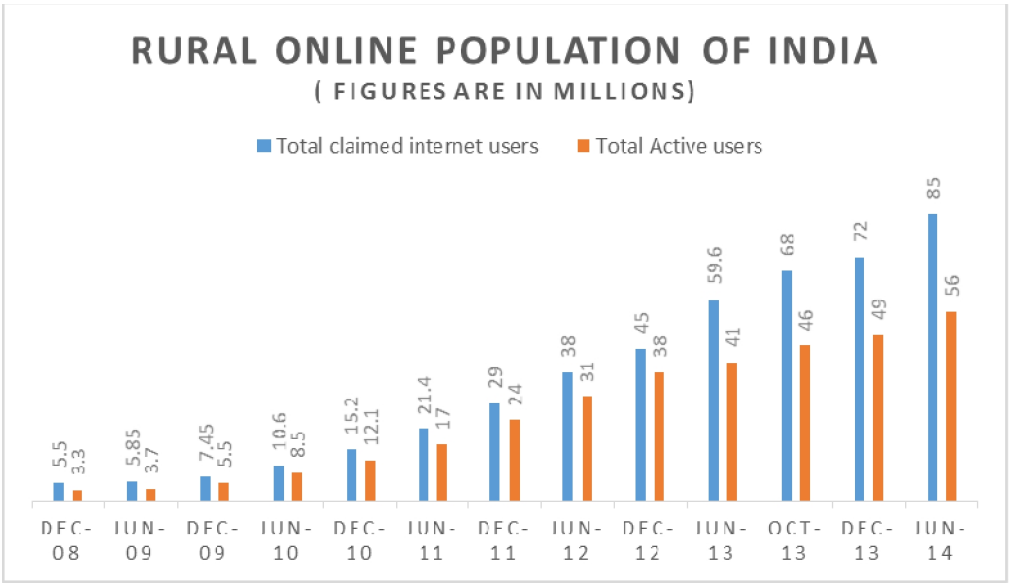


FIGURE 4 – MOBILE USERS VS. MOBILE INTERNET USERS

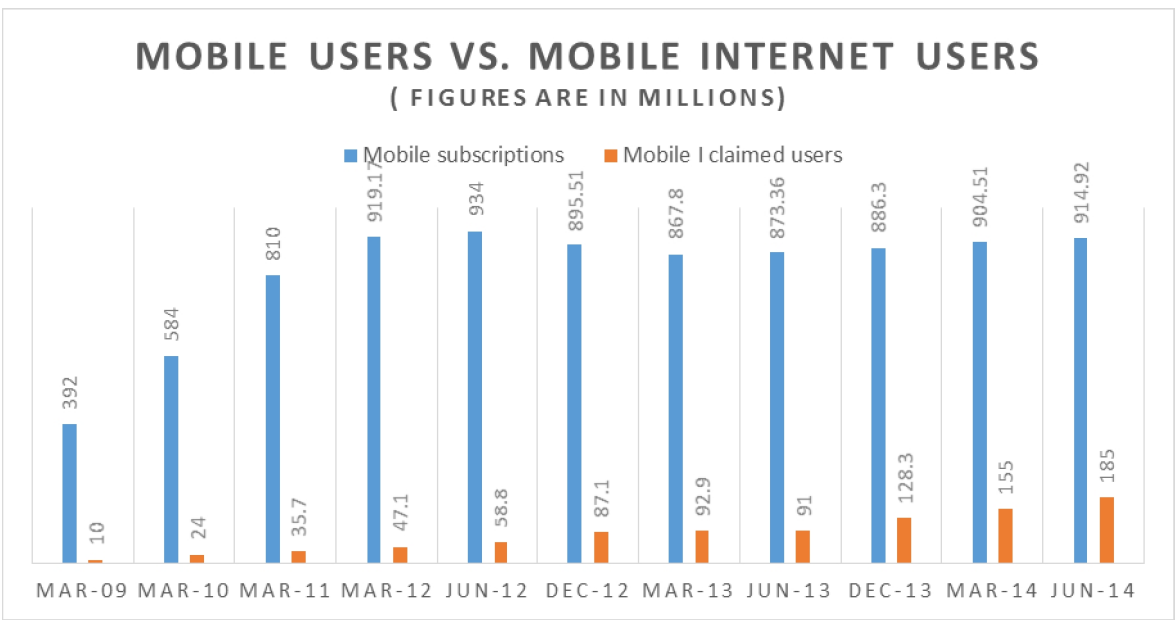


FIGURE 5 – SOCIAL MEDIA USERS IN INDIA

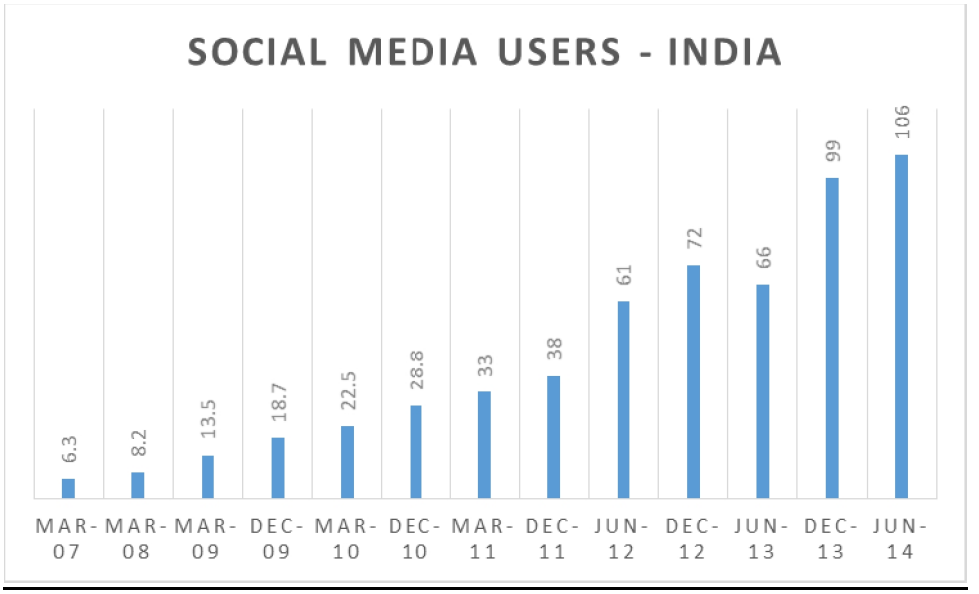


FIGURE 6 – COMPARATIVE STUDY OF TOTAL POPULATION VS. ONLINE POPULATION VS. SOCIAL MEDIA USERS

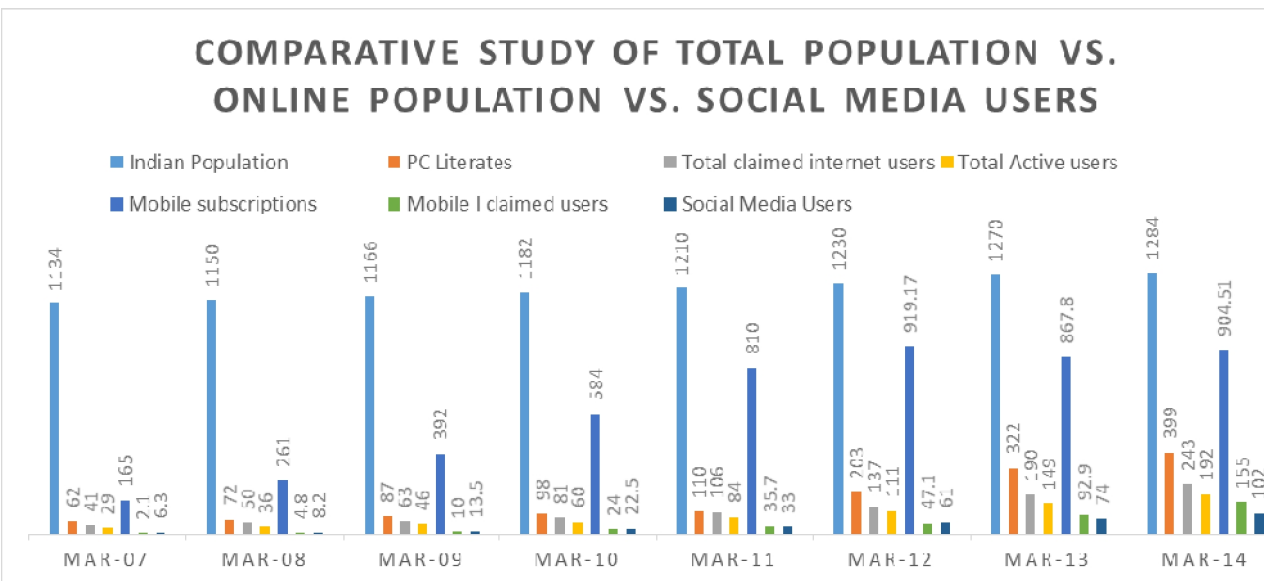


Figure 7
COMPARATIVE ANALYSIS OF GROWTH RATE OF INDIAN POPULATION VIS. A VIS. ONLINE POPULATION, PC LITERATES, MOBILE INTERNET AND SOCIAL MEDIA USERS

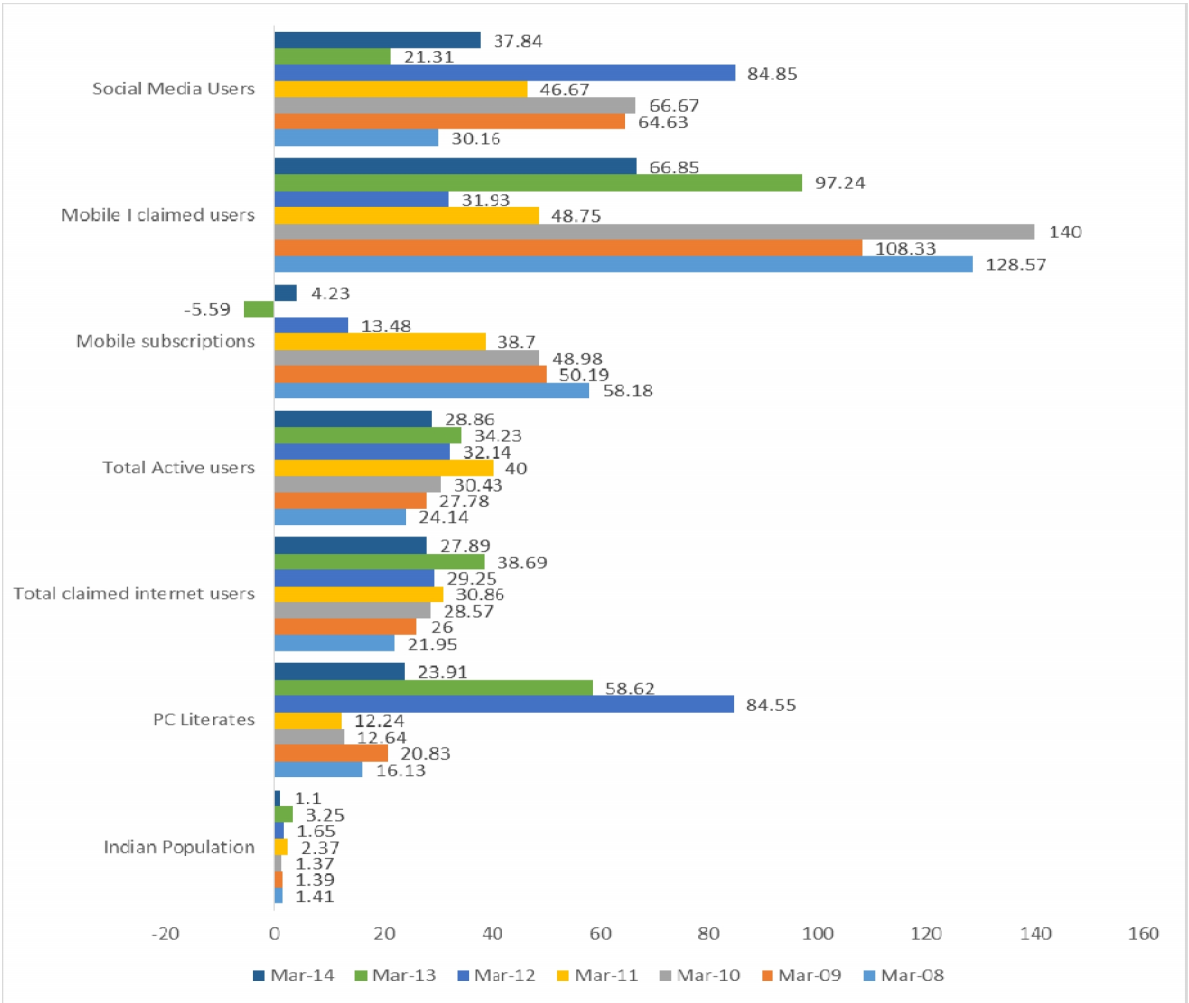
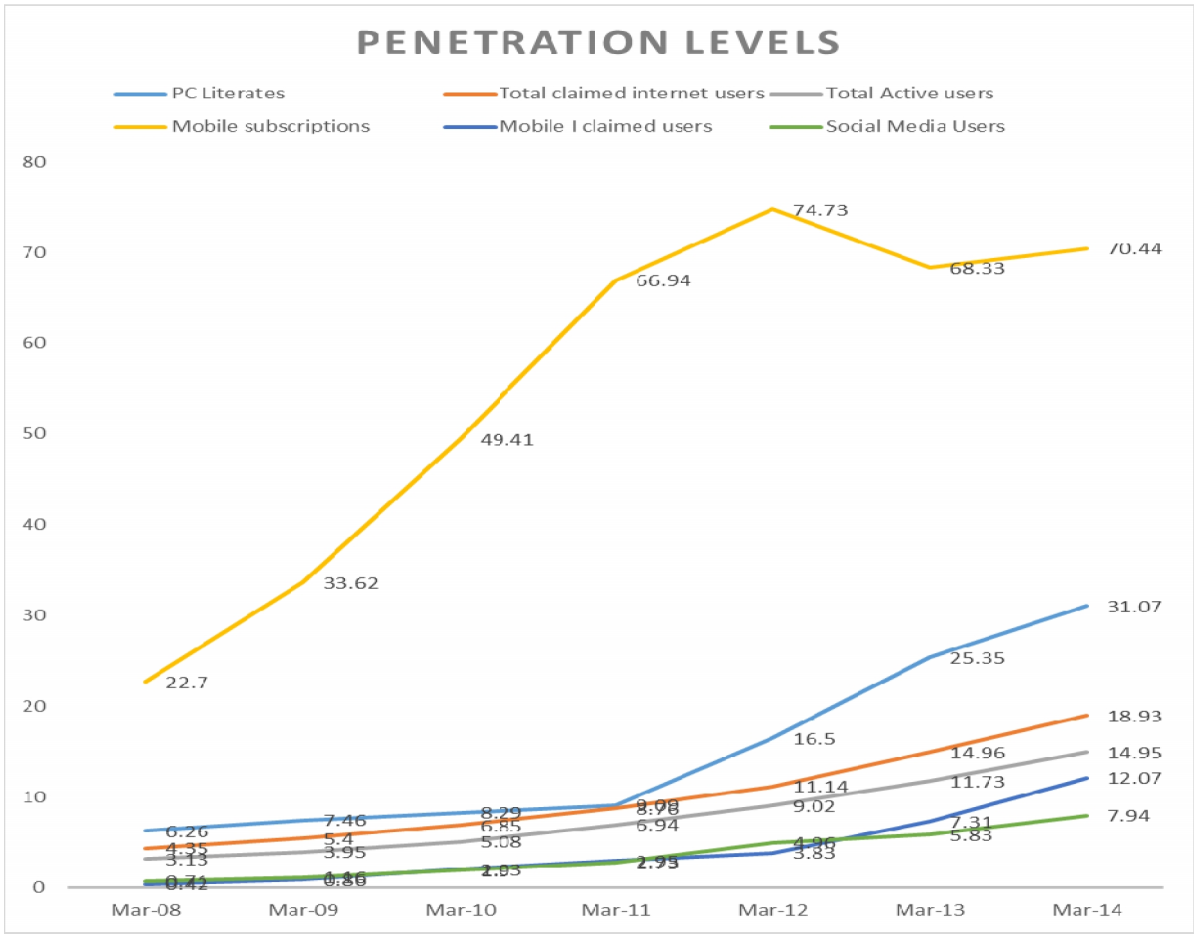


Figure 8
PENETRATION LEVEL OF ONLINE POPULATION, PC LITERATES, MOBILE INTERNET AND SOCIAL MEDIA USERS



TABLES

Table 1 - PC LITERATES, TOTAL CLAIMED AND ACTIVE INTERNET USERS (Figure are in millions)

Year	PC Literates	Total claimed internet users	Total Active users
Mar-07	62	41	29
Dec-07	68	46	32
Mar-08	72	50	36
Dec-08	82	58	43
Mar-09	87	63	46
Dec-09	92	63.4	61.5
Mar-10	98	81	60
Mar-11	110	106	84
Jun-11	119	112	88
Mar-12	203	137	111
Mar-13	322	190	149
Dec-13	372	213	169
Mar-14	399	243	192

Source: IAMAI report

Table 2 -PC LITERATES, TOTAL CLAIMED AND ACTIVE INTERNET USERS – URBAN INDIA(Figure are in millions)

Years	PC Literates	Total claimed internet users	Total Active users
Mar-04	53	16	11
Mar-06	59	32	21
Mar-07	62	42	29
Mar-08	72	50	36
Mar-09	87	63	46
Mar-10	98	73	55
Jun-11	117	85	67
Dec-11	124	92	73
Jun-12	133	99	80
Dec-12	140	105	84
Jun-13	194	111	89
Oct-13	194	137	116
Dec-13	204	141	120
Jun-14	220	158	136

Source: IAMAI report

Table 3 -PC LITERATES, TOTAL CLAIMED AND ACTIVE INTERNET USERS – RURAL INDIA(Figure are in millions)

YEAR	Total claimed internet users	Total Active users
Dec-08	5.5	3.3
Jun-09	5.85	3.7
Dec-09	7.45	5.5
Jun-10	10.6	8.5
Dec-10	15.2	12.1
Jun-11	21.4	17
Dec-11	29	24
Jun-12	38	31
Dec-12	45	38
Jun-13	59.6	41
Oct-13	68	46
Dec-13	72	49
Jun-14	85	56

Source: IAMAI report

Table 4 - MOBILE SUBSCRIPTIONS VS. CLAIMED MOBILE INTERNET USERS(Figure are in millions)

Year	Mobile subscriptions	Mobile I claimed users
Mar-09	392	10
Mar-10	584	24
Mar-11	810	35.7
Mar-12	919.17	47.1
Jun-12	934	58.8
Dec-12	895.51	87.1
Mar-13	867.8	92.9
Jun-13	873.36	91
Dec-13	886.3	128.3
Mar-14	904.51	155
Jun-14	914.92	185

Source: IAMAI report, ITU report

Table 5- SOCIAL MEDIA USERS – INDIA
(Figure are in millions)

YEAR	Social Media Users
Mar-07	6.3
Mar-08	8.2
Mar-09	13.5
Dec-09	18.7
Mar-10	22.5
Dec-10	28.8
Mar-11	33
Dec-11	38
Jun-12	61
Dec-12	72
Jun-13	66
Dec-13	99
Jun-14	106

Source: IAMAI report

Table 6
COMPARATIVE ANALYSIS OF INDIAN POPULATION VIS. A VIS. ONLINE POPULATION, PC LITERATES, MOBILE INTERNET AND SOCIAL MEDIA USERS
(Figure are in millions)

Year	Indian Population	PC Literates	Total claimed internet users	Total Active users	Mobile subscriptions	Mobile I claimed users	Social Media Users
Mar-07	1134	62	41	29	165	2.1	6.3
Mar-08	1150	72	50	36	261	4.8	8.2
Mar-09	1166	87	63	46	392	10	13.5
Mar-10	1182	98	81	60	584	24	22.5
Mar-11	1210	110	106	84	810	35.7	33
Mar-12	1230	203	137	111	919.17	47.1	61
Mar-13	1270	322	190	149	867.8	92.9	74
Mar-14	1284	399	243	192	904.51	155	102

Source: IAMAI report

Table 7

COMPARATIVE ANALYSIS OF GROWTH RATE OF INDIAN POPULATION VIS. A VIS. ONLINE POPULATION, PC LITERATES, MOBILE INTERNET AND SOCIAL MEDIA USERS

Year	Indian Population	PC Literates	Total claimed internet users	Total Active users	Mobile subscriptions	Mobile I claimed users	Social Media Users
Mar-08	1.41	16.13	21.95	24.14	58.18	128.57	30.16
Mar-09	1.39	20.83	26	27.78	50.19	108.33	64.63
Mar-10	1.37	12.64	28.57	30.43	48.98	140	66.67
Mar-11	2.37	12.24	30.86	40	38.7	48.75	46.67
Mar-12	1.65	84.55	29.25	32.14	13.48	31.93	84.85
Mar-13	3.25	58.62	38.69	34.23	-5.59	97.24	21.31
Mar-14	1.1	23.91	27.89	28.86	4.23	66.85	37.84

Table 8

PENETRATION LEVEL OF ONLINE POPULATION, PC LITERATES, MOBILE INTERNET AND SOCIAL MEDIA USERS

Year	PC Literates	Total claimed internet users	Total Active users	Mobile subscriptions	Mobile I claimed users	Social Media Users
Mar-07	5.47	3.62	2.56	14.55	0.19	0.56
Mar-08	6.26	4.35	3.13	22.7	0.42	0.71
Mar-09	7.46	5.4	3.95	33.62	0.86	1.16
Mar-10	8.29	6.85	5.08	49.41	2.03	1.9
Mar-11	9.09	8.76	6.94	66.94	2.95	2.73
Mar-12	16.5	11.14	9.02	74.73	3.83	4.96
Mar-13	25.35	14.96	11.73	68.33	7.31	5.83
Mar-14	31.07	18.93	14.95	70.44	12.07	7.94

Table 9

ANOVA: (GROWTH RATE)

SUMMARY

Groups	*Count*	*Sum*	*Average*	*Variance*
Indian Population	7	12.54	1.791429	0.574081
Total claimed internet users	7	203.21	29.03	26.25523
Social Media Users	7	352.13	50.30429	513.5827

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	8278.742	2	4139.371	22.97897	1.11E-05	3.554557
Within Groups	3242.472	18	180.1373			
Total	11521.21	20				

Table 10

T-TEST: TWO-SAMPLE ASSUMING UNEQUAL VARIANCES (ONLINE POPULATION VS. SOCIAL MEDIA POPULATION)

	Total claimed internet users	*Social Media Users*
Mean	29.03	50.30429
Variance	26.25523	513.5827
Observations	7	7
Hypothesized Mean Difference	0	
Df	7	
t Stat	-2.42255	
P(T<=t) one-tail	0.02296	
t Critical one-tail	1.894579	
P(T<=t) two-tail	0.04592	
t Critical two-tail	2.364624	

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RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

A Study on Research Articles on Hatchback Cars: Customer Perception and Buying Behaviour

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ABSTRACT

In the age of modern technology cars become essential rather than sign of status. Auto Industry is one of the quickest developing industry in our nation. It became a part of our day to day lifestyle and that's why car makers targeting the common men segment at higher level. The decision to buying a car is not an individual choice.

Today's scenario shifted from item marketing to necessity based marketing, buyers gets many choices of selection to make decision. This study determines that customer buying behaviour shows a dynamic role in making 4 wheels for marketers and there is wide possibility of exploration available in this area. This article is an effort of study the consumer buying manner while purchasing a car and to study of their intentions.

By this study it was found that value, resale price, average mileage, easy loans, luxury and security and models of the cars.

SUMMARY

Keywords: Buyer Perception 1, Customer Behavior 2, Purchase decision 3, Influencing factors 4

INTRODUCTION

Individual thoughts in general are very hard to anticipate at the time of choice or decision making. Occasionally, it may get easier and sometimes difficult, to simplify about human behaviour. Predicting the behaviour of people is a very complex task, because it loaded with lots of doubts, possibilities and surprises. Correct estimation or prediction can gives you good returns but incorrect predictions can results in the loss of business. In Today's, scenario consumer is the king of the market at globally. By studying the perception and behaviour of consumer it helps the dealer & manufacture to know how to make changes in existing items or product, which types of items are required/demanded in the market & how to draw buyer's interest to purchase their items.

India is an upcoming market for world auto-giants. As make in India concept initiated and due to low work cost numerous multinational companies are capitalizing their business in India market. As automobile industry grows very quickly from the middle era of liberalization i.e. in 1990's. India is one of the biggest democratic county in the word where its own automobile sector has having a huge demands. This demand also invites the other giant automobile manufacturers across world to come and invest in Indian car industry. Two important characteristics about cars helps us to make better understanding of automobile industry in our county. First, 4 wheels are durable products, we generally buy a car with the expectation of keeping it for an extensive period of time; even, we can also purchase a utilized or a new car, since it is an industry with a well-developed secondary market.

As there is a tremendous market potential available in the Indian Automobile Industry with the increase in population and change in the living standard of a common men as a result of development, it shows a rapid upswing in demand curve for Indian car industry. Due to which Indian car manufacturing industry has become successful like never before in the past few years. This amazing advancement that the Indian vehicles industry had shown the result of a major factor namely, improvement in living standard of the common men and an increase in their disposable incomes.

If we see economy graph of Indian automobile industry then car industry is one of the core industries. To survive in these competitive industry it is very much essential to frame the strategic plan for car manufacturer. As purchasing power of a common man get increase, there is a radical transformation arise in consumption pattern of consumers which attracts major car manufacturers to Indian Market. With the rise in income level, decreases in taxes and easy relax able interest rate on car loans helped to rise in personal disposable income of common men. Even the change in mind set made changes in spending pattern from two wheelers investment to 4 wheelers purchases.

In today's competitive market car manufacturers can authenticate their presence only if they can able to recognize buyer's needs, requirements and also able to satisfy them. Contemporary marketing theory for any successful organization is to study the customer/buyer as the central of their professional activity.

Consumer buying behaviour is one of the very significant aspects for any business because it emphases on how individuals person make choices to spend their existing incomes i.e. (time, money, effort) on consumption of related objects *(1)*. That includes what, why, when, where, how often they buy it, how they evaluate it after the purchase and the impact of such evaluation on future purchase decision *(1)*. The objective to study consumer behaviour is to comprise an examination of variables that affect purchase decisions and items usage by consumer.

Purchase decision of any consumer is most essential tool for any car manufacturer, to recognize how customers makes their purchase decisions helps marketing executives in various ways. For e.g., if a marketer knows through study or survey that design of car and fuel efficiency is the most vital feature for certain target audience, then company can remodel the item to meet their criteria. (E.g. Honda City various Model, Hyundai Verna, Maruti Suzuki balelo – from sedan to hatchback, etc.). Even if manufacturer can't modify the design in short time, then they can use advertising as a tool to change buyer's decision making criteria. For e.g., 4 wheeler or home gives more satisfaction than a toothpaste to consumer in their decision making. 4 wheeler can satisfies the requirement of buyer for their conveyance. As Consumer similarly acquire mental satisfaction by obtaining ownership of products like a car.

With the end of this study, the individual variables like age, income, qualification, profession etc., have been chosen and the impact of these individual variables on the on the consumer's perception, their purchasing behavior and the decision making process have been analyzed *(1)*.

REVIEWED ARTICLES

- Dr. H. S. Adithya (January 2013) describe consumer behaviour of making purchase decision is based on all human behaviour *(1)*. By understanding the concept of consumer behaviour marketer can make marketing decisions which helps them to suit with consumer needs and wants *(1)*. There are four major classes of consumer behaviour factors are involved namely, cultural, socio-economic, personal and psychological *(1)*. The socio-economic factors of consumer behaviour consist of age, marital status, profession, education, income, family size etc. *(1)*. Understanding the importance of passenger car industry in the current economic condition, the researcher has examined the perceptions and behaviour of consumers related to this product *(1)*.

- Shailesh K. Kaushal (March 2014) analysis the buyer behaviour in reference to car purchase intentions and automobile marketing strategies in Uttar Pradesh *(2)*. The paper identified five dimensions of car buyers' purchase objectives which are labelled as safety & security, quality, performance, value and technology *(2)*. The car buyers purchase intention influenced by several factors *(2)*. But from this study it will help the automobile manufacturer and car dealers to understand the buyers buying behaviour and help them to make their marketing strategies accordingly on the following factors (*2)*.

- Pooja G. Luniya and Dr. Manoj Verghese (August 2013) mention from their study that there are various factors which are responsible and influence the consumer to make their purchase decision like Mileage, Easy mode of financing and model *(3)*. As with the rapid and consistent growth in the price of the fuel consumer are more conscious about the mileage and accordingly companies are also modify their engines *(3)*. So they highly prefer mileage while buying a car *(3)*. The study also found that consumer also prefer easy mode of financing while purchasing a car *(3)*. With the growing competition in automobile sector, companies and various financial institutes are proving easy financing facility to grab the maximum consumers *(3)*. It was also found that consumer also prefer model while purchasing a car *(3)*. As we know that there is a wonderful change in the standard of living of the consumers, people are more conscious about the interior and exterior look, style-shape and amenities of a car which varies from model to model in cars *(3)*.

- Shiv Prasad Joshi (February 2013) study exposes that purchasing of car is mainly been influenced by the advertisements and secondly by recommendation of family and friends *(4)*. When they measured the level of satisfaction, it revealed that nearly fifty percent consumers are fully satisfied from their brand which they have selected, while around sixteen percent of respondents are not satisfied from their purchasing decision *(4)*. The study also describe that price factor is also one of the important factor in selection of car and in India safety measures in car are least preferred criteria *(4)*.

- R. Ganapathi, S. Subadra and S. Anbu Malar (March 2011) study describe Behaviour comprises of all human conduct that goes in settling on purchase decision *(5)*. Four main category of consumer behaviour elements and desires are specifically called, social, financial, individual and psychological *(5)*. Understanding the significance of the passenger car industry in the present circumstance, the analyst has to divide the judgments, and behaviour of the customer related to this product *(5)*. "It is rightly said yesterday's luxuries are today's necessities" *(5)*. From the study made from the paper, there are certain product characteristics which are distinguished in the study as affecting the buying decision and fulfilling the customer's requirement *(5)*. Car automakers should need to focus on these attributes as per the choice of potential consumers *(5)*.

PROPOSED RESEARCH METHODOLOGY

- **Objective of the Study:**
  1. To assess car holders' perception and behaviour concerning to the buying and usage of cars.
  2. To recognize and examine the features persuading the buying of cars.
  3. To study the level of fulfilment between the respondents and to recognize the shifting over product choice.
  4. To study the reasons for purchasing a specific brand of four-wheeler.
  5. To study the various features that trigger consumers to buy specific four-wheeler brand like Hatchback Cars in Saurashtra region.

- **Significance of the Research:**
  - **Sample Design**
    a. Sampling Population: Hatchback car owners of Gujarat
    b. Sampling Method: Non Probability Judgment and Convenience sampling
    c. Sample Size: 500
    d. Research Instrument: Questionnaire consist of both open and closed ended questions

- **Hypothesis of the Study:**
  1. There is no significant difference towards preference of hatchback cars among different age groups.
  2. There is no significant difference towards preference of hatchback cars among different income levels.
  3. There is no significant change towards product choice towards country of origin.
  4. There is no important change among the factors influencing product choice.

- **Data Collection:**
  Researcher will collect Primary Questionnaires consist of open and close ended and on Secondary data for the verification of objectives. Secondary data shall be collected from various published sources i.e. journals, newspapers, national and international publication, internet, personal books and libraries.

- **Tools of Analysis:**
  - Graphs, Charts,
  - Chi-square Test & Independent Test
  - Factorial analysis – Coefficient Correlation
  - KMO Model, ANOVA Technique
  - Data Reduction, Data Classification,
  - Data Extraction and Data Tabulation

CONCLUSION

The above study describes that Indian car manufacturing industry in one of the giant automobile sector in the world and one of the reason is county's population. So, to established the business in India marketers need to study the consumer behaviour and its perception for purchasing decision for any product specially cars. As above study expresses that car is a durable product and consumers when make purchase decision for such product they made it for long period of time. Most of reviews and articles suggest that consumer buying behaviour consists of all individual behaviour that goes in making acquisition choices. There are various factors which marketers need to take in to consideration to full fill the needs and desire of customers, various factors plays a vital role in decision making i.e. age, income, qualification, brand preference, models, family and friends suggestion, past experiences, etc. So, car manufactures need to study all these factors and according to the base of study they need to enter in the market.

As Indian economy is getting stable and with the initiative of Making India Project world giant automobile leaders are stepping their foot prints in our county. At the same time the standard of living of common men is also getting improve so they are investing their durable income in making the purchase decision of buying cars. While making this decision various factor they are infusing like, mileage, exterior, interior, easy mode of payment in terms of availability of loans, taxes and lots more. So it is been rightly been said "yesterday's luxury is becoming today's necessity".

ACKNOWLEDGEMENT

This paper is made possible through the help and support from everyone, including: guide, parents, family, friends, and in essence, all responsive beings. Especially, please allow me to dedicate my acknowledgment of gratitude to all advisors and contributors. I would like to thank Dr. Chetna Parmar for his most support and encouragement in preparing my paper.

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Cradle to the Skies – A Journey of a Successful Female Entrepreneur

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ABSTRACT

"NaariTuNarayani" is a sentence that define so many aspects of a Woman's life. Nature or rather God has gifted a woman with various talents and made her both very strong and tender soft. This versatility in a woman, her basic multitasking talent, her being a born manager are some of the key features that definitely add to a success journey for a female entrepreneur. Here in this paper I have presented a success journey of a very young and dynamic female entrepreneur originally from Rajkot and currently settled in Ahmedabad, but associated with many institutes in Rajkot, who has really put in the best of her efforts, and is a very well-known public figure also. She has proven herself the best in all the roles they she has played in her professional and personal life. It is said that behind every successful man there is a woman and behind every successful woman there is an entire family and many a times the father and husband as the strongest pillars of support.

SUMMARY

It is a success journey of female entrepreneur from Rajkot itself.

Keywords: Female entrepreneur, Multitasking, Versatility, Born Managers

INTRODUCTION

Gone are the days when women were considered no match for all powerful men in this world.

The male dominated world was always reluctant to not only accept her or to even acknowledge the fact that women were as good as men on parameters of hard work, intelligence quotient (IQ) and leadership traits.

But this new generation women across the world have proven them all wrong and rather they have proved themselves beyond doubt in all spheres of life including the most intricate and cumbersome world of entrepreneurship.

Yes, there is a section among women who believe in short-cuts but at the same time there is no dearth of women who are confident ,believe in themselves and have enormous fire in their bellies to take on the best in the business and beat them at their own game.

India too has its own pool of such bold and fearless women who have made a mark for themselves both within the country as well as overseas. Here in Gujarat, Business resides in the blood of every Gujarati, so presenting before you one such success story of a young female entrepreneur.

Their relentless zeal, incessant quench for success and willingness to walk the extra mile have broken all myths about their inborn limitations that were supposed to be major roadblocks on their success expressways.

Nisha Punjabi Thakrar – A Young and Dynamic Female Entrepreneur

After finishing her Grade 12th studies her Principal Mr.KiranPatel called her for a meeting and asked her to join his education institute as a teacher for the new afternoon shift he was starting in June 1998. Simultaneously she also took up admission in Christ College under sports quota, which was the first Co. Ed College of the city, but the fulltime graduation demanded attendance, which would not be possible for her with a full time job. The choice was tough but according to her guess that's when your passion speaks up from inside & she chose to be a Teacher join TGES and opted for Kundalia College as attendance was

not a compulsion there. 5 Years she worked in TGES as a Pre School Teacher and also was given responsibility of children with Special needs. During these 5 years she completed her B.Com & Masters in Interior Design.

Then she got a chance of working with a dream brand TATAs. She joined Tata Teleservices Pvt. Ltd & was associated with it for almost 4 years plus. In that time she got married to her best friend. She left Tatas as she was blessed with an angel in her life, Venisha. She took a complete break from work for 2 years as her daughter was an infant. Once again she was ready to go back and she had to choose again between corporate job orher passion, Education- Teacher – Children all around. Her obvious choice was School – Children – Education.

She got back to work with TGES & this time engaged herself to a different aspect of Education – Human Resource. TGES hires huge lot of employees every year & she got to experience a solid recruitment process. For this she had to travel extensively across India to hire Candidates. Out of 20000 + profiles, they selected approx. 100 teachers. This was again a good learning time for her. Today when a candidate sits in front of her, she can immediately have her instincts working automatically. In that 1 year she completed her B.Ed & Diploma in Montessori Teaching program adding up more to her knowledge pool, skills set as well as her CV.

After 1 year of that she had to shift back to Ahmedabad. There she got an opportunity to associate in the management team of a chain of Preschool namely “Shanti Juniors”, by Shanti Educational Initiative. The again a new profile awaited her, she worked on Curriculum Development & Franchisee Management. She was handling & Monitoring 5 preschools at time. This taught her the entire understanding of franchisee network & business partner management & Audits. The preschool curriculum was based upon Play Way Approach which she got to master with the experience.

One more timea new opportunity knocked her door through getting associated with Edvance Preschools Pvt. Ltd. It was a company started by Ex-President Times of India group, India & had tie up with Eton House Singapore. She was appointed as School Support Manager for West Zone (Gujarat, MP, Chhattisgarh and Rajasthan). Here she got to handle just not only the preschools but also had a huge task of setting up Preschool Premises. Process included Selection of Premises to Renovation as per company & educational standards, to Selection of appropriate Educational resources as per city dynamics, to Recruitment process, to Training, to Marketing Planning, to Operational planning and setup to Financial

Audits, to Curriculum Support. It was an amazing roller coaster ride of associating with so many people, cultures, premises, and parents, Children, Marketing events, workshops, educators and lots of travel!!!! She was thoroughly trained for Reggio Emilia Approach towards curriculum as that was the base & highlight point of the preschools. This was a real time learning and hands on experience of learning while actual in field doing.

Then she was pulled by Klay. Klay has been the most memorable part of her life. VBHC (Value Budget housing Corporation) Education services was opening a High End Prep School in Ahmedabad named KLAY (Kids Learning And You) and she was pouched by them to head their preschool. She joined them as Principal. The journey started same as she had to setup the premises & launch it. But this it was different! It was made with international standards matching to the guidelines of NAEYC (National Association for the Education of Young Children) It was the most expensive Preschool of Ahmedabad & she had taken the challenge of giving live access of entire school's cameras to all parents. It was just like staying in Big Boss's home!!! Well that was one of her strong marketing strategies of genuinely caring for kids at the centre and giving an assurance to parents by building a completely transparent approach. It worked! Miracles and she turned to be a good marketing strategist to make her product launch and be successful in the market. In 3 years' time duration, they reached 200 admissions in the most expensive preschool. Media tried to label expensive negatively to Klay but with 100% dedication & giving more than best to every child they could easily justify and live up to the expectations of everyone. During this time she did her course with Asian International College, Singapore on "International Practices in Preschool Teaching". Klay was based on Multiple Intelligence approach on its curriculum & pedagogy. After this experience she could say she was a trained professional for

1. Montessori Approach
2. Play Way Approach
3. Reggio Emilia Approach
4. Multiple Intelligence Approach

After 3 years the company was keener on focusing only metros for placing Klay centres. They passed a decision of shutting down Klay Ahmedabad & Diverting funds from there to Hyderabad. For NishaKlay was her second baby, she had put in her soul for nurturing it like her own baby. She had given complete energy & focus on it & made it a brand in Ahmedabad. This emotional connect made her decide to take over Klay& not let it shut down. And with this the Entrepreneur was born, she created her own company to take over Klay. VeNisha Education Pvt Ltd came into existence. Again in business every time it is not

a cake walk. They were almost done when they faced many huddles over the transition with the company. Many of the clause were not acceptable and overall the deal of takeover didn't work out. The result was she detached herself from Klay& with her 20 to 30 parents & fifty percent staff left Klay. It was a difficult phase for all. Since all other stake holders had left to support her and have faith, she was feeling more responsible for settling them. She then connected parents to another centre & personally went there to settle parents & kids, for teachers she made sure all of them were placed in proper places. This makes an Entrepreneur so much responsible as a citizen of a country. Here she has emerged as a real Hero taking up the responsibility of so many people and their career as well as life.

During that time she was invited by World Forum Foundation for a Global Conference in Puerto Rico, USA to represent, India. She attended the conference & was declared as Chairperson Gujarat for Early Childhood Association, India by the President of ECA, India. That was a huge responsibility to take up in the sector. Though the times earlier were a bit tough to go through, here it was again new dawn waiting for one more opportunity.

She came back and as she had already launched her company, she took that task ahead & started taking projects for Preschool setup, curriculum development, onsite Operational support, Trainings, Workshops, Audits and so much more. Work started pouring in. She started making a team, but she did not want full time workers. So she recruited teachers who can connect to her work in their extra time. This made them feel pressure less, and gave them an environment to get connected well, and no worries of reaching office, they could easily work from home, coordinate and meet as and when required.

Today she has projects running in Ahmedabad, Gandhinagar, Indore & Rajkot. She is also personally connected with Sri SriRavishankarVidyaMandir, Rajkot in the Management team. They are also setting up 5 preschools to start with and many more in pipeline in Rajkot under the Sri Sri banner. She is also a partner in the company Educational Skills and Concepts Pvt. Ltd. They set up customized trainings, Workshops, Hand holding for Educational Institutes. ESC teams educators who come with specialized experience in Education Sector. They are going to organize a largest training fest in Ahmedabad in February 2016 which will have a large spread of trainings laid out for all people as well as educators. She also offers her honorary services to Early Childhood Association.

A Brief introduction of Nisha Punjabi Thakrar, her both families of orientation and pro-creation

She belongs to a business family of Rajkot. Her father has was the one to introduce Greeting cards in Gujarat and start gift and card shops in Rajkot. They were the first ones to establish wedding cards & framing in the city too. Her mother was also a business women handling Archies Gallery in Rajkot. Her sister has been a wonderful human being emitting love & care for all and she is currently busy setting up her husband's dream project of school "The North Star School".

Nisha's Family of Pro-creation – Her husband, Mr.YameshThakrar, holds a dynamic personality with great leadership qualities. He is associated with Education sector catering to the international markets & trainings. He is on advisory board with AIESEC Lucknow, and holds a responsible position in society serving Shree LohanaMahaparishad. He too comes from a business family from Porbander but born & brought up in Rajkot. They are blessed with a beautiful and talented daughter, VeNisha who is 10 years old.

Hereditary features - Acquired Skills for being a Successful Entrepreneur

According to her the strong business genes that come from Sindhi Punjabis' and all their generations have been into business. Her father is a very creative & out of box thinker in his way of doing business and according to her, it flows in the blood.

Role of Family

Yes definitely her father has never stopped her from doing anything in life or making any choices & always treated her equal to a son. More than a son but never less.
From there her husband was the one to help her focus on a vision & move in that direction. He never bonded her to any restrictions nor set any limitations for her. In fact he made her take challenges & played a major role in supporting her decisions. He motivated her to keep adding to her education and travel to get real experiences and learnings of life.

Role of Education

According to Nisha, she was very lucky to be a part of a very well-known Educational Institute of Rajkot known as TGES. One cannot deny the fact that the pillars or the roots need to be very strong enough for a building or a tree to achieve its heights of Success. It has given her all values & decorum of life. Many of which one actually applies during work and life from what the educators demonstrated and what was valued in school. After school rest of the education was more content based which added to knowledge but not to the experience or values of life.

The Idea and the Person who made you feel that spark in you as an Entrepreneur

The way 3 years she was associated with Klay& the way she was able to establish it as a brand with quality services.

It was her husband Mr.Thakrar who felt that spark in her and supported her to launch her own company into Education sector.

Your inspiration and Your Role Model

Inspiration was the way a quality set up & team can bring changes to life of so many in the society. Its all about doing the work you really like doing, only then you can give 100% to it and bring the change. For her, the role model is definitely her husband in this journey.

Overcoming the greatest Obstacles

She described her worst time was when she was going to take over the preschool and the deal got cancelled. Socially there is a lot of pressure that they had to face as many useless questions are raised all around which could break you down. But again her husband proved to be her biggest strength was continuously supporting her and was there beside her as a rock solid pillar to face all the hurdles. So together they gave a new vision and direction to the company they had set.

Characteristics of Successful Female Entrepreneurs

The nature's blessings of making females Multi taskers which males are not, is one such characteristic. The way they can handle Home-children-work-socializing-all of rest, it is just miraculous. They are best jugglers, juggling between different roles as versatile personalities also.

Role of Culture

Culture does play a part as when she says, she looks back at her family culture, she belonged to was the one without any rigidness and was considering females as equal in the house. Women had a say in family and were allowed to pursue what they wanted to, be it daughters or daughter in laws. The same environment was also there in her husband's family. If she was raised in a restricted culture she would not have even thought of pursuing her passion. And if her in-laws side culture would not have been supportive she would not have got all the gains. That is the reason it is said well that if behind every successful man there is a woman, then behind every successful woman there are strong and supportive families.

Keys to Professional Success for women in India

Staying Focused is the Key, never get diverted or walk on any sort of short cuts. Follow your passion to enjoy your work. These are best keys to professional success for all those women who are in the making of Successful female entrepreneurs.

Advice to the Young Women

Early Childhood Education is a beautiful sector and has a lot more to explore and focus in our country. She believes and would like to share the mantra of "No Passion No Action". Have that burning fire in your belly to achieve something in life that you love the most, your passion in life and do anything for achieving it with, being ethical and not compromising on your self-belief and values.

Conclusion

It is not an easy task for any entrepreneur to be successful at the very initial go itself. But for Women Entrepreneurs in India it is even more challenging. They have to face the challenges of cultural bias and lack of public safety at times in addition to the pressures of balancing work, home and family. Just to summarize all in a Nut shell, these things can be a carry home for every young women out there if she wishes to be a successful entrepreneur.

Treat your work and profession very seriously, or you will bring a bad name to women in general. Take pride in what you do, don't slip on quality. Build your own sense of instinct and gut feel, which will take you from something ordinary to something else extraordinary.

Work hard and be patient, even those who initially oppose you may support you later. Keep a positive mind, and don't take 'no' for an answer. Don't be deterred by failure. Don't believe that women are 'less' than a man: a woman is a womb plus a man!

Value your integrity, and be honest to your customers and employees. Make 'clean' money rather than 'tainted' money. Honesty will give you good sleep. Stay healthy and fit. Be friendly, but being 'too friendly' is easily mis-interpreted by unscrupulous men. Give back to society, there is more to life than money.

Work on your relationship with yourself. Keep mental and physical space for yourself to regularly think, plan, mediate and dream. Learn how to have different kinds of dreams – near term and long term. At the end of the day, keep your sense of humor!

And lastly don't forget the special blessing given by God only to the Woman "Sarva Gun Sampan".

ACKNOWLEDGEMENT

I would definitely Thank the Almighty for always guiding me right in all walks of Life. Heartfelt Thank you to my esteemed Institute for organizing this conference and giving me an opportunity for presenting my work on this platform. I am very much Thankful to My honorable parents, sweet sister and my husband as well as my sweet son for always supporting me in all my works. Special Thanks to Ms. Nisha

Punjabi Thakrar who took out the time from her too much busy schedule and had that faith in me for sharing her valuable lessons for other budding female entrepreneurs in this region.

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RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

A Behavioral Influence Of Investors In IPOs

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ABSTRACT

As we all know IPO – INITIAL PUBLIC OFFERING is the hottest topic in the current industry, mainly because of India being a developing country and lot of growth in various sectors which leads a country to ultimate success. This report talks about the Behavioural Influence of Investors in IPO's. How investors behave while investing in IPO's. What are risk factors in IPO's? What are pros and cons of IPO's? Parameters to judge an IPO's. What is behavioural finance, behavioural biases. Our objective for this study was to know how the behaviour effects investment decision and to understand the different dimensions of behaviour on investment decision. Our questionnaire is based on how people frequently investing in IPO's, for how many years they are investing, purpose of investment,kind of stocks do theyinvest, Kind of risk appetite do they prefer, etc. The findings and conclusion is based on questionnaire.

SUMMARY

Different Factors of Behavioral finance effect on investor's decision pattern.

Keywords: IPO, Behavior Finance, Investment Decision, Risk Appetite

INTRODUCTION

IPO stands for **Initial Public Offering** and means the new offer of shares from a company which was previously unlisted. This is done by offering those shares to the public, which were held by the promoters or the private investors prior to the IPO. In the case when other investors or Promoter held the shares the stake holding comes down to the extent their shares are offered to the public. In other cases new shares are issued to the public and the shares, which are with the promoters stay with them. In both cases the share of the promoters in the total capital comes down.

BEHAVIOURAL FINANCE

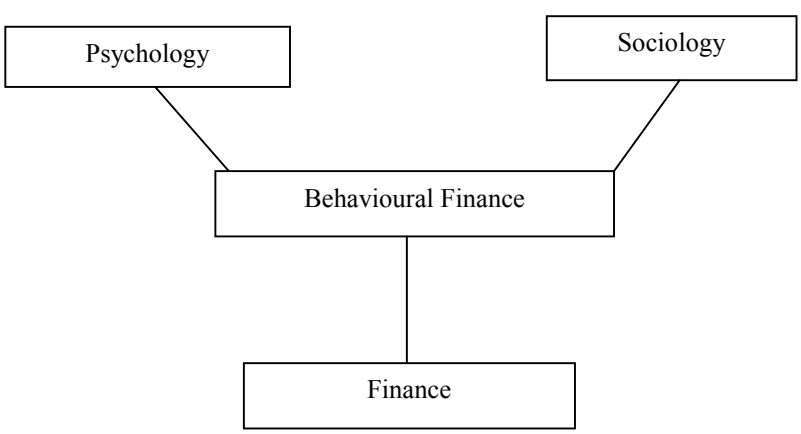
Introduction

"Behavioural finance is the study of the influence of psychology on the behaviour of financial practitioners and the subsequent effect on markets." The science deals with theories and experiments focused on what happens when investors make decisions based on hunches or emotions.

Thus, Behavioural finance can be defined as a field of finance that proposes explanation of stock market anomalies using identified psychological biases, rather than dismissing them as *"chance results consistent with the market efficiency hypothesis."(*Fama, 1998). It is assumed that individual investors and market outcomes are influenced by information structure, and various characteristics of market participants (Banerjee, 2011).

Prospect Theory

The Prospect theory distinguishes two phases in the choice process: the early phase of framing (or editing) and the subsequent phase of evaluation. In essence, the theory explains the apparent irregularity in human behaviour when assessing risk under uncertainty. It says that human beings are not consistently risk-averse; rather they are risk-averse in gains but risk-takers in losses.



MATERIALS AND METHODS

❖ **Objectives**

- To study how the behaviour effects investment decision.
- To understand the different dimensions of behaviour on investment decision.

SCOPE OF THE STUDY

The Scope of the study is based on behavioural finance of investors how each individual react while they invest their hard earn money in IPO's. As IPO is initial public offering so each demographic factor has an impact on their decision. By this study we came to know which demographic factor has most impact while taking the decision on investments. The study was restricted to Kutch region Investors only.

HYPOTHESIS OF THE STUDY

This study is based on the following hypothesis:

- ✓ Decision has an Impact of Age Group and Gender of Investors.
- ✓ Each Investor has different behaviour Dimension while investing in IPO.

Population:-

- The respondent for our research are investors who are investing in IPO's.

Data collection method

- **PRIMARY DATA**- is through QUESTIONNAIRE wherein 17 questions were set to know the how the investor's behaviour influences while investing in IPO's.

Sample Size

- The sample size taken is of 100 investors of Adipur and Gandhidham.

Research design

- ✓ The research design for our topic "A BEHAVIORAL INFLUENCE OF INVESTORS IN IPO's" is Exploratory Research Design because exploratory is to enhance our understanding the different dimensions of investor's behaviour while taking investment decision.

Sampling design

In sampling design we had taken stratified random sampling. A random sample from each stratum is taken in a number proportional to the stratum's size when compared to the population.

RESULTS AND DISCUSSION

gender * frequently invest in IPO's Cross tabulation

			frequently invest in ipo's					
			monthly	quarterly	half yearly	annually	as an when ipo is offered	Total
Gender	Male	Count	10	5	2	20	30	67
		Expected Count	14.1	4.0	2.7	18.8	27.5	67.0
		% within gender	14.9%	7.5%	3.0%	29.9%	44.8%	100.0%
		% within frequently invest in ipo's	47.6%	83.3%	50.0%	71.4%	73.2%	67.0%
		% of Total	10.0%	5.0%	2.0%	20.0%	30.0%	67.0%
	Female	Count	11	1	2	8	11	33
		Expected Count	6.9	2.0	1.3	9.2	13.5	33.0
		% within gender	33.3%	3.0%	6.1%	24.2%	33.3%	100.0%
		% within frequently invest in ipo's	52.4%	16.7%	50.0%	28.6%	26.8%	33.0%
		% of Total	11.0%	1.0%	2.0%	8.0%	11.0%	33.0%
Total		Count	21	6	4	28	41	100
		Expected Count	21.0	6.0	4.0	28.0	41.0	100.0
		% within gender	21.0%	6.0%	4.0%	28.0%	41.0%	100.0%
		% within frequently invest in ipo's	100.0%	100.0%	100.0%	100.0%	100.0%	100.0%
		% of Total	21.0%	6.0%	4.0%	28.0%	41.0%	100.0%

Analysis:-

MALE:- The above table shows that out of 67 males there are 10 males who invest in IPO monthly. There are 5 males who invest in IPO in half yearly. There are 2 males who invest in IPO Quarterly. There are 20 males who invest in IPO annually. There are 30 males who invest in IPO as an when IPO is offered.

FEMALE:- The above table shows that out of 33 females there are 11 females who invest in IPO monthly. There is 1 female who invest in IPO in half yearly. There are 2 females who invest in IPO Quarterly. There are 8 females who invest in IPO annually. There are 11 females who invest in IPO as an when IPO is offered.

- **Interpretation:-**

  From the above analysis we can interpret that both males & Females frequently invest on MONTHLY, ANNUALLY & AS AN WHEN IPO IS OFFERED. As per the sample we can interpret that there is no knowledge gap between both genders regarding IPO.

age group * years of investment Crosstabulation

			years of investment				
			less then 1 year	1 - 3 years	3 - 5 years	above 5 years	Total
age group	16-25	Count	19	17	10	1	47
		Expected Count	14.1	20.7	9.4	2.8	47.0
		% within age group	40.4%	36.2%	21.3%	2.1%	100.0%
		% within years of investment	63.3%	38.6%	50.0%	16.7%	47.0%
		% of Total	19.0%	17.0%	10.0%	1.0%	47.0%
	26-35	Count	11	16	7	2	36
		Expected Count	10.8	15.8	7.2	2.2	36.0
		% within age group	30.6%	44.4%	19.4%	5.6%	100.0%
		% within years of investment	36.7%	36.4%	35.0%	33.3%	36.0%
		% of Total	11.0%	16.0%	7.0%	2.0%	36.0%
	36-50	Count	0	9	2	0	11
		Expected Count	3.3	4.8	2.2	.7	11.0
		% within age group	.0%	81.8%	18.2%	.0%	100.0%
		% within years of investment	.0%	20.5%	10.0%	.0%	11.0%
		% of Total	.0%	9.0%	2.0%	.0%	11.0%
	51 ABOVE	Count	0	2	1	3	6
		Expected Count	1.8	2.6	1.2	.4	6.0
		% within age group	.0%	33.3%	16.7%	50.0%	100.0%
		% within years of investment	.0%	4.5%	5.0%	50.0%	6.0%
		% of Total	.0%	2.0%	1.0%	3.0%	6.0%
Total		Count	30	44	20	6	100
		Expected Count	30.0	44.0	20.0	6.0	100.0
		% within age group	30.0%	44.0%	20.0%	6.0%	100.0%
		% within years of investment	100.0%	100.0%	100.0%	100.0%	100.0%
		% of Total	30.0%	44.0%	20.0%	6.0%	100.0%

Analysis:-

Age 16-25:- The above table shows that out of 47 investors within the age group of 16-25 there are 19 investors who have invested in IPO less than 1 year. There are 17 investors who have invested in IPO from 1-3 years. There are 10 investors who have invested in IPO from 3-5 years. There is 1 investor who invested in IPO above 5 years.

Age 26-35:- The above table shows that out of 36 investors within the age group of 26-35 there are 11 investors who have invested in IPO less than 1 year. There are 16 investors who have invested in IPO from 1-3 years. There are 7 investors who have invested in IPO from 3-5 years. There are 2 investors who invested in IPO above 5 years.

Age 36-50:- The above table shows that out of 11 investors within the age group of 36-50 there is no one in our respondent who have invested in IPO less than 1 year. There are 9 investors who have invested in IPO from 1-3 years. There are 2 investors who have invested in IPO from 3-5 years. There is no one who invested in IPO above 5 years.

Age above 51:- The above table shows that out of 6 investors who have age above 51 there is no one in our respondent who have invested in IPO less than 1 year. There are 2 investors who have invested in IPO from 1-3 years. There is 1 investor who has invested in IPO from 3-5 years. There are 3 investors who invested in IPO above 5 years.

- **Interpretation:-**

  From the above table we can interpret that most of all age group investor who invest in IPO they most of invest in IPO either less than 1 year or up to 1-3 years.

$selectipo*agegroupCrosstabulation

			age group				
			16-25	26-35	36-50	51 ABOVE	Total
select ipo[a]	tips from friend	Count	16	10	1	0	27
		% within $selectipo	59.3%	37.0%	3.7%	.0%	
		% within agegroup	34.0%	27.8%	9.1%	.0%	
		% of Total	16.0%	10.0%	1.0%	.0%	27.0%
	Newspaper	Count	21	21	4	4	50
		% within $selectipo	42.0%	42.0%	8.0%	8.0%	
		% within agegroup	44.7%	58.3%	36.4%	66.7%	
		% of Total	21.0%	21.0%	4.0%	4.0%	50.0%
	insider info	Count	7	3	1	3	14
		% within $selectipo	50.0%	21.4%	7.1%	21.4%	
		% within agegroup	14.9%	8.3%	9.1%	50.0%	
		% of Total	7.0%	3.0%	1.0%	3.0%	14.0%
	published articles	Count	14	5	3	1	23
		% within $selectipo	60.9%	21.7%	13.0%	4.3%	
		% within agegroup	29.8%	13.9%	27.3%	16.7%	
		% of Total	14.0%	5.0%	3.0%	1.0%	23.0%
	street talk	Count	7	0	1	0	8
		% within $selectipo	87.5%	.0%	12.5%	.0%	
		% within agegroup	14.9%	.0%	9.1%	.0%	
		% of Total	7.0%	.0%	1.0%	.0%	8.0%
	brokers advice	Count	14	14	4	2	34
		% within $selectipo	41.2%	41.2%	11.8%	5.9%	
		% within agegroup	29.8%	38.9%	36.4%	33.3%	
		% of Total	14.0%	14.0%	4.0%	2.0%	34.0%
Total		Count	47	36	11	6	100
		% of Total	47.0%	36.0%	11.0%	6.0%	100.0%

Percentages and totals are based on respondents.

a. Dichotomy group tabulated at value 1.

Analysis:-

Age 16-25:- The above table shows that there are 16 investors who select an IPO by taking tips from friends/relative. There are 21 investors who select IPO by newspaper. There are 7 investors who select IPO by getting insider information. There are 14 investors who select IPO by study of company from any published article or website. There are 7 investors who select IPO for investment by street talk/rumours. There are 14 investors who select IPO by going with broker's advice.

Age 26-35:- The above table shows that there are 10 investors who select an IPO by taking tips from friends/relative. There are 21 investors who select IPO by newspaper. There are 3 investors who select IPO by getting insider information. There are 5 investors who select IPO by study of company from any published article or website. There is 0 investors who select IPO for investment by street talk/rumours. There are 14 investors who select IPO by going with broker's advice.

Age 36-50:- The above table shows that there is 1 investor who selects an IPO by taking tips from friends/relative. There are 4 investors who select IPO by newspaper. There is 1 investor who select IPO by getting insider information. There are 3 investors who select IPO by study of company from any published article or website. There is 1 investor who selects IPO for investment by street talk/rumours. There are 4 investors who select IPO by going with broker's advice.

Age above 51:- The above table shows that there is no investor who selects an IPO by taking tips from friends/relative. There are 4 investors who select IPO by newspaper. There are 3 investors who select IPO by getting insider information. There is 1 investor who select IPO by study of company from any published article or website. There is 0 investor who selects IPO for investment by street talk/rumours. There are 2 investors who select IPO by going with broker's advice.

- **Interpretation:-**

  From the above analysis we can interpret that all age investor who invest in IPO either by seeing newspaper or by taking brokers advice. The investor who has age above 51 they also go with insider information for investing in IPO. From the above analysis we can also that the investors who are above 51 age they take less risk with their income and they are risk averse so they take detail information before investing.

$selectanipo*gender Crosstabulation

			gender		
			Male	female	Total
select an ipo[a]	tips from friend	Count	16	11	27
		% within $selectanipo	59.3%	40.7%	
		% within gender	23.9%	33.3%	
		% of Total	16.0%	11.0%	27.0%
	Newspaper	Count	37	13	50
		% within $selectanipo	74.0%	26.0%	
		% within gender	55.2%	39.4%	
		% of Total	37.0%	13.0%	50.0%
	insider info	Count	8	6	14
		% within $selectanipo	57.1%	42.9%	
		% within gender	11.9%	18.2%	
		% of Total	8.0%	6.0%	14.0%
	published articles	Count	11	12	23
		% within $selectanipo	47.8%	52.2%	
		% within gender	16.4%	36.4%	
		% of Total	11.0%	12.0%	23.0%
	street talk	Count	5	3	8
		% within $selectanipo	62.5%	37.5%	
		% within gender	7.5%	9.1%	
		% of Total	5.0%	3.0%	8.0%
	brokers advice	Count	25	9	34
		% within $selectanipo	73.5%	26.5%	
		% within gender	37.3%	27.3%	
		% of Total	25.0%	9.0%	34.0%
Total		Count	67	33	100
		% of Total	67.0%	33.0%	100.0%

Analysis:-

Male:- The above table shows that there are 16 males investor who select IPO by taking tips from friends/relatives. There are 37 males investor who select IPO by reading newspaper. There are 8 males investor who select IPO by taking insider information. There are 11 males investor who select IPO for investment by doing study of company from any published article or website. There are 5 males investor who invest in IPO by hearing street talk/rumours. There are 25 males investor who select IPO by going with broker's advice.

Female:- The above table shows that there are 11 females investor who select IPO by taking tips from friends/relatives. There are 13 females investor who select IPO by reading newspaper. There are 6 females investor who select IPO by taking insider information. There are 12 females investor who select IPO for investment by doing study of company from any published article or website. There are 3 females investor who invest in IPO by hearing street talk/rumours. There are 9 females investor who select IPO by going with broker's advice.

- **Interpretation:-**

  As per the above analysis we can interpret that male investor go with reading NEWSPAPER or by taking BROKERS ADVICE. Males are the logical investors. While here the female goes with FRIENDS/RELATIVES or by reading NEWSPAPER they invest in IPO. While female having sign of Herd behaviour while investing the money in IPO.

age group * avg annual investment Crosstabulation			avg annual investment				Total
			below 1,00,000	1,00,000 - 2,00,000	2,00,000 - 3,00,000	above 3,00,000	
age group	16-25	Count	23	17	5	2	47
		Expected Count	22.1	16.5	5.6	2.8	47.0
		% within age group	48.9%	36.2%	10.6%	4.3%	100.0%
		% within avg annual investment	48.9%	48.6%	41.7%	33.3%	47.0%
		% of Total	23.0%	17.0%	5.0%	2.0%	47.0%
	26-35	Count	20	11	4	1	36
		Expected Count	16.9	12.6	4.3	2.2	36.0
		% within age group	55.6%	30.6%	11.1%	2.8%	100.0%
		% within avg annual investment	42.6%	31.4%	33.3%	16.7%	36.0%
		% of Total	20.0%	11.0%	4.0%	1.0%	36.0%
	36-50	Count	4	6	0	1	11
		Expected Count	5.2	3.9	1.3	.7	11.0
		% within age group	36.4%	54.5%	.0%	9.1%	100.0%
		% within avg annual investment	8.5%	17.1%	.0%	16.7%	11.0%
		% of Total	4.0%	6.0%	.0%	1.0%	11.0%
	51 ABOVE	Count	0	1	3	2	6
		Expected Count	2.8	2.1	.7	.4	6.0
		% within age group	.0%	16.7%	50.0%	33.3%	100.0%
		% within avg annual investment	.0%	2.9%	25.0%	33.3%	6.0%
		% of Total	.0%	1.0%	3.0%	2.0%	6.0%
Total		Count	47	35	12	6	100
		Expected Count	47.0	35.0	12.0	6.0	100.0
		% within age group	47.0%	35.0%	12.0%	6.0%	100.0%
		% within avg annual investment	100.0%	100.0%	100.0%	100.0%	100.0%

	% of Total	47.0%	35.0%	12.0%	6.0%	100.0%

Analysis:-

Age 16-25:- The above table shows that there are 23 investors within the age group, who do average annual investment in IPO below 1, 00,000. There are 17 investors who do average annual investment in IPO within 1, 00,000 – 2, 00,000. There are 5 investors who do average annual investment in IPO within 2, 00,000 – 3, 00,000. There are 2 investors who do average annual investment in IPO above 3, 00,000.

Age 26-35:- The above table shows that there are 20 investors within the age group, who do average annual investment in IPO below 1, 00,000. There are 11 investors who do average annual investment in IPO within 1, 00,000 – 2, 00,000. There are 4 investors who do average annual investment in IPO within 2, 00,000 – 3, 00,000. There is 1 investor who do average annual investment in IPO above 3, 00,000.

Age 36-50:- The above table shows that there are 4 investors within the age group, who do average annual investment in IPO below 1, 00,000. There are 11 investors who do average annual investment in IPO within 1, 00,000 – 2, 00,000. There are 4 investors who do average annual investment in IPO within 2, 00,000 – 3, 00,000. There is 1 investor who do average annual investment in IPO above 3, 00,000.

Age 51 above:- The above table shows that there is no investor within the age group, who do average annual investment in IPO below 1, 00,000. There is 1 investor who do average annual investment in IPO within 1, 00,000 – 2, 00,000. There are 3 investors who do average annual investment in IPO within 2, 00,000 – 3, 00,000. There are 2 investors who do average annual investment in IPO above 3, 00,000.

- **Interpretation:-**

  From the above analysis we interpret that the annual investment of investors is as per their annual income and experience also because the investor who have 16-35 they are doing investment up to 2, 00,000 only and the investor who are age of above 51 they invest up to 3,00,000 also. We assume that because the other factor also effect like they has Age experience or they also most of go with insider information so they invest their money.

gender * avg annual investment Crosstabulation

			avg annual investment				
			below 1,00,000	1,00,000 - 2,00,000	2,00,000 - 3,00,000	above 3,00,000	Total
Gender	Male	Count	32	23	9	3	67
		Expected Count	31.5	23.5	8.0	4.0	67.0
		% within gender	47.8%	34.3%	13.4%	4.5%	100.0%
		% within avg annual investment	68.1%	65.7%	75.0%	50.0%	67.0%
		% of Total	32.0%	23.0%	9.0%	3.0%	67.0%
	female	Count	15	12	3	3	33
		Expected Count	15.5	11.6	4.0	2.0	33.0
		% within gender	45.5%	36.4%	9.1%	9.1%	100.0%
		% within avg annual investment	31.9%	34.3%	25.0%	50.0%	33.0%
		% of Total	15.0%	12.0%	3.0%	3.0%	33.0%
Total		Count	47	35	12	6	100
		Expected Count	47.0	35.0	12.0	6.0	100.0
		% within gender	47.0%	35.0%	12.0%	6.0%	100.0%
		% within avg annual investment	100.0%	100.0%	100.0%	100.0%	100.0%
		% of Total	47.0%	35.0%	12.0%	6.0%	100.0%

Analysis:-

Male:- The above table of gender and average annual investment by the investors shows that there are 32 male investors invest their money in IPO below 1,00,000. There are 23 males investor who do annual investment in IPO between 1, 00,000-2, 00,000. There are 9 males investors who do annual investment in IPO between 2, 00,000-3,00,000. There are 3 males investor who do annual investment in IPO above 3, 00,000.

Female:- The above table of gender and average annual investment by the investors shows that there are 15 female investors invest their money in IPO below 1,00,000. There are 12 females investor who do annual investment in IPO between 1, 00,000- 2, 00,000. There are 3 females investors who do annual investment in IPO between 2, 00,000- 3, 00,000. There are 3 females investor who do annual investment in IPO above 3, 00,000.

- **Interpretation:-**

From the above analysis we can interpret that both male & female investor invest in IPO below 1,00,000 or between 1,00,000 – 2,00,000 only. With the above analysis we can say that both genders have less trust and they thing it is risky so they have fear of loss in investing in IPO.

LITERATURE REVIEW

1. GauravKabra, Prashant Kumar, Mishra, Manoj Kumar Dash (2010). "A study on impact of market movements on Investment decision India". The study concluded that modern investor is a mature and adequately groomed person. In spite of phenomenal growth in the security market and quality Initial Public Offerings (IPOs) in the market, the individual investors prefer investments according to their risk preference. A majority of investors are found to be using some source and reference groups for taking decisions.
2. Shyan – Rong Chou, Gow- Liang Huang and Hui-Lin Hsu (2010). "Investor attitudes and behaviour towards inherent risk and potential returns in financial products" China.Empirical results found no difference by gender to investor propensity to take risk, nor in cognitive perception of such. However, higher and lower perceptions of risk were indicated by investors according to their personal investment experience. Variance analysis also found that less experience dinvest or have lower risk propensity and higher risk perception. The opposite is true for more experienced investors.
3. Dhiraj Jains &NakulDashora (2012). "A study on impact of market movements on Investment decision" India.The present study has shown that investors prefer investing in both primary and secondary market instruments .Most of the decision are rational and influenced by the various information available in market. It was also found that investors prefer the wait and watch policy for taking their decision, and are very cautious and their decisions are influenced by various psychological factors and behavioural dimensions.
4. Syed Tabassum Sultana (2010). "A study on impact of market movements' on Investment decision" India.The study concludes that the individual investor still prefers to invest in financial products which give risk free returns. This confirms that Indian investors even if they are of high income, well educated, salaried, independent are conservative investors prefer to play safe. The investment product designers can design products which can cater to the investors who are low risk tolerant and use TV as a marketing media as they seem to spend long time watching TVs.

LIMITATIONS OF THE STUDY

There are certain limitations of the study which are as under:

- Due to limited resources only 100 respondents were taken.
- The study is limited to the selected as people who only invest in IPO.
- Analysis and Interpretation may not be that strong due to small sample size.

CONCLUSION

We conclude that there are every individual is different with different perception and their risk appetite and investing decision also different and there are many other factors which influence them while investing their own money in IPO's. They chose different industry/company by seeing latest news and by what their broker advise them. We also came to know that decision of investor will mostly deviate by their age as more age they have they are more risk averse & think twice while investing their money and they also use their experience also while investing their hard earned income. While we came to know that there is not much difference in thought process of Male & Female they both go rationally while investing in IPO.

Questionnaire

1) Please Specify your age group.

a. 16 – 25 years

b. 26 – 35 years

c. 26 – 50 years

d. 51 years Above

2) Please specify your gender:

a. Male b. Female

3) Please specify your annual income. (in rupees)

a. Below 1,20,000

b. 1,20,000 – 3,00,000

c. 3,00,000 – 5,00,000

d. Above – 5,00,000

4) **How frequently do you invest in IPO's?**

a. Monthly

b. Quarterly

c. As an when IPO is offered

d. Half Yearly

e. Annually

5**) Since how many years are you investing in IPO's?**

a. Less than 1 year

b. 1 – 3 years

c. 3 – 5 years

d. Above 5 years

6)How do you select an IPO for investment? (Mark all that applies)

a. Tips from friends / relative.

b. Newspaper

c. Insider Information

d. Study of company from any published article or website

e. Street Talk / Rumours

f. Broker Advice.

7) What is the purpose of your investment in IPO's (Mark all that applies)

a. To get listing benefits

b. To get regular dividend

c. For short term appreciation in stock

d. For long term appreciation in stock

8) What is your average annual investment in IPO? (in rupees)

a. Below 1,00,000

b. 1,00,000 – 2,00,000

c. 2,00,000 – 3,00,000

d. Above – 3,00,000

9) According to you, what are the factors leading Indian Stock Market to rise? (Mark all that applies)

a. FII

b. FDI

c. Government Policies

d. Rise in National Income

e. Gold price movement

f. Oil price movements

g. Foreign exchange rates movement

10) Do you invest in industry specific IPO's? if yes please thick the appropriate industry.

a. Telecom

b. Automobiles

c. Banking

d. Pharmaceuticals

e. FMCG

f Infrastructure

g. Steel

h. Metal

i. Media

j. No any specific industry

11) In what kind of stocks do you invest?

a. Large Caps

b. Mid – Caps

c. Small Caps

d. Mixed

12) What kind of risk appetite do you prefer?

a. High Risk – High Return

b. Low risk – High Return

c. Low Risk – Low Return

d. No Risk / Safe Investements

13) Are you panic about stock price movements of IPO are invested in by you?

a. Yes

b. No

c. Indifferent

14) Tick the IPO's where you made investment in last 2 years.

a. Bharti Infratel

b. PC Jewellers

c. credit analysis & research

d. Eco friendly food processing park

e. Tara Jewel

f. Bronze infra Tech

g. Monarch Health

h. Jupiter Informedia

i. BCB Finance

j. Bhartiya Global informedia

k. Future venture India

I. Power finance corporation

m. VMS Industries

n. SRS Limited

O. Readymade steel India

References:

1. Dhiraj Jains &NakulDashora (2012), A Study on impact of market movements on investment decision.

2. E. Bennet, Dr. M. Selvam, Eva Ebenezer, V. Karpagam, S. Vanitha (2011), A study on impact of market movements on investment decision.

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4. Haroonshafi, Relationship between Risk perception and employee investment behaviour.

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6. Syed Tabasssum Sultan (2010), A Study on impact of market

Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th& Jan. 6th, 2016)

(Proceedings available for download at rku.ac.in/icre)

RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

Comparative Study Of Practices Adopted By Government To Enhance Affordability Of Cancer Drugs (With Regards To Some Developing And Developed Countries)

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ABSTRACT

The problem of the cancer gets increasing, and it increases the monetary spending at global level. As per the statistics here we understand the situation of developed and developing nations. In the research paper researcher had made an attempt to draw some light on initiatives taken by government of few developed and developing countries. With that researcher also tried to compare the policies adopted in developing and developed countries with a view to find an efficient policy adopted by government for the cancer drugs. The schemes and system followed by British country are somewhat better option for to adopt. Some strategies are also briefly discussed in this paper depending on the accessibility with reference to information on the basis of available information benefits of polices in developed and developing countries. The suggestion and recommendation are quite subjective to the knowledge and capability of researcher.

SUMMARY

Best policy to implement by the government for the cost of cancer drugs.

Keywords: Cancer Drugs, Affordability, Schemes, Government, Developed country, Developing country

INTRODUCTION

In the year 2008, the global expense of cancer because of early death and debility (excluding direct medicinal costs) and it was valued as around US$895 billion. Cancer patients who have the life-threatening problems are also facing the problem of out-of-pocket costs for cancer therapies (treatments) which can create financial problem. Health economists are worried for both by the rising costs for the treatment and because the cost of drugs seem to be rising faster than the health the compensation against the problem at least in some cases. As in improvise the treatment of the care, simultaneously cost for the care also becomes higher, which creates every treatment progress in less cost-effective than the one that preceded it.

Many of the cost value relations between the cost and quality-adjusted life-year (QALY) added, shows better value per money. So as the cost or money spent for the treatment in that what quality of life patients are getting. In different countries there are different systems for the Health Policy. In most of the developed countries they normally followed National Health policy or National Insurance policy from the government system.

Like in Europe, the most of expense of cancer treatment comes under the third party payers. And this party can be fund created by government, or sickness funds. So, the important part here in the study is like what support the patients get for the cost of the treatment and to decide for the payer for the new therapies are worth or not, for their high rate. In most of Europe countries budgets for the drugs are regulated. They follow the system of Ramsey pricing for the regulation control the price. It is commonly use approach. In reference pricing they set the price of drugs, either on the basis of an international standard, or on the basis of the same group drug common price. Through hospital formulary they control the cancer drug expense system at hospital level. Through the available information, show wide difference in the European countries for accessing to cancer drugs. In this variation it shows some points like, differences in fund allocation for the research, the system or the process for the approval of the drugs, some issues on the budget allocation and for the decision taking the economy play the role. National Institute for Health and Clinical Excellence (NICE) in the United Kingdom plays the important role for the proper and systematic examination for the decision. This could really because of the worth of the health condition, value for the condition. Also there is scarcity in the optional or alternative therapy for cancer treatment.

And if we talk about U.S., oncologists some time prefer for personalized cancer care, which helps patients to treat as per their physiology and better for their financial conditions as they said. There are two different systems in both the countries, U.S. and Canada. But their oncologists face challenges because of the rising costs of cancer drugs. They do have the similar problem of the cost effectiveness and their medication. In the support of cancer survivor and their family guide or educate the doctors to be helpful for cancer patient by providing the less costly therapy. There is also the problem in country like Malaysia, for the death rate because of lung cancer is 30 000 annually.

REVIEW OF LITERATURE

Peter B. Bach,(N Engl J Med 2009)*(1)* explain with some examples of cancer drugs price increase. They have talk about the higher prices of some drugs like paclitaxel, Nitrogen Mustard etc. They try to explain through control of utilisation, control of prices and both. They try to make give some information time difference 1965

to 2008 for the FDA drug approval. Also suggest the Medicare's strategies to keep down the cost, like through control of price, control of utilisation and both.

Milton C. Weinstein, Jonathan A. Skinner,*(2)* discussed about comparative effectiveness Vs cost effectiveness. Discussed example of the colon-cancer, where there is no connection in inclusive costs and outcomes. This paper talks about the using inappropriate chemotherapy. And sometimes use costly chemotherapy treatments. Refining management for it has tried to decrease replication system, decrease administrative excess, and improvise effects.

Michael F. Drummond, Anne R. Mason *(3)* explain about the how NICE playing role for the decision of price of the drugs.

Ramon Luengo-Fernandez, Jose Leal, Alastair Gray,*(4)* paper results show the differences between countries. They try to contribute data to public health and policy intelligence, it's essential to bring sensible cancer care systems and tries to notify impactful public research reserves portion. Also estimate the formal and informal expenditures for the cancer care. They try to find out the data from 23 countries of the European Union.

Scott R. Berry, Chaim M. Bell, Peter A. Ubel, William K. Evans, *(5)*The paper explain to get help from the oncologist to use cost effective treatment for the patients as support.

Richard Sullivan, Jeffrey Peppercorn, Karol Sikora *(6)* Author found the rate of the death patient are increase not only due to the cancer problem but also because of the higher prices of cancer drugs. Something which affect to cost are like, more-use of the drugs, fast growth of it, and all time new technology can be the responsible for the cancer with higher cost, the deficiency of appropriate clinical research. Also explain more self-protective practice is not fair for the patients of cancer. They suggested about the solution through reverse engineering for cancer costs.

Paul Farmer, Julio Frenk, Felicia M Knaul *(7)* suggest formed some force at global level like Global Task Force in developing nations, which made of best from the cancer care communities, and give some advance agenda. Such Global task force formed in 2009. Also explain to suggest about the national health insurance.

F.M.Scherer, Jayashree Watal *(8)* they discussed about the tax benefits for the drug industry to provide drugs for the providing fare price for the low income nations.

Winnie Yip, Ajay Mahal *(9)* they suggest that money alone could not , they try to suggest for better insurance policies and infrastructure for the developing countries like India, China etc. for the affordability for the healthcare and heavy financial risk.

Jakub Adamski, Brian Godman, Gabriella Ofierska Sujkowska *(10)* author find there are only some circumstances where schemes like 'risk sharing' can be appropriate. There are some factors that should be measured by payers in prior of implementation.

Donald J. Birkett, Andrew S. Mitchell, Peter McManus *(11)* explains about the government of Australia, offers its citizens to select the list of pharmaceuticals for subsidization and include in a list when only drug will be cost and clinically effectiveness. Author tries to suggest for providing the transparent system and consistency in decision making.

K Sikora *(12)* explain and show the worry about the higher prices of the cancer drugs and think about good policy to help patients. They talk about the novel screening technology, which helps a lot to detect problem.

Sanjoy Kumar Pal, Balraj Mittal *(13)*Author said the benefits of the specialized cancer centres. They talk about the less possibility of developing super specialized centres because of financial constraints in developing nations and resources availability, improper planning and management.

James Raftery *(14)*some other countries try to follow the system just like NICE are Germany, France. In their structure NICE had published 86 guidance on the use of health technologies and 39 guidelines on the treatment of diseases.

Steve Williamson *(15)* talk about some schemes like PAS (patient access schemes) or risk-share schemes, some price relaxations which helps to National Health Service (NHS) in UK.

Ruth R. Faden, Kalipso Chalkidou *(16)* discussed about the both, British as well as American health care systems. They said as the impression for all the resident people can have accessibility and affordability in America is not true. Whereas in British country its far better coz of British NHS.

Robert Steinbrook *(17)* they said there are different financial level in the same country some can afford costly drugs and others cannot the countries like, China and India. So for them the option suggested is Compulsory licensing, means to allow the use of patent drug with the patent holder's permission can be helpful for the health care contexts.

Kalipso Chalkidou, Patricio Marquez, Preet K Dhillon *(18)* Said there must be strong system for the concern as opposed to technology based solution to help for the affordable the drug.

Paul E Goss, Kathrin Strasser-Weippl, *(19)* Try to suggest about for preference of traditional medicine to treatment for the cancer.

Xuedan You, Yasuki Kobayashi *(20)* in this author talk about the New Cooperative Medical Scheme (NCMS) which is a heavily subsidized voluntary health insurance program established in 2003 and helps to reduce the catastrophic cost.

Jun Gao, Shenglan Tang, Rachel Tolhurst,Keqing Rao *(21)* the paper discuss the economic development in India and the health system is at a junction today.

Michael Kent Ranson, *(22)*The findings in the paper are about to Insurance schemes for community-based in India and elsewhere.

Winnie Chi-Man Yip, William C Hsiao *(23)* Author discussed and suggest about Incentive structure for the provider reform it in China and its public hospitals.

STATEMENT OF THE PROBLEM

This has been the question that, can middle section of the society afford the expensive drug medicine for cancer. It has been concern for various countries government to make the cancer drug reasonable (affordable) to all needy patients. Affordability of the drugs is always concern of the government. Various countries

governments try some initiatives to reduce the price of cancer drugs or through other way make affordable by various polices.
Researcher narrowed the focus to developing and a developed country to find overall polices and schemes adopted by government some developing and developed countries.

NEED OF THE STUDY

Globally, at every year diagnose the new 10 million cancer patients and probabilities to increase in the number are like 20 million till the year 2020. Now it becomes the most critical disease to cure at a relative cost. There is billions of money spent annually for cancer research in R&D department by pharma industries. Not only drug industry but also by the government and some charity funds spend good amount of money. But till it do not resolve the problem. The WHO cancer programme are working for the identify significances in cancer detection, prevention and treatment economically as well as epidemiological scenery and working for the priorities as per the cancer disease.

China and India have newly devoted to inserting funds for the public into health care. Both the nations are now determining that better way the added funds to help their populations. There is the Ramsey pricing option through which best coverage of R&D can be done; it is multinational drug pricing strategy. In this prices strategy the price must much as lower as possible in nations with low ability to pay and/or high price elasticities of demand than compare to wealthy nations.

Prices could be decrease through some options, like increase or improvise purchasing competence, removing some taxes and regulate some mark-ups. More than Three forth (¾) of the world's population or the 85% are of the developing nations - but in these only one about one third part of the countries have latest technology like radiotherapy facilities for treatment In some developing country they do not have expert team of enough numbers of specialist doctors or machines. Through some hospital networks and service helps to improve cancer care in developing country.

Affordability of cancer drugs has been the burning point since few years in each country. With the growing needs of cancer drugs, the concern for government of every country to make cancer drugs affordable also started growing. Though every country's government started fighting against rising prices of cancer drugs with the help of various policies, still more efforts are required from the side of government to achieve the desired results. The main aim of every country's government is to provide the cancer drugs at affordable prices to all the needy persons, which requires more efforts and policies from their side. Some research paper attempted to find the policies and practices adopted in their own country. But with this research paper researcher is going to consider few developed and developing countries , the research sample here is not just limited to one country but it will enhance the study towards few selected developing and developed countries with a view to compare the policies by government of various countries to make cancer drugs affordable. After comparison a subjective conclusion also had been given with regards to efficient policy adopted among the selected sample to make cancer drugs affordable.

SCOPE OF THE STUDY

Present study is restricted till the year 2002-2014 from available data.
This paper can be helpful to Government of India, health sector, policy makers, strategy decider and for further research work as well. After conducting a research researcher will come to know about real situation in health

sector as well as find good alternatives of polices for the cancer treatment and the support they have as well as require.

LIMITATION OF THE STUDY

The limitations of the study are as follows:

- The study includes the data to year till 2002-2008 and some of till 2014 only.
- The study is related to some developed and developing countries only.
- This study is based on the secondary source of data from the published reports, some good papers, articles and some from journals. The dependability of the information and result are mainly depending on the data from available source.
- This study is restricted to only schemes announced by government and some declared regulatory systems. Hence it becomes the limitation of the study.
- There are different tactics of the qualitative analysis. So it's less possible the consistency among experts.

MATERIALS AND METHODS

RESEARCH OBJECTIVES

- To know the measures adopted by government of developing and developed countries to make cancer drug affordable
- To compare polices adopted in developing and developed countries for making cancer drug affordable.
- To find the best option adopted by government to make cancer drugs affordable.

RESEARCH METHODOLOGY

For above mentioned objectives researcher will take the help of secondary data, which contains research papers, government websites and related articles. Researcher will analyze the data and try to achieve the said objectives.

The sample:
For considering developing countries researcher selected India and China as a Representative of developing country category. As the same way US and Canada, Europe had been considered as a representative of developed countries category. Try to cover countries which can help to make strategies for policy framing better for the cancer patients (to survive) and their families (financial conditions).

RESULTS AND DISCUSSION

DATA COLLECTION & ANALYSIS

Data collected in this paper are from the available information. Try to collect the data as per the objectives of the research paper. And also analyse it in a way to satisfy the objectives of the paper. First in this part of the data collection mention some information about the cancer rate in India as well as global level. Where mention from the authentic report information like world cancer report gives some numbers and rate of cancer disease spread in India and globally. They also predicted the future perspective the rate will increase as of some analysis study comparative current perspective. In the paper information include at the time duration before 30 years what was the scenario and why all the country has to worry about the said disease.

The report issued by the International Agency for Research on Cancer (IARC) name as World Cancer Report on page 351 show the cancer rates are set to increase too much higher level globally (Stewart and Kleiues 2003). As in the paper it discuss about the rate risen tentatively in future.Since last more than a decade increase in CIR of all cancer in both men and women in India. There is increase in the rate of the cancer problem in India from 12% to 57%. The total number of new cases, which stood at 5.3 lakhs and care cost have risen to more than 8.3 lakhs. Tobacco consumption support to risen the number of the cancer patients. Two decades ago the numbers of cancer case were 1.94 lakhs, whereas in 2003, there were 3.85 lakhs. In which now at the second position there is a lung cancer which was prior at fifth position.

As per all information some farer schemes comes forward for the better option and afford the cancer drugs for both developing as well as developed countries. As some number shows the status of the cancer, and analyses some suggestions given which may be good to implement by system.
Here the first objective is to find out the adopted policies by the government in different countries like developed as well as developing. Why this objective sets as a first because we find out the status of the schemes helps better option for the worldwide, and to some middle and low income countries. They can also adopt such polices for the better health of their nation. So here if we find some strategies or some policies than, we talk about the off patent drugs. Off patent drugs means expiry of the patented drugs, it can also manufacture by the other than patent holder. Normally the patent register drug can be manufacture by the patent holder so to recover the expenditures of the R&D to recover it they can do in these 20 years. But the issue is like they charge too much for those drugs which can not possible to afford by all. Some countries follow the off patent drugs. But the issue in this option is like in cancer drugs the advance drugs are more helpful for the treatment. Some low income countries if they cannot afford advanced drug than it can be suitable. Some other strategy which got was global as well as regional procurement and financing system. Like other new way of the system that the country provide some tax benefits to the drug manufacture company which provide new drugs to the world. So they get motivate and have some financial expenditure relaxation. We can say they will perform well in their R&D to discover the new drug for the cancer they have to spend more and more money and time also. So to support it make some regulatory system transparent and try to work on it as well in time. But with this the quality must be at good level for the work performance. The strategies found are Ramsey pricing and regulatory mark ups. In Ramsey pricing system, prices are much lower in nations with low ability to pay and higher price bounciness of demand than in the wealthy nations. Than another strategy found is National Health Insurance, in which Insurance coverage responsibility by nation itself. So they can help the people to make them free from the higher cost burden.

Here almost the found strategies and policies adopted by the countries give us the option to adopt best among them. So already in previous para discuss the strategy option available and their explanations. So it satisfies the second objective to compare all above. Through the above explanations author try to find the comparison among those and suggest best or good from it.

So these are the options in this paper like some regulatory related steps to use of off-patent drugs, and the another option like global mechanisms system providing financial support. Something like Ramsey pricing, tax benefits to the drug industry for the R&D of new drugs, regulating mark ups etc. Several middle-income countries for people who living in poverty for them include the treatment in the nationalise scheme of insurance system. These strategies can helps to minimize the costs. Also helps in service access by health system which helps to make the health system strong against the cancer disease. The compulsory licencing is the proper remuneration paid option under the TRIPS.

So below findings are some measures adopted by different developed and some developing countries as of the first objective.
So as per the discussion some findings are like,

- To use off patent drugs
- Follow National Health Insurance
- Make regulatory system transparent
- Decision taken by the government or regulatory body should be cost and quality effective
- With proper data evidence permit the drug or company as per price control
- Tax benefits for the new drugs of R&D to drug industry
- Regulating mark ups
- Public education on health must be stronger.

Europe country has recently tried to apply the risk sharing schemes which helps to provide proper healthcare through taking part by hospitals and pharma companies. But there are limited numbers of situations where it's compatible.

So by second objectives some findings are like to compare all the above data and give some idea which helps to decide the next objective is best policy or the system among all. So, out of all above findings if we compare the better option than its compulsory licencing, follow National Health Insurance or Re- engineering though some of all have its own pros and cons.

Now all the alternatives have their own advantages and disadvantages. So as per the countries requirement and suitability follow the alternative which helps people to access and afford it for the treatment. As we all know the out of the pocket expenses are the burden for the family of the cancer survivor. So try to follow the government system or regulatory system which support to the residents of the country. NICE is the system which is really good in Europe.

As all knows about the new founded drugs are always charge higher price for it with little variation in global prices. That's why the UK they don't except the new drug immediately because of its higher prices. They except after some formalities pass through the National Institute for Clinical Excellence (NICE). One more finding that is about the system followed by British and America. Some important elements structured well in British system compare to America. Than even both the system have some financial, ethical and organizational problems to face the challenges against the higher expense.

Try to make some mark ups or control on the prices of the cancer drugs. There must be a strong body which can work for the price control for cancer drugs which directly helps to country people.
Now after all this information as per the objective in the paper tries to analyze and show one model is impact analysis.

8.1 Impact Analysis

First objective derives the different schemes are followed in different developed and developing countries. It is already mentioned above. Now the thing is how it can impact on different segments like, patients, doctors, Drug industries, specialist hospitals, supply chain like distributors and lastly to the government and policy makers.

Health is the prior most of the any nation in the world. So for the government they have to set some budget for the health system in which they give the benefit for the critical or some major incurable disease. So the cancer is problem of the all countries in the world and other than that accessibility as well as its cost. Treatment of cancer is too costly because of the higher cost of drugs. The pharma companies charge higher prices for the cancer drugs because of their high expense in research and development (R&D) for the drug.

For the comparison of the schemes or strategies is there in developed and developing countries the one alternative off patented drugs. For patients its benefits in the expense of the treatment, but if we see the benefits as compare to advance drugs than its less. Now, for the doctors in developed countries it can less affect but, if in developing countries doctors mostly prescribe branded and advance drugs to treat the patients. For multi-specialist hospitals they try to use mostly the advance and branded drugs. But in public hospitals normally off patented and generic drugs are preferred. For the supply chain it directly not impacted but indirectly affect, because they keep supply the drugs as per the demand of the prescription.

The other alternative is national health insurance, advisable for the countries which always helpful for the patient. They have advance treatment with less worry of cost. In which doctors also not face the problem to treat the cancer patient. But for the government has to keep some good amount for the insurance funds of such expensive treatment. Here companies don't have to bother about the higher charges they are charging for the drugs, but government may keep some regulation for the price also to provide insurance schemes benefits for the patients. But in this regulation and rules must be very strong so it can not harm the government system budget. Here again the distribution system cannot much affected, but private hospitals have to make system as per the government's policies.

Now the other alternative is make regulatory system transparent and strong so it directly helps to the drug industry. Here it will benefit to patients indirectly. If the system is transparent so it will helps to drug manufacturing company. They do have idea about the time duration taking approval for the drugs, as the time will be less to approval for the qualitative drugs as per the regulatory system requirement, it automatically reduce the cost. Pharma companies normally talks about the expense for the R&D and as the regulatory are not transparent so taking more time for the drug approval and so they are charging more for the new drugs.
Now, in this the regulatory system also has to take care about the quality and cost before the approval for the market. So drugs must pass the entire test required by the health safety and other.So giving fast approval cannot compromise in the quality and effectiveness of the drugs. So this alternative directly, indirectly impact to the patients and companies. But here hospitals and dealers or supply chain system least affected.

With this also strategy makers go for the price regulation with some tax benefits for new drug inventor. In this option it can motivate to the inventor for giving some tax benefits for those new invented effective drugs, so they may be ready for some price regulations. If government keeps only price regulation than companies can be delay the research of the new drugs. So if government keeps benefits of tax than kind of financial support with the appreciation.

Lastly the option for the education or increase the awareness of the disease, reasons for the disease, regular checkups to know the health status, diet regulation awareness and the smoking can be the most impactful things

for the disease. Keep educate the people for insurance systems so they will not fall into trouble. This alternative can be only initiates by the resident of the countries or population themselves. Government can arrange programs for the public for the awareness of the disease.

CONCLUSION

Strategies are required to make narrow down the difference in between developed and developing countries in cancer patient's treatment and accessibility for the drugs as well as affordability. In nations where the constraint of the resource as well as less specialised service so some steps to take could be like prevent and treat cancer through the positioning of primary and secondary caregivers, and some other options like using off-patent drugs, and through the global mechanisms for financing (provide benefits for the insurance) and procurement. Like where the policy use could be like PBS (Patient benefit scheme) which really helpful for the patients.

Than other options like NICE (National Institute for Clinical Excellence), the system they are following, again good to implement for other countries and regulatory body.

Then, smoking restricted or nicotine/ tobacco consumption restricted food or diets supplementary are some steps to help for the next generation. Early detections and diagnose are the most important one. For which make people more aware about it.

Try to find out some drugs which are not necessary and as possible as to decrease the use of such drugs for ex. ESA drugs decrease the sale compare to prior. The compulsory licensing is the best and better than other alternatives of the strategy or some policies.

As per researcher analysis above are the good options to make the cancer drug available at a fair or affordable price. Among all above suggestions better one is compulsory licensing for the required nation. Also there are PBS kinds of schemes beneficial for the cancer patients. As like Germany and France other countries can be follow the systems are like as NICE.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

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Building APT Model for Sensex Considering Indian Macro-Economic Factors

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ABSTRACT

In this new era, different pricing model are used to study the specific trend stock market depicts. These models helps to build a formula to understand the stationary of data and the level of its impact on other factors, which can be helpful tool for the investors dealing in stock market. Among various model available for pricing, this study uses APT Model for SENSEX considering different Indian macro-economic factors. The study will analyze the impact of various Indian macro factors on SENSEX and the reliability of APT Model by considering data of last 24 years by using various statistical tools like Granger Causality Test, Multiple Regression with help of some software to measure the same and to even know the scope of other factors to be added in the study.

SUMMARY

Building APT Model for Sensex Considering Indian Macro-Economic Factors

Keywords: APT Model, SENSEX, Inflation, GDP, Interest Rate

INTRODUCTION

In mid 1970s the Arbitrage Pricing Theory (APT) was developed by Stephen Ross. It's merely reasonably intuitive in comparison to CAPM (Capital Asset Pricing Model) which allows multi risk factors and requires minimum assumptions. APT only assumes that capital market is perfectly competitive and also investors do always prefer more wealth to less wealth into certainty.

APT also helps investors to identify an assets like share price of some stock which may be incorrectly priced. Herein the investor continuously thrives for bringing down the price of security back to actual value.

THE APT FORMULA as developed by Ross is mentioned below:
Expected Return = rf + b1 x (factor 1) + b2 x (factor 2)... + bn x (factor n)
Herein,

- rf = the risk free interest rate, which is the interest rate the investor would expect to receive from a risk-free investment.
- b = the sensitivity of the stock or security to each factor
- factor = the risk premium associated with each factor

The APT model describes about the two factors on which the risk premium is dependent:
1. The association with each and every factor stated above.
2. The sensitivity of stock toward each factor which is similar to beta concept.

Risk Premium = r - rf = b(1) x (r factor(1) - rf) + b(2) x (r factor(2) - rf)... + b(n) x (r factor(n) - rf)

By using the above formula, if the calculated risk premium on a stock are higher than the expected one, then in such case investor may sell the stock and also if in case calculated risk premium on a stock are lower than the expected one then investors would surely buy stock until both sides are in equilibrium. Henceforth, arbitrage is defined to be the way investor strives to get this formula back to equal.

USING APT

As with CAPM, herein also the factor specific betas are determined via a linear regression of historical security returns on the factor in question.

Chen, Roll and Ross (1986) mentioned the below macro-economic factors as significant in explaining about security returns:

- Changes in inflation.
- Changes in GNP.
- Un expected shifts in the yield curve.

Even spot or future market prices and indices can be used in place of these macro-economic factors. More direct "indices" that can be used are:

- Currency exchange rates
- Interest rates based on short terms investments
- Precious metal and oil prices
- Diversified stock index

MACRO-ECONOMIC FACTORS

1. INFLATION: It is termed to be a situation in economy wherein there is too much money chasing too few goods. Hence here the scenario can be stated to be that goods command a higher price than its actual price as people are willing to pay that increased value to get that good.

2. GDP: In a year, the total amount of finished goods produced within country's boundary regardless of ownership is termed to be Gross Domestic Product. It implies to only the final goods or services which are consumed by the end user and are not used as input in any other goods.

3. INTEREST RATE: The percentage of amount charged on principal amount by the lender of money to the borrower of money for the use of asset is called Interest Rate and they are mainly calculated on annual basis known as Annual Percentage rate (APR). The assets which can be borrowed may include goods, cash and fixed assets (building or machinery).

4. SENSEX: The "Sensitive index" is treated to be an indication of share price of major companies listed on Bombay Stock Exchange which are shortlisted from different sectors by a committee on basis on some defined criteria, which give us an idea of economy whether it's on boom or in depression and also provide an overview of the capital market.

MATERIALS AND METHODS

1. OBJECTIVES OF THE STUDY

A research is conducted to build APT Model for SENSEX considering Indian Macro Economic Factors. The main objectives of the study are:

- To know the effect of macro-economic factors (GDP, Inflation and Interest rate) on Indian Stock Market.

- To know which factor affect the most on Indian Stock Market.

2. SCOPE OF THE STUDY

This study on building APT Model for SENSEX considering Indian macro-economic factors will analyze the impact of factors on SENSEX and the domination factor among them by considering 24 years data only which can even be useful for investors to invest in share market while taking into account this study for their analyses of market.

3. HYPOTHESIS OF THE STUDY

This study is based on the following hypothesis

- Impact of various macro-economic factors on the SENSEX.

- The dominating macro-economic factor affecting SENSEX.

4. SAMPLE SIZE

The sample size taken into consideration is 24 years (1991-2014) for the macro economic factors and for BSE SENSEX.

5. DATA COLLECTION

There are mainly two sources of data; primary and secondary. This study is based on secondary data has been collected through different websites like RBI, Index Mundi ,Trading Economics, from journals, newspapers, business magazines, and other web sites.

6. TOOLS AND TECHNIQUES

The technique used in this study for analysis of data collected is through Stationarity Test of Augmented Dickey Fuller, Granger Causality Test and Multiple Regression.

RESULTS AND DISCUSSION

In this study on APT different tests and models are used which are explained simultaneously:

(1) The first step is in this research, like any other research, is to organize the data required. This is done by collecting data from relevant web sites and databases.
(2) Secondly, is applying the APT model which is on the assumption that SENSEX is a dependent variable and macro-economic factors are independent which is done through multiple regressions.
(3) For checking the reliability of the model different test are conducted like unit root test, Granger Casuality Test and Cointergration.

Multiple regression: For checking whether there is any correlation between SENSEX and other macro-economic factors, Multiple Regression test is conducted with the help of Gretl Software through Ordinary Least Square model (OLS).The multiple regressions is nothing but the APT Model. The output after running the OLS model in Gretl Model: OLS, using observations 1991-2014 (T = 24)

Here Dependent variable is SENSEX, while the macro-economic factors GDP, Inflation and Interest are taken to be independent variable.

	coefficient	p-value
Const	1923.55	0.7999
GDP	1460.87	0.0146 **
INFLATION	739.879	0.0513 *
INTEREST	-1078.58	0.1227

R-squared 0.499131
The regression can be build i.e.
SENSEX=1923.55(const) + 1460(GDP) + 739.879(Inflation) – 1078.58(Interest)

Interpretation for R-squared means that 0.5(0.499131) i.e 50% of the macroeconomics affect the SENSEX while the rest 0.5 i.e 50% is caused due to the error terms which means other factors which have the effect on SENSEX and which are not taken into consideration.
Further, for testing whether this model is reliable or not different test have been conducted which are explained as follows.

(A) UNIT ROOT TEST

For converting the non-stationery time series into stationery time series unit root test is being conducted for each factor and SENSEX. Stationary explains rationale behind checking for stationary of variables and Unit root tests used to detect its presence.

Unit root: Different financial and economic time series present trending behaviour or non-stationary in the mean. Few examples can be said to be exchange rates, asset prices and macroeconomic aggregates. The most important econometric task is of knowing the appropriate form of trend which the data consists of. The unit root test helps in understanding whether a time series variable is non-stationary using an autoregressive model and the well-known test is the augmented Dickey–Fuller test.

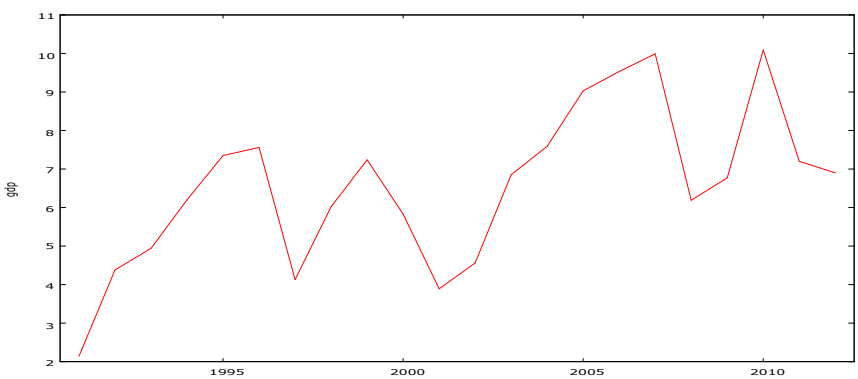
If p-value is greater than 0.05 than we will accept the null hypothesis which means that series contain trend so we have to remove the trend. Detailed explanation is given at below with graph.
For unit root test first of all we have to define hypothesis that is mentioned at below.

Ho (Null hypothesis): series of GDP contains trend
H1 (Alternative hypothesis): series doesn't contain trend

For unit root test, Gretl programme is being used.

- FACTORS

1. Gross Domestic Product



(Graph GDP 1.1)

Here, from time series plot we can say easily that data is not stationary, also the statistics value present the same thing.

Interpretation: Here the p value for the factor GDP comes out be 0.07079, which is more than 0.05, so we accept the null hypothesis that the series contains trend. so to remove the unit root we have to take difference for the series, which means p1-p0. After taking out the difference will apply the statistic again and then analyse the trend.

In Gretl program the difference command is directly available so we do not have to take difference separately.



(Graph GDP 1.2)

After taking the first difference we got p-value 5.871e-005, which is closer to 0 which is less than 0.05 so we reject the null hypothesis so we can say that now data is stationary.

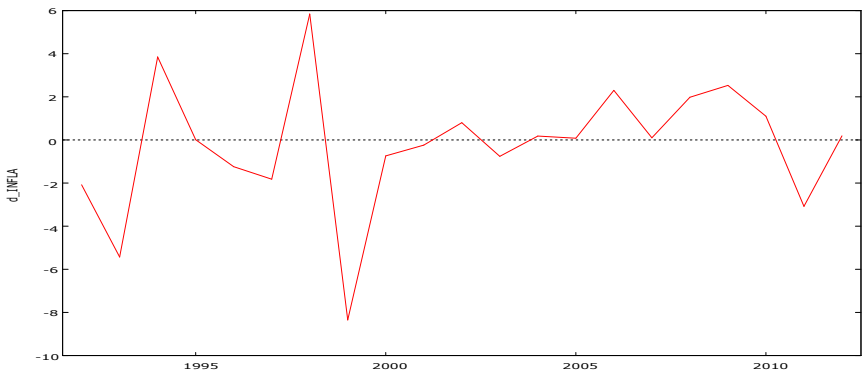
2. Inflation



(Graph Inflation 2.1)

Here, from time series plot we can say easily that data is not stationary, also the statistics value present the same thing.

Interpretation: Here the p value for the factor INFLATION come to be 0.2067, which is more than 0.05, so we accept the null hypothesis so to remove the unit root we have to take difference for the series, which means p1-p0



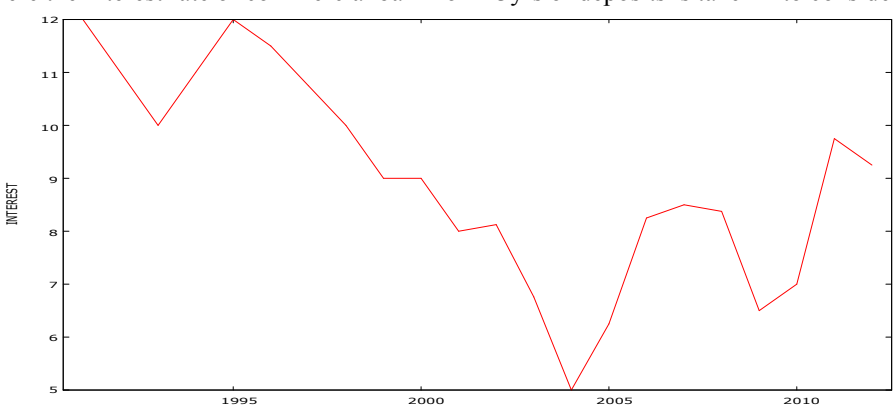
.

(Graph Inflation 2.2)

Interpretation: After taking the first difference we got p-value 0.0001368, which is less than 0.05, so we reject the null hypothesis and accept the alternative hypothesis. Hence, so we can say that now data is stationary.

3. Interest

Here the interest rate of commercial bank for 1-3yrs of deposits is taken into consideration.



(Graph Interest rate 3.1)

Here, from time series plot we can say easily that data is not stationary, also the statistics value present the same thing.

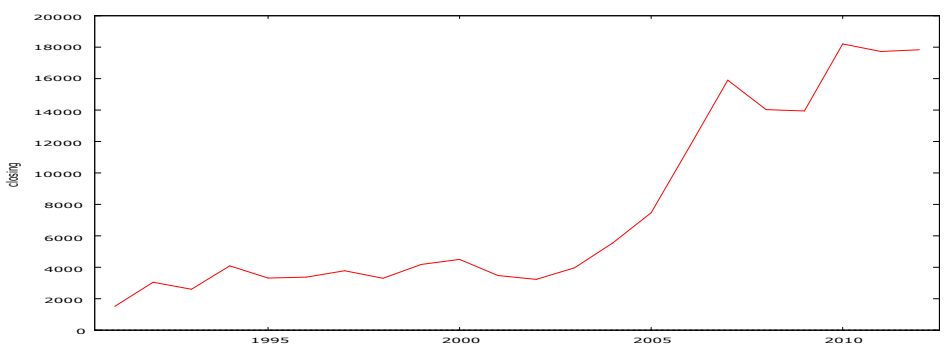
Interpretation: Here the p-value for the factor GDP comes to be 0.3352, which is more than 0.05, so we accept the null hypothesis so to remove the unit root we have to take difference for the series, which means p1-p0.



(Graph Interest rate 3.2)

After taking the first difference we got p-value 7.662e-005, which is closer to 0, so we reject the null hypothesis and accept the alternative hypothesis. Hence, we can say that now data is stationary

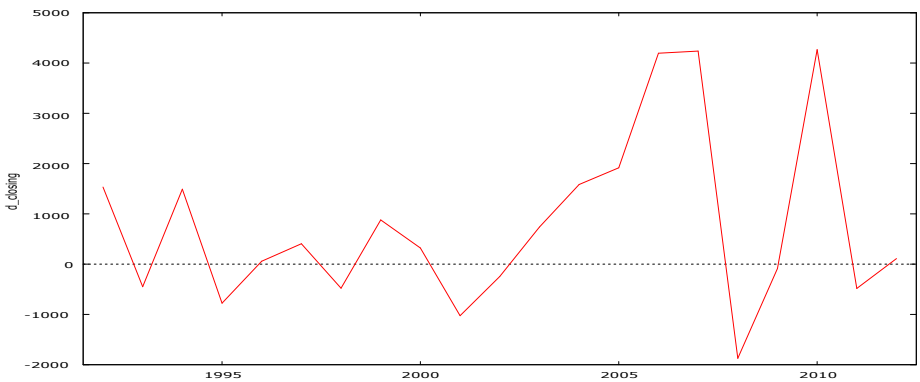
4. SENSEX



(Graph SENSEX closing 4.1)

Here, from time series plot we can say easily that data is not stationary, also the statistics value present the same thing.

Interpretation: Here the p value for the factor GDP comes to be 0.9672, which is more than 0.05, so we accept the null hypothesis so to remove the unit root we have to take difference for the series, which means p1-p0.



(Graph SENSEX closing 4.2)

After taking the first difference we got p-value 0.01199, which is less than 0.05 so we reject the null hypothesis and accept the alternative hypothesis. Hence now we can say that now data is stationary.

(B) GRANGER CASUALITY TEST

This test is conducted to know the dependent and independent variable. Engle-Granger was developed in 1969 as a causality model which is used to examine the causality between the Indian stock market and macro-economic variables. It's a statistical hypothesis test for testing whether a time series is helpful in forecasting another.

For this test, Shazam programme has been used. For causality test, first of all it is necessary to decide on selection of lag. So to decide upon lag VAR lag selection has been used. Here for this test, hypothesis is applicable which is mentioned below. If p-value is less than 0.05 than null hypothesis will be rejected.

	Hypothesis	P value	Decision
GDP – SENSEX	Ho: sensex does not Granger-cause GDP	0.0626	Accept Ho
	H1: sensex does Granger-cause GDP		
	Ho: GDP does not Granger-cause sensex	0.3808	Accept Ho
	H1: GDP does Granger-cause sensex		
INFLATION-SENSEX			
	Ho: sensex does not Granger-cause Inflation	0.3791	Accept Ho
	H1: sensex does Granger-cause Inflation		
	Ho: Inflation does not Granger-cause sensex	0.7483	Accept Ho
	H1: Inflation does Granger-cause sensex		
INTEREST-SENSEX			
	Ho: sensex does not Granger-cause Interest	0.1746	Accept Ho
	H1: sensex does Granger-cause Interest		
	Ho: Interest does not Granger-cause sensex	0.0984	Accept Ho
	H1: Interest does Granger-cause sensex		

In above statistics, in all case null hypothesis is accepted as p value is greater than 0.05. That means BSE SENSEX does not granger cause various macroeconomic factors which means none of the factors have an effect on the other factor i.e dependent- independent variable from this test are not known.

Hence the regression of 0.5 fails in this test.

(C) COINTEGRATION:

For checking whether there is any cointegration between two variables. Herein if two or more series are individually integrated but there exists lower order of integration in some of linear combination, then series are termed to be cointegrated.

Here the null hypothesis (Ho) is that the residuals are nonstationary, which explains that the series are not cointegrated and rejection of this leads to the conclusion that the series are cointegrated.

The cointegration is to be considered when selecting a technique to test hypothesis which is related to relationship between two variables having unit roots.

The two methods that allow examination of relationships through cointegration are (1) the Engle and Granger's (1987) error correction model that can be used in the multivariate context and (2) Johansen's (1991) vector error correction model (VECM) but in this study Engle and Granger's method is being used.

	Hypothesis	**P value**	**Decision**
GDP and SENSEX			
	testing for a unit root in GDP	0.07079	unit root hypothesis is not rejected for closing of GDP.
	testing for a unit root in closing	0.9672	unit root hypothesis is not rejected for closing of SENSEX.
	testing for a unit root in uhat	0.05005	GDP and closing SENSEX are not Cointegrated
SENSEX-GDP			
	Testing value for Sensex and GDP as same as mentioned above		
	testing for a unit root in uhat	0.9282	closing SENSEX and GDP are not cointegrated

Inflation-SENSEX			
	testing for a unit root in Inflation	0.229	unit root hypothesis is not rejected for closing of inflation
	testing for a unit root in closing	0.9881	unit root hypothesis is not rejected for closing of SENSEX.
	testing for a unit root in uhat	0.5828	closing SENSEX and Inflation are not cointegrated
SENSEX-Inflation			
	Testing value for sensex and inflation are same as mentioned above		
	testing for a unit root in uhat	0.9748	closing SENSEX and Inflation are not cointegrated
Interest -SENSEX			
	testing for a unit root in INTEREST	0.5176	unit root hypothesis is not rejected for interest.
	testing for a unit root in closing	0.9881	unit root hypothesis is not rejected for closing of SENSEX.
	testing for a unit root in uhat	0.7715	closing SENSEX and Inflation are not cointegrated
Closing – interest			
	Testing value for SENSEX and interest is same as mentioned above		
	testing for a unit root in uhat	0.9287	interest and closing SENSEX are not Cointegrated

From the above table it can be said that none of the factor and SENSEX are cointegrated which means that other factor i.e the error term may be having an effect on the relationship between factors and SENSEX.

CONCLUSION

The objective of the study is to know the impact of various macro-economic factors on the SENSEX. From the APT model regression it can be said that macroeconomics factors have 50% effect on the SENSEX while 50% is due to the error term. Further for checking the reliability of the model different test are conducted i.e. from unit test, Granger Causality Test and Co integration. From different test conducted for reliability check of model failed which may be due to limited factors taken into consideration and due to the limited time frame. Hence the error term can be thought upon by considering different factors like poverty; exchange rate, population etc. can be considered. We can also include NSE (National Stock Exchange) with SENSEX.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

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RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

A study to identify role of soft skills in improving service quality and enhancing consumer satisfaction in service organization – with special reference to stock broking industry

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ABSTRACT

For consumers in service organization specially in stock broking companies, level of satisfaction depends upon the quality of service and quality of service, in turn, is determined by many factors, one of them being the skills possessed by the people providing the service. Skill set is determined by primary and secondary sources. The skills that the service providers should possess are categorized as hard skills and soft skills. Hard skills are technical skills whereas the survey revealed 25 factors that comprise soft skills. Effort has been made to extract the important factors that lead to improve service quality. Primary data were collected from 50 respondents dealing in stock market through a structured questionnaire and factor analysis was used to extract the factors which affect service quality. On the basis of the survey and study a consumer satisfaction model for service industry has been developed

SUMMARY

The study focuses on importance of soft skills in service organization to improve service quality and customer satisfaction.

Keywords: soft skills, hard skills, service quality, consumer satisfaction

INTRODUCTION

India has been considered as service economy for last many years as 57.9% part of GDP of the nation comes from service industry. In service industry, could it be banking, finance, stock/commodity broking, mutual funds or insurance a tough competition has been observed. Each company from any of these sectors is putting hard efforts to maintain and enhance its market share. In most of the areas all companies are providing core and supportive services at par. Henceforth competition lies at augmented level only. The success of the efforts of the company largely depends upon the people who directly deal with the clients. Hence the skills that the people, associated with the organization, possess are of immense importance to fetch the business. However it largely depends on the nature of the business as what skills are important for what kind of business. Though product/service knowledge is must to serve the clients well, many other skills are equally important to improve service quality that would in turn enhance satisfaction level of clients and make them lifelong customers of the company.

Skill Set:

The skills can broadly categorize as hard skills and soft skills.

Hard skills are related with the IQ of a person. These skills are tangible and quantifiable. The examples of hard skills are math, accounting, physics, programming, reading, writing, typing etc.

Soft skills are more related to the EQ of a person. These skills are less tangible and not quantifiable. The examples of soft skills are self-management skills and communication skills.

Soft Skill Variables:

The soft skill variables identified are as follows:

Acceptance, Positive Attitude, Emotional intelligent, Creative thinking, Decision making, Problem solving, Confidence, Stress management, Anger management, Time management, Empathy, Work ethics, Dedication, Enthusiasm, Patience, Effective communication, Courtesy, Clarity of thoughts, Presentation skill, Socializing skill, Body language, Corporate etiquette, Persuasion, responsiveness, reliability.

Review of Literature:

1. Parasuraman, Zeithaml and Berry (1988) have developed A SERVQUAL scale. SERVQUAL intended to develop a quality scale that would be applicable across different services. The authors suggested that the service quality is an antecedent of customer satisfaction. The five dimensions were named and described as of SERVQUAL given by the authors are tangibles, responsiveness, assurance, and empathy.

2. Prasad Kaipa et al. (2002) in his study on the role of soft skills development in the entrepreneurial success. They categorized important soft skills as leadership, decision-making, conflict resolution, negotiation, communication, creativity and presentation skills, and observed that soft skills are essential for entrepreneurial success

3. Zeithaml, Bitner and Gremler, (2009) found that perceptions are considered in relation to expectations. The authors argued that because perceptions may shift over time and therefore it is necessary for companies to continually assess customer perceptions.

Research Objective

The objective of the study is to identify the soft skills that the people in the service organization should possess while dealing with clients. The study is intended to explore the skill set that would improve service quality and consumer satisfaction.

In this study we try to identify which skills are of great value for clients dealing with stock broking houses that would give them good service experience.

Research Methodology and Design

The methodology and design used for the study is as follows:

Sampling and data collection: The data was collected from the sample of 50 respondents dealing in stock market using convenient sampling.

Research Instrument: A structured questionnaire was used to collect the data. Respondents were interviewed personally to get the data.

Statistical tools and techniques: The collected data was analyzed using SPSS. Moreover, factor analysis was performed to extract the factors – skills – influencing the service quality and customer satisfaction.

Analysis and Interpretation:

Demographic Profile of the respondents:

Table 1. Age group analysis

age group	Frequency	Percent	Valid Percent
26-30	8	16.0	16.0
31-35	6	12.0	12.0
36-40	8	16.0	16.0
41-45	7	14.0	14.0
46-50	9	18.0	18.0
51-55	7	14.0	14.0
56-60	2	4.0	4.0
61-65	3	6.0	6.0
Total	50	100.0	100.0

From the above age group analysis, it has been found that maximum (approx. 90%) number of people dealing in stock market falls under the age group ranging from26-55 and 10% of people are above 55.

Table 2. Gender analysis

		Frequency	Percent	Valid Percent	Cumulative Percent
Valid	Male	37	74.0	74.0	74.0
	Female	13	26.0	26.0	100.0
	Total	50	100.0	100.0	

Gender analysis reveals that 74% of clients in stock market are male. And only 26% female are availing the servicesof stock broking company.

Table 3. Qualification of respondents

		Frequency	Percent	Valid Percent	Cumulative Percent
Valid	Hsc	2	4.0	4.0	4.0
	bachlor degree	35	70.0	70.0	74.0
	master degree	13	26.0	26.0	100.0
	Total	50	100.0	100.0	

Considering qualifications of, 70% of clients are holding bachelor degree, remaining 13% are having master degree and only 2% are HSC.

Table 4. Since how long availing services from stock brokingfirm

		Frequency	Percent	Valid Percent	Cumulative Percent
Valid	1.00	1	2.0	2.0	2.0
	2.00	2	4.0	4.0	6.0
	3.00	1	2.0	2.0	8.0
	4.00	2	4.0	4.0	12.0

5.00	8	16.0	16.0	28.0
6.00	4	8.0	8.0	36.0
7.00	2	4.0	4.0	40.0
8.00	2	4.0	4.0	44.0
10.00	Ta8	16.0	16.0	60.0
11.00	3	6.0	6.0	66.0
12.00	2	4.0	4.0	70.0
13.00	2	4.0	4.0	74.0
14.00	1	2.0	2.0	76.0
15.00	1	2.0	2.0	78.0
20.00	3	6.0	6.0	84.0
21.00	1	2.0	2.0	86.0
22.00	2	4.0	4.0	90.0
23.00	1	2.0	2.0	92.0
25.00	3	6.0	6.0	98.0
26.00	1	2.0	2.0	100.0
Total	50	100.0	100.0	

Above analysis reflects number of years clients availing services of stock broking firm.

Factor Analysis

Importance of factors in improving the service quality and in turn satisfaction level of a client in stock market.

Table 5. Total Variance Explained

Component	Initial Eigenvalues			Extraction Sums of Squared Loadings			Rotation Sums of Squared Loadings		
	Total	% of Variance	Cumulative %	Total	% of Variance	Cumulative %	Total	% of Variance	Cumulative %
1	3.17	12.681	12.681	3.17	12.681	12.681	2.582	10.328	10.328
2	2.252	9.009	21.69	2.252	9.009	21.69	2.111	8.445	18.773
3	2.197	8.786	30.477	2.197	8.786	30.477	1.992	7.967	26.739
4	1.985	7.939	38.416	1.985	7.939	38.416	1.828	7.314	34.053

5	1.889	7.557	45.973	1.889	7.557	45.973	1.762	7.047	41.1
6	1.512	6.049	52.022	1.512	6.049	52.022	1.639	6.556	47.656
7	1.469	5.875	57.897	1.469	5.875	57.897	1.567	6.267	53.923
8	1.363	5.454	63.351	1.363	5.454	63.351	1.543	6.17	60.093
9	1.18	4.72	68.071	1.18	4.72	68.071	1.532	6.127	66.22
10	1.039	4.155	72.225	1.039	4.155	72.225	1.501	6.005	72.225
11	0.996	3.985	76.211						
12	0.919	3.678	79.888						
13	0.806	3.225	83.113						
14	0.701	2.804	85.917						
15	0.614	2.456	88.373						
16	0.574	2.297	90.669						
17	0.474	1.894	92.564						
18	0.386	1.544	94.108						
19	0.319	1.276	95.383						
20	0.296	1.184	96.567						
21	0.28	1.121	97.688						
22	0.214	0.856	98.544						
23	0.138	0.553	99.097						
24	0.122	0.489	99.586						
25	0.103	0.414	100						

Extraction Method: Principal Component Analysis.

Table 6. Rotated Component Matrix[a]

	Component									
	1	2	3	4	5	6	7	8	9	10
Acceptance	-0.16	-0.201	0.099	0.201	**0.671**	0.15	-0.037	0.057	-0.004	-0.138
Positive attitude	0.697	0.028	-0.028	0.21	-0.056	-0.061	0.04	-0.403	0.298	0.075
Emotional intelligence	0.786	-0.004	0.037	-0.246	0.251	-0.019	0.123	0.037	-0.23	0.035
Creative thinking	0.453	0.183	0.039	0.248	0.524	-0.113	-0.37	-0.119	0.05	-0.192
Decision making	0.59	0.057	0.592	0.242	-0.033	0.059	0.099	0.255	0.097	-0.019
Problem solving	0.814	-0.065	0.066	0.05	-0.205	0.097	-0.009	0.128	-0.008	-0.226

Confidence	0.044	0.106	0.068	0.79	0.075	-0.16	-0.235	-0.062	-0.165	-0.056
Stress management	-0.16	0.386	0.223	-0.21	0.416	-0.022	0.341	-0.033	0.011	-0.413
Anger management	0.213	-0.236	0.41	0.069	0.315	0.043	-0.28	0.079	0.114	0.235
Time management	0.017	-0.041	0.088	0.202	0.172	0.008	-0.242	-0.129	-0.769	0.123
Empathy	0.037	0.152	0.806	0.031	-0.094	-0.324	-0.068	-0.077	0.171	-0.08
Work ethics	-0	-0.296	0.7	-0.182	0.124	0.062	0.058	0.009	-0.085	0.116
Dedication	0.133	0.326	0.164	-0.507	0.175	-0.484	-0.173	-0.023	0.063	0.025
Enthusiasm	0.095	0.068	0.019	0.044	0.052	0.766	0.008	0.179	0.061	-0.031
Patience	0.037	0.214	0.043	0.074	0.129	0.013	-0.096	0.858	0.063	-0.063
Effective communication	0.127	0.17	-0.172	-0.309	0.586	0.115	0.148	0.33	0.077	0.159
Courtesy	-0.05	0.768	-0.049	0.035	-0.019	0.075	-0.038	0.181	-0.076	-0.082
Clarity of thoughts	0.247	0.119	0.103	0.221	0.144	0.234	0.547	-0.37	-0.115	-0.221
Presentation skills	-0.16	-0.045	0.032	-0.039	-0.02	-0.073	0.099	0.021	-0.224	0.737
Socializing skill	-0.04	0.494	0.153	0.113	-0.146	0.146	-0.042	-0.212	0.072	0.611
Body language	0.141	0.069	-0.171	0.599	0.158	0.179	0.297	0.253	0.082	0.284
Corporate etiquettes	0.002	-0.097	0.3	0.041	0.33	0.039	-0.074	-0.047	0.76	-0.107
Persuasion	0.056	-0.083	-0.044	-0.091	-0.054	-0.161	0.768	-0.019	0.178	0.128
Responsiveness	0.059	0.803	-0.107	0.014	-0.005	-0.072	0.01	0.057	0.025	0.102
Reliability	-0.05	-0.009	-0.158	-0.144	0.152	0.681	-0.167	-0.288	-0.035	0.042

Extraction Method: Principal Component Analysis.

Rotation Method: Varimax with Kaiser Normalization.[a]

a. Rotation converged in 21 iterations.

Table 7. Factor loading

	Component									
	1	2	3	4	5	6	7	8	9	10
Problem solving	0.814									
Emotional intelligence	0.786									
Positive attitude	0.697									
Decision making	0.59									
Responsiveness		0.803								
Courtesy		0.768								
Empathy			0.806							
Confidence				0.79						
Body language				0.599						
Acceptance					0.671					
Effective communication					0.586					
Creative thinking					0.524					
Enthusiasm						0.766				
Reliability						0.681				
Persuasion							0.768			
Clarity of thoughts							0.547			
Patience								0.858		
Corporate etiquettes									0.76	
Presentation skills										0.737
Socializing skill										0.611

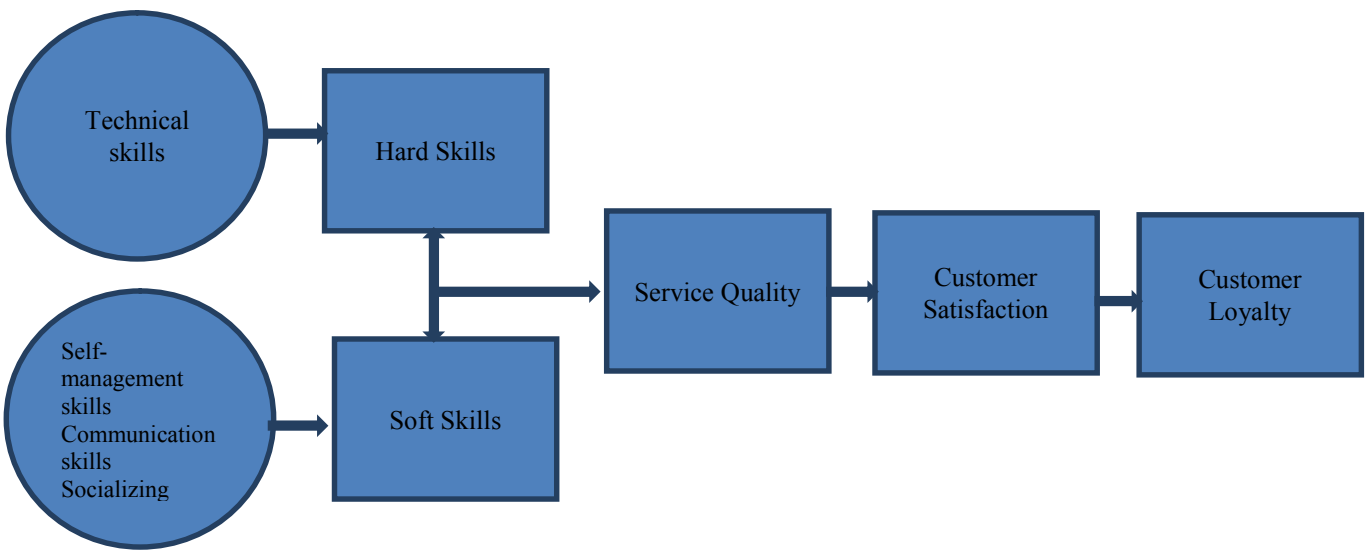
The outcome of factor analysis indicated ten factors that contribute to service quality in stock broking companies. The first factor can be named as self-management skills. The second factor can be named as responsiveness- the factor comprises of courtesy (.768) and responsiveness (.803), Third factor is empathy (.806). Forth factor is body language (.599), fifth factor can be broadly named as effective communication sub components being acceptance (.671) and effective communication (.586), sixth factor is reliability comprising reliability(.681) and enthusiasm (.766), seventh factor is persuasion (.768), eighth factor patience (.858), ninth factor corporate etiquettes (.76) and tenth factor socializing skill (.611)

CONCLUSION

From the above analysis ten major soft skill factors named self-management, responsiveness, empathy, body language, effective communication, reliability, enthusiasm, persuasion, patience, etiquettes and socializing skills are identified that affects service quality in stock broking industry. However the most important soft skill variables are self-management skills, communication skills and socializing skills.

On the basis of the survey and study, following customer satisfaction model is developed for the service industry:

Customer satisfaction model



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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

(Proceedings available for download at rku.ac.in/icre)

RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

Experiencing Experiences - Between Addictive Mobile Phone Delight and Menace: Consumer Behaviour Insights Through Qualitative Research

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ABSTRACT

The marketing paradigm "people buy experiences, not products" has assumed significance in recent times. Mid eighties onwards, much of acclaimed non-positivist research focus in consumer behaviour shifted from creating products to creating experiences. To create experiences, it is imperative to gain deeper understanding into what kind of experiences consumers experience, how experiences are experienced and what experiences do consumers seek and in which situations - the focus of this research paper. An integral part of an ongoing Doctoral Thesis, this paper incorporates an exploratory, qualitative research methodology using purposive, theoretical and judgemental sampling, gathering empirical data and validating it through triangulation of primary sources i.e. in-depth interviews, focus groups and literature review, drawing on grounded theory framework for data analysis. The paper concludes with essence of meanings of consumer experiences with mobile handsets, delineating antecedents and consequences thereof, representing potential implications and insights into marketing, consumer behaviour and entrepreneurship.

SUMMARY

The research objectives are to delineate situational experiences of urban mobile phone consumers, experiences which are remembered, interesting, repeated and valued with respect to their pre-purchase,

purchase, consumption and post-consumption 'situational' experiences, to delineate antecedents of such experiences in terms of their affective, cognitive, behavioural and environmental aspects and to represent them in a textural, structural and diagrammatic form, akin to a grounded theory.

Keywords:
Keyword 1 *experiencing experiences*
Keyword 2 *situational experiences*
Keyword 3 *qualitative research / focus groups / depth interviews*
Keyword 4 *grounded theory / antecedents / consequences*
Keyword 5 *mobile phones*
Keyword 6 *consumer behaviour / marketing*

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1. INTRODUCTION

Background and Rationale

The idea for the *objectives and r*esearch questions for this study originated from the Research Priorities for 2012-14 of Marketing Science Institute[1] (MSI). The aims of the MSI[1] are to bridge the academia-industry knowledge and practice gap through relevant research that focuses on real life marketing challenges faced by some of their member companies.

Every two years, Research Priorities are identified by a team comprising reputed academicians and senior industry representatives. **Marketing Science Institute[1]** (MSI) located in the US. The aims of the MSI[1] are to bridge the academia-industry knowledge and practice gap through relevant research that focuses on real life marketing challenges faced by some of their member companies.

Writing on 'sources of innovative opportunity', Peter Drucker[2] (1985), way back then, highlighted the importance of *'changes in meaning and perception*', 'changes in demography' and 'industry structure' amongst others, and this is relevant even today. A majority of success stories from the domain of Retail sector ('why we buy' - Paco Underhill)[3], banking and other services sector have been witnessed in recent years.

- Question is how far, if at all, such experiences can be created in *Marketing of products.*
- *What accounts for consumer experiences that are remembered, interesting, repeated and valued?*
- How can we gain *relevant i*nsights into *positive consumer experiences*?

All the above questions, together with fundamental concern of gaining deeper insights consumers' behaviour, are research priorities 2012 -14 laid out by MSI[1].

The proposed study addresses these marketing concerns by turning to the concepts of consumer behaviour, a complex phenomenon and an eclectic field. Arguably the most authoritative authors on the subject, Peter Paul and Olson Jerry[4] in their classic work *Consumer Behaviour & Marketing Strategy* (2005) lay down the framework of the wheel of consumer analysis, comprising reciprocally determined elements namely affect and cognition, behaviour and environment – and marketing strategy.

The proposed study takes mobile phones as a product category, Gujarat as the region of research and an exploratory, qualitative research approach, using a combination of in-depth interviews and focus groups, to dig deep into the consumers' underlying motivations.

Mobile phones (cellular **handsets**) as the product category have been chosen since it is a pervasive, symbolic and functional possession in today's world, cutting across all segments, arguably one of the most prized possessions of individuals today.

The Indian Mobile handset market is dominated by leading brands like Samsung, Micromax, Microsoft (Nokia), Intex and Lava amongst several others. Year 2014 saw 247 million handsets being transacted with about 30 % accounting for smartphones, the faster growing category of mobile phones, mainly due to availability of wide range of models and brands in the less than Rs.15,000 price band, influx of online sales, increasing mobile data consumption and decline in data tariffs and operators extending their data networks. As per a recent Cybermedia report[5], "while many new brands emerged in the India market, **the focus continued to be on offering the best experience at the right price-point."**

2. OBJECTIVES, PURPOSE AND LITERATURE REVIEW

2.1 This research paper seeks to address three objectives:

a) To explore consumer experiences as experienced by mobile phone consumers, probing what a mobile phone actually means to people, what kind of situational experiences they have had in recent past and are presently having, that is, right through the buying process and consumption life cycle and what kind of experiences consumers actually seek.

   After pilot-testing and refining the probing script questions, respondents were carefully chosen using theoretical and judgemental sampling with regard to their ability to share their experiences in depth and frankly. Delineating the core phenomena and narrating the essence of meanings of consumers in the Findings part of this paper fructifies this objective.

b) One of the main themes of this conference is Innovative Research Methods and Tools and as a towards this end hence, the second objective and a strength of this research paper is that it seeks to highlight that the emerging insights and findings have been made possible by using relatively innovative and still nascent Qualitative research methodology in India, and is inspired by its much respected status and growing popularity amongst researchers in the more developed Western countries of US and Europe.

   This consumer research makes a bold attempt using an exploratory and qualitative research methodology with audio recorded narratives, incidences and observations from focus groups, depth interviews for gathering rich, free flowing data which has been gathered, analyzed and represented here qualitatively, supplemented by literature review.

   The sections on Qualitative methodology, justification for choice of methodology, literature review of methodology especially relating to depth interview, focus groups, sampling, grounded

theory frameworks using constant comparative method for data analysis are hence included to validate the choice of methodology.

c) The third objective is to conclude the findings and insights by delineation of antecedents and consequences i.e. establishing cause and effect relationships, in a textual and diagrammatic representation associated with the essence of common descriptions and shared meanings of consumers' mobile phone experiences; the central theme - especially the addictive experiences ranging from delight to menace, antecedents and to explore and describe the emerging coping strategies and consequent marketing implications for business.

   These are explained in a theoretical process-flow diagrammatic form in the Concluding part of Findings and analysis and may be construed as this paper's humble contribution to consumer behaviour literature to propose a theoretical explanation of experiencing experiences with mobile phones, presented in a model framework relating to experiencing experiences.

To use the terminology of consumer behaviour, the research objectives are to delineate situational experiences of urban mobile phone consumers, experiences which are remembered, interesting, repeated and valued with respect to their pre-purchase, purchase, consumption and post-consumption experiences, to delineate antecedents of such experiences in terms of their affective, cognitive, behavioral and environmental aspects and to represent them in a textural, structural and diagrammatic form, akin to a grounded theory.

2.2 Literature Review – Key Concepts, Theories and Analytical FrameworksOn Consumer Experiences as in Marketing

This research paper has benefitted from the review of literature relating to the subject of the paper, on consumer behaviour, consumer experiences with regard to the field of marketing. The works of Abraham Maslow (1962)[6] in defining 'peak experience' as 'moments of happiest happiness and fulfillment' deserve foremost mention, as do the 'wheel of consumer analysis (WOCA framework) by Peter & Olson which explains dynamic interrelationships of consumer affect, cognition, consumer behaviour and consumer environment as a hub and wheel framework, especially his concept of situations as 'a sequence of goal directed behaviours alongwith affective and cognitive responses in the environments in which they occur' and the concept of 'reciprocal determinism' amongst all three elements is indeed a landmark contribution. Per Peter & Olson, a situation is defined as 'a person acting in an environment for fulfillment of a specific purpose or goal'.

Richard Oliver (1997)[7] gave a useful expectancy disconfirmation approach linking positive or neutral or negative disconfirmation satisfaction to post purchase performance experience of the product/brand.

Other noteworthy concepts and contributors to consumer behaviour and experience include means end chain linkages (Edward Tolman 1933)[8], laddering interview as an interview technique (Peter & Olson)[4], consumption experiences in Holbrook and Hirshman's seminal works (1982)[9] on hedonic consumption and Brian Lofman[10] on instrumental vs. hedonic consumption and experiential perspective of consumer behaviour as an alternative to the information processing and decision making approach.

The Thought-Emotion-Activity-Value (TEAV) Model by Hirshcman[11] (1986) encompasses all forms of consumption, including those included in the traditional Consumption-Affect-Behaviour-Satisfaction (CABS) Model (Engel, Kollat and Blackwell, 1968, Howard and Sheth[12] 1969, Nicosa 1966).

Concepts of symbolic meaning given by Levy[13], the seminal HBR article of Pine and Gilmore[14], 1998 in 'The Experience Economy' and by Berndt Schmitt[15] on experiential marketing 1999 catapulted the significance of focusing consumer behaviour thought and practice of the 'experiences' dimension to centre stage.

Another definition of experience marketing has been defined by Gortes[16] (2004) as aiming 'to provide consumers with compelling and memorable experiences that emotionally connect them to a brand each time they buy a product'.

Relevant to the scope of this research is 'product experience' as defined by Desmet and Hekkert[17] (2007), as "the entire set of affects that is elicited by the interaction between a user and a product, including the degree to which all our senses are gratified (aesthetic experience), the meanings we attach to the product (experience meaning) and the feelings and emotions that are elicited (emotional experience)."

Yet another view of experience is that 'experience is not what happens to you but what you make of what happens to you", say Aldous Huxley, Texts & Pretexts, Harper and Brothers[18] (1993). This reflects the interpretive side of what an experience may mean to a consumer.

Moneesha Pachauri[19] makes the point that it may be more worthwhile to study consumption than buyer behaviour whereas Eric Hauser[20], Director of the International Experiential Marketing Association (IXMA) asserts that 'traditional marketing theories are rapidly changing, experiential marketing is leading the way into the new marketing paradigm'.

More than an year after this research was undertaken, in Nov. 2014, the Outlook Magazine dedicated its 19th Anniversary Issue with a welfie photo on its cover (group selfie) entitled "Hello, Beautiful – How the Mobile Phone, also born in 1995, changed the way we live, love, work, play, think – and look at ourselves", in a nationwide survey of known personalities, calling it a 'predatory technology'.

3. MATERIALS AND METHODS

Research Philosophy, Methodology, Tools

3.1 Research Philosophy

This research in sync with the ontological perspective of non-positivism; in that multiple realities do exist. As opposed to the positivist worldview that there is one single truth and the researcher's job is to find it, this paper holds the view that the nature of truth or reality varies from person to person and is subjective.

In fact, consumer experiences and the meanings they internalize and interpret from their experiences with a particular product or brand are different – and to each, the truth is how they individually perceive it. Qualitative and interpretive research recognizes this complexity and rather, the focus of research is to understand the multiple views and subjective experiences.

3.2 Research Methodology, Tools & Techniques

Keeping in mind the exploratory design and objectives of the study, the following multi method qualitative study data collection methods and sampling techniques have been chosen, which have been recommended by research methodology authors:

a) Face to face semi-structured in-depth interviews to collect primary data from respondents. 'In an exploratory study, depth interviews can be very helpful to find out what is happening and to seek new insights' (Robson 2002:59 as in Saunders[37] et al.) and to probe consumers' explanations of their meanings to capture their narration/stories of experiences.

b) Focus groups discussion (Kruger & Kasey[38] 2000:25) being an information – rich source of interviews (Malhotra & Dash[40] 2010 : Mobile Phone script)

c) Field notesof researcher (Saunders[37] 2011 ibid) includes memos, transcripts and documented records and diary notes from learnings from interviews, secondary sources, academic and/or scholarly journals and interactions, textbooks, published management literature, industry conferences, newspaper articles, websites, other sources.

3.2.1 Sample Size

While selecting a suitable sample size for qualitative studies, Saunders et al. (2011) emphasizes the relationship between sample selection technique and purpose and focus of research questions where, generalizations are made to theory and not to the population.

Sample size depends upon the research objectives, upon what is useful, credible, and feasible in terms of available resources of time and cost, and the validity of insights gained from the data have more to do with the data collection and analysis skills of the researcher than the sample size (Patton2002)[9].

Guest[26] et. Al (as in Saunders 2011) suggest a sample size of 12 respondents for depth interviews within a homogeneous group. If the sample is drawn from a heterogeneous population or if the focus of the research question is wide ranging, John Creswell (2007)suggests a sample size of 25 to 30 interviews until data saturation.

At the time of writing this research paper as in December 2015, qualitative data from a sample of 12 respondents in depth interviews and 8 respondents (total 20 consumers covered) in Focus group have been collected and analysed, apart from field observations, adequate enough judging from constant comparision method, to propose authentic findings and analysis for the purpose of this paper.

3.2.2 Sampling unit

Sampling unit is a person residing in urban Gujarat region, male or female, in the age group 15 to 70 years, belonging to urban higher middle class family and above,having educational background of 10^{th} standard or above,a personwho is willing and reasonably experienced with mobile phones having used it for atleast for last 3 years, to be interviewed for this purpose. Consumers of all product categories and

price points – feature phones, smartphones and I-phones/notepads/hand-held devices (excluding laptops) form part of the sampling population.

Basic characteristic influencing the sample choice is the respondent's potential to possess information-rich responses relevant to the topic, willingness to give some time (45 minutes to 1 hour), participate in free sharing of their past, present and expected affective and cognitive responses with mobile phones with the researcher including their antecedents, with a reasonably positive degree of involvement on part of participant.

3.2.3 Sampling Procedure

Purposive, Judgemental sampling (non probabilistic) is ideally suitable for qualitative studies (Newman, 2005)[21] especially when they are relevant to the focus of research objectives and questions, for gaining insights from small samples, which is the purpose of this study, as mentioned in above section on sample size.

Though such samples are not statistically representative of the total population, Patton[22] (2002) contrasts the need to obtain information-rich cases in purposive samples with the need to be statistically representative in probability sampling, advocating that purposive sampling strategies are actually a strength where objective-specific choice of samples is made which are heterogeneous (maximum variation sampling), homogeneous, unusual or special, critical-case and typical of sample characteristics which is considered a strength in validity and sample representativeness (Saunders et al. ibid).

This view is further reinforced by Marshall and Rossman (1999)[23] '*where the researcher is able to relate his research project to existing theory, the researcher will be able to demonstrate that the findings will have a broader significance than the cases that form the basis of such research work.'*

Further, such empirical findings can be quite useful as a starting point for formulating meaningful hypothesis to be tested statistically or otherwise in further research studies (Saunders et al. ibid).

3.2.4 Data Collection and Analysis

On appropriate research methods for qualitative studies, 'the interpretive approach is relatively new in the field and has become quite influential, based on theories and methods of cultural anthropology to understand consumption and its meanings, using long interviews and focus groups' (Peter & Olson 2013)[4].

a) Interviews and Focus Group discussions would be audio recorded, with consent of respondents.
b) Combination of narrative, story-telling, projective techniques and probing through laddering technique would be used for collecting the data required for addressing all three objectives and research questions.
c) Grounded theory : In combination with and to give shape to narrative analysis, this study will also draw upon the works of Strauss and Corbin (2008)[24]and Glaser and Strauss(1967)[25];in aninductive-deductive combined approach to analysis may be used which validates purposive sampling to select critical cases 'to further the development of concepts and categories and to explore relationships between these two to develop a theory which is thoroughly grounded in the data, by constantly comparing the data being collected with concepts and categories being used'(as outlined in Saunders et al. ibid).

The analytical framework and stages of grounded theory involve:

- Open coding (process of disaggregation of data into units)
- Axial coding (recognizing relationships between categories) and
- Selective coding (integration of categories to form a theory)

3.3. Applying Grounded Theory to Fieldwork

The data generated is qualitative in nature and so are the methods for data analysis and interpretation. On qualitative data collection methods in Qualitative Research and especially grounded theory, Guest et. Al and Emily[26], writings of Strauss & Corbin, John Creswell and various articles by Berndt Schmitt and Holbrook and published news reports were referred specially to ensure methodological and content validity.

3.3.1 Data Reduction – Open, Axial and Selective Coding

The data analysis in this research follows the grounded theory approach where audio interview data is converted to textual transcripts and from that narrative text, categories of relevant data are formed – first open codes, then axial and then selective codes – to explain the flow of an explanation of a process or theory which is grounded in primary source data compiled from participants' narratives or observations.

The process of naming or labeling sets of data into categories, which posesses certain specific characteriistics is called Coding. The audio recorded data are transcribed into text and then data are broken down into segments and then examined for "what is common', that data which makes sense when it is named into *categories or concept themes*.

After the data are segregated or thus *categorized,* they are then examined for *properties*that characterize each category. So O*pen coding* is a data reduction process where data is broken down to a small set of concepts or meaning clusters that seem to best describe the phenomenon being reserached.

3.3.2 Incoming Fieldwork Data and Constant Comparative method

After collecting primary data though Depth Interviews and Focus Groups (, the analysis from above sources – at open coding, axial coding (inter-relationships between categories) and selective coding (delineation of core phenomena) can be considered as valid enough so as to explore and establish causal relationships amongst categories and derive a description of the essence of meanings of experiences experienced by research participants so as to make a meaningful contribution to theory, which is the purpose of this study.

From the categories so far identified, the incoming data were commpared and anaylysed it so as to check if it is fitting into or confirming with data categries or inter-relationships being examined. Thus, constantly, data analysis and the researcher, sort of move back and forth among analysis of categories and fresh data collection, this guides and narrows the focus of questioning which characterises qualitative research design. Hence the analysis gradually moves from coding to developing more meaningful conceptual categories, and contributes to theory development.

To quote Glaser and Strauss, the "basic,defining rule for the constant comparative method" is that, whilecoding an incident, the researcher should compare it with all previousincidents so coded, a process that "soon starts to generatetheoretical properties of the category '' (as in Sehriban Bugday –Slideshare Dec 2013).

4. RESULTS AND DISCUSSION

Findings and Analysis

Following the prescribed format of qualitative research, the findings of this research are presented in a textural and structural description by analysing qualitative data from participant observations and focus group interactions, by forming open categories from recorded interview transcripts, coding and categorising verbatim statements of respondents and then relating them to and associating them with meaning-units and related statements on specific experience dimensions of other categories and then establishing antecedents and consequences i.e. cause and effect relationships so as to conclude the experiences as experienced by mobile phone users from an experiential, consumer behaviour and marketing implications especially in terms of the huge entrepreneurial opportunity that such insights hold for all business entities associated with the mobile phone.

4.1 'In Vivo' Quotes from Depth Interviews & Focus Group

True to Qualitative research tradition, the emerging themes and incoming data statements are represented in the following pages overleaf, verbatim – in the words and statements of respondent participants themselves. This is the first stage of qualitative data analysis, to list out the Open Coding Categories – as relevant to the research question. From a closer analysis and constant comparision with data from further interviews, Axial Coding i.e. establishing relationships amongst categories and finally, selective coding (integrating the data) for describing the core phenomena and developing the theoretical explanation of the phenomenon would be undertaken.

Qualitative research regards verbatim exact statements and textually transcribed sentences of research respondents from audio focus group discussions and depth interviews as most authentic, empirical, valid units of data. These various sentences are categorized i.e. coded into common meaning units and then subjected to data analysis with related statements for establishing cause and effect relationships and selecting the essence of those shared and common meanings and experiences by all research participants in light of research objectives.

These statements are called In Vivo codes, which literally means, 'in the words of respondents themselves.' To give an idea, some open categories & emerging themes which were constantly compared to incoming data of successive Depth Interviews are mentioned below:

Quote

a) *My mobile is my constant companion, it's my life. I can't imagine living without it*

b) *It's my personal moments, my most cherished memories, music, friends, family, all into one place*

c) *"In between breaks" …what's app is always ON. Whenever you get time, in between work breaks, you simply open FB, what's app, music….and temporarily escape in your own world…it's a stress buster in today's fast life. It recharges you in few minutes…then back to work. And at night…*
d) *Availability of latest technology and fast obsolescence*
e) *All my friends had it and I did not (feeling left out, must catch up, be IN)*
f) *I want peace in life (asked from a CA final year student)*
g) *Aspirational craze for I Phone / Apple*
h) *Temperament / individual nature antecedents*
i) *Basic use, superior functionality - for calling, basic features, apps*
j) *Narratives having situational and contextual linkages:*

i. *lost in marriage, emergency*
ii. *fresh to college, birthday party, sharing, feeling left out, peer choice*
iii. *software crashed, blackout, emergency*
iv. *'must be the latest technology'*
v. *given to me by company, it's a nuisance yet entertaining*
vi. *its MY mobile, bought with my own money, context - first job*

These categories seem to emerge as relevant and interesting, and would be compared to incoming data from successive interviews and for forming open codes i.e. categories of interest to the researcher and then be analysed for more accurate interpretation. As discussed earlier, the data generated is qualitative in nature and so are the methods for data analysis and interpretation.

The dominant, more shared, common experiences depending upon data insights were identified as the research progresses and as the interview questioning sharpened with each successive interview.

4.2 Select Sample Transcript - interview interaction between researcher and participants

The meanings, experience essence of the data reproduced in the following pages and the insights and conclusions drawn from them have been summarised in the Findings and Analysis section of this paper.

Quote
Researcher: Today our topic for discussion is on Experiences with mobile phones…so tell me, what is the best thing about having mobile phones?

Participants' responses:

Connecting people, in short time.

It has made our life easy.

Nowadays we have everything in our phones, you want a bus ticket, we can do that on our phones, check our e mails, talk to our friends which are not in the same area, we can talk to them. We can do anything…

Video calling is the best thing. When we are in India and our relatives are in US, we are closer, can talk to our relatives in US.

Internet, surfing internet. Connecting with people.

Best thing I think is that we are available – at any time and everywhere.

Now things are present in application. We easily use…or tasks…interacting with the outer world.

Researcher: What is the worst thing about having mobile phones?

P**articipants' responses:**

Addiction!

What's App chatting!

Depends on us, it depends on us. If our choice is bad (for unproductive use), then it is addiction.

Continuously chatting…

It's a nuisance! Anyone can call you anytime, anywhere

It's turned out to be like a drug addiction…something that you just can't live without. You have accepted the technology, as a dependent source that you just can't live without…

There is one more bad thing about mobile phone, it does bring us to people who are far away from us but you forget the people who are next to you!

If you are sitting with family, then also we are in the mobile only, although they are sitting next to us, but we don't talk to them.

Initially we used to run behind local calls and it used to be used only for making calls and those kind of calling schemes. But one thing since we've noticed that because of this Android things, what's app, all these social media activities have come over, now do we really receive calls, let's be honest…I mean people think, he who will spend money to make calls, you have even free calling schemes if you are using 3G internet maybe, chatting is the only mover so call rates have reduced like that (indicates 'down' motion).

Main use is for messaging, not calling. Ya. Call rates have gone down, drastic change

Researcher: To me, my mobile phone means….describe in few sentences…

Participants' responses:

World of knowledge

I hold it like a diamond ring. It is precious! Because papa has worked very hard to get it for me!

It is useful for my work.

It's like clothes, everybody wears clothes, everybody needs a mobile. You can't live without it.

It is like a friend. When I am alone, I don't feel alone even if I am alone.

Updates, knowledge, time pass
Backbone of my life
Besty (best friend)
Belief (a trust)
People change with time, but mobile does not change

It is like my friend

It is always there with me, whenever I need

It's a safe, a personal safe where all our secrets are hidden; without telling anybody.

There are some secrets which you can't tell friends and there are some secrets which you can't tell parents!!

About I Phone

In US it's totally different scenario; people are using less of Androids and more of the I Phones.

If I am using Android I can connect my laptop with it but if I have I Phone then I cannot use the data on my laptop which has another system; it has to be compatible with I Phone system only…security purposes

But then that is a problem, right? One can't share things with others…with my friends and family who have different devices…

It is for security purposes….these people have done it

Thing is whatever you do, virus should not enter (I Phone system).

You can conquer the world, but if you use I Phone, you HAVE TO use <u>our</u> I Phone compatible devices <u>only</u> (software, data platforms, apps that use Apple's high security norms). They have created this thing for themselves.

That's what. It is a disadvantage of I Phone and that's why I don't like I Phones because its only I Phone to I Phone and people cannot really afford it and cannot use it.

Very costly, mobile is very costly, that is another reason for being possessive.

Researcher: Narrate your Experience with your past mobile phones

Participants' responses:

My past phone did not have an Android system, plus camera and battery life were an issue and was feeling bit odd taking it out in front of friends, teasing, like hey are you still using that old mobile phone?

But I will tell you, those old dabba like mobiles were so tough, nothing would happen if they fell to the ground, how much ever hard. But these new phones, I Phone 6, for example…the glass display casing broke, it was just kept in my friend's pocket and the display broke, so delicate!

It needed such a lot of charging, I tell you…
Samsung….always used to hang!
If you don't use it then also the battery drains out!

Normal phones all hang. I Phone, I have 64 GB memory, you take out apps and all and I am left with clean 20 GB memory and space …that's enough, it will not hang. Games, movies…videos…

I would like to add something. First we had normal phones; we used to communicate normally. These phones went out of vogue. Communication became difficult. I upgraded because, see, now exams will come, we can share our photos and notes and all ….this had become very difficult with my previous mobile phone, so I upgraded to Android. (Situational experience, specific goals, means-end chain, category functionality)

And all pictures did not open in my old Samsung. There were some Samsung versions which did not support quite a few image formats sent through what's app or Bluetooth…so I needed to go for a model where this was possible.

For me, whenever I used to study, I always used to sit with a dictionary. Now, it gave me a reason to give to papa to buy an Android phone, that look, it has an inbuilt dictionary and I will be able to use it for my studies!

Until January I used to use E3 Nokia. Then company stopped making new applications and are not updating what's app applications. I was not getting new apps, not getting new updates so I bought new Android phone.

See, I buy the latest phone available in the market. I have two I Phones. I thought it was a superior phone but its battery broke and melted from inside (check this). Such a thing could not have happened even with Samsung.

When I had first bought my I Phone I had repented but the second one I bought it gave me everything that I wanted but then the display broke….I had not even used it much

Category: Post Purchase, consumption experiences (in words of participants)

I don't even brush but first check messages…

No, I first brush and then check my mobile for messages!

Charging, charging…charging…first check the charging…

First I get up at 5 am, then I check what is the time? So I can judge how much longer I can sleep, then I check charging, if not charged properly, put it in charging, then when I go to the gym, at times I take it with me or if I feel then I let it be in charging only if it needs charging. Then return home, take it with me – the charged phone with me – and set out for college. I listen to songs on the way (it takes one hour of commuting time on the bus) and then once I enter the college it is in my pocket until I leave the college, it stays in my pocket only.

Facebook….if more people give 'likes' to you, there are more people who are jealous of you! People are double faced!

Checking the result on the mobile, I got no backlog! (memorable experience)

Capturing personal moments, when doing mischief and teasing with friends, these are wonderful moments which we capture on video

Category: Experiences 'Sought' by participants: what experiences do you seek with your mobile phone?

A 3 D experience!

What we want, is to control (or limit / reduce) my what's app usage. So I should be able to tell my mobile to control it in some way, and my mobile should actually control it for me! Like its exam time, and I don't want to use what's app now and want to stay away from it…

I seek inner peace. If I am going out somewhere; I just keep it to one side…

Unquote

4.3. Key Findings, Emerging Themes and Insights

The validity of the key findings that have emerged has been established through three sources of incoming data; depth interviews, focus groups and researcher's observations and field notes further validated with published new-reports. In other words, the insights analysed through the analytical process of qualitative data reduction, constant comparision method and grounded theory may be considered to be reasonably reliable.

The findings in this research paper focus entirely on understanding the subjective lived experiences of mobile phone consumers at an affective, cognitive and behavioural level. As such, though the mobile experiences do relate to product features, mobile and internet technology, content, popular apps, social media, android versus feature phones, aesthetics, pricing, purchase decisions, online and offline channels, leading brands, advertisements, this paper focuses primarily on understanding the meanings and situations of consumer experiences.

To a considerable extent, the findings relate to common 'Situational Experiences' Experienced by Mobile consumers and represent core phenomena, central theme box in the concluding diagram. The kind of experiences one has, depends upon the total personality nature of person and the kind of experiences that the person has had in his/her life and with mobile phone brands generic – and specific brands - in the past. The key findings represent paradigm shifts in themselves – not just to do with mobile phones but with relationships, work, enjoyment, entertainment and oneself, these mobile experience concepts are

pervasive enough to each well be the topic for a separate study. These are summarised below and are further represented in the section on conclusion.

Key Findings, Paradigm shifts and Insights of Experiencing Mobile Experiences:

1. **Concept of mobile being an all inone most personal, individual survival possession**, with narcissist craze about the 'selfie', the mobile is now a personal self-extension by default, for work efficiency, instant knowledge, networking, work performance, career confidence and facilitating everything connected to doing business, marketing and relationship management through social media, news, music, videos, games, apps, voice and data services. Insight – it is an extension of one's own self, a self identity, a kind of status symbol or expression of oneself, an alter ego of sorts.

2. **Concept of Addiction, Self Control and Conflict:** Constant Self Control Conflict between extreme addiction on one hand and extreme nuisance and goal-diverting disturbance. One photo shows a human wrist bound with iron chains holding onto a mobile phone – it epitomizes one of the key central themes and could be used in a diagrammatic representation. Insight – the extent to which consumers are addicted and chained to it that is alarming. Constant daily inner conflict is being experienced, resulting into self control crisis and work productivity crisis.

   Most consumers experience severe time pressures with so many groups, mails, calls and especially what's app and experience a feeling of being overwhelmed by the media and coping with issues and the people they interact with. On one hand consumers seek ways to control and cope with the over indulgence and on the other it is so habit forming they are glued to it and seek even more thrilling and specialized apps, features, services through mobile phone and hence can't stop.

3. Concept of the mobile meaning a '**Safe Deposit Locker which holds all secrets, being an emotional love bank of fondest, memorable experiences (videos, sounds, images, messages), favourite Music**, **videos**. So much so that consumers guard it with strong passwords and takes care to hide the content from rest of the world.

   Some people prefer and/or aspire for Apple's I Phone over Android system due to the data security aspect. Insight is that people have lot of cherished memories and secrets both to hide, and assumes crisis and panic, disaster situation if it is lost, most importantly it should not reach wrong hands. Means – end chain may be established with Apple's I Phone and some features and facilities for data safety conscious consumers.

4. Concept of **paradigm shift in relationships – it brings far people near and takes near people far.** Insight – the dynamics of relationships and social interaction have changed. Attention deficit during personal face to face interaction with family and friends sitting next to us has suffered and possibly caused interest deficit albeit temporary, whereas people or groups located far away, perhaps even abroad with whom chatting is going on, is enjoyed. Many consumers

experience experience a feeling of pride at having so many social groups, whereas it is a nuisance for some, pleasure for some. Social media, Group friendships, texting, sexting, sharing, keeping in touch has changed relationships and how people keep in touch.

5. Concept of everyone from tiny tots to illiterate people to hawkers to homemakers to elderly people **learning how to use smartphones,** become 'smarter' and more connected, using what's app to **"be included, not get left out"** wrt latest technology, apps, features, speed, online behaviour suiting to their goals (situational aspects). Feeling 'in' and included and happening immediately after purchase, after experiencing apps and especially what's app and sharing pictures, is a key finding.

6. **Concept of 'experiencing experiences'** – consumers experience some of the most joyous experiences and consider the mobile as the best way to spend leisure time, listen to music, or chat with loved ones or read news or work mails during commuting time, spending any waiting time in offices, and during the work-day, getting re-charged by catching up on messages and what's happening "in between break times". First thing in the morning and just before going to bed, rediscovering private space and individual joys as also and the sense of belongingness that comes with group sharing n social media – which is not without its downside. One insight is that for every 'like' that one gets on FB, there are many more people within your circle who are actually jealous. So all that appears on social media is not necessarily a true picture or opinions about people or issues..

   In addition, consumers also experience being locked into the same current mobile phone experiences, unable to purchase the aspirational phone/brand immediately due to various reasons ranging from cost to others. Having to wait for the next dream mobile purchase is a time horizon that could last from few days to an year or more, is a stage in one's life and mobile usage termed in this research as 'experiencing experiences' – that is, a feeling of compromising, being left out in some respects, watching others have it, experiencing at times, shame or inadequacy until the purchase is made.

   It is during this stage 'that various experiences are experienced, at cognitive and affective level and during which consumers plan their next purchase because for many, with changing technology, time and situations which are co-created (like peer pressure, advertisements, online portal offers, people and situations in work environment), their present mobile phone is not giving them what they want and consumers seek an upgrade to a better mobile phone hoping for special experiences unique to themselves.

Thus, mobile phone experiences hold several memorable and potential joys and benefits for most consumers which at most times outweigh the hazards and the flip side of living with it, since the cellphone has become an obsession of dangerous habit forming proportions that eats away into already limited time that people have to accomplish their daily goals.

The cellphone has a powerful impact on overall quality of life both positively (eg. apps for health and walking, maps for reaching new places) and negatively (several examples of relationships being spoilt, individuals wasting their time in unproductive activities like chatting, gaming).

Many of these experiences are found to be raising self esteem, work confidence and social confidence as well, keeping in touch with long lost friends, the negative side found to reduce quality time with loved ones, with some institutions banning its use during work sessions. At a macro level, antecedents and triggers for such experiences are environmental forces of technology, culture, shared values and peer pressure from friends and family – alongwith a desire to be happening and 'in'.

For some it's a nuisance, for some it's a delight. For most of the consumers, it's a delight in several situations but in some situations it's a menace and unwanted addiction that diverts from goals and creates time pressures, conflicts and stress.

For those whom it is a nuisance, are sincere students and busy professionals who caring mothers who switch off their whats' app or mobile data or even shut off their phones for some periods. Professionals find it stressful to cope with social media overload, constant phone calls and mails (*'one of the best things – and one of the worst things is that it can reach you anywhere, anytime')* andtend to switch it off, or put it on silent mode or switch off the data.

Extreme cases of work diversion due to android smart-phones' addiction are also there, where, in order to focus on exams or work goals, some consumers start hating the experience and as a consequence, resort to behaviour like de-activating internet and data services, stopping to use smartphone itself, deleting apps like games, what's app, Facebook and even switching to a regular feature phone and restrict the usage by children. Such consumers shared their positive experience of feeling of peace and a greater sense of work focus which helps the achieve their goals.

The research strongly suggests that the temperament or human nature, personality type of the person concerned is responsible for the subjective meanings and resultant consequences with respect to their mobile phone behaviour. This aspect is still being researched at the time of writing this paper, pending further analysis.

5. CONCLUSION

The objective of this paper is to explain how experiences are experienced by mobile phone consumers with all its situational antecedents and meanings, what causes such experiences and what are its consequences. Since qualitative studies do give importance to clarity of final purpose (Creswell) about developing a diagrammatic explanation of the process, judging from preliminary incoming saturation point data, the following diagram is represented for the sake of better understanding, pending ultimate analysis, since this research paper is part of an ongoing Doctoral Study and final data is yet coming in of what the final theory may look like.

5.1 Developing A Theoretical Framework of Grounded Theory

For an explanatory process flow diagram like the one presented here, a word of interpretive caution - generalizations are to theory and not to the sample population, though the proposed model uses empirical primary data, the theory is meant to contribute towards better understanding of consumer behaviour and experiences and to conclude a valid hypothesis and starting point for further research rather than an ultimate conclusion in itself.

Antecedents (sub-parts of main) ➡ Core Phenomenon ➡ Consequences (impact outcomes) lead to marketing implications for contribution to Consumer Behaviour theory and marketing practice, which is the contribution of this Qualitative Thesis Research study.

Antecedents are "why it is happening" would have dimensions of contextual and situational factors, as also those of individual's temperament / human nature and earlier lived experiences and what it means to consumers today – and now, at this point in time.

Core phenomenon is the essence of meanings of "what is happening out there", as per data analysis and textual interpretations of narratives duly validated by research participants. This is also known as core or central phenomenon, a description of a relevant insight or main finding of relevance, representing main phenomena which this research has set out to explore.

Consequences are "outcomes – desired or otherwise, they constitute an end result (at least for the time being – here and now), an impact of what is happening out there – they are present-state descriptions of participants' states of being". **Diagrammatic representation is presented overleaf.**

5.2.1 Textural and Structural description of essence of meanings

The exploratory and open ended nature of the research, patient listening and probing of consumers indeed fructified into throwing up some interesting insights that may not have been possible with a very structured, fixed answer choice kind of question format. The interactive nature of the research and the flexibility to change the script and narrow it down to more relevant shared meanings and experiences enabled the uncovering of some very interesting insights, which possibly some of the readers of this paper may possibly identify with.

The Core or Central Phenomena is theessence of shared meanings as shown in the Diagram box emerging from the data is that most people seem to be experiencing a conflict situation between the joys and the pains of having the mobile as their constant companion in life, and are trying hard to cope with mixed feelings, on one hand, the sheer joy of experiencing all that the mobile stands for – connectedness, relationships, discovering long lost friends, social confidence, a magical world of apps, music, work productivity, a faithful loyal friend who captures moments and memories and keeps all secrets safe, and on the other, coping with the seemingly unavoidable frustration against an enslaving mobile technology which has already developed into extreme addiction so much so, that some participants shared a concerted effort to control and limit its usage wrt unproductive time wastage in order to focus on more worthwhile goals in life and not let it control oneself at any cost.

The structural description has been described in the Findings section, and can be inferred from the in vivo codes, sample transcripts and interpreted in the light of the content of Antecedents and Consequences shown in the diagram.

These findings, analysis and concluding diagram proposing the theory of Experiencing Experiences have direct entrepreneurialand marketing implications.

As this research has progressed over depth interviews conducted in 2014 and most of 2015 the new technologies and features introduced reflected the same findings of this study – in other words, this research validated the experience and features which consumers valued and sought (front selfie-taking camera phones, power banks – to help resolve the battery life problem and narcissist selfie desires).

Whatever points this research uncovered, actually became an innovative offering in a new model in the market about 6 to 9 months later, validating the process, the script and the findings in that the research is progressing in the right direction.

Not even a decade back, the then market leader Nokia heavily paid the price for not listening to consumers – and quickly lost the market to players like Samsung, Micromax and a host of others who delivered what consumers wanted, because they understood the consumer – and the experience they wanted, better.

Emerging insights for entrepreneurs and marketers in the mobile handset space

Flowing from the analysis of grounded theory research findings and relevant insights from the diagram on experiencing experiences with respect to mobile handsets, entrepreneurs in the mobile phone space would benefit from planning and executing the following product-market strategies:

1. Investing more into market research time through qualitative research to delve into what experiences consumers seek – features, benefits - the front facing camera selfie, solutions aimed at longer battery life or more memory space or speed; 'the next best innovation - affordable', whether it is 4G experience or whatever it is that consumers fancy.

2. Innovations aimed at helping to resolve the conflicting situations due to addictive usage would go a long way in promoting a more valued experience. As shared very well by one consumer – 'the mobile can have some feature or app that tells me when to stop so I can regulate its usage'. It could be some app or some timing clock or online activity sensing software that regulates addictive use of say, social media or selected groups or select websites or some other apps, which could help people to manage their precious time to devote to more worthwhile goals.

3. To address negativity towards mobile phones being a menace, Entrepreneurs could plan marketing and awareness programs advocating safe and judicious usage of mobile handsets. Consumers do need to be taught how to – and how not to use the mobile phone so that it gives all benefits which it should – and at the same time, does not significantly compromise quality time with loved ones or be a distraction from more important goals.

4. Entrepreneurs can focus on designing consumer experiences based on the findings of the research question as to what consumers are seeking from their present or their next mobile phone purchase, is the finding that consumers seek those kind of experiences which are symbolic of what they personally want from life itself – for most, it's THE big question. While some consumers hope, expect and dream that technology will one day, empower them with respect to their customized unique and dream wish-list ranging from 'I just want peace in life' to 'to enjoy and spend quality time with loved ones' to 'reach heights of career success through its apps and secure data technology', to 'it should be a trusted work assistant that connects office data and images irrespective of where I am', others seek 'peace' and 'time with family' to 'a gadget which has a self control facility to prevent excess waste of time. A host of customized apps to cater to distinct segments can be explored.

5. Another critical area is data safety and the anxiety over keeping one's secrets and data safe from prying eyes and viruses or data loss due to theft, breakage, damage or handset loss. Here again is an opportunity for entrepreneurs to come up with some protected data or security package solutions that can be accessed only by the consumer – even should the mobile be lost. Apple's devices are too costly and there is a big opportunity in this 'safe deposit vault' concept, if it is made more affordable.

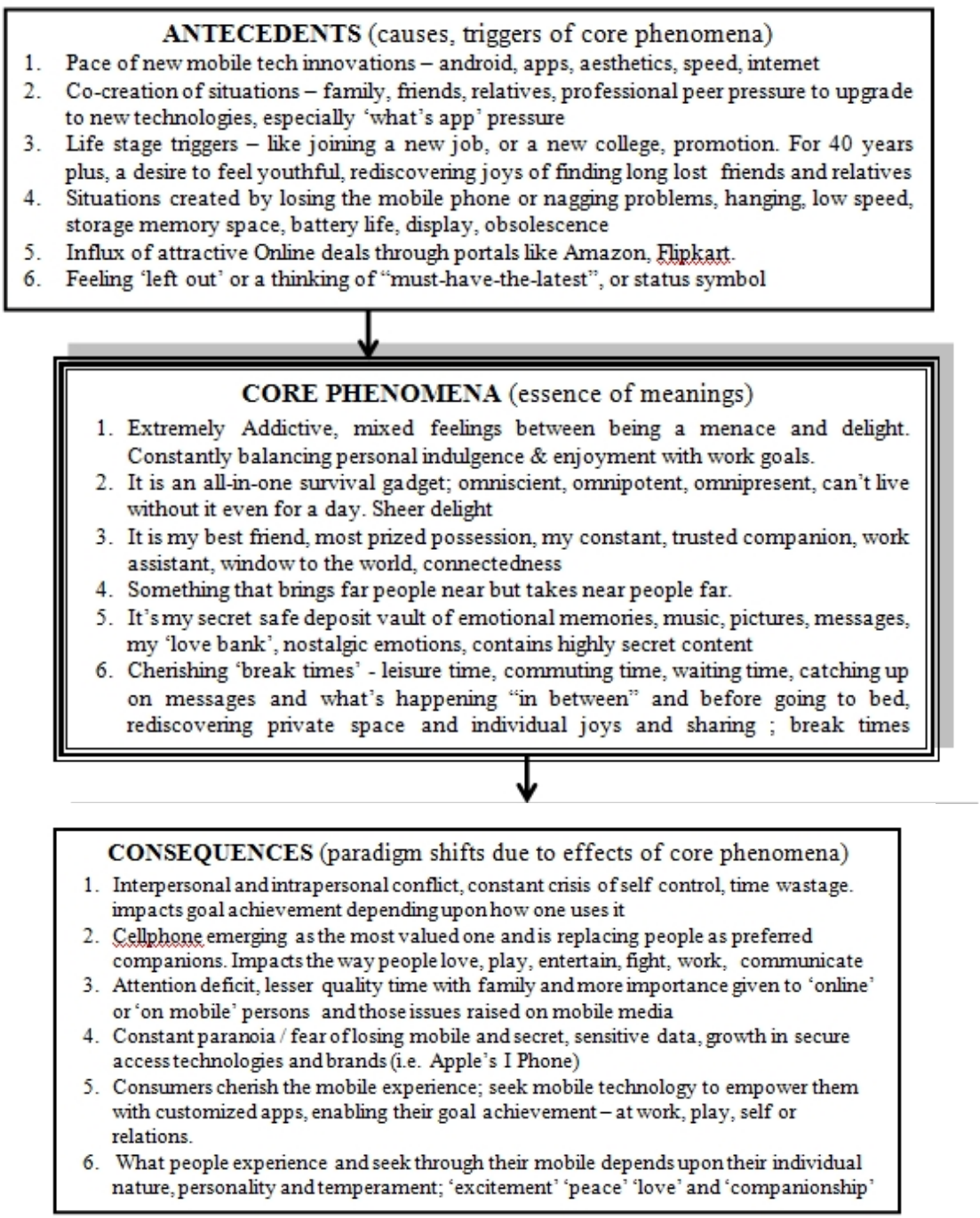
The limitations of this research are that it is restricted to the consumers of Gujarat, India, that the findings are not generalisable to the population as in quantitative studies – (though in this research paper, they are not meant to) and that the final analysis is pending since this is part of a still ongoing doctoral research. Interpretive bias has been addressed by using 'bracketing' i.e. a qualitative research process where the researcher is aware of and sets separately aside his or her own notions about the topic, though in qualitative studies such as this one, it could still be a limitation.

Amongst the positives of this research is that it is conducted with Qualitative inquiry in natural settings of consumers after taking them into confidence and trust, for open ended and free sharing ; especially grounded theory has this advantage of being intrinsically validated since the proposed theory as in this paper (as per the diagram on experiencing experiences) has developed from consumers' responses themselves and is not based on past theories or researches which may or may not fit today's dynamic marketing environment for a particular industry, product category, culture or geography.

It is hoped that this paper motivates further qualitative or quantitative research with respect to the topic, the methodology, analytical approach and/or the findings which may be useful as a hypothetical starting point.

FIGURES

5.2 'Experiencing Experiences – A Grounded Theory Framework of Experiences of Mobile Phone Consumers (Diagrammatic Representation)



ACKNOWLEDGEMENT

I would like to acknowledge the contributions of my wife and son (Harsha and Yatharth – my inspiration), my parents and family, my close friends, my PhD guide (Dr. Raashid Saiyed who motivated me for qualitative research), the various authors, researchers (refer References) whose excellent work I have referred, my organisation Saffrony Institute for their support, RK University and Dr. Nikhil Gokhale and last but not the least, my research participants for making this research paper publication possible.

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(Proceedings available for download at rku.ac.in/icre)

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Innovation, Restructure, Reorganize: Challenges for Indian Incorporation

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ABSTRACT

As the whole world is limping in the current recession, now all of us need to re-think about how to gain the glory achieved in the past. We all need to change ourselves to survive successfully during this time. There are major challenges in our country which are inevitable and Indian incorporation have to curb them and provide the solutions to enhance the productivity of employees, enhancing the business life cycle and reviving the economy as such. This paper describes on the three major challenges namely Innovation, Restructure and Reorganize which are ahead of our nation and by concentrating on these things; our country can reach to greater heights.

SUMMARY

Indian Incorporations are facing lots of challenges out of which this paper tries to cover the discussions on three major challenges mentioned above.

Key words: Innovation, Restructure, Reorganize

INTRODUCTION

More and more countries have become increasingly multicultural. Increasing differences in environment including globalization, changing regulations and innovation coupled with changing stakeholder expectations, brings risks and opportunities for Indian Inc.

In words of Jack Welch, "Globalization requires reinventing everything – to think of ourselves as compared to others". Hence in the era of Globalization, being competitive is a necessity.

Indian Inc. is headed by gigantic business houses like TATA, RELIANCE, BIRLA, JINDAL GROUP, ADANI etc. which are ruling the roost of our Indian Economy and it also comprises of Small & Medium Enterprises which form the backbone of growth. There are about 3.3 million SMEs in India which contribute nearly 7% of our GDP. Indian juggernauts having their kitty full with money can cope up with the emerging trends and paradigm shifts. It is possible for a company to move its plant from Singur to Sanand if the name is TATA, had it been any SME the incident would have ruined many expectations of society. The top layer of Indian Inc. is better placed to adapt to changing environment since they have resources and expertise at their disposal. In contrast the SMEs have to risk their ideas. Innovation for them is a gamble; it can either pay off or fail miserably.

The future is bright for those who think ahead of the times and move with confidence. The challenges that come on the way should not be seen as obstacles but they should be considered as opportunities. Similarly in this world of snowballing rate of change, the opportunities for Indian Inc. lies in excelling by managing change through Innovation, Restructuring and Reorganizing.

Innovation may be defined as, "Competing against the Present for a better Future". Innovative firms can develop and get new market opportunities. Product introductions have increased in recent years. Innovative companies create a positive attitude toward innovation and risk taking, generalize their business process, practice teamwork, and allow their people to experiment and even fail. [1]

Our approach towards innovation is rather unique. Today, Indian companies have taken over the role of providing patronage to creative individuals – in return the individual's creative pursuits are aimed for overall benefit of the organization and society. When anyone thinks of innovation the companies which come to mind are like Apple, 3M, Google, and Sony or Perhaps DuPont, Starbucks and Virgin. Rarely does the name of any Indian company come up at first thought, or even as second thought. When prodded, most people will scratch their heads, think and guess names of one or two Indian companies. 1980s-1990s were the decades of *Japan & Quality*; can 2010s-2020s be the decades of *India & Innovation*? The question is can we use Innovation as a spring board to tackle the challenges India facing today? India is known as a country of "Jugaad" as they say in Hindi. But that has begun to change. There are enough examples in this country that has achieved breakthrough innovation and turned industry norms upside down to pull off impossible in their field. [3]

Indian manufactures have focused on delivering low cost products to previously untapped markets by innovating to lower costs and create new delivery mechanisms- as with Dainik Bhasker delivers the Newspaper in different languages, in desirable prices and in top quality paper and color combination and it is India's largest selling Newspaper from day one. E-Choupal, an initiative by ITC has integrated those who are at the bottom of India's pyramid. [4]

In a changing world, no enterprise can expect to gain and maintain its success, without constant innovation. Past, or present success, is no insurance against future failure. Continued renewal is the key to success.

MATERIALS AND METHODS

Exploratory research approach was adopted to explore various cases of the conglomerates. Secondary sources of data like journals, books and case studies were referred to explore major three challenges of current organizations: Innovation, Restructuring and Reorganizing.

RESULTS AND DISCUSSION

Innovating Culture

The essence of competency lies in its sustainability, so innovation should not be restricted to new product development but it should be a core process of the organization. The first step towards innovative culture is to develop trust within the organization. Organizational structure is based on command and control whereas in innovative culture the perquisite is free flow of ideas.

The key to innovative culture is the development of tactic knowledge in an organization. The experience and skills of employees cannot be documented rigidly. Thus the emergence of a culture where employees are free to express and experiment will ultimately lead to innovation. Employees should not be severely punished for their mistakes but at the same time achievement of employees should be praised. Even a small tap on the back does wonder for the confidence of employees. This creates bonding of an employee with the organization.

Company can think about developing a culture of personal and corporate innovation by *Jim Carroll's* "Six C's of Innovation." i.e. Curiosity, Creativity and rebellion, Collaboration, Change, Courage and Creating excitement every day. [5]

JIM CAROLL'S 6 Cs OF INNOVATION



Creative organizations develop the system where curiosity increases they also motivate their people and to look for new opportunities everywhere. Those companies develop a collaborative culture in which information sharing is appreciated. They are familiar that success comes from change, not shying away from it. They also believe that true, real, sustainable success can only come from doing things differently, and that this in and of itself requires courage, because change involves risk. And last but not least, creative companies make work exciting and challenging as the staff within the organization many

times gets bored by their work. And, all this help an organization to build up a strong and focused culture of Innovation and creativity.

Restructure: Adapting changes in the world industrial scene

Organizational restructuring brings changes in decision making, information flow and management style. It requires changing the belief of their personnel. This is called learning. Organizational performance ultimately rests on human behavior and improving performance requires changing behavior. Since the world is changing, continuous organizational learning is necessary to stay up to date. Organizations that cannot learn will become obsolete. Leaders should periodically examine the organizational structure of their enterprise to assure that it continues to provide an environment for organizational learning. It also includes centralization or decentralization of organization, training and redeployment, changes in HR policies, rationalization of pay structure, financial restructuring, etc.

Basic business processes reengineering, in an enterprise, is crucial for achieving breakthrough improvements in customer-oriented performance. Its objective is to uphold quantum leaps in performance of the process, in terms of time, cost, output, quality, and responsiveness to customers. Reformation of Indian Fresh Vegetable Industry have made survival of Mom & pop shops or Kiranawala's stores almost impossible into Metropolitans and cosmopolitans and farm to fork system adopted by Reliance Fresh is eating out the businesses of wholesalers and many more intermediaries.

Reorganize: Aligning resources for growth

Reorganizing will assess business processes and policies. The activities which are redundant or do not add value will be eliminated during reorganizing. Only then all activities will be directed towards achieving a common goal and there will be alignment

of resources. The organization needs to unfreeze, change and be dynamic to accommodate changes.

The important analysis before reorganizing include understanding current processes, market needs, identifying bottlenecks, study of successful and failure cases of organizations which have undergone reorganizing. Reorganizing will be instrumental for rightsizing the organization, improved and speedy business processes, better relations and synchronization of the supply chain and improved product experience to customer.

Resistance to change is a critical concern for organizations before committing themselves for a change. The actual problem for resistance arises from the belief of people that change will give them some discomfort, so they confront it. In order to bring change in an organization first of all the employees must be taken in confidence that change is for their betterment and in the interests of the organization.

There should be goal congruency between the Corporate Vision and Business Activities. The personal goal of employees should be in line with the organizational goal. The top management must regularly review its strategic plans and assess its conformity with current business activities.

The first step towards reorganizing the core as well as support processes includes documenting the supply chain activities and analyzing which activities add value to the process. If need be decisions about outsourcing certain activities can be taken. The external parties should be given a chance to express their views about the organizational activities and their suggestions to improve them. There should be free flow of information in the supply chain of an organization.

Managing *YO-YO* situation and snowballing rate of change

In order to sustain in the competitive world the Indian companies will have to transform the common into unique. The companies just cannot rely only on core competencies but focus on building edge competencies. Organizations today, have no choice but to try to be competent in capabilities that are appropriate for coping with environmental turbulence, complexity, ambiguity and uncertainty in a regular manner. Organizations need to acknowledge and accept high level of volatility and uncertainty and live with stress of unstable and unclear situations. They have to accept errors and use them as learning opportunities as well as to anticipate and visualize the future, and map it against their desired vision.

Today, knowledge, technology, and managerial practices, that provide competitive edge, are becoming short-lived. Many of techno-scientific, geo-political, and socio-economical trends and forces affecting organizations are increasing. But how organizations respond to this fast-paced change, will largely determine their survival and success.

To achieve an advantage, firms can outsource material from one country, process it in another, and market it, anywhere in the world. They can create tie-ups, and networks, with enterprises across industries and nations, to build on each other's strengths; and improve, or, sustain their competitive position. To be competitive, a company would increasingly need to know what is changing. To remain competitive over the long term, the company would further need to know, how and why change is occurring, and understands the relationship between change and competitiveness.

An enterprise has many dimensions – culture, strategy, education, information systems, infrastructure, technology, human resource and they all need to cohere together. If parts of the organization system are not considered in concert, they will inevitably clash. When the entire enterprise system is not balanced, organizational change is reduced to a mapping exercise. Organizational change should be future oriented. It should increase organizational capacity to meet future challenges. It may require both short and long term measures to galvanize the organization, and measures to consolidate and build on change-induced success. [5]

CONCLUSION

In the past, managerial competency implied the possession of specific skills and abilities. Today it involves the development of a mindset that enables managers to confront, understand, and deal with a wide range of factors and forces, within and outside their organizations. The enabling mindset, in turn, rests on curiosity, creativity, learning, and a continuing critical examination of implicit assumptions and beliefs that guide decisions and actions.

People give their own meanings to their environment in terms of their mindsets. The second part consists of know-how, beliefs, frames of reference, and learning. Mindsets of people develop overtime, and tend to become stable or fixed. People need a new mental direction and amplified conceptual skills accustomed to environmental complication, ambiguity, and quick change.

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Psychological Contract Fulfillment and Employee Satisfaction

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ABSTRACT

Psychological Contract consists of perceived mutual obligations of employer and employee. **The purpose** of this research is to explore relationship between Psychological Contract fulfillment and Employee Satisfaction. Unilateral approach of measurement is adopted. Data is collected using **structured questionnaire**. Samples are 100 employees all belonging to white collar jobs of power generation sector. This study has been conducted in the region of Kutch (Gujarat) .**The results** of this research study indicated that there is as such no significant relationship between Psychological Contract fulfillment and Employee Satisfaction for these particular employees of this particular organization. The obligations are not being fulfilled fully by both the sides, i.e., employer and employee sides but employees are more or less satisfied. Researches done in Indian context and Indian Industries is relatively lower than foreign context.

SUMMARY

The research study aims to explore the relationship of Psychological Contract Fulfillment and Employee Satisfaction in special context of Power Sector of Kutch region.

Key Words: Psychological Contract, Employee Satisfaction, Indian Context

INTRODUCTION

The concept of Psychological Contract emerged almost fifty years ago as addendum while understanding OB. The concept refers to the perceived implicit mutual promises and obligations between employee and employer.

According to Conway &Briner, psychological contract gained accolades in past 20-30 years. These recent changes witnessed major shifts in the expectations and beliefs of employee as well employer in organization setting. Traditional transactional relationship was put to a test. And concept and its importance were illuminated. Denise Rousseau in his publications in 1989, 1990 and 1995 emphasized on unilateral approach of Psychological Contract and focused on employee's perception about mutual obligation and expectations between employer and the employee. Psychological Contract being relatively neglected area in Indian context[1], the study has its own importance in the arena of employment relationships.

This study arises due to the change in the nature of employment contract from relational to transactional. Gone are the days when employees were loyal to organizations and were willing to spend their entire career life in those organizations. Nowadays employment contract has economic exchange framework. It is necessary to understand what employees and employers expect from each other to increase employee retention, satisfaction and commitment.There are many matters which are uncovered in the formal contracts. This gap is fulfilled by Psychological Contract, which is base for employment relationship. Fulfillment of this contact in a way leads to employee satisfaction, commitment and intent to stay.

Fulfillment of Psychological Contact is essential for harmonious employee relations. Many of the researchers have tried to explore and establish the linkage of Psychological Contract fulfillment with various aspects like employee motivation, job satisfaction, work-life balance, organizational citizenship behavior, retention, etc. [2-4]

The purpose of this study is to explore whether Psychological Contract fulfillment has any relationship with employee satisfaction or not.

MATRIALS AND METHODS

As stated above, the research study aims to explore the relationship between Psychological Contract fulfillment and Employee Satisfaction. Employee Satisfaction is a construct that consists more than one concept. So the study would also analyze whether Psychological Contract fulfillment has any relation with various factors of Employee satisfaction or the relation is not significant.

Employee satisfaction has positive and significant correlation with the following six factors: (1) Autonomy/Influence, (2) Challenge, (3) Performance Measures, (4) Feedback, (5) Instrumentality, and (6) Stability/Security. [5]Many of the researches have included 'Working Environment' as one of the classic factor of employee satisfaction. Many other researchers say that this factor is very much influencing on employee satisfaction. Performance measures and feedback factors are combined as one factor named 'Performance Management System' and following hypothesis are framed:

Hypothesis 1: Psychological contract fulfillment has no relation with employee satisfaction.

Hypothesis 2: Psychological contract fulfillment no relation with employee autonomy/influence.

Hypothesis 3: Psychological contract fulfillment has no relation with job challenge.

Hypothesis 4: Psychological contract fulfillment has no relation with fair Performance Management System.

Hypothesis 5: Psychological contract fulfillment has no relation with job instrumentality.

Hypothesis 6: Psychological contract fulfillment has no relation with job stability/security.

Hypothesis 7: Psychological contract fulfillment has no relation with Working Environment

After collection of secondary data, primary data is collectedfrom managerialand non-managerial employees of power generation sector in the region of Kachchh (Gujarat). The sample size is 100 employees and the data is collected with the help of structured questionnaire. Sampling method adopted is convenience sampling. Questionnaire contains two parts. First part Psychological Contract fulfillment are taken from the 'Psycones, 2005', which is one of the four measurement instrument for Psychological Contract prescribed by Freese and Schalk(2008).The questions regarding Employee Satisfaction are derived from the Immediate Manager's Index (IMI) and the Overall Satisfaction Index (OSI) [6-7]

After developing the questionnaire, first Pilot Survey was done. The respondents were from different departments of the organization. The sampling method used was convenience sampling. As respondents were able to fill the questionnaire without any difficulty and no queries came up, final survey was carried out. All the respondents were associated to the group of white-collar jobs.

RESULT AND DISCUSSION

The Cronbach's alpha of reliability test of the instrument is 0.856. To test the hypothesis mentioned above correlations are found. The results are shown below.

	Employee Satisfaction	**Autonomy/ Influence**	**Challenge**	**Fair Performance Management System**	**Instrumentality**	**Stability/ Security**	**Working Conditions**
Psychological Contract (Pearson Correlation Sig- 2 tailed)	.021	.020	0.126	-.073	.128	.095	-.172

The results of the correlations indicate that there are no enough evidences to reject all the null hypothesis mentioned earlier. Hypothesis 3, 5 and 7 can be rejected as there is partial relationship between Psychological Contract fulfillment and Employee Satisfaction factors, i.e., Challenge, Instrumentality and Working Conditions respectively, but the correlation is insignificant.

Researches support that Psychological Contract Fulfillment has positive relationship with Job Satisfaction. The Purpose of this study is to explore the relationship between Psychological Contract and Employee Satisfaction.

The responses of the employees indicate that the obligations and promises from both, employer and employee side are not fully kept, still employees are more or less satisfied. This is also reflected in the responses by employees regarding emotions related to employment relationship. Most of the respondents rated high on positive emotions. This supports the argument by

Rousseau and Schalk that Indian white collar employees are less likely to express their perception of violation even if they feel that the employer has not kept the deal.

The results of the study contradict with the literature. The results indicate that there is as such no significant relationship with Psychological Contract with Employee Satisfaction for these particular employees and organization.

The study shows the results for Indian context, industry and employees are different from foreign context mentioned in the literature.

CONCLUSION

Most of the other related researches are exploring relationship between Psychological Contract and Job Satisfaction. Not much literature base is available specifically for linkage with Employee Satisfaction. The number of researches done in the Indian context is comparatively lesser than foreign countries. There are some prescribed instruments to measure the Psychological contract fulfillment. Most of the instruments are from foreign researchers, used in foreign countries and industries. The use in the Indian context, in Indian industry with Indian employees can have different result than that is in foreign context.

AKNOWLEDGEMENT

For success of the research study, I first and foremost would express my sincere gratitude towards the organizations that granted me the permission to take the responses and the respondents,without whom this study would nothave been possible. On above that I would thank my research guide and RK University for continuous support.

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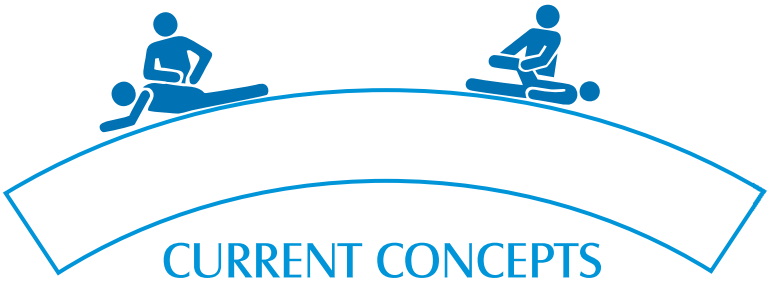
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SECTION NO. 2: MEDICINE RESEARCH

Physiotherapy is a speciality that remedies impairments and promotes mobility, function, and quality of life through examination, diagnosis, physical intervention, and prognosis. Just as many aspects of the modern health care system are changing, physiotherapy too is subject to significant shifts that will present both challenges and opportunities for patients and practitioners alike. Physiotherapy practice is currently achieving new heights because of the technological boom. Physiotherapists are now able to take advantage of the technological breakthroughs in determining the best therapeutic techniques for patient treatments. Physiotherapists in India have started getting better day by day and are steadily transitioning into world-class service providers of international repute. As research in physiotherapy is progressing, it is very important to keep ourselves updated with all of the recent advances, so as to be able to deliver the best to our patients and clients.





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ISBN: 978-93-5254-061-7 | www.rkuconference.org | JAN. 5 & JAN. 6, 2016

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A study to find the effectiveness of Mulligan's mobilization along with conservative exercises on pain and functional activity in patients with knee osteoarthritis: A randamized controlled trial

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ABSTRACT

AIM OF STUDY
To compare the effect of mulligan's mobilization along with conservative exercise versus conservative exercise alone to reduce pain and improve functional activity in patients with knee osteoarthritis.

• PURPOSE OF STUDY
It is the need for the physical therapist to know the effectiveness of mulligan's mobilization for the management of OA knee

STUDY DESIGN:-It is an experimental study with randomized control trial.

STUDY DURATION:-4 weeks

OUTCOME MEASURES
i. Western Ontario McMaster University Scale [WOMAC]
ii. Timed get up & go test

iii. 6 minute walking distance

METHODOLOGY
This study is to be conducted with 50 individuals of knee osteoarthritis & subjects are randomly assigned into two equal groups. The pre-test measurement is taken for both the groups with WOMAC scale, Timed get up & go test and 6 minute walking distance. Group A received Mulligan's technique and group B received conservative treatment. At end of 4 weeks post test measurement were done.

SUMMARY

This study concluded that Mulligan's Mobilization along with conservative exercise is proved to be more effective in reducing pain and improving functional activities in subjects with knee osteoarthritis.

Keywords: Mulligan's mobilization, osteoarthritis, WOMAC scale, Timed get up and go test.

INTRODUCTION

Osteoarthritis (OA) is a degenerative, non-inflammatory condition causing disability in older age[1-4] and can affect mainly weight bearing joints[5-7]. OA is characterized by degeneration of articular cartilage, subchondral sclerosis and formation of osteophytes[8-10]. These changes often lead to pain, limitation of range and muscle function, restriction in activities of daily living, and decreased quality of life.

Osteoarthritis most commonly affecting the synovial joints[11]. Osteoarthritis or degenerative joint disease is most frequently seen in clinical practice. Patients with knee OA are managed in primary care, and they represent a large group seen by physiotherapists[12, 13].

Thirty three per cent of adults aged 53 to 85 years show radiology evidence of knee OA. According to Pennix, the knee is the second most affected joint. An estimated 12 to 45 million adults are suffering from osteoarthritis[14]. The prevalence of OA is

increasing, mainly due to increasing life spans, and the consequences of OA have a significant impact on society[15-17].

Radiographic changes are seen over majority of OA patient above 65 years of age. But the data from autopsy study indicates almost all the patients above 65 have evidence of osteoarthritic changes. [18].

The problem in OA mainly occurs during weight bearing activities such as walking and stair climbing and it improves at rest.[19]. As the disease progress, additional impairments are loss of joints mobility, decrease of muscle strength, postural deformities and instability[20,21]. Especially in elderly patients, OA has a major impact on their ADL activities and frequently leads to limitations of function.[15, 19, 22]

OA is described as a condition of cartilage degeneration, stiffening of the underlying sub-chondral bone (sclerosis), and active new bone formation (osteophytes)[23,24]. In later stages of disease, the pathological changes in cartilage and bone are followed by pathological changes in other tissues of the joint and its surroundings, such as synovial membrane, capsule, ligaments and muscles. This may lead to capsular restriction, instability of the joint and muscle atrophy[25-27]. These changes will often lead to a reduced load ability of joints, which will often result in functional disability.

For the rehabilitation of OA patients, exercise, life style modification, joint protection technique, medication and surgery are all treatment modalities that should be considered. Pain is the primary symptom of arthritis, and multiple treatment option are available to relieve pain and improve function[28]. The treatment designated for each patient may reduce pain, improve joint mobility and reduce functional limitation[29].

Some researchers have showed that physiotherapy treatments are effective in controlling pain and improving activity in people with knee pain. These studies have included combination of patella-femoral tapping, muscle stretching, wax therapy, various pharmacological drugs, strengthening and coordination exercises along with techniques, various exercise protocols and recent advances like Manual therapy aimed

at decreasing tightness of the lateral structure and are individually responsible for the improvement of the joint[30-35]

Exercise is a commonly prescribed and effective treatment for patients with knee OA[36]. Exercise therapy helps to improve muscle strength, increase of joint range of motion, decrease of pain, and increase in functional ability[37-41]. A Cochrane systemic review of hip and knee osteoarthritis[42] defined exercise therapy as activities performed actively, passively, or against resistance. Participation in regular exercise is helpful in improving function and preventing disabilities. An exercise program for patients with knee osteoarthritis has been seen to improve quality of life by 40 to 60 per cent[12, 36, 43].

Few trials have investigated the relative benefits of differing approaches: systemic reviews of exercise therapy for the osteoarthritis knee have included randomize controlled trials, which are clinically diverse, with variability in the interventions employed[42,47,49].

Paraffin wax application may also be helpful in relieving pain and increasing range of motion of the joint[35].Heat therapy in form of wax and moist heat pack is used in osteoarthritis of knee to reduce pain and stiffness, and to increase circulation to the affected area, thus reducing pain and stiffness[50]. Heat may work by improving circulation and relaxing muscles, thus heat relieves pain and stiffness in arthritic joints[51-53].

Manual therapy is particularly aimed at the improvement of elasticity of the joint capsule and the surrounding muscles[54].

Mobilization with movement is the concurrent application of a pain-free accessory mobilization with active and/or passive physiological movement. Passive end-range overpressure may now be applied without pain as a barrier[56, 58, 59].

This new approach has been used to restore functional movements in joints (often in one treatment session), even after many years of restriction[57]. There are number of evidences supporting the clinically beneficial effects of Mulligan's mobilization with movement techniques. The most frequent reported effect is that of an immediate and substantial pain reduction accompanied by improved function[55].

Many studies have provided the evidence that accessory mobilization of an osteoarthritis knee joint immediately gives pain relief and restore function. It may therefore be an effective means of reducing pain in this population[60]. Claims of immediate reduction of pain free movement are associated with MWM techniques generally[56-58].

The purpose of the present study is to compare the effectiveness of Mulligan's Mobilization along with conservative exercise verses conservative exercise alone on subjects with osteoarthritis of knee in reducing pain and improving function.

AIM OF STUDY

1. To know the effect of Mulligan's mobilization in reducing pain and improving functional activity in patients with knee osteoarthritis.
2. To know the effect of conservative exercise in reducing pain and improving functional activity in patients with knee osteoarthritis.
3. To compare the effect of Mulligan's mobilization along with conservative exercise versus conservative exercise alone in reducing pain and improving functional activity in patients with knee osteoarthritis.

NEED OF STUDY

As osteoarthritis is the second most common rheumatologic problems, it is one of the five leading cause of disability among elderly women & men. So it is the need for the physical therapist to know the effectiveness of Mulligan's mobilization along with conservative management to reduce pain and improve functional activity in patients with knee osteoarthritis.

HYPOTHESIS

NULL HYPOTHESIS:-

There is no significant difference in reducing pain and improving functional activity in subjects with Mulligan's mobilization group as compared to conservative treatment group with knee osteoarthritis.

ALTERNATIVE HYPOTHESIS:-

There is significant difference in reducing pain and improving functional activity in subjects with Mulligan's mobilization group as compared to conservative treatment group with knee osteoarthritis.

MATERIALS AND METHODS

RESEARCH DESIGN

STUDY DESIGN: It was an experimental study with Randomized Control Trial**.**

SAMPLING DESIGN: Simple Random Technique

SAMPLING PATTERN: Single Blinded Study

INCLUSION CRITERIA:

AGE: 40-70 years

Unilateral/Bilateral knee OA

Exposure of osteoarthritis more than 3 months and less than 2 years

Grade II and III Kellgren and Lawrence radiological classification

EXCLUSION CRITERIA:

Recent soft tissue injury around knee joint

Any neurologic disorder

History of rheumatoid arthritis

Hip/knee joint replacement

History of knee surgery/ major knee trauma

Intra-articular joint injection in 4 weeks

STUDY POPULATION: Subjects who fulfilled above criteria are selected & taken as study population.

STUDY SETTING: Shree B.G. Patel College of Physiotherapy, Anand.

STUDY DURATION: 4 Weeks

OUTCOME MEASURES:

i. Western Ontario McMaster University Index (WOMAC)
ii. Timed Get Up & Go Test
iii. 6 Minute Walking Distance

APPARATUS:

i. Stop watch
ii. Plastic pylon marker
iii. Inch tape
iv. Armrest chair
v. Towel
vi. Sand bag
vii. Mobilization belt

METHODOLOGY

The study was conducted by taking 50 physically active subjects of knee osteoarthritis fulfilling inclusive criteria. Informed consents were taken from each subjects and were divided into 2 equal groups by using simple random sampling technique. The baseline measurement were taken to check pain and functional status of knee by using WOMAC Scale, Timed Get Up & Go Test and 6 Minute Walking Distance.

Group A (Experimental Group)

Group B (Control Group)

After baseline measurement Group A (Experimental Group) received Mulligan's Mobilization with conservative treatment for 5 days in a week for 4 week period and Group B (Control Group) received only conservative treatment for 5 days in a week for

4 week period. At the end of 4thweek post-test measurement were taken for both groups as similar to the pre-test measurements. The collected data were compared and analyzed.

- **MULLIGAN'S MOBILIZATION FOR EXPERIMENTAL GROUP A**

  GROUP A received Mulligan's Mobilization technique along with conservative management:

  Mulligan's Mobilization with movement techniques:

  Lateral and Medial glide in prone lying with mulligan belt with active flexion of knee. Subjects were instructed to lie in prone position on the treatment table. Belt was placed at the patient's lower leg so that the proximal edge is at the tibial joint margin on one side and the other side was tied with the therapist's waist.

  For applying the MWM medial glide, the therapist stands on the contra lateral side of the affected knee. The lower leg was supported with one hand and the thigh was supported above the knee joint with the other hand. The glide was performed by gliding the knee medially with the help of the belt and the patient was asked to flex the knee actively.

  For applying MWM lateral glide, same method was used, but the therapist stands on the same side of the affected knee.

  When the patients were able to flex further without pain, the overpressure was applied through the hands of the therapist. Based on the suggestions of Mulligan, Three sets of 10 repetitions were applied with 1 minute rest between each session. Five treatment sessions were given in a week and total of 20 treatment sessions were given in four weeks.

- **CONSERVATIVE TREATMENT FOR CONTROL GROUP B**

I.CALF STRETCHING EXERCISE:

Subject stands against the wall and asked to extend the hip and knee to be stretched. The other limb rest on the floor is comfortably flexed. then subject is asked to reach forward towards the wall without raising the foot of extended limb till the subject felt stretch discomfort in the calf muscles. The same procedure should be repeated on other limb.

Exercise Dosage/Progression:

2-3 repetitions each of 30 seconds duration.

II.HAMSTRING STRETCHING EXERCISE:

The subjects lie on supine, and therapist stabilize the contra lateral limb and **moves the stretching limb with knee extended position till the stretch** discomfort felt by subjects in the hamstring muscle. This procedure should be repeated on the other limb.

Exercise Dosage/Progression:

2-3 repetitions each 30 seconds duration.

III. PRONE QUADRICEPS STRETCHING EXERCISE:

The subjects lie on prone and the therapist stabilizes contralateral limb and instructs to extend the other limb with knee slightly flexed position till the subjects felt stretch discomfort in the quadriceps muscle. This procedure should be repeated on the other limb.

Exercise Dosage/Progression:

2-3 repetitions each 30 seconds duration.

IV. LONG SITTING KNEE FLEXION AND EXTENSION EXERCISE:

The subjects positioned in long sitting and asked to flex the knee by sliding the foot from the table towards pelvis and holds for 3-5 seconds and then asked to extend the knee by sliding the foot and hold the end position for 3-5 seconds.

Exercise dosage/ progression:

Repetitions are increased from a minimum of 10 to a maximum of 30 repetitions.

V. STATIONARY BICYCLE EXERCISE:

Subjects cycles on a stationary bicycle at a self-selected pace beginning at 1-5 minutes duration and progressing to 15 minutes, as tolerated.

Exercise dosage/ progression:

When the subject reaches 15 minutes on the stationary bicycle, the walking speed should be increased, as tolerated.

VI.STANDING TERMINAL KNEE EXTENSION EXERCISE:

The subject is in standing position facing towards suspension frame. The therapist position should be comfortable sitting position in chair or using step stool. The theraband applied around patient's knee & holds by therapist towards opposite of resistance direction. Then therapist hold the theraband and asked to extend the knee maximum as possible for 30 seconds and do it with other side.

Exercise dosage/ progression:

One set with 30 seconds hold, increase the resistance of theraband as tolerated by subject.

VII. SEATED KNEE EXTENSION ISOMETRICS EXERCISE:

The subjects is positioned in long sitting with knee rested on last degree knee extension board. The subject is instructed to press the knee against the knee

extension board and keep the knee in extended position for 3-5 seconds. The exercise should be repeated on the other limb.

Exercise dosage/ progression:

Exercise is progressed from 10 contractions to 30 contractions .

VIII.STEP UPS:

The subject is positioned in step standing. The therapist instructs the patient to hold this position for 30 seconds. The exercise should be repeated on the other limb.

Exercise dosage/ progression:

One set of 30 seconds hold the position. Increase step height as tolerated by subject.

All the exercises were given for minimum of 30 minutes duration for 5 session in a week for a period of 4 weeks. subjects were also advised to do home exercise program of the above mentioned exercise, except stationary bicycle, once a day, 10repetitions each.

RESULTS AND DISCUSSION

This study was done taking 50 subjects, 25 each in the experimental and control group. The pre and post test data within the groups were compared using paired't'test at 95% confident interval.

The post test data between the groups were compared using unpaired't'test at 95% confident interval

- **GROUP A : EXPERIMENTAL GROUP**

MULLIGAN'S WITH CONSERVATIVE EXERCISE GROUP				
OUTCOME	***PRE/POS***	***MEAN***	***t VALU***	***p VALU***

MEASURES	***T***		***E***	***E***
WOMAC	*PRE*	*59.7200*	*24.165*	*0.0000*
	POST	*26.1200*		
TGUG	*PRE*	*18.0744*	*16.22*	*0.0000*
	POST	*15.1672*		
6MWD	*PRE*	*15.0400*	*-29.837*	*0.0000*
	POST	*20.5200*		

The P-value for WOMAC scale is less than 0.05, so there is significant difference within the group and the null hypothesis is rejected.

The P-value for TIMED GET UP AND GO TEST is less than 0.05, so there is significant difference within the group and the null hypothesis is rejected.

The P-value for 6 MINUTE WALKING DISTANCE is less than 0.05, so there is significant difference within group and the null hypothesis is rejected.

Similarly the data of the control group were compared using paired‘t’ test**.**

- **GROUP B: CONTROL GROUP**

ONLY CONSERVATIVE EXERCISE GROUP				
OUTCOME MEASURE	***PRE/POST***	***MEAN***	***t VALUE***	***p VALUE***
WOMAC	*PRE*	*46.4000*	*23*	*0.00000*
	POST	*33.1600*		
TGUG	*PRE*	*18.3268*	*16*	*0.00000*
	POST	*16.8572*		

6MWD	*PRE*	*17.0400*	*-21*	*0.00000*
	POST	*18.2800*		

The P-value for WOMAC scale is less than 0.05, so there is a significant difference within the groups and the null hypothesis is rejected.

The P-value for TIMED GET UP AND GO TEST is less than 0.05, so there is a significant difference within the group and the null hypothesis is rejected.

The P-value for 6 MINUTE WALKING DISTANCE is less than 0.05, so there is a significant difference within the group and the null hypothesis is rejected.

To compare the effectiveness between the two groups, an unpaired't'-test were applied with 95% confidence interval.

- **PRE TEST VALUES OF GROUP A & GROUP B**

PRETEST OUTCOME MEASURES	*GROUP*	*MEAN*	*STANDARD DEVIATION*
WOMAC	*GROUP A*	*46.4000*	*46.40±13.40*
	GROUP B	*59.7200*	*59.72±11.15*
TGUG	*GROUP A*	*18.0744*	*18.07±3.94*
	GROUP B	*18.3268*	*18.32±4.09*
6MWD	*GROUP A*	*15.0400*	*15.04±1.83*

	GROUP B	*17.0400*	*17.04±2.87*

- **POST TEST VALUES OF GROUP A & GROUP B**

POST TEST OUT COME MEASURES	GROUP	MEAN	t VALUE	p VALUE
WOMAC	GROUPA	26.1200	-2.471	0.021
	GROUPB	33.1600		
TGUG	GROUPA	15.1672	-1.505	0.145
	GROUPB	16.8572		
6MWD	GROUPA	20.5200	3.114	0.005
	GROUPB	18.2800		

The p-value for WOMAC scale is less than 0.05, so there is significant difference between the groups and null hypothesis is rejected.

The p-value for TIMED GET UP AND GO TEST is not less than 0.05, so there is no significant difference between the groups and null hypothesis is accepted.

The p-value for 6 MINUTE WALKING DISTANCE is less than 0.05, so there is significant difference between the groups and null hypothesis is rejected.

RESULTS

The P-value for WOMAC SCALE, TIMED GET UP & GO TEST AND 6 MINUTE WALKING DISTANCE in experimental group is less than 0.05, so there is significant difference within the group and hence the null hypothesis is rejected.

The P-value for WOMAC SCALE, TIMED GET UP & GO TEST AND 6 MINUTE WALKING DISATANCE in control group is less than 0.05, so there is a significant difference within the group and hence the null hypothesis is rejected.

The p-value for WOMAC SCALE & 6 MINUTE WALKING DISATNCE between groupsis less than 0.05, so there is significant difference between the groups and hence the null hypothesis is rejected.

The p-value for TIMED GET UP & GO TEST between groups is not less than 0.05, so there is no significant difference between the groups and hence the null hypothesis is accepted.

DISCUSSION

The purpose of the present study was to compare the effectiveness of Mulligan's Mobilization and conservative management on subjects with osteoarthritis of knee.

Osteoarthritis commonly affects joints and leads to functional limitation. The main impairment with knee OA patients are pain and decreased functional activity. As disease progress additional impairments are loss of joint mobility, decrease of muscle strength, postural deformities and instability.

Many studies have been concluded to discover better treatment protocol or preventive method for the management of osteoarthritis of knee. Commonly used approaches were of patella-femoral-tapping, muscle stretching, wax therapy, exercises and manual therapy etc.

The result of this study rejected the entire null hypothesis and proved that the treatment with MWM technique significantly increases improves functional activity and decrease pain in the patients with osteoarthritis of knee. Similarly Chin Lee 1996 in his study found that the MWM technique has demonstrated greater improvement in pain sensitivity and functional abilities, where total 6 treatment sessions were given within 3

weeks of time. Also Moss.k.Sluka, A.Wright 2007 in his study found that accessory Mobilization of an OA knee joint immediately produces both local and widespread hypoanalgesic effect.

The reason behind the effectiveness of MWMs are based on mechanical dysfunction and therefore positional fault correction. More recently studies have investigated further mechanisms and effects that may underpin MWM techniques, including hypoanalgesic and sympathetic nervous system (SNS) excitation effects, and increasing joint range of motion, enhancing muscle functions.

The present study analysis also found that conservative treatment (exercise therapy) have significant improvement in reducing pain and improving functional activity in patients with OA of knee.

Exercise therapy is effective in reducing pain and disability in OA knee (M E VAN BAAR et al). The treatment period for the study was 12 weeks, with an ensuing 24 weeks follow up.

The findings of the present study confirmed that MWM technique is more effective then conservative treatment in patients with OA knee. The rate of improvement from MWM technique is similar to the other study (Chin Lee 1996).

Therefore this study reject null hypothesis and proves that MWM technique is effective than conservative treatment in patients with OA knee. As the statistical analysis of the study showed that there is more significant improvement in reducing pain and improving functional activity in the group treated with MWM technique.

CONCLUSION

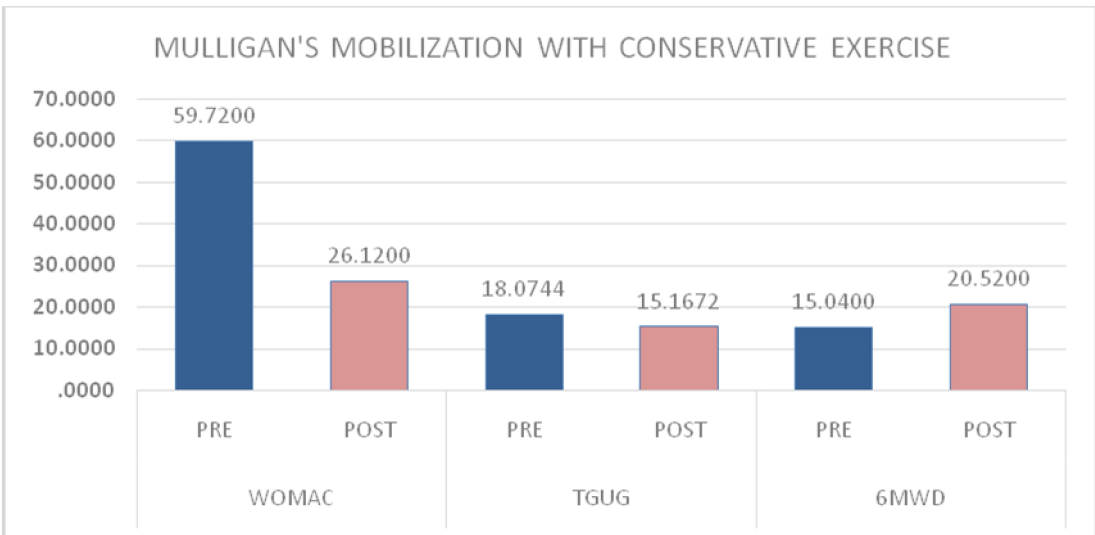
This study concluded that Mulligan's Mobilization along with conservative exercise is proved to be more effective in reducing pain and improving functional activities in subjects with knee osteoarthritis.

Thus, in patients with OA of the knee, Mulligan's Mobilization with Movement technique seems to be a suitable treatment option.

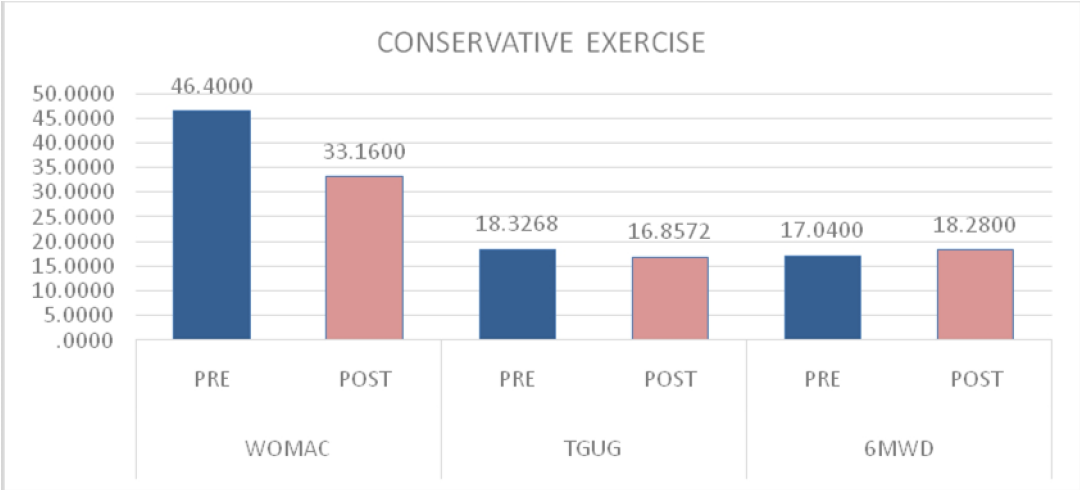
FIGURES

BAR DIAGRAMS

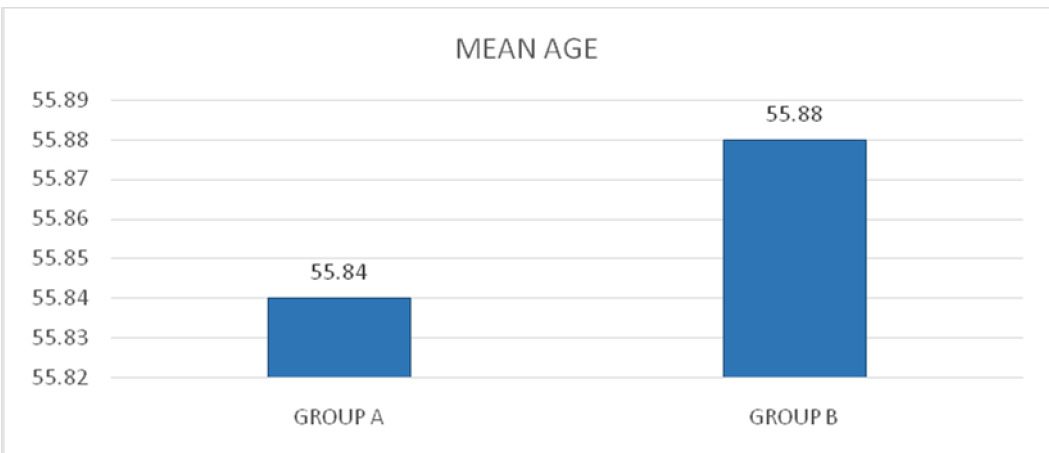
i. EXPERIMENTAL GROUP:-



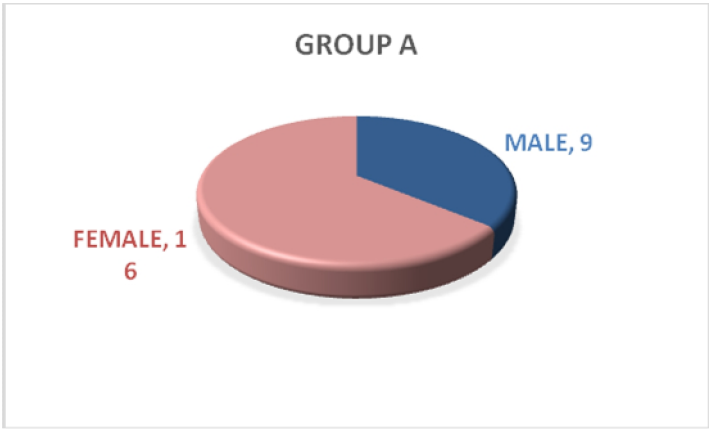
ii. CONTROL GROUP:-



iii. MEAN AGE OF GROUP A & GROUP B:-



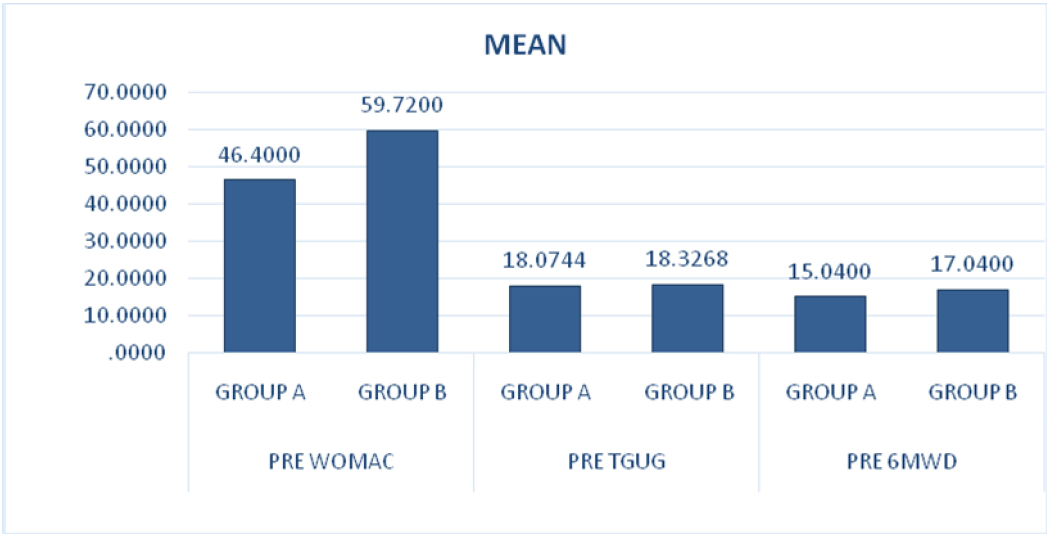
iv. GROUP A:MALE/FEMALE RATIO



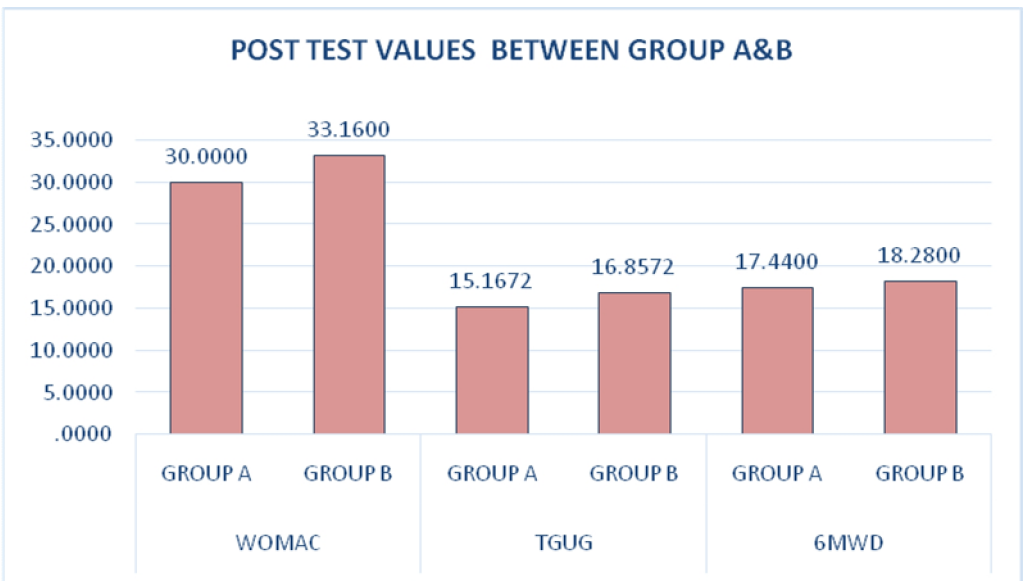
v. **GROUP B:MALE/FEMALE RATIO**



vi. **PRE TEST VALUES BETWEEN GROUP A & GROUP B:**



vii. POST TEST VALUES BETWEEN GROUP A & GROUP B:-



TABLES

- **MEAN AGE OF GROUP A & GROUP B:**

GROUP	*MEAN*	*STANDARD DEVIATION*
GROUP A	*55.8400*	*55.84±8.41*
GROUP B	*55.8800*	*55.88±8.34*

- **GROUP A & GROUP B: MALE/FEMALE RATIO**

GROUP	*FEMALE*	*MALE*
GROUP A	*16*	*9*
GROUP B	*12*	*13*

1. WOMAC	2. QUESTIONS	3. PRE TEST MEASURE 1ST DAY	4. AT THE END OF 4TH WEEK
PAIN	**1.**		
	2.		
	3.		
	4.		
	5		
STIFFNESS	**1.**		
	2.		
5. PHYSICAL FUNCTION	**1.**		
	2.		
	3.		
	4.		

	5.		
	6.		
	7.		
	8.		
	9.		
	10.		
	11.		
	12.		
	13.		
	14.		
	15.		
	16.		
	17.		

TIMED GET UP AND GO TEST:

PARAMETERS	PRE TEST MEASURE 1ST DAY	AT THE END OF 4TH WEEK
STARTING TIME		
FINISH TIME		
REST TIME		
ASSISTIVE DEVICE		
COMMENTS		

6 MINUTE WALKING DISTANCE:

PARAMETERS	PRE TEST MEASURE 1ST DAY	AT THE END OF 4TH WEEK
STARTING TIME		
ENDIND TIME		
REST TIME		
ASSISTIVE DEVICE		
DISTANCE (in meter)		
NO.OF ROUNDS		
COMMENTS		

ACKNOWLEDGEMENT

I am grateful to **Dr.Manojkumar**, Principal of Shree B.G Patel College of Physiotherapy, Anand, for his invaluable support and guidance throughout this study.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

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RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

A Research Paper on Women's Health

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ABSTRACT

Women's health has been so far one of the most neglected area of health. Actually women of our great nations do not consider their health as vital and simply shun such aspects saying we will see when time comes. There are more misconceptions about a women's health then the clarity regarding it
This paper aims at presenting the correct understanding about the women's health, current scenario of it. There are so many different perspectives about a women's health. Like wise there are so many misconceptions as well. Sadly enough the women of substance take their health as granted and simply overlook the aspect. All those different takes of women on women's health, men on women's health, working women on their own health and other women's health, house wife's take on the same along with some experts has been imbibed to present a varied still composed stand on women's health in India.

SUMMARY

Indian women are negligent about their health and probability of finding a healthy women out of 100 is less then 10%.

Keywords: women,health, fitness,BMI,ailments

INTRODUCTION

Today the situation has although taken a head turn, the concept of women's health till date remains as illusive as a mirage. Despite of having all modern and technological advancement the concept remains still untouched. The contention here is not to undermine the health care initiatives of government or any other organization but to deliberate the callousness of WOMEN OF SUBSTANCE about their own health.

After independence government of India ensured free primary health care facility to each citizen. Each village, each area, each locality has its own health care centre that looks after the general health of locals. Such facilities are available across the length of India and even in remote parts.

Free check ups of pregnant women and free vaccines and boosters to infants and children have incredibly increased the health standard of both- the girl child and boy child as well.

Increasing level of education, eradication of backward social stigmas, changing family pattern have cemented the place of a girl child as at par with boy child. Nowadays it is not the health of a girl which is questionable. Modern parents ensure that their girl child is given all the necessary medical as well as other types of attention and care.

The issue becomes critical after the girl attains women hood. That is when she gets married and starts working &/OR both. Her own health is the last priority or even a non existent priority of women of 21st century. Because they think that there is no need for putting in any extra efforts for their own health until any thing acute happens to them.

Secondly the life style hazards are so much there that they do not want to change it. For example all those women who have been working professionally also need to spend at least 30 minutes in morning sun. The reason being sheltered office life makes body deprive of sunlight which results in vitamin D deficit. Ask any women about this and an eternal excuse of 'not having time' would prop up.

There might not be any visible signs of ailment still an observant individual can sense a gradual corrosion of Indian women's health. From following classic instances the reader can relate to at lest one symptom or more for the women folk of his/her family.

1) Anemia
2) Tiredness
3) Breathlessness
4) Headache
5) Backache
6) Sleeplessness
7) Migraine
8) Joint pain
9) Knee pain
10) Difficulties in conceiving

MATERIALS AND METHODS

Personal interviews with respective experts on Indian women's health, book and article reviews, health bulletin and a questionnaire filled up by 100 females ageing 18 onwards.

RESULTS AND DISCUSSION

The result of the questionnaire survey strengthens the general notion of the experts that 1) Indian women are negligent al most careless about their health, 2) Education and professional background has not helped to increase women's health because ratio of unfit professional and literate women is same as that of unfit house wives and illiterate women. 3) Modernization, better standard of living has not made any impact in improving women's health.

CONCLUSION

Fitness just means going to gym. The overall concept of wellbeing which has healthy physical and cheerfully positive outlook towards life escapes Indian women like running water. The findings are alarming which states that hardly 10 women out of 100 can be said to be fit. Most of the women are either suffering from anemia, vitamin deficiency, bone corrosion. Finding a woman who has proper BMI is rare in Indian society. Even the page 3 celebrities who frequent gyms and yoga classes have some chronicle and sleeping and breathing disorders. Most of the house wives have same kind of issues and probably that is why in Gujarat women sleep in the afternoon to catch up on the night sleep.

FIGURES

"**Fig. 1**. A survey of women from different walks of life".

Insert here.

age	*literate*	*illiterate*	*married*	*unmarried*	*professional*
18-30	*10*	*10*	*05*	*05*	*08*
31-40	*10*	*10*	*10*	*00*	*07*
41-50	*10*	*10*	*09*	*01*	*05*
51-60	*10*	*10*	*09*	*01*	*04*
60 onwards	*10*	*10*	*10*	*00*	*00*

TABLES

Table 1. Quantitative health estimation of 100 females.

Females	Diabetic	Having pressure	Obese	Arthritis	Other ailments
18-30	3	3	4	-	12
31-40	5	5	7	2	01
41-50	11	6	8	7	02
51-60	16	12	14	16	05
60 onwards	17	15	14	14	11

Table 2: Qualitative health analysis of 100 females

Females	Improper BMI	Anaemic	Unfit	Depressed	Insomniac
18-30	7	20	16	11	12
31-40	19	19	7	11	09
41-50	16	19	8	7	14
51-60	16	12	14	16	19
60 onwards	17	15	14	14	11

ACKNOWLEDGEMENT

The author expresses her gratitude to all those experts from medical field who have given their invaluable insight into this research paper. Especially

Mrs. Rita Khatri, senior supervisor, Health & Drug Department, Gandhinagar,
Dr. Monica Bedi, Gynaecologist, Lilavati Foundation,
Mr.Khushwantsigh Sodhi, Alembic Drug Corporation, Women's Drug division,
Dr.Ujjwala Khengar, Nutrition consultant, National Health Care Division, Pune
Mrs.Shehnaz Pastonji, Co founder, VLCC,
Dr.Aarzo Zorabian, Department of cultural heritage of Gujarat, Bhuj, Asmita Gujratni-arvachin,
Dr.Anirudhha Chaudhery, head of the centre for rehabilitation of tribals,
Dr.Arbindo Ghosh, Indian Medical Institute for Research, Bhubenshwar,
Miss Kala Kishor, Yoga expert and teacher, Surat,
LT.COL. Naval Sharma, rt.col. of Indian Army, Tracking Coach and Adventure Sport Guide,
Mrs .Nilofer Rao, Psychiatrist, Agakhan Trust for Women's Welfare,
Mrs..Gulnar Rangrez, President of Women's Council for Food & Nutrition, Delhi.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

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A Validated 11-Item Multi-Component Exercise Program on Dizziness Caused By Benign Paroxysmal Positional Vertigo Individuals: A Pilot Study

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ABSTRACT

Background of the study: The concept of CNS and vestibular plasticity forms the physiological rationale for the multi component exercise program. To my knowledge the additional components which will add advantage on treatment of dizziness. Objective: The study aimed at investigating the effects of 11-item validated multicomponent exercise program on dizziness in benign paroxysmal positional vertigo patients. Methodology: The study was approved by the ethics in research committee of Dr.D.Y.Patil Vidyapeeth, Pune with ref no .DYPV/EC/361/15,and the study was register clinical trail registry of India with no: CTRI/2015/10/006308. The 21-items were screened by the expert panel and 11-items have statistically qualified and appeared to have a high potential utility as a treatment tool. And thus the 11-item validated multi component exercise program was used in study, treatment of dizziness caused by benign paroxysmal positional vertigo .Results: The obtained table values of dizziness handicap inventory and berg balance score when observed statistical values of T-test, it shows a significant. Conclusion: The study concluded that 11-item validated multi component exercise program shows statistically effective tool in the treatment of dizziness caused by benign paroxysmal positional vertigo

SUMMARY

11-item multicomponent exercise program was alternate treatment tool on dizziness caused by benign paroxysmal positional vertigo (BPPV) individuals of Indian Population. And it was cost effective and economic tool

Keywords: 11-items,Exercise Program ,Dizziness ,Benign Paroxysomal Positional Vertigo (BPPV) INTRODUCTION

INTRODUCTION

The use of visual and vestibular information for the control of posture is complicated by the fact that these sense organs are located in the inertially unstable head. Because the center of gravity of the head is located above its axis of rotation, any movement of the body results in head motion [1]. Uncontrolled head motion complicates the use of vestibular information to make estimates of body motion and position Proprioceptive feedback reaches the CNS from the receptors located in muscles and joints, vestibular apparatus in the inner ear and eyes, muscles and joint receptor are stimulated by movements of musculoskeletal system .The vestibular apparatus provides information on whole body position and is stimulated when upright posture changes ,The eyes help orient the head and body with respect to environment [5,6] .The concept of CNS and vestibular plasticity (compensation) forms the physiological rationale (logical basis) for the multi component exercise program [5-7]. When I did a literature survey it was found that in the most of the studies the exercise protocol commonly used was cawthrone-cooksey and Brandt daroff exercises, the exercises were performed at least three to five times a day and visit the therapist weekly or thrice weekly

To my knowledge the additional components which will add advantage on treatment of dizziness are neck movement, mental imaginary techniques, proprioception exercises, occulomotor exercises this was because every component is having a strong physiological connection with the vestibular system

Hence based on the literature of visual, proprioceptive and vestibular components, I have validated a multi component exercise program on dizziness in benign paroxysmal positional vertigo.

Objective of the study The study aimed at investigating the effects of 11-item validated multicomponent exercise program on dizziness in benign paroxysmal positional vertigo (BPPV) patients

MATERIALS AND METHODS

The study was approved by the ethics in research committee of Dr.D.Y.Patil Vidyapeeth, Pune with ref no.DYPV/EC/361/15,and the study was register clinical trail registry of India with no:CTRI/2015/10/006308.The exercises consists of 21 items based on retrospective of vestibular rehabilitation, the 21-items was screened by the expert panel and 11-items have statistically qualified and appeared to have a high potential utility as a treatment tool. And thus the 11-item validated multi component exercise program was used in study ,treatment of dizziness caused by benign paroxysmal positional vertigo .Total 8 patients were included in this study ,all the patients were explained about the study and informed consent forms were taken .

The inclusion criteria was age group is 18-65 years ,Both Males and females ,Subjects were diagnosed as benign paroxysmal positional vertigo (BPPV) by ENT or Physician ,Able to experiencing the dizziness for longer period of 3 months ,Able to transfer independently from sitting to standing and move independently ,Able to tolerate the exercises, The subjects whose dizziness handicap inventory score was16-52

Exclusion Criteria: Any neurological deficits that could influence posture or balance (like stroke, parkinsonism, ataxia etc.) , Any musculoskeletal disorders which affect the balance (like lower extremities fractures ,lower extremity muscle sprains etc.) ,Perceptual disorders ,The subjects whose dizziness handicap inventory total score was More then 52

And all the 8 patients were treated with the validated multi component exercise program which consists of 1.Straight head bend downwards and upwards 2.straight head ,and then turn head ahead 45 degrees towards the right 3.straight head ,then turn head ahead 45 degrees towards the left 4.close eyes and imagine the blank back ground 5.close eyes imagine the checker board or busy back ground 6.close the left eye ,and fix the right eye as the object moves 7.close the right eye and fix the left eye as

the object moves 8.single leg stance right side 9.single leg stance right side 10.heel and toe raises 11.pertubation (Figure 3)

The total duration of the treatment was 12 weeks; the patient was visit to the outpatient department 0, 3, 6, 9, and 12 weeks. During the first visits 0 weeks the patients was explained about the purpose and benefits of the program and explain what is expected from them exercises are to be done daily two times five days a week and the baseline score was noted by using the Dizziness Handicap Inventory and Berg Balance Scale ,and during next visits i.e. 3 ,6,9,12 weeks to the outpatient department the patient or attendant ask to demonstrate the exercise program to therapist ,therapist first observe the exercise program and made necessary adjustments to exercises and again therapist assess the patient by using Berg Balance Scale and Dizziness Handicap Inventory (i.e. 0,3,6,9,12 weeks).This study was blinded therapist and outcome assessor .

The effects of validated exercise program were assessed by using the statistical T-test spss 20.0 version. And the relationship between of Dizziness Handicap Inventory and Berg Balance Scale was assessed by using the spss version 15.0.

RESULTS AND DISCUSSION.

The mean values of age, height and weight of participants were 54.647, 165.38, and 73.00. Table 1 shows the mean and standard deviation of dizziness handicap inventory during the visits to the outpatient department i.e. 0,3,6 ,9,12 weeks were 51.15 (1.933) ,46.95 (2.23) ,43.5 (2.72) ,39.6 (1.61) and 35.9(1.77) (Figure 2).The mean and standard deviation of berg balance score during the visits to the outpatient department i.e. 0,3,6,9,12 weeks were 24.6 (21.95) ,29.6 (2.06) ,32.3 (3.32) ,35.82 (3.21) ,44.6(3.36) (Figure 1).The obtained table values of dizziness handicap inventory and berg balance score when observed statistical values of t-test, it shows a significant (Table -2) Hence it was recommended the 11-item validated multi component exercise program was statistically qualified tool in the treatment of dizziness caused by benign paroxysmal positional vertigo.

The study was performed to know the effect of multi component exercise program on dizziness in benign paroxysmal positional vertigo patients.The study have been shown the significant improvements of Dizziness Handicap Inventory Scores and Berg Balance Score ,the reason the improvement was validated exercise program was designed based on visual ,vestibular and proprioceptive components physiologically it plays a major role with connections of vestibular system[4,6,7].In the exercise program we have included head movements at various directions which stimulates the otolithic organs which was influence in maintain the balance, by repeatedly performing leads to vestibular adaptation.

The post treatment score of BBS showed improvement reason ,for that improvements in BBS outcome measure was addition of somato –sensory exercises.(Single leg stance and perturbation), that the somato-sensory system can detect changes and theoretically reestablish balance without vestibular information.

According to Horak states that at mid frequency ranges, the vestibular system is maintain balance but higher and lower frequency ranges of tilt, the somato sensory system is able to help[7]. It was also observed that the multi component exercise protocol was safe without any adverse effects for the patients presenting with dizziness caused by Benign Paroxysmal Positional Vertigo.

By using this multi component exercise program it was also observed that balance was also improved, markedly also observed the Dizziness Handicap Inventory-Emotional component .The reason was this exercise program was included the component of mental imaginary technique which helpful in relaxation and somato sensory component which helpful in improving the balance [2-4].

CONCLUSION

The study concluded that 11-item validated multi component exercise program shows statistically effective tool in the treatment of dizziness caused by benign paroxysmal positional vertigo. Hence it was also observed that protocol .This multi component exercise program was easy to perform, and also helps in balance training which prevents the falls prevention

FIGURES

Figure 1: Pre and Post Score of Dizziness Handicap Inventory, during 0, 3, 6,9,12 weeks of treatment

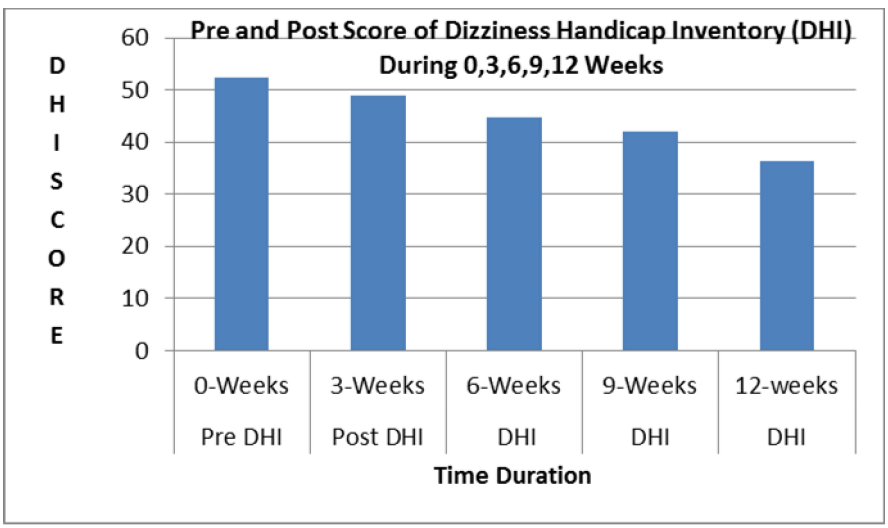


Figure 2: Pre and Post Score of Berg Balance Score, during 0, 3, 6,9,12 weeks of treatment

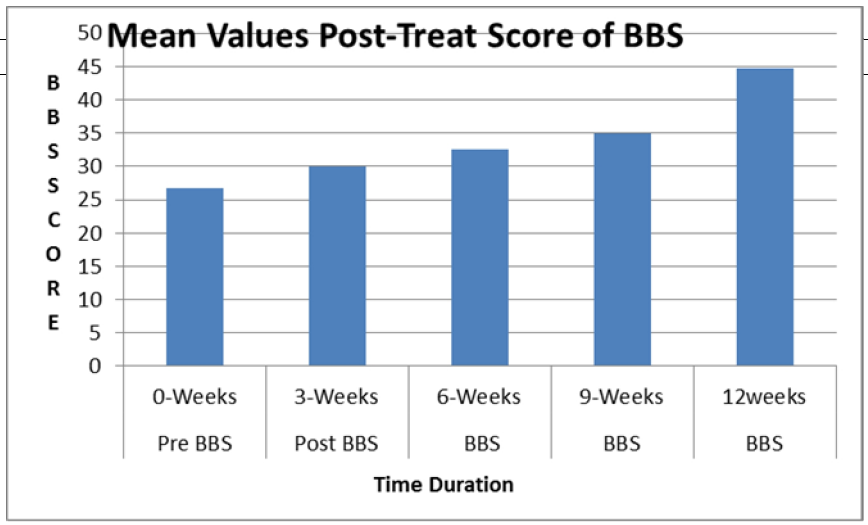
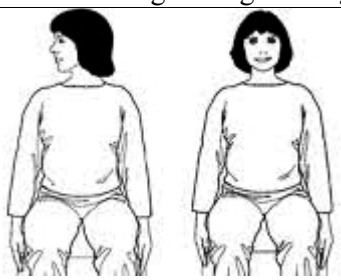
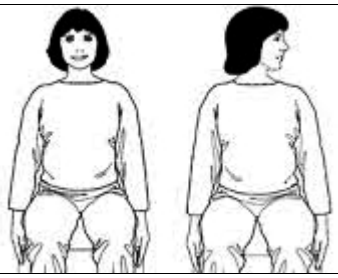
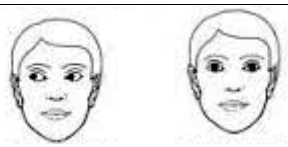
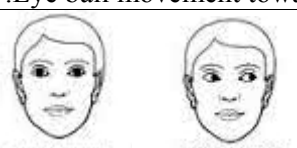
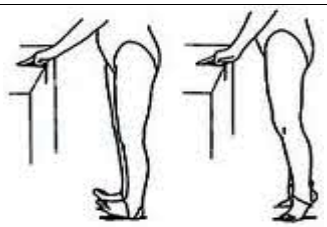


Figure 3:11-Item Multi Component Exercise Program used as potential tool for the treatment of dizziness caused by BPPPV Individuals

<table>
<tr><td>1.Straight head</td><td>2.Head rotating-45 degrees-Right</td></tr>
<tr><td></td><td></td></tr>
<tr><td>3.Head rotating-45 degrees -Left</td><td>4.Eyes closed imagining the blank background</td></tr>
<tr><td></td><td></td></tr>
<tr><td>5.Eyes closed imagining the busy or checker back ground</td><td>6.Eye ball movement towards right</td></tr>
<tr><td><table><tr><td>White</td><td>Black</td><td>Black</td></tr><tr><td>Black</td><td>White</td><td>Black</td></tr></table></td><td></td></tr>
<tr><td>7.Eye ball movement towards the left</td><td>8.Single leg stance: Right</td></tr>
<tr><td></td><td></td></tr>
<tr><td>9.Single Leg Stance -Left</td><td>10.Heel and Toe raises</td></tr>
<tr><td></td><td></td></tr>
<tr><td>11.Pertubation training : frontal plane</td><td></td></tr>
</table>

TABLES

Table 1:

Mean, Standard Deviation of 11-Item Multi Component Exercise Program BBS and DHI Score during 0, 3, 6,9,12 weeks of

	N	Mean	Std. Deviation	Std. Error Mean
Pre DHI 0 weeks	**08**	**51.15**	**1.933**	**0.336**
Pos DHI 3 weeks	**08**	**46.95**	**2.233**	**0.389**
Pos DHI 6 weeks	**08**	**43.5**	**2.721**	**0.474**
Pos DHI 9 weeks	**08**	**39.6**	**1.619**	**0.282**
Pos DHI 12 weeks	**08**	**35.9**	**1.77**	**0.308**
Pre BBS 0 weeks	**08**	**24.26**	**2.195**	**0.382**
Pos BBS 3 weeks	**08**	**29.6**	**2.066**	**0.36**
Pos BBS 6 weeks	**08**	**32.3**	**3.326**	**0.579**
Pos BBS 9 weeks	**08**	**5.825**	**3.215**	**0.56**
Pos BBS 12 weeks	**08**	**44.65**	**3.363**	**0.585**

Table 2: BBS and DHI Score Values 0,3, 6,9,12 weeks by using the statistical T-test

	Test Value = 0 .05					
	t	df	Sig. (2 taild)	Mean Difference	95%Confidence Interval of the Difference	
					Lower	Upper
Pre DHI 0 weeks	**110.194**	**7**	**.0**	**51.15**	**50.39**	**51.76**
Pos DHI 3 weeks	**103.335**	**7**	**.0**	**46.95**	**45.37**	**46.95**
Pos DHI 6 weeks	**93.174**	**7**	**.0**	**43.5**	**42.17**	**43.1**
Pos DHI 9 weeks	**170.35**	**7**	**.0**	**39.6**	**38.44**	**39.58**
Pos DHI 12 W	**165.859**	**7**	**.0**	**35.9**	**34.47**	**35.73**
Pre BBS 0 W	**114.601**	**7**	**.0**	**24.6**	**23.02**	**24.58**
Pos BBS 3 W	**135.369**	**7**	**.0**	**29.6**	**28.94**	**29.41**
Pos BBS 6 W	**94.48**	**7**	**.0**	**32.3**	**31.53**	**32.89**
Pos BBS 9W	**110.86**	**7**	**.0**	**35.825**	**34.9**	**35.18**
Pos BBS 12W	**118.457**	**7**	**.0**	**44.65**	**43.15**	**44.54**

ACKNOWLEDGEMENT

Nil.

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Patients' expectations: a necessary focus of care in physiotherapy

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ABSTRACT

Background: Patients' expectations have become a dominant component in patient management in health care now-a-days. Hence, this article aims to review patient expectancy constructs and items, modes of data collection and related studies.
Methodology: Articles on patients' expectations were collected from electronic database.
Results: We found that commonly used expectancy constructs in the literature were ideal, realistic, predictive, normative, structure and process, treatment outcome and self-efficacy. Expectancy items were related to functioning, complications and return to normal activities/work. Various modes of data collection are use of specified questionnaires such as PEQ and QEPP and interviews (telephonic/focus group). There is lack of studies on patient's expectations and physiotherapy care in India.
Conclusion: Necessary focus should be given to patients' expectations for physiotherapy care in India. Hence, a research was proposed to understand patients' expectations of physiotherapy care for patients with paraplegia in India.

SUMMARY

A research was proposed to understand patients' expectations of physiotherapy care for patients with paraplegia in India.

Keywords: Patients expectations, Patients expectations and physiotherapy, Patients expectations' and physiotherapy care in India, Patients expectations in physiotherapy

INTRODUCTION

Emphasis on patients' expectations had its impetus in late 1980's and 1990's. Expectations are defined as anticipation that given events are likely to occur during, or as an outcome, of health care. They are complex beliefs or values resulting from cognitive processes and influenced by previous experiences. They are also dependent on experience and social learning. Expectations can include wants, hopes, desires and anticipation (*1*).

Expectations could be desires, predictions, or related to health care structures like building, equipment or staff, or related to processes (the way physiotherapist interacts with the patient) or related to health outcome. It is a complex and multidimensional entity (*1*).

There are various taxonomies/classifications of expectations. One such taxonomy was proposed by Thompson and Sunol in 1995. According to them, there were four types of expectations: Ideal (desires, preferred outcome), Normative (what should happen), predicted (expected outcomes) and unformed (unarticulated). But this taxonomy was based on less integrated models. Additional taxonomy included value expectations (what patients would like to happen), predicted or expectancy probability (judgement about the likelihood of event occurring), process expectancy and outcome expectancy (belief that a specific behaviour will lead to a certain outcome). Key determinants of behaviour are outcome expectations and self-efficacy (*1*).

Expectations are generated based on personal characteristics, previous experience, role models/interaction with friends/relatives, physiologic factors and socio-cognitive factors.[1] Internet plays an important role in generating expectations now-a-days (*2*). This is depicted in figure 1.

Patient centred care has gained its importance in physiotherapy. It has been reported that physiotherapists must pay attention to patients' social and psychological aspects along with biomedical/diagnostic one. Physiotherapist must be an active listener and should be ready to discuss the issues raised by patients as they may have varying degrees of expectations (*3*).It has been reported thatpatients' expectation is an important predictor of patients satisfaction. Patients' satisfaction has said to be achieved when his/her expectations have been met or exceeded (*4*).

Physiotherapy profession not only requires the technical knowledge or knowledge regarding patients' condition but also to contact with the patient and understand what he/she expects from physiotherapy. Biomedical model alone is insufficient to understand the patient, but we ought to use bio-psycho-social model to understand him/her completely (*5*).Studies on patients' expectations of physiotherapy care in India are Scarce. Thus, the aim of this article is to review expectancy constructs, items, modes of data collection and studies related to patients' expectations.

MATERIALS AND METHODS

Data was collected using electronic database (google, sciencedirect, researchgate, pubmed). Included articles were on patients' expectations, patient's expectations for physiotherapy care and patients' expectations for physiotherapy care in India.

RESULTS AND DISCUSSION

The result and discussion regarding patients' expectations are as follows:

1. Expectancy constructs and items

Expectancy constructs are generalised, specific, ideal, predictive, normative, unformed, realistic, structure and process, outcome, treatment and self-efficacy. Expectancy constructs with their meanings are as shown in table 1.

Expectancy items include expectations for physical functioning, mental functioning, cognitive functioning, sexual functioning, functional independence, complications, health improvement, and return to normal activities/work etc (*1*).

2. Modes of data collection for patients' expectations

Modes of data collection for patients' expectations are questionnaires and interviews (telephonic/focus group interview). Bowling et al (*6*) in 2014 explored the development of patients' expectation questionnaire (PEQ) and examined its psychometric properties. Three scales were developed: pre visit ideal expectations, pre-visit realistic expectations and post visit expectations (met expectations). This questionnaire had 6 domains and 29 items. Domains of this questionnaire were structure of health care, process of health care, doctor-patient communication style, consultation and treatment/procedure performed, doctors' approach to information and treatment outcomes. This questionnaire was found to have good reliability and validity.

Tekielska et al (*5*) in 2012 constructed a questionnaire of expectation patient-physiotherapist (QEPP). This questionnaire has 12 statements and response is given in 5 point scale. Key variables of this questionnaire are understanding, support and informing.

Semi-structured interviews can be done telephonically or open ended interviews by means of focus group interview method. Focus group research is more helpful for participants to discuss their beliefs, perceptions and expectations.

Focus group research is a way of collecting qualitative data which essentially involves engaging a small number of people in an informal group discussion/s focused around a particular topic or set of issues.[7] Initially this method was used by market researchers to understand consumers attitudes and opinions but now in the past 20 years, it has gained its importance in the field of social science and recently in health care. An advantage of using a focus group research is that it is economical, fast and efficient method. It provides socially oriented environment and a sense of cohesiveness. It consists of 6-12 participants (preferably 8) and lasts for around 1-2 hours (*7*).

Online focus group has gained its importance in last 5 years. Main advantage of online focus group is that any patient in any part of India/World can participate with the help of computer especially the one who are living in the community. It saves travel time for physiotherapist and patient as well as finances associated with that. There are various softwares to conduct online focus group interviews such as itrack, visionlive, Zoom software etc (*8*).

3. Studies on patients' expectations and physiotherapy care

Wiles et al in 2002 found that communication strategies of physiotherapists need to be improved to encourage the realistic expectations of the patients with stroke. In their study, they found that there was a lack of encouragement from physiotherapist to achieve expectations while patients maintained high expectations for first three month after the insult (*9*).

Metcalfe et al in 2003 investigated patients' expectations in peripheral musculoskeletal conditions and its effect on treatment outcome. This was a two phase study and the findings suggested that the physiotherapist must be aware of the psychological aspect of their patients. Physiotherapy interventions may have an effect on patients' beliefs, cognition and perceptions (*10*).

Foster et al in 2010 while evaluating physiotherapists and patients expectations in case of knee osteoarthritis did not find any relationship (*11*). Hush et al in 2010 conducted a systematic review of patients' satisfaction with musculo-skeletal physiotherapy care. They found in their study that patients were highly satisfied with musculo-skeletal physiotherapy care in north Europe, north America, United Kingdom and Ireland. Key determinants of patients satisfaction were process of care and interpersonal attributes. An important finding was that patients' satisfaction was not consistently associated with treatment outcome. It becomes important for a physiotherapist to understand the determinants of patients' satisfaction (*12*).

Harvey et al in 2012 found that physiotherapists' and patients' expectations varied for walking at one year post SCI. This was really problematic, so there is a need to have better strategies (*13*).Dealing with patients' expectations improves rehabilitation adherence and helps clinicians to provide more individualised care (*14*).

Systematic review in 2015 found that physiotherapists give more preference to treat the mechanical aspect of pain, rather than dealing with the psychological factors also that improves recovery in case of low back ache. That's why they feel unprepared to deal with the multidimensional presentation of pain in case of low back ache (*15*).

There is lack of studies on patients' expectations and physiotherapy care in India. Hence, there is a need to have more such studies for better treatment outcomes. Limitation of this article is that only electronic database was searched and articles in language other than English were not included.

CONCLUSION

Modern science emphasizes not only at the diagnostic part of the condition but also at the psychological and social aspect of the patient. Studies have shown that dealing with patients' expectations improves patients' satisfaction and treatment outcome. Necessary focus should be given to patients' expectations for physiotherapy care in India. Hence, a research was proposed to understand patients' expectations of physiotherapy care for patients with paraplegia in India.

PROPOSED RESEARCH

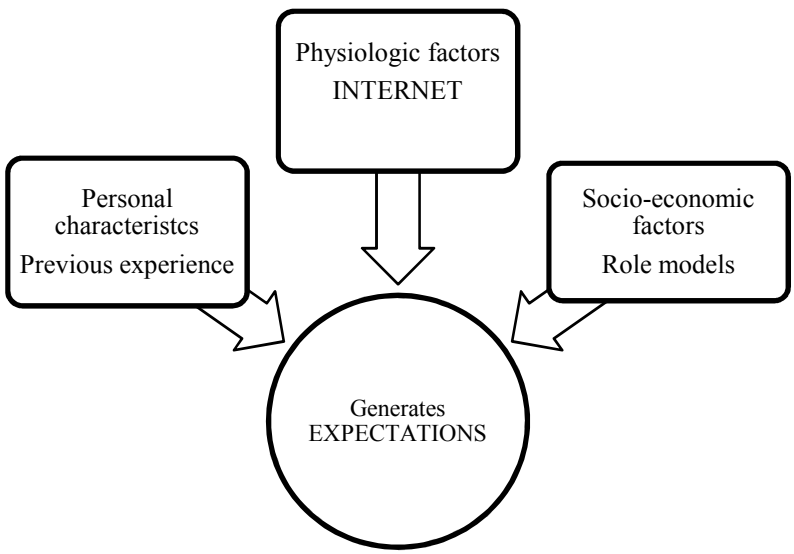
This study will be done by using a focus group research method. Focus group includes patients' and caregivers. Inclusion criteria of patients will be adults with paraplegia of traumatic cause (T6 below with adequate balance). Focus group must have at least 8-10 patients with paraplegia. Patients will be taken from the community. They will be asked for their consent. Once it is received, they will be asked for their demographic details and invited for group discussion. They will be interviewed in a group (online software for group discussion may also be used). Open ended questions will be put forward and responses of the individuals will be recorded.

Role of the researcher will be to facilitate the discussion. Field notes will also be taken during the interview. Summary of the interview will also undergo "Member Checking." Same procedure will be applied till data saturation. Once the data is available, it will undergo open coding, focussed coding and

generation of themes. Data analysis software like NVivo or ATLAS.ti may be used if easy to apply and budget permits. Report on patients' expectations will be compiled using narratives. Implication of this study would be patients' satisfaction and better physiotherapy treatment outcome.

FIGURES

"**Fig.1.** Factors in generating expectations"



TABLES

"Table1. Expectancy constructs"

Expectancy constructs	**Meaning**
Generalised expectation	where individual has little or no previous experience
Specific expectation	where individual has previous experience
Ideal expectation	desired or preferred outcomes
Predictive expectation	actually expected outcomes
Normative expectation	what should happen
Realistic expectation	what patients think will happen
Unformed	unarticulated
Structure and process expectation	related to buildings, equipment, staff and the way physiotherapist interacts with patient
Outcome expectation	belief that specific action will lead to certain consequences
Self-efficacy	belief in one's capability to perform to attain certain outcomes
Dispositional expectation	Relatively stable optimistic/pessimistic expectation regarding future outcome

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EFFICACY OF PLYOMETRIC TRAINING IN FOOTBALL PLAYERS

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ABSTRACT

BACKGROUND: Football is a sport that involves varying degree of kicking ball to score goal that requires high speed, agility and power. Plyometric is a Training that involve a muscle to reach maximum force in the short period of time.The training include movements consist of eccentric, amortization and concentric phases..**AIM**: To find out efficacy of Plyometric training in football players.**METHODOLOGY**:30 subjects were divided into two group using odd and even random method. Group A is experimental were given various plyometric exercise and Group B were given running and stretching exercise. **RESULT AND DISCUSSION**: Outcome measures T-Test, Hexagon Test and 10 m shuttle run Test were taken pre and post to plyometric training sessions. For within group analysis and between groups analysis was done. Analysis showed significant improvement in experimental group. These improve in agility and

speed. **CONCLUSION**: Plyometric training improves speed and agility in football players.

SUMMARY

Plyometric training should be included in season practice session to enhance performance.

Keywords: 1-Plyometric training,2-Football players,3 -agility,4-speed

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INTRODUCTION

Football is a sports game in that player should have a capacity to produce various powerful movements during 90 minutes of play. However, the movements including sprinting, jumping, changing direction, throwing, or kicking a ball occurring during football. Many of these activities also a highest rate of power development considering the short period span on the ground to produce power, such as sprinting, jumping or changing direction (100 milliseconds). Efforts to improve football performance often focus on techniques and tactics with the help of fitness and applied human body physiology. In the aerobic context of a football match, the most interesting events during a match are represented by high-intensity work such as Jumping, kicking and changing direction. Power is crucial for performance in sports in which changes in direction, acceleration and jumps are required. Agility is the ability to maintain or control body position while quickly changing direction during a series of movements. Agility training is thought to be an enhance the performance of motor neuron through neuromuscular conditioning and neural adaptation of muscle spindles, Golgi-tendon organs, and joint proprioceptors..Plyometric training involves exercises in which the active muscles are stretched prior to its shortening to increase the performance. Plyometric training complete the basic need of muscles to develop its maximum strength and speed of movement within short period of time.

MATERIALS AND METHODS

Subjects for the study were selected from YCC football Academy, Rajkot. The subjects willing to participate were asked to sign inform consent. Those players fulfilling inclusion and exclusion criteria were randomly divided into two groups. Division of players was done on basis of odd and even method .Group A included even players and Group B included odd players.

Outcome measures were taken before starting of training session. Three outcome measures taken were T-Test, Hexagon test and 10 meter shuttle run test. Training programm was of 2 weeks. Group A was experimental group included 15 players and group B was control group

FLOW DIAGRAM OF STUDY PROCEDURE

EXPERIMENTAL GROUP (GROUP A)	**CONTROL GROUP (GROUP B)**
PRETRAINING STECHING	RUNNING
PLYOMETRIC TRAINING	STRECHING
POSTTRAINING STECHING	-
2 WEEKS	2 WEEKS

RESULTS AND DISCUSSION

Here within Group statistical analysis was done by paired t test shows significant improvement in experimental group for T-test,10m shuttle run test and Hexagon test but for control group only Hexagon test is significant at 5% significant level.

For between group statistical analysis was done by Unpaired t test shows significant improvement in experimental group compared to control group..

TABLE 1. EXPERIMENTAL GROUP SCORE ANALYSIS

#	Mean	SD	t-value	p- value
T-Test pre	13.872	0.851	3.883	<0.001
T-Test post	12.368	1.578		
Hexagon Test pre	4.721	0.424	7.761	<0.001
Hexagon Test post	3.980	0.571		
10 m Shuttle Test pre	5.652	1.031	6.620	<0.001
10 m Shuttle Test post	4.349	1.014		

Table 2. Within group analysis control group

	Mean	SD	t-value	p- value
T-Test pre	**13.619**	**1.032**	**1.482**	**0.161**
T-Test post	**13.473**	**1.051**		
Hexagon Test pre	**6.204**	**1.004**	**3.896**	**0.002**
Hexagon Test post	**4.847**	**1.327**		
10 m Shuttle Test pre	**5.036**	**0.739**	**0.219**	**0.803**
10 m Shuttle Test post	**5.086**	**0.857**		

Table 3.Between Groups Analysis

	Mean	SD	Mean Difference	t- value	p- value
Group A T test	**12.368**	**1.578**	**1.105**	**2.257**	**0.032**
Group B T test	**13.473**	**1.051**			
Group A Hexagon test	**3.980**	**0.571**	**0.866**	**2.322**	**0.028**
Group B Hexagon test	**4.847**	**1.327**			
Group A 10 m shuttle test	**4.349**	**1.014**	**0.736**	**2.149**	**0.040**
Group B 10 m shuttle test	**5.086**	**0.857**			

Discussion:

The study was performed to find out the effect of Plyometric training in football players. The study showed there is a significant improvement in agility and speed of performance with Plyometric training.

All players were given equal training without any bias. Players having history of past injury which lead to instability and further injury were not included in study.

Plyometric is the main part of the most physical activities including running, jumping, hopping and throwing. It is an extremely successful method of transforming strength into power, Agility and Power. Result of our study showed significant improvement in agility and speed of players.

Plyometric exercises work on basis of two models: Mechanical model and Neurophysiological model.

In Mechanical model, elasticity of muscle tissue create a mechanical energy and that release when stretching of muscles is immediately followed by its normal action. This effect is similar like a stretching of spring.

In Neurophysical Model is working on principle of stretch reflex. Stretch reflex improve the activity of muscles which are working eccentrically and allows it to act more forcibly. This reaction result into concentric muscle action followed by eccentric muscle action.If Stretch reflex fail to work muscle action does not ossur immediately after pre-stretch.so both the mechanical model (series elastic component) and the neurophysical model (stretch reflex) improve the rate of force production during Plyometrics training.

All Plyometric movements involve three phases.

1. Pre stretch or eccentric muscle action.
2.time between pre - stretch and start of concentric muscle action or amortization.
3.Actual muscle contraction.

In football players , this last phase movement the players desires – the powerful jump or throw. This sequence of three phases is called the stretch-shortening cycle.

The adaptations result into muscle hypertrophy (increase in the cross sectional area of muscle fibers and number of muscle fibers) thereby increased muscle strength and muscle power. Increase in oxygen level and blood volume resulting in improving endurance and bone mass due to short and speedy mechanical loading.

CONCLUSION

Plyometric training should be included in season practice session to enhance performance .Also Plyometric training improves agility and speed. These are one of crucial components in professional football players.

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Physiotherapy in Disaster Management: Preliminary investigation

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ABSTRACT

Introduction:

Disaster either it is manmade or natural it leads to human, material, financial loss. The affected community needs help from community, government and different organisations to recover and rehabilitate back to pre-disaster status.
Disaster causes mobility impairment and disability. Physiotherapist's role in disaster has not been well investigated. Also it was found that physical therapists were not part of disaster management team.
Objectives:
To find what were physiotherapist's role in disaster?
To find scope of physiotherapy management in disaster
To find physiotherapy management in phases of disaster like preparedness, response, rehabilitation.
Method:
It was cross sectional study using telephonic interview. Participant were 20 physiotherapists working or have worked in disaster.
Results:
There are very few number of physiotherapist worked in disaster. Physiotherapists help in Disaster and their role was not exactly giving physiotherapy. They worked in emergency, cleaning wound, preventing long term disability.

SUMMARY

Role and Scope of Physiotherapy in Disaster management

Keywords: Physiotherapy, Disaster, Management,

INTRODUCTION

Disaster defined by International Federation of red cross and red crescent society as a sudden, calamitous even that seriously disrupts the functioning of a community or society and causes human,

material and economic or environmental loss that exceeds communities or society ability to cope up using its own resources(*1*).

The disaster leads to number if injuries, disability and human loss(*2*). In India both types of disaster are common natural and manmade disaster. In 2001 earthquake Kutch in India many people suffered from Disability. Physiotherapists are the specialist who can help these kinds of patients and help them to become independent functionally. After reviewing disaster and physiotherapy articles it was found that physiotherapists are presently not a member of Disaster medical team(*3*). They are not employed by organisations which are working in disaster. Even it is seen that the role of physiotherapist were not clear. Physiotherapists those worked in disaster worked voluntary. The question is what physiotherapist should do in disaster? Was not clear(*3*)

It some studies it was found that physiotherapists were helping medical team. They also helped to clean wound, transportation of patient. They also did some odd job like cooking and making temporary shelters for injured patient in relief camp(*4*). At present it becomes necessary to find physiotherapy role in disaster. The numbers of studies were suggesting finding scope of physical therapists in disaster.

MATERIALS AND METHODS

A cross sectional study was done. The researcher included physiotherapists working or worked in disaster through convenient sampling. Sample size is 20 Physiotherapists.

Semi structure questionnaire was filled using previous research. The questionnaire was filled by telephonic or personal interviews with participants. Interviews were conducted between May 2013 to June 2013. Participation was voluntary.

RESULTS AND DISCUSSION

Researcher could get 4 participants as the sample size for the pilot study.

Role of physiotherapists,

Past roles:

All four physiotherapists have worked mostly in Natural disaster only and three of them in Earthquake in Gujarat in 2001, where only one work in Geophysical disaster that is tsunami in 2004.

Generally the Physiotherapists who were participant were from different places in India.

Most of them had worked in Disaster management on some special assignment but were not employed by government or NGO.

One of them said “I was finance coordinator and my job was to distribute finances as per need of NGOs”

Whereas other said “I was sending and doing food and accommodation arrangement for PG students and Doctors”

Present role:

It was observed that all of agreed that presently physiotherapist should be part of National Disaster Management Authority, India. They said Physiotherapist should be involved in all Manmade and Natural disasters. One of them exclaimed “Disaster management course should be part of not only Masters but also Undergraduate”

Also one said physiotherapist can presently work as team leader, rehabilitation worker and policy maker in Disaster authority.

Future role:

Almost all agreed other than one participant that, for future Physiotherapists should know well on the Disaster. One participant also told that physiotherapist can also train population in future on disaster preparedness like monk drills, patient transfer etc.

We can also improve our role better and more clearly defined with experiences in disaster management if we are involved. Some suggested going for more research on the relevant topic of disaster management. Almost all agreed and suggested to come out with more awareness in disaster management planning.

DISCUSSION

In Disaster there are number of areas where physiotherapists can have his role. Physiotherapists can rehabilitate patient from acute and chronic injuries.

In this study it was found that physiotherapists did work in disaster but their work was not clear. While comparing with other studies where physiotherapists it was observed physiotherapists were doing management work which is not their role. It was found that their role is mainly in Rehabilitation phase not in Preparation or relief phase(*5*).

Physiotherapy in vital in disaster affected areas and it should be given for long term. Physiotherapists can help in patient examination, musculoskeletal injuries mobility, transportation and vocational rehabilitation. Physical therapy can be important for the patients who already suffering from disability before disaster(*6*). All physiotherapists who want to participate in disaster undergo disaster management training.

CONCLUSION

This study found that there is role of physiotherapy in disaster rehabilitation. The physiotherapists have significant work in rehabilitation phase of Disaster.

ACKNOWLEDGEMENT

I want to specially thank Dr.Priyanshu Rathod, Director, SOPT, RK University Rajkot for continuous guidance and supervision of this research work.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th& Jan. 6th, 2016)

(Proceedings available for download at rku.ac.in/icre)

Computer Based Developmental Pediatric Screening (CBDPS): Evolving Healthcare Entrepreneurship

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ABSTRACT

Background: To practice health entrepreneur effectively in the 21st century, computer is playing a key role to improve Quality of Care and Quality of Life of every individual. Physiotherapy intervention includes long-term re-habilitation where data collection and analysis plays vital role to set the therapeutic goal and prognosis in children with developmental delay. Developing a software to screen the developmental millstone will open new areas for entrepreneurship. **Objectives:** to design computer based developmental pediatric screening (CBDPS) software and review the scope of healthcare entrepreneur within it**. Materials**: Computer, Internet, assessment format, scales–GMFM, SSP. **Procedure:** The paper based developmental pediatric screening format was prepared and converted into software program which was taken for expert review by feedback system. **RESULT AND DISCUSSION:** The interdisciplinary approach and professional requirement of computerization is fertile to screen developmental delay. **CONCLUSION:** An integration of technology in health care system will open new opportunities to serve as health care entrepreneur.
SUMMARY: An integration of technology in health care system will open new opportunities to serve as health care entrepreneur

Keywords: computer, Developmental Screening, Entrepreneurship

INTRODUCTION

To practice health entrepreneur effectively in the 21st century, computer plays a key role to improve quality of care and quality of life of every individual. Physiotherapy interventions includes long-term re-habilitation programs where data collection, analysis and storage has vital role to set the therapeutic goal and prognosis. Frequent assessment and analysis of information from traditionally managed paper based Assessment system is time and cost effective. Computerization where software based assessment is new areas of practice in physiotherapy and rehabilitation science in India. Recruiting software engineer and designing a tailor made software for business will develop entrepreneurship among physiotherapists.

However, there are three major challenges has been observes *first*, shift from traditionally practiced paper based assessment to computerized software based data management system, *second*, develop a skill to design Computer Based Developmental Pediatric Screening (CBDPS) for Indian patients, *third*, high cost of international software and its business practice on Indian patients. In context to meet above three challenges or research questions by developing new era of health care entrepreneur in physiotherapy practice, we will develop a software and assess its applicability over paper based assessment with association of software engineer. However, assessment of "developmental millstone" will be taken as an example to meet the challenges. Review of Literatures: computer and paper *based data management:* Physiotherapists regularly record clinical information during normal daily practice to manage the treatment of patients and to make decisions on treatment most likely to produce expected outcomes.[1,2] Computer as an Information technology is becoming an essential tool for improving the clinical management of patients and assessing outcomes.[3] Researchers has given new terminology "Electronic Medical Records (EMRs)" which are computerize health information systems that collect, store and display patient information.[4] They are a means to create legible and organized recordings and to access health information about individual patients. Moreover, EMRs are intended to replace existing (often paper based) health records which are already familiar to practitioners.[5] Patient records have been stored in paper form for years and over this period of time, they have consumed higher space and notably delayed access to efficient health care.[6] The perceived advantages of EMRs can be summarized as optimizing the documentation of patient encounters, improving communication of information to clinicians, improving access to patient health information, diminished errors, optimizing billing and improving reimbursement for services, forming a data repository for research, quality improvement and reduction of paper.[7,8] Entering clinical information in computers as EMRs instead of handwritten records improves accessibility of the information. When EMRs include standardized data elements, which can be aggregated and stored in a health database from which the data can be exported for analyses.[9] even, researchers has define a clinical database as a collection of information from electronic medical records from many providers one purpose of which is research.[10,11]

Pediatric developmental delay screening: In context to serve interdisciplinary and develop computer based screening software as an example of new era of healthcare entrepreneur, Pediatric developmental delay screening includes Gross Motor Function Measure (GMFM) which is a clinical tool designed to evaluate change in gross motor function in children with cerebral palsy (CP) and short sensory profile (SSP) for sensory assessment has taken for computerisation.[12,13] One of the study on "Reliability and Responsiveness of the Gross Motor Function Measure-88 in Children with CP" and observed that Both are reasonable for measuring gross motor function in children with CP.[14,15] Evolving Healthcare Entrepreneurship: Entrepreneurship is always been an uphill task but never an impossible one if done with the right attitude and ethical values. Physiotherapists are managing wide speared channels of home health care services and developing physiotherapy & Rehabilitation centers as a health care administrator. Managing human resources and marketing remains a part of professional growth. In world of personalized medicine and therapies, technology via Health care entrepreneur remains essential part of the development.[17,18]Study Design: Observational Cross sectional study, Ethical approval: taken from shree Giriraj Hospital Research Ethics Committee, Rajkot (approved by CDSCO, GOI)

MATERIALS AND METHODS

Computer (laptop) with Internet, developmental pediatric assessment form, scales–GMFM, SSP. feedback form, **Minimum standards of experts group:** Paediatric Physiotherapists and Software Developers – With minimum one year of experience. **Method:** The paper based developmental pediatric screening format (PBDPS) was prepared by pediatric physiotherapists and converted into software program (CBDPS). Record of every interaction for reformation with experts and outcomes / solutions in designing CBDPS software was documented to develop a Guideline.To develop the comprehensive feedback form, advantages and disadvantages of both the methods from 20 pediatric physiotherapists were taken after making them to practice computer and paper based screening. To compare the PBDPS practice with CBDPS, feedback form was given to 60 physiotherapists and outcome was assessed. **DATA**

ANALYSIS: The feedback form includes 9 questions regarding the usage of CBDPS. The components are feasibility, reassessment, time, cost effectiveness, administration, use in research purpose etc. Scoring was, 1: Satisfactory (not good as paper based method) 2: Good (same as paper based method) 3: Better (better than paper based method) Mean of each question was taken.

RESULTS AND DISCUSSION: RESULT:

Constructing guideline / troubleshooting in making CBDPS was documented. Managing software designer and physiotherapists to develop CBDPS software and understanding the prospectus of business on CBDPS was major learning outcome of the study. The result of feedback form and their mean scores of CBDPS over PBDPS was analysis. The total mean score is 2.19 which suggest that overall CBDPS is significantly better than PBDPS. We have observed that administration (1.68 out of 3) and time taken (1.7 out of 3) has shown less impact for CBDPS as compared to PBDPS, due to hands on skill to practice CBDPS. Whereas, data analysis (2.45 out of 3), data storage (2.53 out of 3), transferring and editing data (2.63 out of 3), reviewing the condition periodically (2.65 out of 3) has shown efficient scores. Mean years of experience of participants (physiotherapists) is 1.6 years. **DISCUSSION:** It is always time-effective to reassess and compare periodically taken pediatric assessment / screening. CBDPS has an advantage to analysis the data more effectively as compare to data collection by PBDPS. Even with CBDPS, we can reassess only those components which we desire to focus or needed for therapeutic intervention. Hence comparison helps to know the areas of improvement / deterioration. CBDPS provides numerical values as output to get the status and set the goal. Calculation of scale in traditional (PBDPS) method is very lengthy and time consuming, While CBDPS automatically calculates and gives the result. On the other hand, administration, feasibility and time consumption has less impact of CBDPS. However, CBDPS requires computer and run-time assessment may be difficult with children getting distracted or requires paper based and letter-on converted to CBDPS. Average mean scores of the total are 2.19 which suggest that overall CBDPS is comparatively better than paper based method of assessment.

CONCLUSION:

The CBDPS software making guideline must be taken into consideration for physiotherapists who is interested to convert any clinical assessment from paper based format into computer based screening software. An integration of technology in health care system will open new opportunities to serve as health care entrepreneur. In context to broad spectrum of computer applications in medical science and availing advantages of information and technology, this paper is a model and can be taken into consideration to develop entrepreneurship in physiotherapy practice

FIGURES

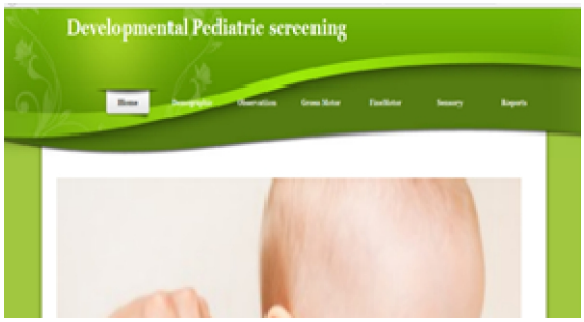


Fig. 1 – home page of web site

TABLES

Table 1. Customer's feedback score

1. Areas of feedback on CBDPS OVER PBDPS	2. Score (mean value - out of 3)
3. To ease the administration	4. 1.68
5. To have time effectiveness - saving	6. 1.70
7. Feasibility of use	8. 1.80
9. To have an applicability in research	10. 2.12
11. To have cost effective – saving	12. 2.17
13. To have data analysis and data mining	14. 2.45
15. To have data storage and retrieval	16. 2.53
17. To be able to edit and transfer the data	18. 2.63
19. Ability to review the condition periodically	20. 2.65
21. Mean	**22. 2.19**

ACKNOWLEDGEMENT

The researchers would like to acknowledge active participation and contributions from groups of expert including paediatricians, Paediatric Physiotherapists and Software Developers in developing a software for social wellness.

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Effects yoga on balance in geriatric population

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ABSTRACT

BACKGROUND:
Geriatric population defined as population aged 60 years and above. Due to aging processes, balance often is impaired, which can lead to dependency in ADL, falls and fractures.
Yoga can address risk factors like poor balance, impaired mobility, reduced strength and flexibility.
AIM: To study the effects of yoga on balance in geriatric population
METHODOLOGY
60 elderly of both genders were included from the old age homes of Ahmadabad and randomly divided in to 2 groups. Experimental and control, Experimental group performed yoga for 6 weeks, 6 days in a week. 5-10 minutes of warm up followed by 25-30 minutes of different asana poses and 5-10 min of relaxation. BBS and TUG scores were taken as pre and post data.
RESULT:
Statistically significant improvement in balance in experimental group
CONCLUSION:
Yoga can be applied clinically for improving balance in elderly

SUMMARY

Yoga therapy can be used to improve balance in geriatric population during clinical practice

Keywords: yoga, balance, geriatric, BBS, TUG test

INTRODUCTION

The geriatric population is defined as population aged 60 years and above [1]. There is no United Nations (UN) standard numerical criterion, but the UN agreed cut off is 60+ years to refer to the older population [2].

It is estimated that presently in 2011, there were around 600 million persons who were aged 60 years and above over all over the world. According to Indian Scenario of geriatric population 2011, 99 million out of 1.21 billion are over the age of 60, which was 77 million in 2001. According to an estimate, by 2021, India's elderly population will cross 137 million. Currently, India has the second largest aged population in the world [3, 4].

Life expectancy for the elderly in developed and developing countries has increased as a result of improvement in public health and medical advances[5]. Due to the increased longevity and life expectancy, the quality of life (QOL) has been considered as an important issue for aging individuals [6].

The performance of all activities of daily living requires good balance control while at rest or when moving from one position to another [7, 8]. Maintenance of the balance function is essential to stay physically active in life [9].

During quite stance balance is defined as the ability to maintain centre of mass (COM) within base of support (BOS) [10]. Achieving effective balance is a multi-system and multi-dimensional task [8]. Coordination of sensory, neural and musculoskeletal system is needed to maintain balance [7, 8]. Many of these systems undergo deterioration as people age [11, 12]. This can affect balance, restrict safe mobility and increase the fall and adversely affect quality of life [11, 13].

Balance problems in elderly are most commonly due to multi factorial condition which may include age related or disease-related declines in the balance system. Causes of reduced balance in elderly could be weakness in the core stabilizing muscles, altered muscle activation patterns, loss of proprioception, and an inability to control normal postural sway. Balance problems and falls are leading cause of institutionalization in this group [14, 15] which may cause major consequences like dependency in activities of daily living (ADL) [16].

The postural and equilibrium components of balance control ensure stability of the body during different activities. The exact demand of the balance control system is determined by both the task and the environment in which it is performed [8].

Balance assessment tools in older people most commonly uses simple clinical measures. Different measures have been designed to assess different aspects of balance or a person's balance ability under different conditions. These included measures of static and dynamic standing balance, utilizing tasks, such as stepping, reaching or leaning, and turning [17]. The Berg Balance Scale (BBS) was developed by Berg et.al (1989) which is widely used as clinical assessment tool for measurement of functional balance in elderly [18, 19]. Timed Up & Go test (TUG) is also commonly used to examine functional mobility, risk of falling, balance and general locomotion in community-dwelling, frail older adults [20, 21].

Yoga as a complementary therapy is thought to be more therapeutic than traditional exercise because it involves active engagement between mind and body [22]. According to Feuerstein yoga therapy aims to promote health and self awareness for the purpose of enlightment[23]. Yoga is an alternative approach to conventional exercise training and it also can be adapted to meet the needs of people with physical

limitations [24]. Yoga is a gentle form of exercise that has positive impact on physical, mental and emotional well being in older adults [25].

Yoga is a commonly practiced, mind-body approach which has major components like meditation, breathing, and activity or postures [22]. Increased muscular strength, flexibility, range of motion, energy, relaxation, and sense of well-being, decreased pain, improved sleep quality, reduction of stress, and control over physiological parameters are the presumed benefits of yoga therapy[26-30]. Yoga can address known fall risk factors (poor balance, impaired mobility, reduced strength and flexibility) and improved balance in older adults [25].

Although yoga is historical a spiritual discipline, it has been used clinically for therapeutic intervention. Since past 3 decades, the number of publications for clinical applications of yoga has greatly increased [31]. In literature there are many articles of use of yoga in variety of condition such as multiple sclerosis, rheumatoid arthritis, breast cancer, low back pain, migraine, epilepsy [26-35]. There are many reviews on the effects of hath yoga in rehabilitation after myocardial infarction, menopausal symptoms, diabetes, and hypertension [36-40].

Yoga and Balance

Yogasanas ranges from simple to complex body postures, along with controlled breathing. These asanas stretches major muscle groups and uses isometric contraction and relaxation of various group of muscles to assume static posture. [22].

Its practice has been associated with increased muscle strength, endurance, flexibility, range of motion and cardiopulmonary endurance. It mainly works on increasing body awareness and proprioception, which will lead to improvement of balance in older adults [28, 59].

There are very few studies establishing the effect of yoga on balance in elderly individuals. However, there is a lack of sufficient evidence that is dedicated to Indian geriatric population. Hence the need of the present study is to determine, whether yoga practice for duration of 6 weeks would lead to change in balance in geriatric population.

MATERIALS AND METHODS

Consent form, Assessment form, BBS scale, Pen, pencil, Paper pad, Scale, Ruler, Measure tape, Stop watch, Steps, Chairs

Method:

Ethical clearance was obtained.

60 healthy elderly volunteers residing at old age homes of Ahmadabad city, aged 60 years and above, both male and female were taken for the study. Sixty elderly were included in study depending on inclusion/exclusion criteria. Inclusion criteria for study were Age 60 years and above, both male and female, Subjects who were willing to participate in the study, Subjects who were ready to sign written informed consent form, and Subjects who were functionally independent that is, score of 100 point on barthel index. Exclusion criteria for the study were History of any recent musculoskeletal problems, serious cardiac and pulmonary condition which may required hospitalization, neurological conditions, psychiatric illness, Serious visual impairments (i.e. cataracts), Self-report of uncontrollable diabetes & hypertension, vertigo, Who were already in another active research study

Subjects were conveniently divided into two groups, 30 into yoga therapy group (study group) or group A and 30 into control group or group B. Total six subjects were drop out from the study, four from group A and two from group B. In group A two found six weeks a long duration, one had some personal issue and one could not completed because of lack of time. In group B two subjects were lost to follow up.
On the first visit a complete assessment was done including history taking. All subjects were explained about the aim and nature of the study and those willing to participate were requested to sign written consent form. Pre participation evaluation form consisted of general assessment including and outcome measures that include Berg Balance Scale (BBS), Timed Up and GO test (TUG). The outcome measures were taken before and after six weeks in both the groups.
Subjects in group A received yoga therapy six days in a week for six weeks. Subjects in group B were asked to report at the end of six weeks duration.

Some important guidelines and precautions for practice of asanas were explained such as:

- Take light snacks 1 hour before yoga class.
- Evacuate bowel and bladder.
- Dress should be loose and comfortable.
- Avoid jerky movements while doing asanas.
- Breathe normally while maintaining the pose.
- Do not force your body to achieve final pose.

Each therapy session starts with pranayama in form of anulom-vilom and 5-10 minutes of warm up focused on slow dynamic muscle movements with shoulder/arm circling, wrist circling and neck rolling. This was followed by 25 – 30 minutes of asanas consisting of following poses: pavanmuktasana, sputa matsyendrasana, setu bandha sarvangasana, bhujangasana, ardh-paschimottasana, paschimottasana, parvatasana, marjarasana, trikonasana, virbhadrasana, uttkatasana, and tadasana. All the asanas were progressed gradually starting from supine to sitting to standing position. Each asana was performed for three to five rounds according to participants' capacity. Each therapy session ends with savasana and pranayama. Entire session of yoga was under supervision. Heart rate, Respiratory rate and Blood pressure were measured at each session.

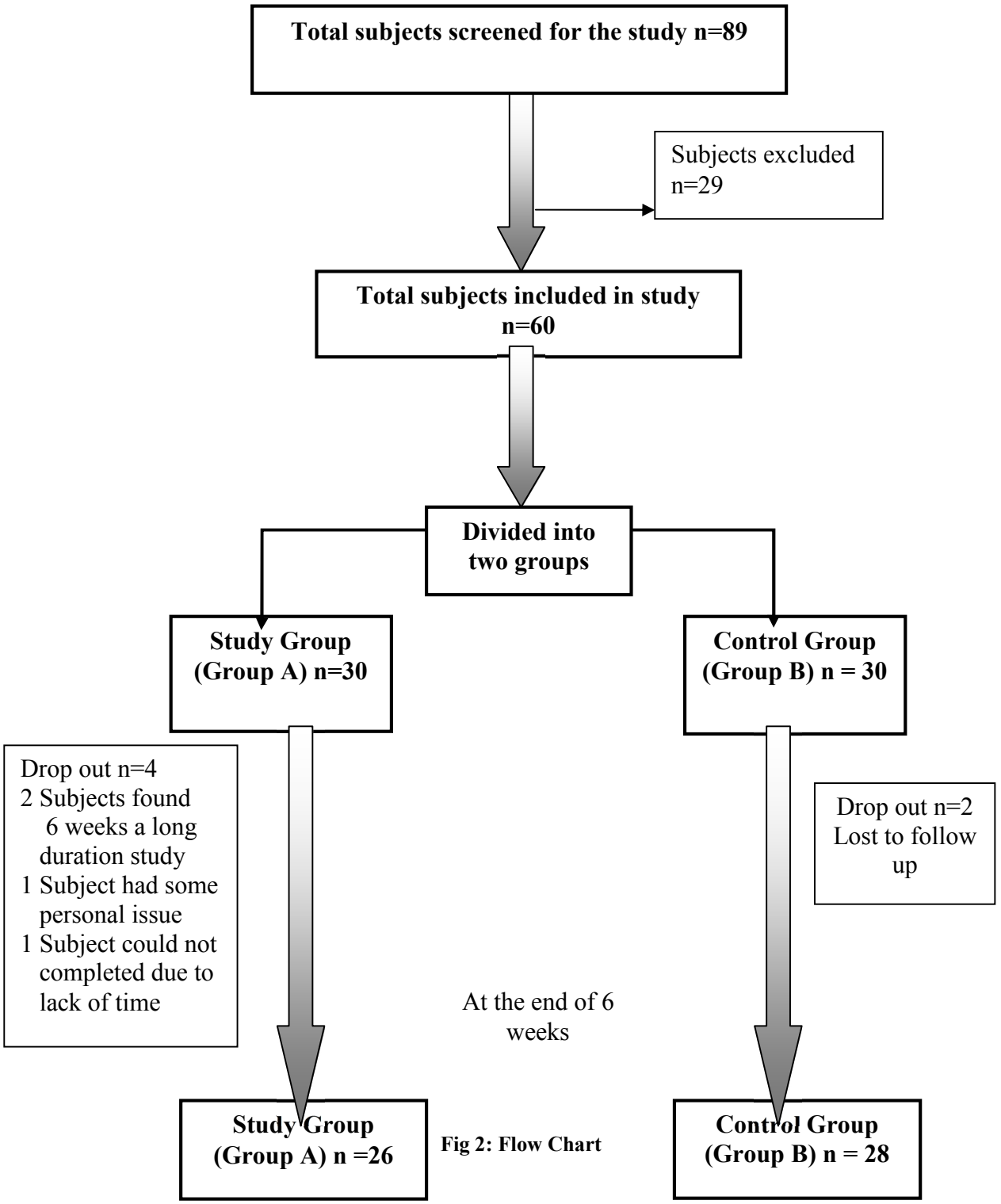


Fig 2: Flow Chart

RESULTS AND DISCUSSION

Before applying statistical tests, data was screened for normal distribution and outcome measures were analyzed using appropriate statistical test using Graph pad Prism-5. Confidence interval was set at 95% & $p<0.05$ was considered as significant. Changes in outcome measures were analyzed within group as well as between groups.

In this study to analyze the difference in BBS score before and after intervention within group A and B "Wilcoxon matched pairs signed rank test" was used. P value <0.05 was considered significant. Here within group A, $p<0.0001$ shows statistically significant difference while within group B, $p=0.23$ was not significant.

For comparing the post-intervention mean difference in BBS score between groups A & B, Mann-Whitney U test was performed. Mann-Whitney $U=22.5$ at $p<0.0001$ was found to be statistically significant.

In this study, to analyze the difference of TUG score before and after intervention within group A and B "paired t test" was used. Degree of freedom was kept at $df=25$ and $t=3.673$ at $p=0.001$ was found to be statistically significant within group A. Degree of freedom was kept at $df=27$ and $t=1.651$ at $p=0.11$ was not statistically significant within group B.

For comparing the post-intervention mean difference in TUG score between Groups A & B, "Unpaired t test" was used. In Unpaired t test, degree of freedom kept at $df=52$ and $t=2.878$ at $p=0.0058$ was found to be statistically significant between group A and B.

From the above findings, null hypothesis was rejected. And above findings conclude that;

- Statistically significant difference was found in BBS score with in group A compared to group B.
- Statistically significant difference was found in TUG score with in group A compared to control group B.
- There is a statistically significant difference in BBS and TUG score between group A and group B

Discussion:

The aim of the present study was to analyze the effects of 6 week yoga therapy program on balance in geriatric population with mean age of 67.6±6 years in group A (yoga therapy group) and 68.63±6.2 years in group B (control group). The outcome measures analyzed were berg balance scale and timed up and go test.

Group A subjects were asked to perform yoga therapy in form of different asanas for 45 to 50 minute a day, 6 days in a week for 6 weeks. Subjects in group B were asked to inform at the end of 6 weeks. BBS and TUG were taken at the beginning and at the end of six weeks in both the groups.

Statistically significant improvement was seen in balance measured by BBS ($P< 0.0001$, $w=-435.0$) and TUG test ($P=0.001$, $t=3.673$) in subjects who have performed yoga compared to those who have not.

The result of the present study is supported by Kathleen K. Zettergren et al [61] who found statistically significant improvement in BBS score but improvement in TUG score was not statistically significant and this may attribute to small sample size (n=16) they took.

Mary L et al [62] have evaluated the effects of modified chair-yoga in 16 elderly where they found improvement in functional mobility by reduction in TUG score.

In a preliminary trial with stroke survivors, Julie V et al [63] found that subjects who adhered to the yoga program experienced the most benefits in terms of mobility and balance.

The improvements in physical measures like balance, directly related to the yoga intervention are not surprising. Yoga poses are very similar to conventional balancing exercises given in routine clinical practice. Asanas or poses in the present study are given in different position like supine, sitting, quadruped and standing in a sequential order and progression was done according to improving balance. It ranges from low COM and wide BOS in supine position to high COM and narrow BOS in standing position.

Mark D et al [28] concluded that muscular strength, muscular endurance, flexibility, and cardio respiratory endurance can be improved after yoga therapy. They also reported that ankle flexibility, shoulder elevation, trunk extension, and trunk flexion was improved significantly after hatha yoga practice. Improvement in range of motion can be due to the static stretching nature of the asanas as stretching is most commonly advisable to improve flexibility. He also reported that increase in isometric and isokinetic muscular strength. The increases in isometric muscular strength in the above mentioned study most likely derive from holding static postures in the asanas. Because staic or isometric contractions do not reliably lead to increases in isokinetic strength measurements, the controlled movement from one asana to the next should also be considered for increase in isokinetic muscular strength.

Mandanmohan et al [69] demonstrated significant improvements in muscular strength in children resulting from yoga practice.

Arlene et al, who found improvement, fear of fall (FOF), balance and lower body flexibility in the population of older adults living and working in a retirement community. Out of all outcome measure, only static balance was found statistically significant. And they revealed that, the improvements in balance scores may be resulted of improvement in flexibility [25].

Flexibility decreases significantly per decade in both male and female after 20 years of age[70]. Especially hamstring and lower back flexibility in sit and reach test reduces about 2.5 centimetres per decade in both genders [71].

In a recent study involving young adults, 9.8% increase in LB flexibility was reported after 2 months (3 times a week) of yoga therapy[72]. On the other hand 6 to 10-week of stretching exercises in the elderly resulted in 25% increase in LB flexibility [73].

Greendale et al [74], reported a case study of a woman with hyperkyphosis who participated in a yoga intervention twice a week for 12 weeks improved significantly in the ability to stand quickly, move faster and reach longer, as well as reported increased physical self-awareness and well being.

According to Jayasinghe S R, Hatha yoga practice will lead to improvement in flexibility, balance, strength and overall fitness. Pranayam component of yoga (slow breathing) is able to improve heart rate variability by improving cardiovascular rhythms [36].

The increase in muscular strength, ROM and flexibility in addition to yoga poses may contribute to improvement in balance and postural control in the present study.

CONCLUSION

The present study concludes that yoga is effective in improving balance in elderly individuals at the end of six weeks compared to control group. Thus it can be used clinically to improve balance in geriatric population**.**

TABLES

Table 1: Mean difference in BBS score within group A & B

Group	**Pre treatment (BBS score)**		**Post treatment (BBS score)**		**w value**	**p value**
	Mean	**±SD**	**Mean**	**±SD**		
Group A	44.08	3.773	48.62	4.867	-325.0	< 0.0001
Group B	45.79	5.006	45.96	5.364	-35.00	0.2388

Table 2: Mean difference in BBS score between group A & B

Group	**Mean BBS score**	**±SD**	**U value**	**p value**
Group A	4.538	1.726	22.50	< 0.0001
Group B	0.5357	0.5762		

Table 3: Mean difference in TUG score within group A and B

Group	Pre treatment TUG (sec)		Post treatment TUG (sec)		t value	p value
	Mean	±SD	Mean	±SD		
Group A	12.23	2.673	11.50	2.717	3.340	0.0026
Group B	12.86	2.851	12.64	3.082	1.651	0.11

Table 4: Mean difference in TUG score between group A & B

Group	Mean TUG score (sec)	±SD	t value	p value
Group A	0.9231	0.6884	2.878	0.0058
Group B	0.4286	0.5727		

ACKNOWLEDGEMENT

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EFFICACY OF MUSCLE ENERGY TECHNIQUE FOR PECTORALIS MINOR IN PATIENT WITH FROZEN SHOULDER

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ABSTRACT

Background and Objectives: Frozen shoulder is a spontaneous onset of shoulder pain accompanied by progressive restriction of both active and passive Glenohumeral movements. Scapular dyskinesis has been associated with frozen shoulder. One specific cause of scapular dyskinesis may be an abnormally shortened Pectoralis minor muscle. Diminished Pectoralis minor flexibility prohibits optimal scapular kinematics. Physiotherapy management is preferred treatment. **Methodology:** 50 subjects were assessed and divided into two groups. In group A, conventional physiotherapy treatments and In group B, conventional physiotherapy treatments along with muscle energy technique for Pectoralis minor was given. Pre and post outcomes measures LSST, ROM, SPADI and Length of Pectoralis Minor were analyzed. **Result:** The result showed that there was significant improvement in both the groups, but there was extremely significant improvement in group B. **Conclusion:** Muscle energy technique for pectoralis minor with the conventional physiotherapy is effective in management of frozen shoulder than convectional physiotherapy alone.

SUMMARY

Muscle Energy Technique for pectoralis miner is effective in frozen shoulder patient

Keywords: Frozen shoulder, Scapular dyskinesis, muscle energy technique, Pectoralis minor

INTRODUCTION

Movements of the human shoulder represent a complex dynamic relationship of many muscle forces, ligament constraints, and bony articulations, static and dynamic stabilizers allow the shoulder the greatest ROM of any joint in the body(1). The shoulder is a complex anatomic structure that allows movement in many planes.

Frozen shoulder describes the common shoulder condition characterized by painful and limited active and passive range of motion. Frozen shoulder was first introduced by Codman in 1934. He described frozen shoulder as a painful shoulder condition of insidious onset that was associated with stiffness and difficulty sleeping on the affected side. Long before Codman, in 1872; the same condition has already been labeled "Periarthritis" by Duplay. In 1945, Nevaiser introduced the term adhesive capsulitis to describe the inflamed and fibrotic condition of the capsulo ligamentous tissue.

Frozen shoulder is defined as an idiopathic condition of the shoulder characterized by the spontaneous onset of pain in the shoulder with restriction of motion in every direction.[1] Its etiology, although unclear is associated with the interaction of constitutional and extrinsic factor among patients who, notably are between 40-60 years of age, stages of freezing, frozen and thawing characterizes the natural history of the frozen shoulder.The condition is a self limiting within one to three years. Frozen shoulder is reported to affect 2% to 5% of the general population(3,4,5,6,7,9,10,12), increasing to 10% to 38% in patients with diabetes(3) and thyroid disease(5,6,7,8,9,10,11,12). Individuals with primary frozen shoulder are commonly between 40 and 65 years old,[13,14,15] and the incidence appears higher in females than males(2,4,10,11,16,17,18).

The Pectoralis minor as a scapular downward rotator, anterior tilter and internal rotator is essentially an antagonist to the necessary scapulothoracic motions during elevation of the arm. A reduced resting length of this muscle has been shown to result in increased scapular internal rotation and reduced posterior tilting as the arm is raised over head(18).

In general, a global loss of active and passive motion present; the loss of external rotation with arm at patient's side is the hallmark of this condition. Various treatment options exist to decrease pain and increase range of motion thereby increasing the quality of life of persons with frozen shoulder. Treatments of frozen shoulder include invasive interventions (intra-articular injections of glucocorticoids), pharmacological treatments (anti-inflammatory drugs, analgesics, Minor tranquilizers/muscle relaxants, Hypnotics), and non-pharmacological treatments (exercise, and patient education). Commonly used treatments include transcutaneous electrical nerve stimulation (TENS) for pain relief and ultrasound with range of motion exercise to restore movement.

MET are soft tissue manipulation methods in which the patient, on request actively uses his muscles from a controlled position in a specific direction with mild effort against a precise counterforce. It is a manual procedure that uses controlled, voluntary isometric contraction for a targeted muscle or muscle group and is widely advocated by authors in the field of osteopathy.

MATERIALS AND METHODS

An experimental study was conducted in RK Physiotherapy Rehabilitation and Research Center, Rajkot. 50 subjects for this study are randomly selected using random sampling technique from Physiotherapy Rehabilitation and Research Center. **Inclusion criteria:** Subjects were clinically diagnosed of Frozen shoulder and age group between 40 to 60 years, Both females and males, painful Restricted ROM in capsular pattern (external rotation >abduction > internal rotation), symptoms lasted at least 3 months,

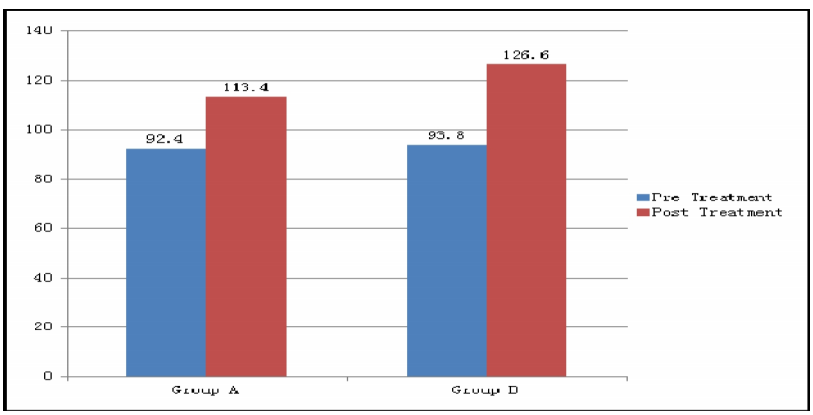
comprehended commands and willing to participate in the study. **Exclusion criteria:** Patients had taken local corticosteroid injection to the affected shoulder within the last 3 months or current corticosteroid therapy, history of hospitalization in last 1 year and Pregnancy.

METHOGOLOGY

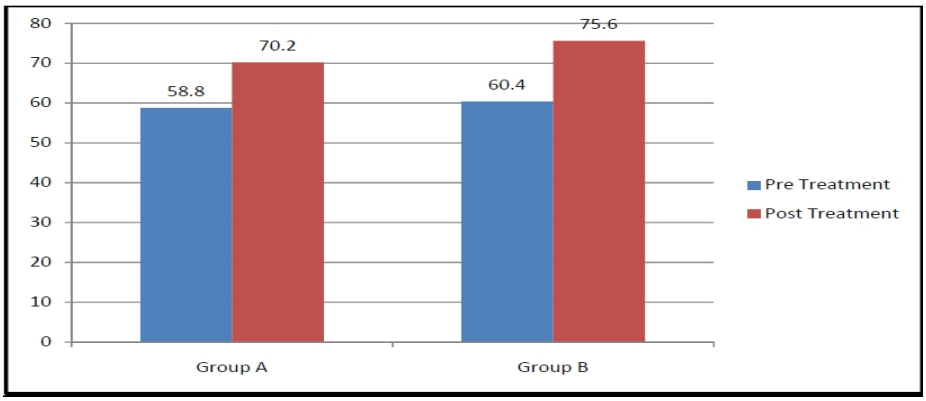
Subjects were screened based on the inclusion and exclusion criteria, and were requested to participate in the study. Prior to the commencement of the procedure, written informed consent was obtained from the participants. After being allotted in the groups demographic data was collected, assessment of ROM, SPADI, and LSST was done at the beginning and at the end of prescribed protocol; a follow up was taken on the 4th week after the commencement of the procedure. Subjects were randomly allotted into two groups; group A and group B were given a treatment protocol for 4 weeks. One group, group A was given conventional physiotherapy treatment i.e. ultrasound therapy, passive capsular stretching, GH mobilizations and shoulder muscles strengthening and the other group , group B was given the conventional physiotherapy treatment along with muscle energy technique for pectoralis minor.

RESULTS AND DISCUSSION

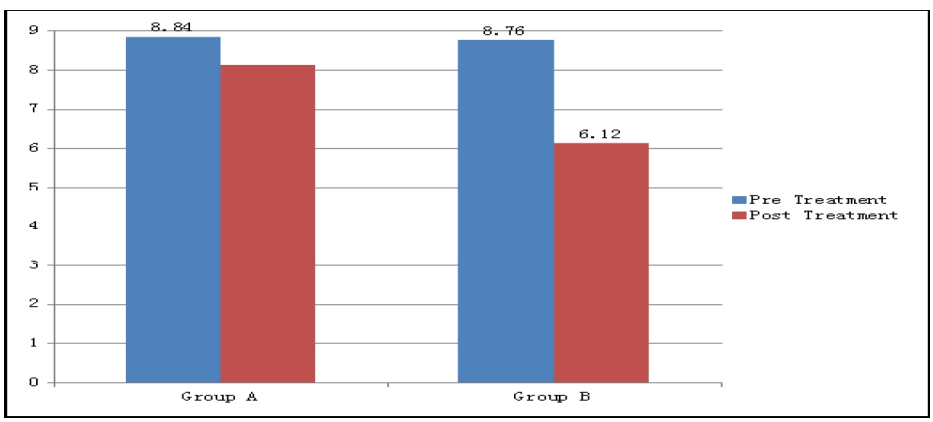
The result of this study was analyzed in terms of improvement in active range of motion of abduction and external rotation which was measured using universal goniometer, improvement in terms of pain and shoulder function were measured using shoulder pain and disability index (SPADI), improvement of scapular asymmetry was measured using lateral scapular slide test and the improvement in length of the pectoralis minor was measured using length of pectoralis minor test. Paired‘t’ test was used for inter group comparison and unpaired‘t’ test was used for intra group comparison. Wilcoxon signed rank test was used for inter group comparison and mann-whitney test were used for intra group comparison to evaluate SPADI scores.



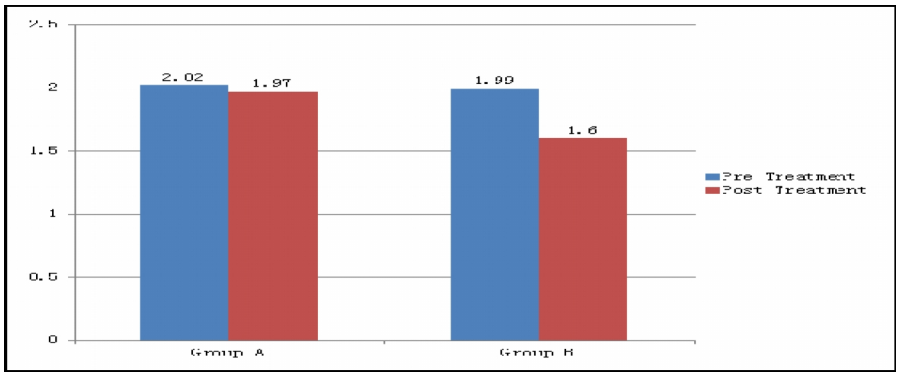
“Graph 1. Pre and Post treatment Intra group comparison of abduction ROM in both groups”



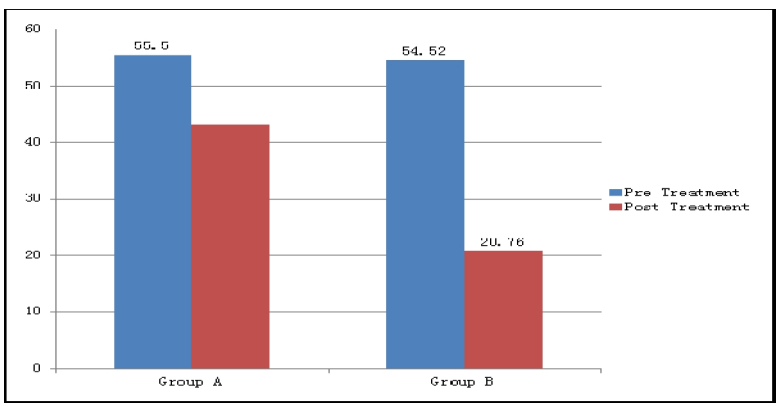
"Graph 2. Pre and Post treatment Intra group comparison of external rotation ROM in both groups"



"Graph 3. Pre and Post treatment Intra group comparison of length of pectoralis minor in both groups"



"Graph 4. Pre and Post treatment Intra group comparison of lateral scapular slide test in both groups"



"Graph 5. Pre and Post treatment Intra group comparison of SPADI in both groups"

Data clearly shows that there is improvement in abduction and external rotation of shoulder, length of pectoralis minor, lateral scapular slide test and SPADI in both groups. When comparing outcomes between these two groups, the result shows significant improvement in group-B

CONCLUSION

The results obtained from this study following data analysis, showed that patients in both the group has shown improvement in ROM and functional capacity but patients from group B (experimental group)

showed significant improvement on comparison between the groups in terms of ROM (abduction and external rotation), function capacity in terms of improvement in SPADI scores and improvement in scapular asymmetry in terms LSST and also increase in length of the pectoralis minor muscle assessed by Length of pectoralis minor test.

Thus, it could be concluded that muscle energy technique for pectoralis minor along with the conventional physiotherapy is effective in management of Frozen shoulder to reduce pain and improve functional capacity than convectional physiotherapy alone.

ACKNOWLEDGEMENT

I thank almighty god and all who have helped me for their all time support and encouragement

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COMPARISON OF EFFECT OF PROPRIOCEPTIVE NEUROMUSCULAR FACILITATION TECHNIQUE VERSUS MOTOR RELEARNING PROGRAMME ON IMPROVEMENT OF TRUNK CONTROL IN HEMIPARETIC PATIENTS

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ABSTRACT

BACKGROUND:

Trunk control is an important factor after the stroke. The literature suggests that trunk exercises are required to achieve good trunk stability which is essential for balance and daily functional activities. By keeping these facts in view present study aims at evaluating the efficacy of PNF techniques & MRP on improvement of trunk control in hemiparetic patients.

METHODOLOGY:

Subjects were randomly assigned to two groups, group I (PNF) & group II (MRP)for trunk to improve trunk control. Trunk control was assessed on day 1 and at the end of 4 weeks interventions, with the help of Trunk Impairment Scale.

RESULTS:

On the T.I.S, there was no significant difference between two groups for static sitting balance, dynamic sitting balance & co-ordination were 1.467, 0.127, 0.766 respectively.

CONCLUSION:

Both the treatment techniques are effective to gain trunk contol in hemiparetic patients.

SUMMARY

The Hemiparetic patients were assessed on T.I.S and assigned in two group of PNF and MRP, after 4 weeks of intervention, it was concluded that both the techniques are equally effective.

Keywords: Keyword 1, Stroke 2, MRP 3, PNF techniques 4, Trunk impairment scale

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INTRODUCTION

According to world health organization stroke can be defined as "rapidly developed clinical signs of focal disturbance of cerebral function of presumed vascular origin and of more than 24 hours duration."[1] Following stroke, loss of motor control leads to multidirectionally **impaired trunk** muscles strength which has a potential to affect functional activities.[13] The PNF technique includes the exploitation of postural reflexes, the use of gravity to facilitate movement in weak muscles, the use of eccentric contraction to facilitate agonist muscle activity and the use of diagonal movement patterns.[22, 23, 24] In the Motor Relearning Program, visual and verbal feedback is given and in addition the patient is encouraged to recognize the "correct movement response" from incorrect ones so that unnecessary activities can be eliminated. [26]

HYPOTHESES

Experimental Hypothesis:

There will be significant difference in effect of Proprioceptive Neuromuscular Facilitation technique and Motor Relearning Programme to improve trunk control in hemiparetic patients.

Null Hypothesis:

There will be no significant difference in effect of Proprioceptive Neuromuscular Facilitation technique and Motor Relearning Programme to improve trunk control in hemiparetic patients.

MATERIALS AND METHODS

MATERIALS

1. Pen
2. Pencil
3. Paper
4. Plinth
5. Stop watch
6. Box
7. Consent form
8. Assessment form

METHOD

- **STUDY DESIGN:** Interventional Comparative study.
- **SAMPLING TECHNIQUE:** Purposive Sampling
- **STUDY SETTING:** Hemiparetic patients from any physiotherapy center, Rajkot.
- **SAMPLE SIZE:** 30 Hemiparetic Patients was taken for the study.(Group 1–15 subjects & Group 2–15 subjects)

SELECTION CRITERIA

INCLUSION CRITERIA

- Age : 40-65 Years
- Gender : Both male and female
- Ischemic or hemorrhagic subacute Hemiparetic patients.
- An independent ability to sit for 30 seconds.

EXCLUSION CRITERIA

- Any previous musculoskeletal problems to trunk.
- Medically unstable patients.
- Multiple strokes
- Patients with impaired cardiovascular status
- Patients with aphasia (sensory)

PROCEDURE

Measurement procedure:

- Prior to the participation, all the subjects were explained the purpose & detail of the study, importance of therapy protocol & thus gaining active participation & co-operation from them.
- Informed consent was signed by the subjects for their voluntary participation.
- Demographic data was obtained from the patients, including name, age, sex, address, side of stroke.
- Subjects were requested to continue their normal activities and avoid any other form of exercise for the duration of the study.
- Before and after the 4 weeks of interventions both the group were evaluated by Trunk Impairment Scale. Total duration of session of Physiotherapy was 60 minutes.

Routine Physiotherapy:

Conventional rehabilitation includes,

- Passive range of exercise
- Active assisted movements
- Mat exercise
- Stretching
- Strengthening exercise
- Relaxation
- Ambulation with assistive device or support

PNF technique for trunk (Group - 1):

- 5 days per week for 4 weeks

  Chopping technique
  Lifting technique
  Bilateral leg patterns for trunk

Motor relearning programme for trunk (group - 2):

5 days per week for 4 weeks
Trunk rotation
Side flexion
Reach forward and sideways
Reaching downwards
Reaching sideways
Reaching forward
Reaching backward

Functional outcome measure:

Trunk Impairment Scale

RESULTS AND DISCUSSION

All the statistical analysis was done by using SPSS 14 version for windows software. Wilcoxon signed rank test was used for Intra group analysis and Mann-Whitney U test for Inter group effect analysis. Table 1 and 2

DISCUSSION

The results of the present study indicate that both PNF and MRP are equally effective in improving trunk control in stroke patients, thereby supporting the null hypothesis. Both these techniques focus on strengthening the weaker muscles and improving the functional level of the patients by performing movement patterns and task that mimic activities of daily living.

The analysis of T.I.S. components in Group 1 treated with PNF showed significant improvement in the trunk control following a 4 week treatment protocol. These results accord with those of **Susan Bennet & James L Karnes** who concluded that spiral and diagonal mass movement patterns of PNF resembling normal activity are designed to address problems such as weakness and lack of stability.

The analysis of T.I.S. components in group 2 treated with MRP showed significant improvement in the trunk control following a 4 weeks treatment protocol. These results are supported by **Dora YL Chan,** Dora concluded that MRP is effective for enhancing functional recovery of patients with stroke. The 'sequential' and 'function-based' design in the clinical protocol for MRP is believed to maximize its effectiveness

CONCLUSION

- This study emphasizes the importance of trunk performance. Results of the study show that PNF technique and MRP both are equally effective in improvement of trunk control in hemiparetic patients.

TABLES

TABLE 1 : AGE DISTRIBUTION OF SUBJECTS

Group	N	Mean (years)	SD (years)

Group 1	15	56.6	7.881
Group 2	15	56.2	7.6457

Interpretation: Table 1 displays the statistics of age distribution of the 30 subjects. Among the 30 subjects, the mean age of 15 subjects in Group 1 was 56.6 years with a standard deviation of 7.881, and the mean age of 15 subjects in Group 2 was 56.2 years with a standard deviation of 7.6457. No significant age difference was seen across the two groups.

TABLE 2 : INTER GROUP ANALYSIS OF T.I.S. COMPONENTS

T.I.S. COMPONENTS	MEAN IMPROVEMENT SCORE		Z VALUE	P VALUE
	GROUP 1	GROUP 2		
STATIC SIITING BALANCE	1.2	1.2	1.467	0.142
DYNAMIC SITTING BALANCE	3.3337	3.8	0.127	0.899
COORDINATION	1.2	1.533	0.766	0.444

Interpretation: As per the Mann Whitney U test, data reflect that p value (probability value) is greater than 0.05 which is the standard value. The "P" value for two tailed is 0.142, 0.899 & 0.444 which is more than the standard value. There is certainly sufficient information to accept the Null hypothesis. So, Null Hypothesis accepted & alternate hypothesis is rejected.

ACKNOWLEDGEMENT

My sincere thanks to my parents and all the contributor.

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A Study to evaluate the Effectiveness of Constraint Induced Movement Therapy on affected Upper Extremity functions in Children suffering from Hemiplegic Cerebral Palsy

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ABSTRACT

OBJECTIVE: A study to evaluate effectiveness of Constraint induced movement therapy (CIMT) on affected Upper extremity functions in children with hemiplegic cerebral palsy.
METHOD: 15 patiens diagnosed as hemiplegic cerebral palsy by neurological consultant , Both Male – Female(age group 5-12 years) and who met with the inclusion criteria . Informed written consent was taken. Before intervention Fugl Meyer Assessment-UE scale(FMA-UE) and Manual Ability classification system(MACS) were taken and data were recorded. Subjects were allocated into 2 Groups.Group A(n=7) Experimental group were taken CIMT and conventional physiotherapy. Group B(8) control group were taken conventional physiotherapy.
RESULT: There is a significant difference on pre and post Fugl meyer assessment, thus within and between group analysis was shows statistical significant difference on upper extremity function.
CONCLUSION: Study concluded that there is a significant difference on affected upper extremity function in hemiplegic cerebral palsy patients.

SUMMARY

To evaluate effect of CIMT on UpperExtremity functions in children with hemiplegic cerebral palsy.
Keywords: Keyword 1Constraint induced movement therapy, Keyword 2Fugl-meyer assessment, Keyword 3Manual ability classification system, Keyword 4Upper extremity function, Keyword 5 Hemiplegic cerebral palsy

INTRODUCTION

Cerebral palsy (CP) the term used to describe a broad spectrum of motor disability which is non-progressive and is caused by damage to the brain lesion.

CP is the most common physical disability in children, Approximately 1/3 of children with CP have spastic hemiplegic CP, in which one side of the body is significantly impaired than the other side of the body.*(1)* Reduced strength and motor control of upper extremity interfere with exploration, play, self-care, and other activities of daily living (ADLs), and thus gives interfere with development in multiple domains.*(1)*

Conventional treatments, including physical therapy, occupational therapy, pharmacotherapy for muscle tone, and surgery which focus on teaching compensatory skills and prevent deformity but none of this has demonstrated success in ameliorating the primary disorder. Recently,a new approach of treatment for hemiplegic CP has emerged from the confluence of behavioral psychology/learning theory and discoveries in neuroscience regarding neuroplasticity.

The new approach, called constraint- induced movement therapy (CIMT), CIMT is an intervention in which a constraint is utilized on the unaffected hand of a person with hemiplegia to improve functioning of their involved upper extremity.CIMT is based on the principles of mass practice and shaping .*(2)* Early reviews (Charles and Gordon, 2005; Hoare and Carey, 2007) have reported positive outcomes in children treated with CIMT.*(3)*

MATERIALS AND METHODS

MATERIALS:

- Paper
- Consent form
- Data collections sheet
- Restraint
- pen

METHODS:

- Study design: Experimental Study
- Sample size: 15 Patients (Rt-6) (Lt-9)
- Sampling Method: Randomized sampling
- Setup : Physio. Department
- Duration of study: 2 months

15 patiens diagnosed as hemiplegic cerebral palsy by neurological consultant , Both Male – Female were included(age group 5-12 years) and who met with the inclusion criteria . Informed written consent was taken. Before intervention Fugl Meyer Assessment-UE scale(FMA-UE) and Manual Ability classification system(MACS) were taken and data were recorded. Subjects were allocated into 2 Groups.Group A(n=7) Experimental group were taken CIMT and conventional physiotherapy. Group B(8) control group were taken conventional physiotherapy.

Group A(Experimental Group):(n=7)

Subjects in Group A were given Constraint induced movement therapy and conventional therapy.Subjects were required to demonstrate at least 10 degree of active wrist extension and finger extension in the affected limb.Group wore a mitt on the unaffected hand 2hr/day therapy session for 7 days per week for 2 months.

Group B(Control Group):(n=8)

Subjects in Group B were given Conventional physiotherapy.

RESULTS AND DISCUSSION

RESULTS:

The present study was done to determine the effect of CIMT on affected upper extremity function of hemiplegic cerebral palsy patients. It consisted of 15 subjects, divided into group A and group B containing 7 and 8 subjects respectively. FMA was analyzed using Wilcoxon Signed Rank test within each group. There was a statistically significant difference within group A ($p=0.0156$) and group B ($p=0.0078$). Difference in FMA was analysed using Mann Whitney U test between the groups. There was significant difference between the groups ($U=46.5$; $p=0.0368$). On MACS was analysed within each group using Wilcoxon signed rank test. There was no statistically significant difference in group A ($p=0.25$) and group B ($p>0.99$), Difference in MACS scale between the groups, analysed using Mann Whitney U test showed no significant difference between the groups ($U=36.5$; $p=0.335$).

DISCUSSION:

Results show significant difference between both group. The results shows improvement on Upper extremity function in children with hemiplegic cerebral palsy**.**

Eliasson et al. [2005] reported on a study in Sweden comparing a CIMT to conventional treatment. 21 children aged 18 months to 4 years with hemiplegic CP of varying etiologies and degrees of severity wore a restraining fabric glove on the less-affected hand for 2 hr a day, 7 days a week, for 2 months. Only the CIMT group showed significant gains in AHA scores immediately post-therapy.*(2)* In Eliasson et al. [2005] studied that children 18 months to 4-years-old, age was positively corre- lated with improvement,*(2)* incontrast to Gordon et al. [2006], and Taub et al. [2007], who found no differences by age in degree of improvement in their samples of children aged 4–13 years and 2–6 years, respectively. *(5,6)*

Susan fasoli et al(2009) studied the upper extremity test of the Fugl-Meyer Assessment (FMA-UE) for six children with hemiplegic CP, ages 5-12 years, who participated in a robot-assisted therapy study.*(7)* They concluded that the Fugl-Meyer Assessment is being used with greater frequency to evaluate changes in upper limb motor impairment in children with neurologic disorders and hemiplegia. Reliable standard administration will allow for outcome comparisons among pediatric and adult research studies(7)

CONCLUSION

There is a significant difference on Fugl-meyer assessment in Constraint induced movement therapy and Conventional therapy on affected Upper Extremity functions in children with hemiplegic cerebral palsy.

FIGURES

Fig 1. Mean age distribution of subjects in both groups

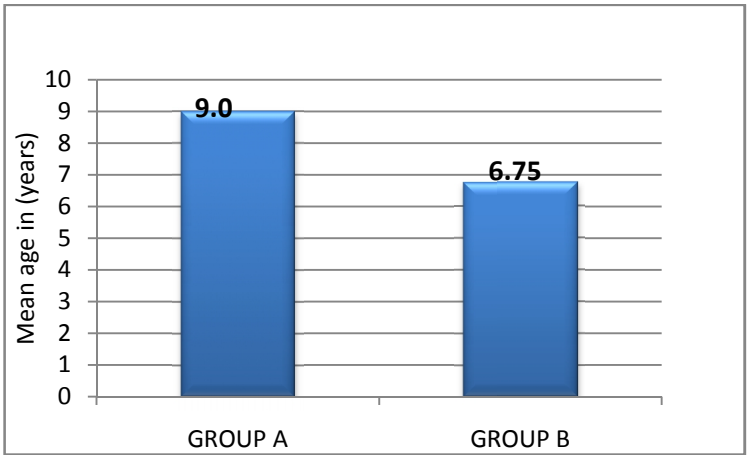


Fig 2. Gender distribution of subjects in each group

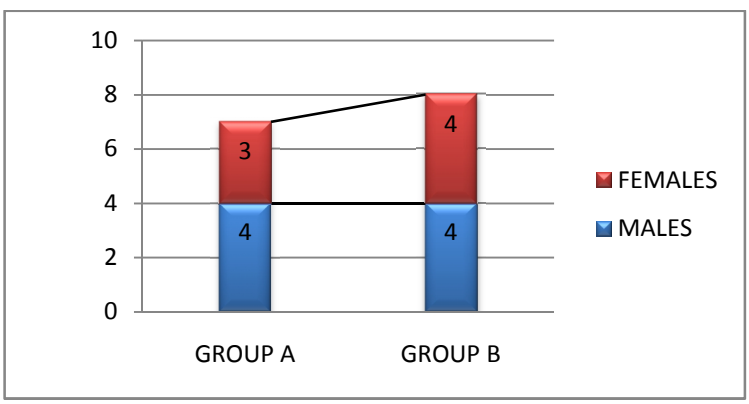


Fig 3. FMA scale within group

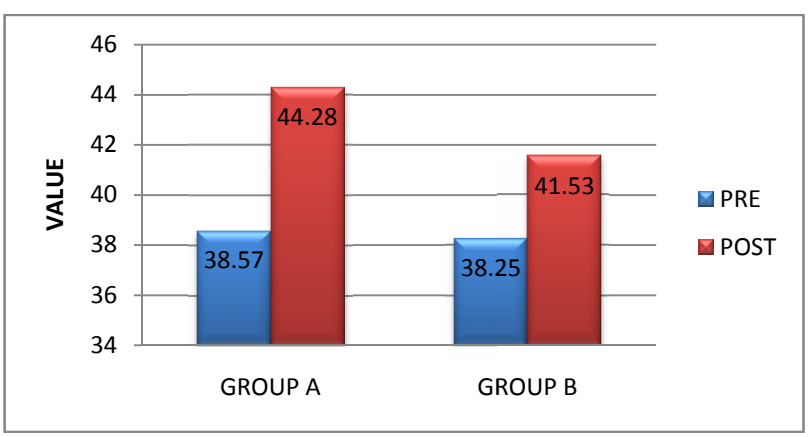


Fig 4. Difference in FMA between groups

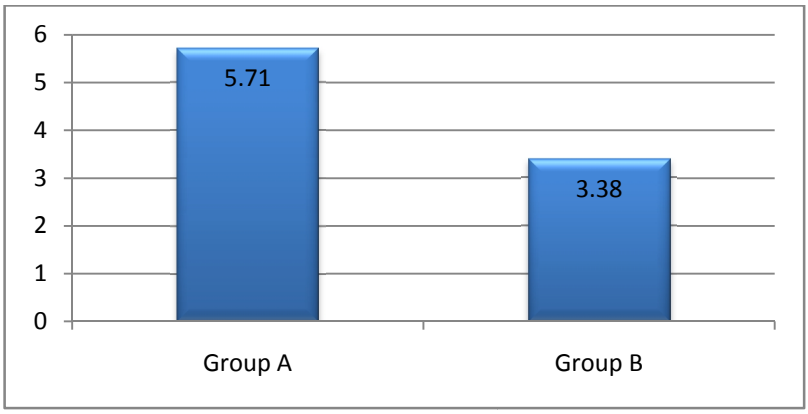


Fig 5. MACS scale within group

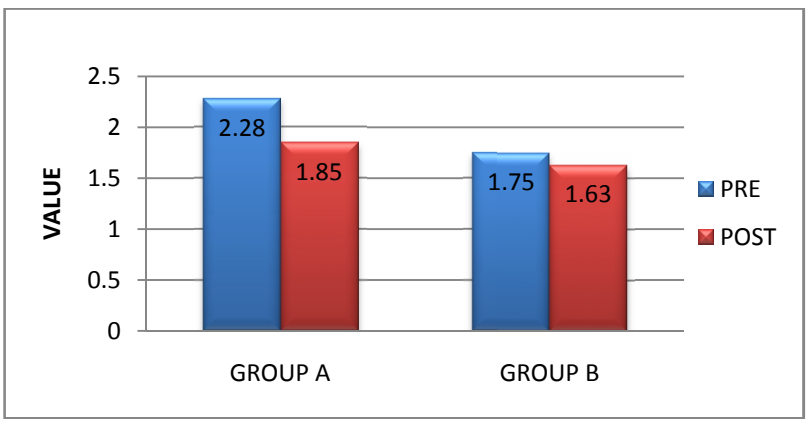
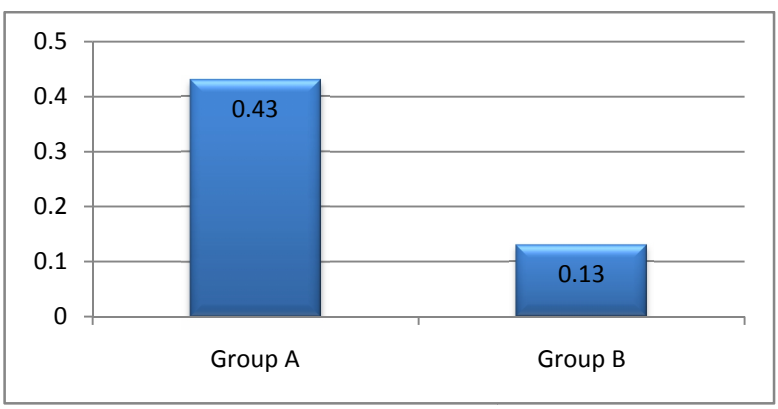


Fig 6. Difference in MACS scale between groups



TABLES

Table 1. Tests used for within and between the groups

VARIABLE	WITHIN GROUP A	WITHIN GROUP B	BETWEEN GROUP A and B
FMA	Wilcoxon Signed Rank test	Wilcoxon Signed Rank test	Mann Whitney U test
MACS	Wilcoxon Signed Rank test	Wilcoxon Signed Rank test	Mann Whitney U test

Table 2. Demographics of subjects

CHARACTERISTICS	GROUP A	GROUP B
MEAN AGE (Mean ± SD) (years)	9 ± 1.41	6.75 ± 2.38
MALES (%)	4 (57.14)	4 (50)
FEMALES (%)	3 (42.86)	4 (50)

Table 3. FUGL Mayer Assessement (FMA) scale within group

	Group A	Group B
Pre FMA	38.57 ± 5.59	38.25 ± 4.83
Post FMA	44.29 ± 5.96	41.63 ± 5.53
p value	0.0156	0.0078

Table 4. Difference in FMA between groups

	Mean + SD	Mann Whitney U value	p value
Group A	5.71 ± 1.60	46.5	0.0368
Group B	3.38 ± 2.13		

Table 5. MACS scale within group

	Group A	Group B
Pre MACS	2.29 ± 0.76	1.75 ± 0.71
Post MACS	1.86 ± 0.90	1.63 ± 0.74
p value	0.25	>0.99

Table 6. Difference in MACS between groups

	Mean ± SD	Mann Whitney U value	p value
Group A	0.43 ± 0.53	36.5	0.335
Group B	0.13 ± 0.35		

ACKNOWLEDGEMENT

I am greatful to my patients to support me during my study and I am very much thankful to the authors of clinic in which I have perform my study.

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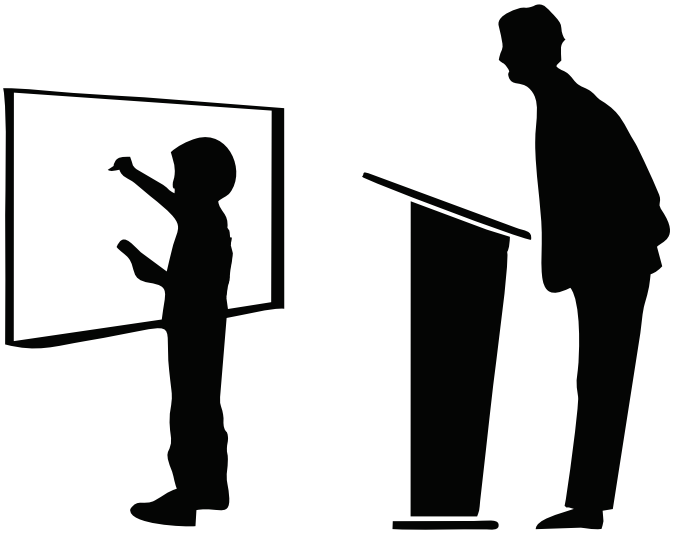
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RKUNIVERSITY

SECTION NO. 3: PEDAGOGY RESEARCH

Effective teaching techniques are indeed necessary for facilitating the process of learning and research. RK University has made pioneering contributions to the field of pedagogy via activity-based classroom teaching and outcome-based learning. Research articles in this section mainly focus on innovations in the field of education.





INNOVATIONS IN PEDAGOGY

ISBN: 978-93-5254-061-7 | www.rkuconference.org | JAN. 5 & JAN. 6, 2016

Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

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RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

Innovations in Pedagogy – "Education in Museum" - Future of Quality Learning Environment

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ABSTRACT

In the era of innovative pedagogical techniquesin the educational landscape, museum education will play a main role. Museum is a perpetual non- profit organization established for the purpose of examining, evaluating, preserving, conversing, sharing information and exhibiting varied collections of objects in terms of history, science, technology, and technical for the purpose of enjoyment and education, which is open to public. The research aims to create awareness regarding the concept and benefits of education in museum with educational purposes among the students and the teachers of schoolsand colleges.An excellent learning environment of museums enables students to develop their core skills of ability, think innovatively and creatively, critical thinking, collaborating, and combining information. By applying the practice of projecting to the field of education, this paper provide indication of prospective futures as seen in the vibrant revolutions in quality education currently taking place outside traditional schools and colleges.

SUMMARY

Include a brief summary of your research work in exactly one sentence.

Keywords: ***Education, Innovative, Learning Environment, Museum, Quality***

Introduction

Traditionally a spot where accumulations of old things are put on a display in galleries and glass cabinet is well known as museums. Museums that teaches students, teachers, and public globally to enhance themselves by instructing and offering them some assistance with grasping the significance, use and significance of the works displayed, thus playing the role of an education institution. In this context, a museum is an establishment where works and things to be acquired from the past are gathered, kept, secured and showed to individuals, students and teachers to understand practically and aesthetically and make appraisals considering the related authentic and social aspects. Further making more descriptive museums can think to be truly living as they make us feel domiciliation in the past and discovers items and things to energize.

Mostly, the first impression of the word museums are considered as a house with collection of coins and fossils , the dirt, aroma and colour of the ancient and convey on about what "historical" really implies. Further exhibiting and presenting the objects and things give data about the occasions and issues individuals truly experienced previously. Be that as it may, museums likewise have the undertakings of showing individuals the approaches to enhance themselves, guiding, motivating, directing, propelling and urging them to make correlations between the information of what they definitely know and what they have recently learnt. In backing of this, (Oğuzoğlu, 2007), is of the opined that museum are to show individuals that advancement is practically unsurprising and what's to come will be unique in relation to the present time.

As indicated by (Paykoç and Baykal, 2000, 103), museum is like an asset to the society within and has a huge part in the social, financial, and political advancement of that society. In museums, students and teachers get the opportunity to see numerous reports about society in an air of social cooperation, which helps them to get a handle on that they fit in with that specific culture and increase self-assurance. (Buyurgan, 2002, 106) trusts that museum empowers individual to be presented to three- dimensional elements which would deliver vastly improved results in their everyday life. Today in developed countries, in the era of transformation in the system of teaching methodology identified with schools and colleges, while teachers are urged to practice social sources, it is gauge that they profit by historical centres, workmanship displays, and particularly from the instructive projects of these foundations. It is hence, the exceptional projects arranged by galleries for schools and colleges are these days turning out to be considerably more imperative than some other time. On one hand, while giving deep rooted quality instruction, formal training by educational museum excursions which on the other hand is of great support. To attain quality in education proper environment and new learning skills are to be used now a days. Consideration ought to be taken to control and relate

the things taught at schools and colleges are connected, in actuality. Indeed, museum is the main fundamental establishments that can go about as a scaffold between this need and to make this quality learning environment.

In today's period of granting quality training, education and learning environment in differed interdisciplinary studies, it is presently a broadly known reality that learning ought not be constrained with the degree it can happen at schools and colleges just as there are incomprehensible changes and advancements in regards to the methodology towards exhibition halls. Because of that awareness of other's expectations to society - individuals, understudies, educators, teachers, and specialist, offering projects to instruct kids has gotten to be one of the essential elements of museum- exhibition halls as of late. Further, many scholars put emphasis on the importance of museums in education as it spotlight on being dynamic in education, gaining experience, learning through new environment, creating correspondence aptitude by interaction and being constructivism. Hence, museum give an ideal domain in which kids can investigate, do trials, workshops and experience articles and things to enhance themselves affectively, physically, socially and subjectively.

Museum and Education

Vision in the goals of education have changed over period, similarly as there is a change in the social and educational environment. A “multicultural education discusses to any form of education or teaching that integrates the ethics, texts, histories, philosophies, and perspectives of people from diverse cultural backgrounds” (http://edglossary.org, 2013). Understanding the likes and dislikes of students and defining their own qualities, now-a-days it is suggested to help children know themselves better further allowing them to make decisions about what they want to do or buy. Students would learn extra and better when given a domain where there is no apprehension, pressure and disappointment and they feel shielded and cheerful (Kansu, 2005). Students who know themselves can convey what needs be better in their associations with others and they are acknowledged for that. Students who know themselves can express themselves better in their relationships with others and they are appreciated for that. A cordial and welcoming environment should be provided to students at home and even at school. There is a close relation between children’s emotional health and effective thinking and learning skills. Instead of using one teaching methodology, and thus one level of intelligence, we hope to access intelligence through the multiplicities of thought. (Dunn & Barijugh, 2014). Our hope and vision has changed by consolidating the discrete ideas of playground, classroom, library, and laboratory under one room. The "museum gives a fabulous domain which engages students to study while living and encountering that

can add to their physical, sentimental, nostalgic, scholarly, and societal improvement" (Doğan, 2010).

The real objective of education either in school or in colleges in the period of cutting edge instruction is to get ready students for higher instructive foundations and a superior individual in life as per their intrigues, abilities and formative and pedagogical qualities with successful direction and advising administrations given beginning from exceptionally youthful ages (Oğuz, 2004). Students take in more through diversions, dialect study and aesthetic works, for example, books and pictures with the guides of innovation and varying media part, which has grown extensively in quick speed.

As indicated by (Buyurgan, 2002) museums compose a few instructive projects for diverse divisions of society and educational department subject-wise and for that they make utilization of inventive and intelligent displays and even dramatization in order to have the dynamic investment of guests, students and academician. To persuade individuals to visit galleries, what makes a difference is the presentation outlines with instructive worries as well as the ones in which instructive elements are highlighted a considerable measure (Atagök B, 1999). This paper provides a summary of innovation in education system referring to selected examples in a broad cross section study of education in museums.

Importance of Museums

According to the modern learning theory, it is apparent that just classroom teaching and exercises would not meet the necessities for children, youngsters studying in colleges, teachers, professors and even researcher to have the qualities they need. Learning requires active participation of all in the current scenario. The choices a learner settle on at ought to be all around coordinated with the substances he or she has created personality a top priority, and the standards another person has set before would not make any difference. It is consequently, there ought to dependably be distinctive thoughts of understanding and dynamic learning courses in an instruction framework. The students and learners ought to be offered need to the learning encounters that give them the chances to do analyses, see the relationship in the middle of articles and life, surmise about things and make inductions. As indicated by (Duru, 2007) going to class and school is an unquestionable requirement however it is not satisfactory all alone by any stretch of the imagination, for it has turned into a basic element of value training that understudy have a few aptitudes and intrigues impossible to miss to them and they have to focus on them with an explanatory methodology.

Enumerating goals and interrelationship of modern museums and education:-

- Enlightening individuals inside of the connection of deep rooted training.
- Propagating science-based data.
- Virtualising and visualising learning process.
- Aims at arousing interest and curiosity instead of giving information about a thing.
- Education through museums should rouse, expand creative energy and enthusiastic mindfulness.

Knowledge gained at school and colleges are rather theoretical and students find difficulty in identifying any links between them and real life. Students learn to look at things from diverse perspectives by visiting so as to meet new individuals and new society's new place. Students confronting issue learning at school could end up being brisk learners in museums since it utilizes knowledge and faculties as a part of every single conceivable way. Learning activities in museums exposes the features of intelligence, the element of insight and abilities of the students which are not noticed at school or colleges, while student who experience issues in learning with conventional showing techniques can take in more effectively and quicker. To improve upon quality in education and learning environment the only way is promoting cooperation between museums, schools, colleges, teachers and professors.

Objective of Research

In the current scenario of educational system to compete globally, the quality learning environment has to be adopted by implementing innovations in pedagogy. Learning through museums is new innovations in teaching. Thus, this paper aims to create awareness regarding the concept and benefits of museum with educational purposes among the students and the teachers of schools and colleges. The paper being a conceptual paper, provides an overview of concepts and applications of various museums and innovations in education system through museums. Further, few case studies on benefits from museums with educational purposes applied in developed nations are cited which can be used as best practise in schools and colleges in India also.

Studies on Museum education

(Kratz & Merritt, 2011) in his paper provides an overview of educational innovation in a broad cross section of U.S. museums, citing selected examples. Museum educator's shows aptitudes like basic deduction and investigative thinking abilities; the capacity to

filter through an enormous measure of information and blend data. To achieve a more national gathering of people, the museum as of late made a progression of educational program for classroom use for learning science, innovation, designing and math while bringing issues to light about the vocations in the assembled environment, for example, building design, arranging, development and building. The National Building Museum has developed a kit called "Bridge Basics" and investigates the universe of bridge through seven key lesson arranges (www.nbm.org). The students of grades 5–9 have to make distinctive types of bridges by utilizing playing cards, wood sticks and string, students must cooperate by building arch, string, cable-stay and suspension bridges. The students later had to test the bridge by using different weights. By doing so the students learnt valuable lessons when bridge falls and determine what went wrong further learnt to imagine for an alternate scenarios. Thus this makes the approach effective as these lesson plans are tied to real world examples. On their path home from school, students see bridges around them and unite these lessons to the present world. . A key component of the system is to organize a basic specialist to visit the class to depict their occupation way, their day by day assignments, and abilities expected to succeed in their employments.

(Dogan, 2010) in his study wants to investigate what primary school students benefits from museums with educational purposes in the schools of Ministry of National Education, within family and school frame. The participants of the study are 50 Social Sciences teachers of schools of Ministry of National Education in the province of Bursa, who deliver Social Sciences course, art historian, archaeologist, and staff that are employed in the Bursa City Museum and Archaeology Museum. Relation was identified between teachers' having libraries that they can enhance themselves skilfully, allotting time for exhibition hall visits, and sense that Social Sciences course makes delight inside them. This adds to social skills and manners, in other words, being concerned about cultural or natural inheritance. There were found to be some relationships between teachers' allowing time for museum visits and students' feeling interested in the topics of social studies lessons. Students attach more importance to learning through games, language study and artistic works such as books and pictures. Thanks to the technology developing fast, the audio visual sector has developed much. On account of the innovation growing quick, the varying media part has grown much. By and by, relations have been resolved between dispensing time for museum visits and the delights inside teachers challenge by Social Studies course.

(Tasdemir, Kartal, & Ozdemir, 2014) aimed to determine pre-service teachers' views about science centres and museums, as out- of-the-school learning environments,

through case study. For that reason, the pre-administration educators were given to take part in out-of-the-school learning situations which were planned structurally, and their states of mind and perspectives were learned toward the end of the procedure. The outcomes demonstrated that they held positive perspectives with respect to the exercises identified with science focuses and museums. In addition, pre-administration teachers' mentality midpoints were sure both in general assessment and under the subject of characters toward science and innovation course. In addition, pre-administration educators' mentality midpoints were sure both in general assessment and under the subject of characters toward science and innovation course. As positive perspectives they said that exercises were visual, perpetual information shaped in result of the exercises, they would have the capacity to utilize the learning they had gotten in everyday life, they had the chance to apply balanced, and that they could procure new information.

In order to improve the creativity of the students, story completion activities are carried out. The first and last sentence about the story of an object in the museum is given, or a story is cut at a certain place, and students are asked to complete it. The real story and students' stories are compared and debated (Oğuzoğlu, 2007).

(Konya, 2012) identifies the primary-grade teacher candidates' views on museum education. In his study 209 primary-grade teacher candidates who were seniors in the Primary-Grade Teaching Program were surveyed A study arranged by the analyst was utilized to get teacher applicants' perspectives on museum and gallery training. Amid information investigation, frequency and rate were utilized. Accordingly, it was found that teacher applicants had positive conclusions about museum training, but a significant number of them (71.30 %) perceived that museum education should be used especially during history lessons. They specified that museum education was examined generally in social studies and development history courses amid their college training. For the teachers who think multi-directionally on this subject, museum training as a successful strategy ought to be utilized as a part of each lesson.

Applications of Museums

Mostly teachers and students are in the perceptions that museums have objects, coins and fossils that can assist as illustrative cases of what is communicated in different subjects. Teachers and students in the developing countries who visit museums and art galleries, realize that there is a necessity to organized unique preparing projects for making mindfulness among the students, teachers, professors and the general population. This will give pleasure and happiness not only to the students but also to the teachers as it take the learners away of the shut classroom or workmanship room air,

which understand that the craftsmanship lessons can be studied in a more invigorate, living and experience expanding environment.
Museum's exhibit objects in a way that would provoke

- Creative intuition and viable learning.
- Serves to accomplish the long haul objectives of instruction.
- Also capacities as labs in which instructors can have better idea about their student.

English pedagogue, Hord, is of the opinion that with mutual and shared targets and results helpful projects in the middle of museums and schools ought to be energized and actualized, within the ideologies of development and working together. The possibility that museums which are being visited yearly or rarely by schools ought to be changed to create extends with the goal that they can be utilized dependably as a part of training on standard premise. The researcher believes that instructors don't have satisfactory data and experience about how they can utilize museum exhibition halls in pedagogy.

In this change of instructive project, which has been orchestrated and shaped by methodology as action centred, adjusting learning and capacities from the social studies angle, giving chance to association because of environment bearing his/her own particular encounters and individual contrasts. According to (Merriman, 2000), various focused solicitations in museums creates diverse impact on viewers by reading and watching written texts and descriptions in the form of story- telling and viewing dramatic performances. The Museum composes addresses and presentations by noted researchers, academicians and creators all the time on a scope of subjects from workmanship, craftsmanship history, structural planning, legacy and some more.

The increasing number of museum programs in multidisciplinary subjects is now spreading throughout the country of India. These programs are provided for very young children to adult, and their families through formal education systems. Given below is the list of all fascinating museums in India that must be visited so as to have a fantastic experience.

1. Academy of Fine Arts
2. Ahmedabad Science City
3. Air Force Museum
4. Archaeological Museum
5. Architectural museum
6. Art Museum
7. Assam State Museum

8. Bangalore – Marine, Law, NIMHANS Brain Museum
9. Birla Industrial & Technological Museum (BITM)
10. Botany Museum
11. Calico Museum of Textiles
12. Delhi Police Museum
13. Government Museum Bangalore
14. Kolkata Science City
15. National Handicrafts and Handlooms Museum
16. National Agricultural Science Museum
17. National Police Museum
18. National Rail Museum
19. National Science Centre Museum
20. Natural National History Museum
21. Nehru Museum of Science and Technology
22. Physic Museum
23. Red Fort Archaeological Museum
24. Science and Technology Museum
25. Shankar's International Dolls Museum
26. The Hindustan Aeronautics (HAL) Aerospace Museum
27. Virtual museum
28. Visvesvaraya Industrial and Technological Museum
29. Zoology Museum

Few of the above listed museums are briefly explained so as to make students and teachers aware of the application of museums for learning and teaching and make use of them as an innovative pedagogy in their curriculum.

1. Birla Industrial & Technological Museum (BITM)

- AT BITM there is Fascinating Physics Gallery, which has 28 interactive exhibits on Mechanics, Gravitation, Light and Electromagnetic Waves. This helps students to understand the concepts of physics easily. Further, students can visualize about coal mine looks like and what is happening deep down below the ground in that dark world.

- BITMS has underground mock-up coal mine Thus students who can't visit to a real coal-mine can visit BITM's Mock up coal mine, the only one of its kind in India. The students can have an idea of the coal cutting methods - both manual and mechanical, how coal is transported out of the mine, why

the tunnel does not collapse after the excavation of coal, what are the safety measures adopted etc. This mock up coal mine might help students to decide about their carrier in this field ahead and even make aware of the uses of coal clearly. (http://bitm.gov.in/.)

2. National Agricultural Science Museum (NASM).

- AT (NASM) situated in the National Agricultural Science Centre campus of ICAR in New Delhi, is the first of its kind in the country. Here child learns a practical demonstration of a water cycle rather than from his science book. The students with a touch of a button can feel there thunder, lightning and then there is rain in the room. Water evaporates from the ground, clouds form and rain follows and after a few seconds everything goes quiet followed suddenly by a rainbow on the top of the room is seen.
- Further as you enter the museum on the first floor, six steel pillars greet you. They symbolise the six factors that agriculture is dependent on—soil, water, climate, tools, farmers and seeds.
- Students who find the difficult to remember from books, for example where black gram are grown in India, can easily remember by pressing the buttonmarked 'black gram' and the map lights up highlighting the states where the pulse is grown. The visual display helps student to remember faster. (http://icar.org.in/).

Thus students, researcher, teachers and professors who are confused either in any particular topic, subjects, about their topics for research, or choosing their carrier in life in particular area can visit respective museum and have their confusion clear and be confidents. Knowledge gained by this visit in museum is long lasting and permanent. Thus, the new educational era focus on the improvement of a fundamental set of skills and to attain quality education with proper learning environment.

Museums…. Encouraging Activities

- The above listed museums has started to attract attention in various fields which necessitates the effective usage of different museum based on the subjects for the aims of enriching the academic programmes of the students,

teachers and making quality learning permanent and entertaining. Nowadays, to attain quality education through museums various types of innovations are used by the museum lecturer. Depending on the topic of research either by the student or the teacher theme based events are organized so as to gain practical knowledge and students and understand better. Museum conducts various encouraging activities as and when required for the schools and college as follow

- Temporary exhibition
    - Water Day
    - Botany Day
    - Wildlife Day
- Mobile Museum
- Workshops
- Research work
- Lecture Series
- Touch and Feel – Concept learning
- Gallery Talk
    - Picture Gallery
    - Coin Gallery
    - Sculpture Gallery

Technology –Internet, Media

Adult education

With the adjustment in the instruction framework the new educational programs motivations parents to effectively take an interest as though they are instructors, and can screen the training and learning procedure of their tyke. In the event that parents are to think more about their kids in their adapting, then the kids will improve in studies. Adult Education goes for spreading instructive chances to those grown-ups, who have either lost the possibility of picking up training for reasons unknown in past or have the period of formal instruction is route back, however now feel a requirement for learning of any sort - fundamental training, learning craftsmanship and art for acquiring reason, ability improvement and equivalency (http://mhrd.gov.in/grown-up training). Museum even sorts out workshops on a scope of craftsmanship and specialties for grown-ups in order to make them practical sound.

Digitization of Museum's collections

In order to achieve the maximum benefit of these museums to the students, the academician and the society and the ultimate aim of bringing the collections of these museums closer to the public from all walks of life, digitalization use in education is made compulsory in the educational landscape. The details of the collections of various museums among various field can be seen by the public from the Digital repository setup, developed by The Ministry of Culture as a web portal www.museumsofindia.gov.in.

Virtual Museum

A virtual museum is an assemblage of electronic artefacts in the form of digitization, growing fast now a days. They offer a way of content, interactivity and visual learning. Students and teachers are motivated to use research-based skills through virtual museum and also help students who are visual learners rather than theoretical based learning in classroom. It sometimes even help the parents to guide the student at home and clear their concepts by learning. The museums help to integrate technology and provide new, meaningful, contemporary opportunities for students to gain management skills. Thus education in museum which innovative pedagogical techniques if encouraged and implemented in the field of all areas of educational landscape can have positively impact in school and even in higher education in India.

FINDINGS

As seen that the innovative themes of each museum listed above will help students, teachers and adults to learn and have knowledge not only in history and archaeology subject but various subjects and courses can be learnt through museum education. Thus from the above literature review , case studies and it is clear that learning through museum educations will focus on the development of a core set of skills of the students in schools and college and will also help teachers in the new pedagogy methods.

The Road ahead…..

Museums ensures the road towards the quality learning environment in the educational landscape, which reflects the success of the learning process. Environment variables influence the nature of school and colleges significantly to attain quality learning. Thus, educating through museum and quality learning environment, the standard of education will definitely increase. In this respect, the students, teachers and professors must create an environment and focus on extreme utilization of educational activities to accomplish quality education and every one of these confirms to the supremacy of museums in education. Lastly, the author concludes that acquiring knowledge through school and college helps students and teachers to start imagination on the topic studied or taught whereas learning through museum helps them to make their imaginations clear.

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(Proceedings available for download at rku.ac.in/icre)

RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

Value-based management education in India

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ABSTRACT

The Management education in India dates back to the ancient times whereas emergence of modern management education began with the establishment of IIMs in India. The boom in management education came in with the approval by AICTE to the private sector to set up B-schools and accolade post graduate degrees for diploma in management. Today there are around 4000 B-schools in India. But the foremost parameter of education that is 'quality' is missing in many of these Institutes. This research paper highlights the missing links in management education and suggestions for remodeling it. The paper aims to examine the major challenges that the Indian management education is grappling with and explore possible ways through which the system could be made robust.

SUMMARY

This research work is conducted to evaluate the reasons for skills gap arising from low academic output and introspective attempts to meet the expectations of industry from the management graduates.

Keywords: Management education, Curriculum, Pedagogy, B-schools, AICTE

INTRODUCTION

In the India, management education has been developing over past fifty years. The Indian landscape of management education consists of the IIMs, (Indian Institutes of Management) University affiliated management institutes, autonomous institutions approved by the AICTE, institutions without the approval of AICTE, and foreign universities are also providing degree and postgraduate degrees in India.

The Union Government, in 1987, accorded permission to set up the AICTE, which is a regulating body for professional education in India. As per AICTE data the number of institutions imparting management education has increased in just five years. The intriguing question accosting all the stakeholders is; why the permission was granted to these many institutions in such a short time?

During the last five years, the AICTE, by granting permission to more and more number of institutions, as well authorized a substantial increase in the intake of students by more than 300%[5]. Therefore, these figures reveal that the AICTE adopted an expansion strategy of quantity over quality of education inculcated. Experts feel that AICTE's policy is liberal in terms of permitting new institutions and increasing intake of students.

Secondly, institutions enter into education sector to make quick profits instead of contributing to the field with some genuine concern. This proves that education has become a business for huge profit making. Thus institutions that are imparting management education have totally ignored the quality aspect and concentrated merely on quantity and profit making. Furthermore, lapses in course upgradation and banking on a few core subjects and niche electives added to the existing issues. The facet of 'skill quotient' is lost in management education. Faculty members having industrial experience or teaching through innovative pedagogues are less in numbers to share their expertise.

PEOPLE SPEAK

Kushankur Dey[2] in an article says that although management institutions in India have increased in number, the quality has been obscure. The management program on its plus side has had a greater outreach among a huge section of the society but one needs to evaluate the program on a value-based system. In the article titled 'Do we need value-based management education in India?' Dey questions the effectiveness of the program, the quality issues, the industry-academia parlance and the role of the regulators of the management education in India.

In the article 'Rethinking the MBA', Datar S.[1], Garvin D. and Cullen P. talks about branding and pricing of the management program. They state that branding of B-schools should be in the context of 'rigor' and 'relevance' of the program. The output of the program should be measured in terms of pedagogy, updating of curriculum, evaluation pattern, application of concepts taught, and feedback of the course structure. The role of the regulator should be that of a 'watch-dog' and not of maximizing revenues and increasing the enrolment programmes year by year. To pursue an MBA in India from a reputed B-school it takes about 8-9 Lakhs course fee for two

years apart from food, hostel and such other expenses. But the real picture of price paid or cost incurred in MBA is tested at the end of two years program.

Tim Westerbeck[7], after attending a global conference in Delhi called 'Rethinking and Rebooting', suggests few major areas of opportunity for India given the distinctive culture, demographics and location. He recommends that engineering education system, business education system and the private enterprises should join hands to mine the rich intellectual capital of India. He advises management leaders to supply MBAs locally but to train them globally and to establish deep-rooted partnership with business houses. B-schools should be evaluated in terms of quality and not on the basis of rankings and accrediting systems.

A study that was conducted by Aspiring Minds, a talent management firm in Gurgaon, found that Indian B-schools don't teach the basic skills like communication, that are vital for facing management job interviews. The firm based its conclusions on an 'employability test' it conducted on 32,000 MBA degree holders from 220 b-schools across India. The test included topics ranging from grammar to quantitative analysis and found that hardly 10% of those tested had skills that recruiters typically look for while hiring management graduates[4].

Earlier, a Bangalore-based education consultancy MeritTac conducted a similar study of about 2264 MBAs in the country and found that barely 21% of the MBA students were employable[4]. Industry experts say that these findings are a proof that India's MBA curriculum is flawed because of its emphasis on mugging up rather than on hands-on experience.

Mr. Mukherjea[4], head of the equity division at Mumbai financial services firm Ambit Capital, states that industry-specific skills and vocational training is "glaringly missing" in most Indian b-schools. The "one-size-fits-for-all curriculum" do not offer students a chance to get an in-depth understanding of their area of specialization.

Mr. Rohit Sarin[4], head of Client Associates, a private wealth management firm in Gurgaon, says that companies like his are opting to hire individuals with a few years of work experience over fresh MBA graduates who have little or no work experience prior to enrolling in business school in India. To make Indian MBA graduates more employable, he says business schools need to "make a conscious effort to bridge the gap between theory and practice."

On the other hand Mr. Nishant Saxena[6], in an article titled 'Missing elements in an MBA' suggests the reasons for the gap between industry requirement and academic curriculum resides in the attitudes, big picture thinking, business communication, grooming/personality/confidence, corporate exposure, domain knowledge, sales and customer service, basic managerial skills and ethics.

Padmashree Bakul Dholakia[3], who is the director general of IMI Delhi, states the major challenges that Indian management education system faces are a lack of focus on quality; an acute shortage of skilled faculty; an inadequate interface with the corporate, failure to upgrade with changing business environment, failure to upgrade curriculum and contextual case material; a scarcity of leaders to drive the necessary change and inadequate focus on robust governance systems.

MATERIALS AND METHODS

The sample size for this exploratory research is 50 faculties from reputed B-schools and 50 HR managers from the corporate world who hire MBA graduates. The research is conducted through questionnaire and discussion method by using non-probability technique of sampling.
The study is useful for reviving the current scenario in management education by testing each and every parameter on the basis of quality pursued. The feasibility of the suggestions made in the paper varies from Institute to Institute but are valuable at large. The observations of the research conducted among management faculties and HR personnel from the questionnaire are represented in Table 1:

Table 1

Sr. No.	Points in concern	Affirmative	Negative
1	Is excluding GD and PI from the screening process of MBA aspirants, a wise decision?	6	94
2	Is merely 'appearing' in entrance tests for MBA enough for a candidate to enroll in MBA or should there be a lowest accepted cut-off limit?	24	76
3	Should the candidates aspiring for MBA be allowed to enroll without appearing for entrance exams for the sake of filling a huge number of vacant seats?	32	68
4	Do you find it reasonable that AICTE has approved so many colleges that today there are umpteen number of seats available compared to the number of students aspiring for MBA?	12	88
5	Majority of the students that enroll for the course join with the right attitude to aspire management as a curriculum and are actively interested to take-up entrepreneurial activities.	29	71
6	There is a huge mass of students in India, paying lakhs of fees to enroll for the course, who are willing to become MBAs. Are these students aware or even willing to develop entrepreneurial aspirations and attitudes for their career?	45	55
7	The MBA program is designed to develop globally competent workforce and moreover it encourages developing entrepreneurs but is the current curriculum matching with the industry needs?	2	98
8	Majority of the focus of faculties and the institutions is not in students' development but in course completion. Their efforts are more in the direction of students' ranking rather than in developing skills or training them for entry level jobs. Is it so?	21	79
9	Given poor soft skills and poor communication skills in majority of the students, are adequate efforts made by the system to polish students accordingly?	3	97

10	The job openings that are available for business management students are also available for other graduates (BBAs, BComs, etc.) having experience. Do you agree?	66	34
11	Are the contemporary MBAs, graduating in huge numbers are equivalently employable by the local industries?	8	92
12	Do you find a skills gap in the demand-supply of Management graduates in the industry?	78	22
13	Do you think there is a downtrend in Business Management Education?	60	40
14	Is there a need to reorient Business Management Education?	84	16
15	Should the Business Management Program be a technical course?	36	64
16	Is the AICTE able to fulfill its duties and responsibilities as a mentor to all the management institutes in India?	24	76

RESULTS AND DISCUSSION

- Student entry pattern for the management program needs to be more accurate in order to test students' verbal and written skills. Not only should that, but also on the job experience be made mandatory for students to enroll for the program. The job experience will discipline them in the corporate practice and students gain required knowledge of the market and maturity to be eligible for the course.

- GD and PI were the only parameters to screen out the students that have weak communication skills. It is expected from any MBA graduate to possess the basic communication skills. He/she must be fluent in communicating his/her thoughts into words to face the job interviews.

- There is a subject in MBA called 'Business/Managerial Communication'. It has been evident that any student cannot fluently read, write or speak in English just by merely passing the subject in the university exams. Extra efforts need to be made by inculcating the learning beyond the subject.

- Extending the duration of practical projects shall be fruitful for developing students and making them job ready. It makes them aware of the market scenario as well as makes them learn theory and its applicability.

- A model of curriculum should be designed wherein a student attends college during the day and works in the evening by which not only the cost of MBA is covered but also acquires the knowledge of four basic functions of management. This will help them to make an accurate decision of choosing their specialization areas as well.

- Upgradation of course curriculum seems no more an option but a key solution for the authorized bodies. A major concern for India is the lack of contextual case materials and the ignorance towards the importance and benefits of research. Instead of covering lengthy syllabus and grabbing theories, students should be encouraged to undertake research activities and prepare their own case studies.

- Objective type of questions and case studies should be the pattern of examining the students rather than descriptive essay writing. To the point answers should be brought in practice. Performance of the students in the practical projects should be evaluated by the concerned industry guide alongwith the institute guide.

CONCLUSION

Management which is more of an art should not be undertaken as a technical course. Efficiency of AICTE is questioned time and again but no concrete steps have been taken yet. A separate association of Management institutes should be formed to effectively understand the market scenario for MBAs. Premier institutes such as IIMs should participate for the greater good. The regulating bodies should make the academic and industrial tie-ups mandatory to put the students into practice of work environment. There is a dire need to make students employable by making them acquire skills needed in the entry-level jobs.

ANNEXURE

The following table shows the B-schools approached for the research study.

Table 2

Sr. No.	Name of the Institute	Website
1	AHMEDABAD INSTITUTE OF TECHNOLOGY (MBA), AHMEDABAD	www.aitindia.in
2	ANAND INSTITUTE OF MANAGEMENT, ANAND	www.aimrksm.org
3	ATMIYA INSTITUTE OF TECHNOLOGY & SCIENCE (MBA), RAJKOT	www.atmiya.net
4	BHAGWAN MAHAVIR COLLEGE OF MANAGEMENT, SURAT	www.bmefcolleges.edu.in
5	C. K. PITHAWALLA INSTITUE OF MANAGEMENT, SURAT	www.ckpim.ac.in
6	C.K. SHAH VIJAPURWALA INSTITUTE OF MANAGEMENT, VADODARA	www.cksvim.edu.in
7	C.U. SHAH COLLEGE OF ENGINEERING AND TECHNOLOGY, WADHVAN	www.ccetvbt.org
8	CHAUDHARI TECHNICAL INSTITUTE, GANDHINAGAR	www.ctimba.org.in
9	CHRIST INSTITUTE OF MANAGEMENT, RAJKOT	www.cimrajkot.edu.in
10	D.L. PATEL INSTITUTE OF MANAGEMENT & TECHNOLOGY, HIMMATNAGAR	www.vidhyanagari.org

11	FACULTY OF BUSINESS MANAGEMENT, MARWADI EDU. FOUNDATIONS GROUP OF INSTITUTIONS	www.marwadieducation.edu.in
12	G.L.S. INSTITUTE OF COMPUTER TECHNOLOGY, AHMEDABAD	www.glsict.org
13	GANDHINAGAR INSTITUTE OF TECHNOLOGY	www.git.org.in
14	GIDC, RAJJU SHIROFF INSTITUTE OF MANAGEMENT STUDIES, VAPI	www.grimsvapi.com
15	GOLDEN JUBILEE INSTITUTE OF MANAGEMENT AND TECHNOLOGY, SIDHPUR	www.gjmba.org
16	GROW MORE FACULTY OF MANAGEMENT	www.growmore.ac.in
17	HASMUKH GOSWAMI COLLEGE OF ENGG.(MBA), VAHELAL, AHMEDABAD	www.hgce.org
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26	MANISH INSTITUTE OF MANAGEMENT (NUTAN SARVA VID. KEL. MANDAL SANCHALIT MBA COLLEGE),	www.nootanmba.ac.in
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ACKNOWLEDGEMENT

We thank Dr. N B Sharma (Director- Shri Sunshine Group of Institutions), Sham H. Sachinwala (S. S. Agrawal Institute of Management & Technology, Navsari), Dr. M Samir Gopalan (Head Marketing- Indus Univeristy), Archan Mehta (Assistant Professor- LJIMS), Nivedita Srivastava (Dean- Oakbrook Business School), Dr. Parul Chotalia (Director- T. N. Rao College of Management Studies), Dr. Samir B. Patel (Principal- Grow More Faculty of Engineering), Dr. Neeraj D. Sharma (Professor- S.N.Patel Institute of Technology & Research Centre), Prof. Surbhi Ahir (Principal- SRK INSTITUTE), Dr Rajesh Khajuria (Director- C K Shah Vijapurwala Institute of Management), Prof Amit Patel (Chaudhari Technical Institute), Prof. (Dr.) Hitesh A. Patel (Director- Patel Group of Institutions) for their valuable time and assistance.

We would also like to show gratitude to Ms. Pooja Vaghela (Officer-Allahabad Bank), Mr. Manish R. (Area Sales Manager- Shubham Housing Development Finance Company), Mr. Pratik S. (Assistant Manager-HDFC Securities), Mr. Parag T. (Territory Manager-Star Health & Allied Insurance Co. Ltd.), Mr. Hemant Joshi (Credit Manager-Kotak Mahindra Bank Ltd.), Mr. Bhavesh Patel (Assistant Manager- Sanghvi Forging & Engineering Ltd.) Mr. Mehul Dave (Admin Dept., Rajkot District Co-operative), Mr. Vishal Dave (Assistant Manager- Federal Bank) who all have provided their insights and expertise that greatly aided the research, although they may not agree with all the analysis of this paper.

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Enhance Classroom Learning Using Innovative Teaching Learning Method for Theory of Mechanisms and Machines

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ABSTRACT

Kinematics of Machines is one of the essential courses of Mechanical Engineering undergraduate curriculum in which mechanical students have to learn relative movements and connection of various parts of machine to get desired output. In this article, the authors have presented innovative hands-on activity approach. This approach has been implemented and has been found that it enhances classroom learning. This method will assist teachers in achieving their teaching objectives. These hands-on activities are very low cost. It helps the students to think hard about how machines work and helps them not only to analyze but to implement real mechanism by understanding line geometry. The study results present here is actually drawn from students' feedback also which are taken on EDMODO, a learning management tool for the course.

SUMMARY

An innovative, low cost and hand on activity approach is design and implemented in the classroom to teach inversion of mechanismof four bar chain mechanism and it is proposed to teach some of the subject content of theory of mechanism and machines.

Keywords: Hands on activity, low cost method, Active learning, Inversion of four bar mechanism

INTRODUCTION

In most of the academic institutions in India, instruction based pedagogy is practiced and institutes are built to support it (1). However many professional bodies have identified deficiency in such delivering methodology and suggested to teach more about "real-world" engineering design and operations by changing such pedagogy to meet requirement of 21st century learner (2). So as a part of that many teachers are looking continuously for new ways to actively involve students in classroom learning which

develops very important qualities like critical thinking, deeper understanding create interest in subject content and enhances their presence of mind during the lecture (3). Learning through hands-on activity has proved a great success to achieve the same goal (4).

The kinematics of Machines is a basic course of mechanical engineering branch which includes describing methods of mechanisms, typical mechanism's analysis, and synthesis of mechanisms and study of moving parts of a machine without considering force effect (5). Now a day to teach one of the topics of this course named "Inversion of Mechanism", lectures are conducted using power point presentation, where images of actual machines, line diagrams and videos which shows construction and working of the machines or mechanisms are used. Compare to the olden days chalk and duster methodology, it seems interesting and less confusing. But in the process of teaching-learning, teacher should pay attention to the teaching of basic theory as well as practical application and connection, so the teacher' stand-up situation is changed. In teaching typical mechanisms, contacting specific case of daily life is very much important, such as crusher, sewing machine, mend shoes machine to analysis (6).

So, conventional method may not give a chance to engage students thoroughly. It gives just an idea and virtual imagination about construction and working of the mechanism. In this paper, the authors have discussed about some innovative approach to teach the said topic using hands on activity and give a chance to the students for learning while doing. The whole article is organized as follow: The next text heading gives the contextual requirement and limitation of existing teaching methods widely used. The following headings present: Designing of Session Plan using new methods, experience during implementation, and finally conclusions are drawn in last.

Contextual Requirement

Active learning strategies allows to make classroom live, by engaging and involving students during the class hours. And it plays a vital role to motivate students towards learning, higher order thinking and getting immediate feedback. Chalk and duster method may not elicit to 21st century learner because it is structured like one way communication and mostly focused on delivery part. Now a day's many faculties are also using power point presentation during the lectures also. However it does not serve the purpose of active learning at all the time though it totally depends on the faculty's instructional approach. At the same time, there is a barrier for the faculties to implement active learning strategies due to lack of high tech equipment and its availability in the classrooms. So this article gives the concept of low cost hands on active learning strategy which can be successfully implemented during the classroom hours as well as laboratory hours to understand theory of mechanisms and machine topic very well. For that one of the topics related to the content is chosen for demonstration which is "Inversion of four bar chain mechanism" because four bar chain mechanism is very fundamental and simplest type of mechanism.

MATERIALS AND METHODS

The hands-on activity is designed for IV semester, mechanical engineering students to construct a four bar chain mechanism using straw and pin which can help them to understand inversion of four bar chain mechanism. Session planning is done for one class period of sixty minute. First step in the session plan is to define prerequisites and objectives of the session. Accordingly activity and other content of the session can be finalized.

1 Objective of the session

a. Identify construction, working and application of the Four Bar Chain mechanism
b. Describe the terminology related to Four Bar Chain mechanism
c. Interpret various inversion of Four Bar Chain mechanism in practical life and differentiate between them.

2 Prerequisites

a. Types of motion of a link
b. Basic terminology of mechanism like link, pair, joint etc.
c. Difference between mechanism, structure and kinematic chain
d. Degree of freedom of a mechanism
e. Kinematic Diagram

After defining objectives, the next step is to define an activity and its timely involvement during the session. For that following changes have been made in the conventional teaching learning process of the one hour class which is discussed in table - 1.

3 Instructional Flows

3.1 Basic Understanding

Start session by linking previous session with current session, and specifies its purpose. To do a kinematic analysis of any machine or mechanism, it is first converted in to line diagram- called as kinematic diagram. Now consider that mechanism having n number of linkages where if one of the linkages is fixed, it can perform desired task. Now, in n linkages mechanism, if we fixed different linkages at different time we can get various types of output motion of linkages. Thus various types of mechanism can be generated by fixing different linkages which called as inversion of mechanism. Four bar chain mechanism has four linkages, so it has four inversions.

3.2 Hands on Activity

To conduct a 22 minutes of hands on session, following materials are required. Step by step instructional approach during the activity is discussed in the table – 2. Material requirement is given for each group: Candy Stick (4 Sticks per group), Pins (4 Pins per group), Blade/Cutter (1 No per group), Activity Sheet (1 No per group). Also one projector and computer/Laptop are required for power point presentation of the topic. Sample copy of activity sheet and take away sheet are also shown in (figure 1) for further reference.

RESULTS AND DISCUSSION

Initially it seems that proposed active learning method reduces the lecturing time which may affect delivery of the content and its quality. But here some observations have been drawn from comparison of proposed method with traditional session before and after implementation.

- As a part of activity, each group has to reflect their observations in activity sheet, which gives an opportunity to critically think and it develops technical writing skills. It also works as an assessment document and faculty can get indirect feedback of the session.
- During model making, students have to understand working of mechanism and find types of links, pairs and joints used, which gives a chance to apply knowledge.
- The team based hands on activity is adjusted in a one hour lecture to clarify the concept which develops the negotiation and teamwork skills.
- The students were found very excited about, and interested to, these activities compare to other class which is reflected from polls and feedback form.
- Assessment norms are defined for in-class teaching learning process using activity sheet. Also take away sheet is given to the individual students and instructed to submit it back before the next lecture. So faculty can judge amount of transfer of knowledge.
- Activity also mapped with different level of cognitive, affective and psychomotor domain. Table – 3 shows relation between various activates and skills developed.

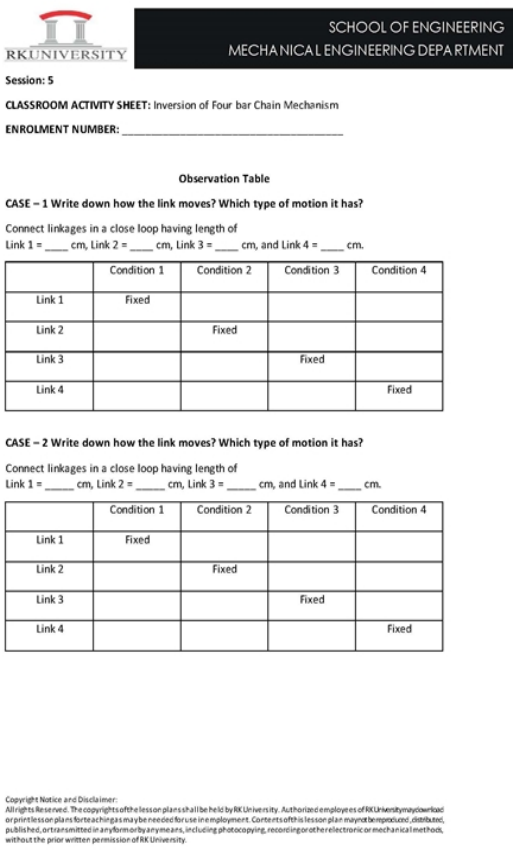
A care should be taken that faculty takes care in giving step by step instructions to create models completely and timely. Otherwise it may happen that student may lose their confidence and interest for further teaching learning process. Here some glimpse of in class activity is shown in figure – 2

CONCLUSION

The study was offered in even semester of 2015 to the group of 60 students. The method improves students' performance and develops skill among students. The students were found very excited and interested to do these activities. The feedback and comments from students were positive and encouraging which is clearly seen from polls (figure **3**) taken on learning management system EDMODO. In future, further research is required to collect the data and interpret effectiveness of proposed innovative approach of hands on activity over conventional method. The methodology presented here can be used as the starting point for the other faculties who wants to offer similar activity or deliver a same topic in class.

FIGURES

Figure – 1 Sample template of classroom activity sheet and take away sheet of proposed activity



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Session: 5

CLASSROOM ACTIVITY SHEET: Inversion of Four bar Chain Mechanism

ENROLMENT NUMBER: _____

Observation Table

CASE – 1 Write down how the link moves? Which type of motion it has?

Connect linkages in a close loop having length of
Link 1 = ____ cm, Link 2 = ____ cm, Link 3 = ____ cm, and Link 4 = ____ cm.

	Condition 1	Condition 2	Condition 3	Condition 4
Link 1	Fixed			
Link 2		Fixed		
Link 3			Fixed	
Link 4				Fixed

CASE – 2 Write down how the link moves? Which type of motion it has?

Connect linkages in a close loop having length of
Link 1 = _____ cm, Link 2 = _____ cm, Link 3 = _____ cm, and Link 4 = ____ cm.

	Condition 1	Condition 2	Condition 3	Condition 4
Link 1	Fixed			
Link 2		Fixed		
Link 3			Fixed	
Link 4				Fixed

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TAKE AWAY SHEET

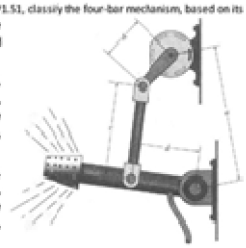
Student Enrolment number: _____

1. A mechanism to spray water onto vehicles at an automated car wash is shown in Fig. Classify the four-bar mechanism, based on its possible motion, when the lengths of the links are a = 12 in., b = 1.5 in., c = 14 in., and d = 4 in.

2. For the water spray mechanism in Fig. P1.51, classify the four-bar mechanism, based on its possible motion, when the lengths of the links are a = 12 in., b = 5 in., c = 11 in., and d = 4 in.

3. For the water spray mechanism in Fig. P1.51, classify the four-bar mechanism, based on its possible motion, when the lengths of the links are a = 12 in., b = 3 in., c = 8 in., and d = 4 in.

4. For the water spray mechanism in Fig. P1.51, classify the four-bar mechanism, based on its possible motion, when the lengths of the links are a = 12 in., b = 3 in., c = 12 in., and d = 5 in.



	Exercise - 1	Exercise - 2	Exercise - 3	Exercise - 4
Which link is fixed?				
Which work as drive and its type of motion?				
Which work as driven and its type of motion?				
Which work as coupler and its type of motion?				
Whether satisfy Grashof's law?				
Name the type of four bar mechanism				

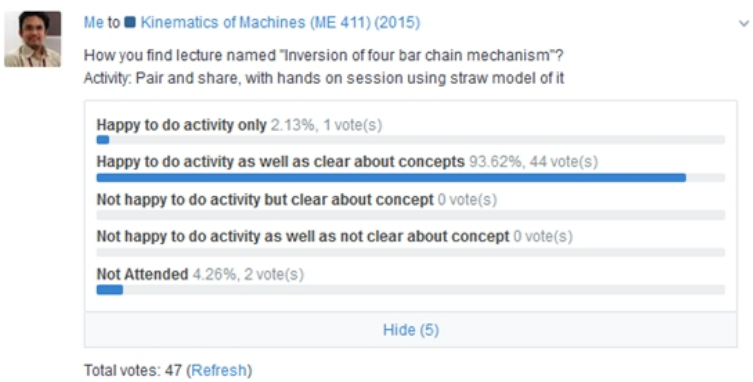
Faculty Sign:

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Figure – 2 Students preparing Models and observing motion of various linkages



Figure –3 Polls results taken by faculty for proposed hands on session using EDMODO



TABLES

Table – 1 Comparison of timely instructional flow structure of one class period for conventional and using active learning strategy

Instructional flow	**Conventional Session (60 Min)**	**Active learning Session (60 Min)**
Basic Understanding	10 Minute	10 Minute
Hands on Activity	-	22 Minute
Knowledge sharing with discussion	40 Minute	18 Minute
Conclusion	05 Minute	05 Minute

Table – 2 Step by Step activity and content for hands on activity

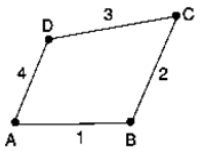
Activity		Content
1.	Group Formation (2 Minute)	Divide the class into small groups of approximately two students each. Keeping constraint of conventional infrastructure for seating in the class. Group may be formed on four to five students also.
2.	Material distribution (1 Minute)	Give all the materials and activity sheet. Roles and responsibilities may be assigned to team members by proper instructional approach to avoid messy classroom.
3.	Model Making  Line Diagram of four bar chain mechanism (3 Minute – Model Making, 2 Minute for observation and filling up activity sheet for each inversion, Total –19 Minute)	Instruct them as per below. Treat straw as link and use pin to join linkages. • Prepare four linkages as per given length (in cm): 3, 5, 6, 9, 7 cm each. • Case 1: Connect linkages in a close loop having length of link 1 = 6 cm, link 2 = 9 cm, link 3 = 5 cm, and link 4 = 3 cm. Also show them a line diagram. • Keep fingers on link 1 first so that it cannot move. (we can say that fix link1) • Now try to give motion to link 4 and observe the motion of link 3 and link 4. Write down answer of the following question in activity sheet. ○ Are the linkages moving? How? Which type of motion it has? • Repeat step no 4 and 5 by keeping finger on link 2, 3 and 4. • Now remove link 2 which having length of 9 cm. Instead of that use link having length of 7 cm. • Case 2: Connect linkages in a close loop having length of link 1 = 6 cm, link 2 = 7 cm, link 3 = 5 cm, and link 4 = 3 cm.

Table – 3 Mapping of classroom activity with skill-set

Activity	Skill Set developed during activity
Group Formation	Negotiation, Team work
Material Distribution	Assign responsibility
Model making	Application of knowledge, technical writing, willingness to learn, problem solving, critical thinking
Knowledge sharing with discussion	Knowledge, relate theory and practice, verbal communication,

ACKNOWLEDGEMENT

Authors would like to thankful to learn modern teaching learning process from Mr. Mohit Patel- Executive vice Precedent of RK University during LED course. Authors would also thankful to Dr. Veenakumar- coordinator of IEEC program and to Prof. Krishna Vedula-IUCEE for giving a wonderful opportunity to interact with various experts and educators across the globe during IEEC – pilot- I program held at BMSCE, Bangalore. Authors would also like to thank students and staff members of RK University–Rajkotfortheir co-operation and support.

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After All, It's Just a Written Assignment!

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ABSTRACT

The case study is about an innovative pedagogical technique adopted by a leading post graduate management institute. The protagonist, Shruti is a first year MBA student. She is contemplating on her progress as a learner in the course titled, 'Managerial Analysis and Writing'. The institute has developed this subject to polish the analytical skills of the future manager. The mode of teaching is case study method of analysis. But the twist is, rather than solving the issue in the case, the course puts emphasize on the framework to solve any issue identified. The other distinctive feature of this course is that it provides both, summative assessment as well as formative assessment to the students. The readers will get an idea about the rigor put on the formative assessment by the faculty (Facilitator) and academic associate (assistant to the faculty) for the ultimate benefiters, students (learners).

SUMMARY

The case study is about an innovative pedagogical technique adopted by a leading post graduate management institute.

Keywords: Formative Assessment, Learning-by-doing, Pedagogical Effectiveness, Self Analysis, Written Assignment

Hey Aisha! What's up? Shruti asked as she was settling down for the last session of the day. She extended, "Tomorrow too many assignments' deadline is there. You seem to be tired now, Aisha."

Na, na! I am just not interested in this subject. I don't know why are we studying it at post-graduate level? I mean, why is this subject even exists? And why is this scheduled as last session all through the term? Why? Why? Why? Aisha spoke in a single breath.

Stop! Stop Aisha! Shruti said. OMG! I got your point. Wow! You are in deep love. Both the girls winked and smiled. Shruti continued, Okay, tell me seriously, what's the matter? It's the last session of the term of this subject, no marks, but only grades given, no books, no mid-term exams, no end-term exams, and after all it's just a type of written assignment!

What? Just a written assignment! It's a skill building course, written communication skills, Aisha blabbered.

The conversation was interrupted as the professor along with her associate entered the class.

Prof. Asha and her associate, Annie, had brought evaluated assignments to the class. It's a feedback class today. The professor (known as Facilitator during session) presented a general feedback to the class. She demonstrated areas of improvement and progression of the class through examples taken from our assignments. The queries were resolved as and when they arose during the class. Following this, Annie distributed the assignments individually to each student. Shruti was very excited to see the grade. She had worked very hard for this assignment. Coming from a regional medium school, expressing the thoughts in a new language was a Roadies task for her. She got a 'B' grade, Aisha - 'B plus' and Sharman had secured the highest grade, 'A minus'. The Facilitator resolved few queries and the session was dismissed.

Shruti and Aisha also left for the hostel. While walking, Shruti again asked curiously, 'Aren't you happy with the grade?

See Shruti! Aisha replied, Point no. 1 -'B plus' is a good grade. Point no. 2 - But seven other people have received the same grade this time, and Point no. 3 – I have a 'B plus' again. I had received the same grade in the previous assignment too. So basically, I have no idea where am I standing; whether I have moved ahead or not. I really don't understand why don't they give us marks instead of grades?

Shruti listened quietly patiently; however, her opinion differed. She spoke, "the grades are just a part of the evaluation, which shows 'assessment of learning'. The most unique feature about this course, i.e. Managerial Analysis and Writing (MAW), is the individual feedback written on each page. This shows us the real path to improve upon, i.e. 'assessment for learning'."

Aisha was stunned at Shruti's this discourse. Both the girls continued moving towards the hostel.

Before going to bed, Shruti reflected on the day's happening. Somehow she was still stuck with the conversation which happened at a later part of the day with her roommate, Aisha. "What was she talking about? Are grades everything? What about learning, the self analysis? She has received an upper grade" She started pondering and her thoughts went back to the first day in the nation's top MBA Institute.

- **<u>3 months before</u>**

"After the exhaustive first session (One session = 75 minutes!), this 15 minutes are like icing on the cake", said Sharman to me, his neighbour. This informal chat came as a surprising blow to me. I just smiled and gave a nod. Sharman continued, "The first day at all places remains the same; ice-breaking session, all types of introductions happen – faculty, students, blue print of the course, syllabus, types of assessment, expectations of/from the students, etc. Nevertheless, this session seems to be interesting. What do you say?" Without waiting for me to respond, he restarted with his one-sided conversation, "Economics, Marketing, Human Resource, and Finance, are subjects which we heard somewhere at the

under-graduate level, from our elders, coaching classes; but nobody told us about this MAW. I found this to be something new. So, what do you think Shruti?"

Meanwhile, a senior female faculty accompanied by a young woman entered the class. Thank God! I mumbled.

The faculty introduced herself as Prof. Asha Sinha. The young woman silently moved towards the last row and settled down. Without wasting any time, she came directly to the point, "Managerial Analysis and Writing- What do you expect from this subject?"

The responses came from various directions of the class as business writing, letter writing, and how to write professional e-mails.

Prof. Asha nodded positively at each reply showing both her thumbs up alternatively. "Okay! Let's comprehend this subject title word by word. We all understand the meaning of 'Manager Tasks' and 'Writing' and few of us have explored these terms here too. Still, all of us have skipped a word. Which one?" she inquired. The class pronounced in a chorus, "Analysis". "Wow! This one was easy." Sharman and I whispered to each other. "Bingo" the professor replied. She spoke in a composed tone, "This is what we are here to learn, understand, execute and reflect. Analysis – Its technical definition, please Google it. Here, inside these four walls, we will study it as a skill to be developed, polished and nurtured. As managers, we have to lead a team, bring profit to organisation, take troubleshooting steps and most importantly identify the core issue. Managers receive segregated data, dynamic statistics, limited information, incorrect information; few things are kept officially hidden; with all these restrictions, they are intended to visualise a clear picture of the situation and take quick decisions for the future of the organisation. And 'You' have decided to put your feet in that shoe."

She took a breath. There was a pin drop silence. The whole class was listening attentively.

A few seconds later, Sharman raised his hand and said, "Skills are in-built. So how is this subject going to help build our analytical skills?"

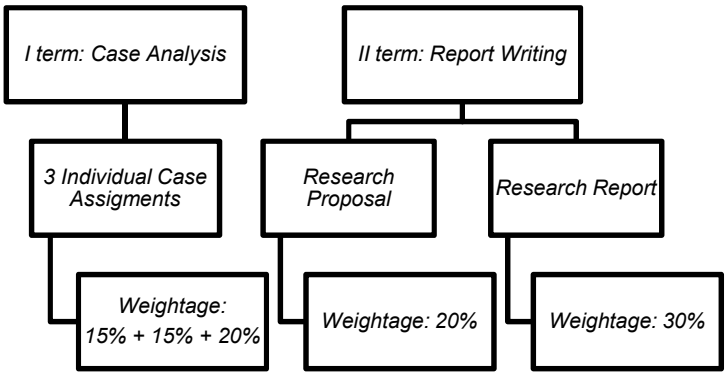
The professor seemed to be happy with this open dialogue. She countered, "With rigour practice. No books, no exams, no marks; only practice, continuous practice. The ultimate objective is to polish the skills which all of us possess. For this, we have chosen case-study method of analysis. The situations are pre-determined in case study format ranging from a half page to 20 pages.

The receiving team now gave a completely blank look. They appeared confuse now. Prof Asha was expecting the same reactions. Since the first year of teaching this course, all learners had the same facial expressions. "Why not?" she thought, "since childhood, the learners are scrutinise with tests, half-yearly exams, final exams, competitive exams, marks, distinction; these are the measurement of their learning curve, benchmark for their next goals. Surviving in the competition is the only goal of existence; ethics, empathy, impartiality, open-mindedness, reflection and cool headedness are mere terms of spiritual discourse for them."

One of the students dared to question, "Ma'am, can you please elaborate on the blue print of this subject? Everything seems to be alien - case study, skill building. We are unable to figure out anything."

The professor responded affirmatively and subsequently she drew a chart on the blackboard. She was glad that the students have built an open channel of inquiry.

MAW – 2 Credits Course



Addressing the students, Prof. Asha specified that MAW, being a skill course, adopts a rigour process of learning-by-doing approach. The term 1 is based largely on the **case methodology.** She pointed out, "In this institute, each course will have a few case studies as teaching pedagogy. Everyone would give you a case study to read, understand and solve the explicitly stated problem. But in the 'real' world, one needs to identify the core problem from the available information. Here, MAW will come to your rescue. We will discuss and analyze each case in the class, try to identify the core issue(s) and search for alternatives to resolve the identified issue.

The faculty plays a role of facilitator who encourages open discussion and explores different perspectives on single situation. Based on the class discussion, the learners, i.e. 'you' write decision reports for each case and submit these as individual assignments.

The course lays stress on the responsibility of the learner in the learning process. Therefore, each assignment submitted by every learner is evaluated and detailed written feedback is provided. Further, each assignment is also followed by a feedback class to share the observations of the evaluators and bring out the learning areas more specifically."

Aisha interrupted and questioned, "This means no marks? How would we get to know our growth? There must be some measurement tool for assessment."

Prof. Asha stayed unfazed with this unexpected break. She answered calmly, "As I have informed earlier, there would be no mid-term, end-term for this subject. The course evaluation is based entirely on assignments, individual and group, as assigned in the class. There are no hall examinations for this course. Each assignment is an exam in its own nature."

She added, "Broadly speaking, assessment falls in two categories. The first one is assessment **of** learning with which everyone is familiar. In this one gets marks, grades; the one which tries to measure the extent of achievement with respect to defined expectation. This is also known as summative evaluation. The second type of assessment is assessment **for** learning, also called formative evaluation. While all assignments are graded, detailed feedback is provided (with pencil) on the blank side of each page so that the learner can track his/her progress. The evaluation parameters are designed is such a way that the students can self analyse the learning progression. It is, therefore, the learners are urged to work on learning agenda and action plans for learning after each feedback. "I hope all things are clear now Ms Aisha" Prof. Asha took a pause to take any queries from students.

After resolving few queries, Prof Asha resumed her lecture, "MAW is taught by a **team of faculty members and Academic Associates (AAs)**. The Academic Associates assist the faculty members in class preparation, assignment evaluation, student communication, managing the course logistics and handling the back office. All assignment submissions are made to the AA attached to one's respective section. Further, the Academic Associate can be contacted for queries (if any) related to the evaluation, class conduct, and receipt/non-receipt of course material." By the end of the session, she introduced the young woman sitting in the back row as Annie D' Souza, one of the AAs.

This session was succeeded by few practice sessions on alternate days, so that future managers are exposed to each and every aspect of this new pedagogical technique and get trained on it. During breaks, the students discussed about this course only. Every time some or the new element was tossed, e.g. there could be no one right answer while analysing any case study. Each student could come up with a different problem as per his/her knowledge & experience and, provided it has to be justified objectively using case facts.

- **A week later**

A small case of three pages was distributed two days before the case discussion session. The class discussion was average – the Facilitator drew a mind map on the board and threw some open-ended questions, the learners attempted to answer those questions from the case. Then the Facilitator filled the blocks of mind map, gave structure to segregated facts and concluded the lecture with the first written assignment. The assignment had to be submitted in the hard copy as well as soft copy format within four days. By the evening, a formal communication regarding assignment submission details was mailed by MAW team.

For the next three days, no one from the student community, even looked at that case. On the night ahead of submission date, all of them remain awake for the whole night to complete the report. It was fun; both boys hostel and girls hostel were full of life - 120 students asking the same question to each other: "What is the problem?" Aisha and I, too put a lot of efforts in analysing the case study to the best of their understanding and writing the analysis report.

Report done. Online submission done. Now only need to get a hard copy of the report and then party! Sharman thought.

- **After 2 weeks**

First individual case assignment feedback session held. Prof. Asha entered with a smiling face and wished everyone cheerfully. Annie brought the pile of evaluated assignments. Everyone was excited to see the grades. The professor presented a general feedback ppt on the overall class performance. The presentation was informative, but it was long and boring. She applauded the genuine efforts made by the students in the first assignment itself. In the last 15 minutes, Annie distributed the assignments. Aisha got 'B' grade, Sharman and I acquired 'C minus' grade.

But wait! That's not enough. The assignment was full of something scribbled with pencil. Actually the text in pencil was more than what we had typed. This is what Ma'am was saying about. The formative assessment! Specific comments were given to the content, language, presentation and formatting with an 'overall' remark. Overall remark written in my assignment was as "*Need to work more on conceptual clarity regarding case analysis. Keep learning!!!*"

Prof. Asha wrapped up class by announcing about next case discussion after two days. The circle of case discussion, assignment submission and feedback class was re-lived.

Annie, our Academic Associate, has a cool personality. She is neither as strict as the professor nor too casual with the students. But they can informally chat with her about issues related to the submission, 'raatri jagran', how long was the queue at the printer, or request being a bit lenient with submission time deadlines. During one of those conversations, she explained us about the evaluation pattern. She told us that each assignment is screened thrice before the final grading. It takes almost 45 minutes for an AA to go through one complete assignment! All the remarks are then cross-checked by the faculty.

Aisha, Sharman and I achieved 'B plus', 'B' and 'C plus' grades, respectively, in II assignment. Two students got 'F' grade as they copied the assignment from the online sources. The improvement is clearly visible; still there is a long journey to travel, I convinced myself.

The III individual assignment was the final case analysis assignment. This was the last attempt to improve the grade of this term. This time I started making report two days before the final submission. Aisha had finished her assignment within four hours only this time.

- **Today**

Sharman received the top grade, 'A minus'. Aisha and I achieved 'B plus', and 'B' grades, respectively. Sharman felt on the top of the world. The overall remark in my assignment was, "*Reflects efforts are put! Good presentation! Keep Learning and Improving!!!*" With this, Shruti went to sleep with satisfaction in her heart and a smile on the face.

The next day Prof. Asha asked Annie and Suyash (the other AA) to prepare an AOL report on the basis of results of term 1.

Annie prepares the following note:

Program: **MBA** *Subject:* **Managerial Analysis and Writing (MAW)**
Batch: **20XX-20XY** *Section:* **1A**

Managerial Analysis and Writing (MAW) is a 2 credit course, and is taught across two terms in an academic year. Term 1 includes three individual assignments and weighs 50% (15% + 15% + 20%). Term 2 includes two group assignments, namely the research proposal (20%) and research report (30%) which amounts to 50%.

This report is a compilation of result of first term, i.e. Term 1. It links the learning, learning goals, its objectives, traits and evaluation methodology to ensure assurance of learning (AOL).

AOL Evaluation for Term 1

For the purpose of AOL, 50% of the overall course evaluation has been used. This includes outcome of three individual assignments. The rubric maps the evaluation component to the trait, defines standards and target population for the learning objectives of the learning goal stated below:

L.G 1: To equip students with analytical thinking skills, critical reasoning skills and systematic report writing skills for decision making

- *L.O.1.1: Learning rigorous and logical process of problem solving and decision making*
- *L.O.1.2: Developing the written communication skills (Term 2)*

The AOL Matrix

S. No.	Trait	Method of Evaluation	Below Expectations	Meet Expectations	Exceeds Expectations
		Grade	**D- to C**	**C+ to B**	**B+ to A+**
1	Learn to apply a systematic and logical process for diagnosing business situations, problem solving and decision making	Three Individual Case Assignments	Demonstrates an inadequate understanding of the issue(s) involved; Unable to apply a systematic and logical process for diagnosing business situations.	Demonstrates a reasonable understanding of several (or most) issues involved; An acceptable understanding about applying a systematic and logical process for diagnosing business situations, problem solving and decision making;	Demonstrates a thorough understanding of the issue(s) involved; Presents an insightful and thorough analysis of all the issues identified by applying a systematic and logical process for diagnosing business situations, problem solving and decision making
	Target Population		**20%**	**60%**	**20%**

Sr. No.	Trait	Method of Evaluation	Particulars	Below Expectations	Meet Expectations	Exceeds Expectations	Grade F	Total Students

			Grades	D- to C	C+ to B	B+ to A		
1	Learn to apply a systematic and logical process for diagnosing business situations, problem solving and decision making	Case Assignment No. 1	*No. of Students*	30	33	1	0	**64**
			% of Students	*46.15%*	*52.31%*	*1.54%*	*0.00%*	
		Case Assignment No. 2	*No. of Students*	16	38	8	2	**64**
			% of Students	*25.00%*	*59.38%*	*12.50%*	*3.13%*	
		Case Assignment No. 3	*No. of Students*	10	41	12	1	**64**
			% of Students	*15.63%*	*64.06%*	*18.75%*	*1.56%*	
Compared against target population				**20%**	**60%**	**20%**		

Interpreting the AOL Matrix

(I) Trait 1: Learn to apply a systematic and logical process for diagnosing business situations, problem solving and decision making

Only one trait is being evaluated over three individual case assignments. The target population defined in the matrix is compared with the result of case assignment no. 3. This is done to reflect the gradual progression of the students during the course of four assignments. The performance of the students is assessed in grades.

- The **"below expectations"** category ranged from grade D+ to C. In the first case assignment, 30 participants *(46.15%)* fell in this category. Showing gradual progression by case assignment no. 2, 16 participants *(25%)* received this score. By the end of the first term, in case assignment no. 3, 10 participants ***(15.63%)*** obtained these grades, which were lower than the set target of 20% showing positive learning.

- The **"meet expectations"** category ranged from grade C+ to B. In the first case assignment, 34 participants *(52.31%)* fell in this category. By case assignment no. 2, 38 participants *(59.38%)* received this score, reflecting a reasonable progress. This was followed by 41 participants ***(64.06%)*** getting this score in the third case assignment. The set target (60%) was met.

- The **"exceed expectations"** category ranged from grade B+ to A+. In the first case assignment, only 1 participant *(1.54%)* fell in this category. A significant growth was visible by case assignment no.

2, where 8 participants *(12.50%)* received this score. Continuing the trend, the number of students who achieved this high score by the last case assignment increased to 12 ***(18.75%)***. The defined target (20%) was, however, missed marginally.

Closing the Loop - Suggestions for Improvement:

- As a first step taken towards achieving learning objective 1.1, can include caslets based on critical thinking at the beginning of the course. This will expose the students to the rigor of course gradually.
- To capture the students learning, especially for those performing below expectations, one-on-one feedback sessions can be organised.

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NOTE

All the names and figures mentioned in the case are fictional and are used purely for the purpose of identifying different case characters and analyzing the information for teaching.

RKUNIVERSITY

SECTION NO. 4: PHARMACY RESEARCH

The field of Pharmacy has witnessed turbulence in recent times, but has nevertheless stood its ground and prevailed in the rough weather. In fact, it has remained nearly unaffected by the adversities during these times of depression and inflation. The inter-disciplinary nature of the pharmaceutical domain is one of the main reasons behind its survival and growth. Be it research or academics, the interdisciplinary roots of this domain have anchored deep into the ground. Untapped potentials and unexplored horizons continue to beckon us to play our part, small or big, in contributing to the research in this field. In fact, there's yet another unique aspect of this domain that needs further exploration. It's called "entrepreneurship." The central theme of the first international conference of RK University focuses on research and entrepreneurship. The pharmaceutical sector is undoubtedly going to have a strong say in it! Accordingly, the sub-theme, in order to cover the broad spectrum of our domain, has been simply and aptly titled "Latest Trends in Pharmacy."



ISBN: 978-93-5254-061-7 | www.rkuconference.org | JAN. 5 & JAN. 6, 2016

Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th& Jan. 6th, 2016)

(Proceedings available for download at rku.ac.in/icre)

RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

Design and evaluation of tadalafil fast dissolving film for treatment of erectile dysfunction

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ABSTRACT

Tadalafil is one of the most effective agents for treatment of erectile dysfunction which acts by inhibiting the cGMP-specific phosphodiesterase type 5. Film formulations are popular in patients because of quick onset and user friendliness. The aim of present investigation was to develop fast dissolving films of tadalafil which provides rapid onset of poorly water soluble drug. Film formulation can be taken within the pocket and patient can take it without need of water without any grittiness. The developed formulation will disintegrate within minute with enhanced solubilization which ultimately provides good bioavailability and quick onset. Different polymers grades like HPMC E15, HPMC E6 and maltodextrin DE-10 were evaluated at different concentration level during screening. The optimized formulation (batch OT9) containing HPMC E6, propylene glycol and poloxamer 407 showed greater drug dissolution , satisfactory in vitro disintegration time (20.07 sec) and physicomechanical properties that were suitable for mouth dissolving film.

SUMMARY

Fast dissolving films of tadalafil for treatment of erectile dysfunction provides rapid onset of poorly water soluble drug by enhancing the solubility and provides the formulation which can be taken within the pocket and patient can take it without need of water.

KEYWORDS:
Keyword : “Fast dissolving oral film”; “Tadalafil”; “Solvent casting”; “Quality by design”, “Solubility enhancement”

1. INTRODUCTION

Erectile dysfunction is defined by national institute of health as the inability to achieve or maintain an erection required for satisfactory sexual performance. As per a population based survey, Erectile dysfunction is commonly reported in 12% patients younger than 59 years, 22% patient between 60 to 69 years and 30% elder than 69 years (1). Phosphodiesterase Inhibitors such as tadalafil inhibits the cGMP-specific phosphodiesterase type 5 (PDE5) which is responsible for degradation of cGMP in the corpus cavernosum located around the penis. The inhibition of phosphodiesterase type 5 (PDE5) by these drug increases the amount of cGMP, causes relaxation of smooth muscle and increased blood flow into the corpus cavernosum. Tadalafil has been successfully proved effective to cure erectile dysfunction. Patients are able to have sexual activity at 30mins and up to 36 hrs after taking tadalafil (2). Tadalafil is practically insoluble in water and very slightly soluble in some organic solvents. The extremely limited solubility of tadalafil causes major difficulties and challenges in formulating a dosage form that demonstrates acceptable bioavailability. Satisfactory drug loading with acceptable content uniformity is itself a big challenge when water insoluble active is in suspended form (3,4).

The oral route is most preferred route of administration for drug delivery due to the aspect of patient compliance. Fast dissolving films is ultrathin strip, which is similar to postage stamp in shape and size, with actives and mostly water soluble excipients mainly film forming polymers and plasticizers. The fast dissolving films films have larger surface area compared to orodispersible tablets (ODTs) that leads to rapid disintegration in oral cavity. Unlike the Orodispersible tablets which are fragile and brittle, films are flexible enough with adequate ease of transport and handling. Like the other liquid dosage forms, precise dosing and unit dose formulation is possible with fast dissolving films. Films provide ease of swallowing and patient can take it without need of water (5,6).

The aim of the present investigation was to develop oral films of tadalafil which not only provides rapid onset but also provides unique product differentiation from other marketed products such as film coated tablets, chewable tablets and effervescent tablets. The formulation development was carried out as per the concept of quality by design with proper identification of critical quality attributes, quality target profile of product, criticality in selection of process parameters and materials. A 32 full factorial design was used for optimization and selection of final formulation. Present investigation will provide the formulation which can be taken within the pocket and patient can take it without need of water by simply putting it on tongue. The patient will not feel any discomfort and grittiness during and/or immediately after dissolution frequently found during disintegration of orodispersible tablets. The developed formulation will disintegrates in fraction of minute with enhanced solubilization of poorly water soluble drug by means of solubility enhancer which ultimately provides good bioavailability and quick onset compared to widely consumed film coated tablets. Present investigation will provide formulation which is formulated by simple continuous process (mixing and homogenization, casting, drying & cutting) compared to film coated tablets involving many unit operations.

2. MATERIALS AND METHODS

2.1 Materials

Tadalafil (MSN Organics Pvt. Ltd, Andhrapradesh, India), Methocel E15 and E6 (Colorcon Asia Pvt. Ltd, Goa, India), Poloxamer 407 (supplied as Kolliphor P407) and cremophor RH 40 (supplied as

KolliphorRH 40) (BASF, Mumbai, India) and Maltodextrin having dextrose equivalent value 10 (Supplied as Star-dri 1005A) (Maize products, India) were received as gift samples. Mannitol (Supplied as Pearlitol 200SD) (Signet chemicals, Mumbai, India), Sunset Yellow FCF (Roha dye chem., Mumbai, India), Mixed fruit flavor Liquid (SK Flavors and fragrances, Ahmedabad, India) and Sucralose (Jk sucralose Inc., India), Polyethylene glycol (PEG 300) and Propylene glycol (Finar Chemicals Ltd, Ahmedabad, India) were procured. All other materials and chemicals used were of either pharmaceutical or analytical grade.

2.2 Drug excipient compatibility study

Possible interaction of drug with various excipients proposed for use in final formulation was checked by using differential scanning calorimetry (DSC). DSC study of pure drug, excipients and their combination used in final optimized formulation OT9 was carried out using DSC instrument (DSC-60, Shimadzu, Kyoto, Japan) at Shri S. K. Patel College of Pharmaceutical Education and Research, Kherva, Ganpat University. In this process, samples (3-5 mg) were put into aluminium cell and scanned at 50- 300 °C, at 10°C per minute rate under nitrogen atmosphere against blank DSC aluminium cell as a reference.

2.3 Analytical method development

Calibration curve of tadalafil was taken in demineralized water. Accurately weighed 50 mg of tadalafil was transferred to 100 mL volumetric flask and dissolved in methanol. The volume was adjusted up to 100 mL with methanol to get 500 µg /mL stock solution of drug. The stock solution (500 µg/mL) was further diluted to get concentration of tadalafilin the range of 7.5-20 µg/mL. These solutions were scanned for the maximum absorbance using Shimadzu UV 1800 double-beam spectrophotometer (Shimadzu, Kyoto, Japan). The absorbance of these drug solutions were estimated at λmax.

2.4 Formulation strategy as per quality by design elements

Before designing the experimental work, Quality Target Product Profile (QTPP) and Critical Quality Attributes (CQA) of product are necessary to identify for better understanding of target formulation and summary of QTPP and CQA was outlined in Table 1. QTPP is a prospective summary of the quality characteristics of a product that ideally will be achieved to ensure the desired quality, safety and efficacy of the drug product. CQA is a physical, chemical, biological, or microbiological property that should be within an appropriate limit to ensure the desired product quality (7-9).

2.5 Preliminary Screening of components for placebo film formulations

Hydroxypropylmethyl cellulose (HPMC) is known for its excellent film forming property and well-acceptability for solvent casting method. So, Different grades of HPMC, mainly HPMC E15 and HPMC E6 were evaluated as a primary film former. For the fabrication of film, PEG 300 was used as plasticizer and sucralose was used as sweerner. Sugar alcohols are generally added to film because they dissolve quickly by creating pores in film and fasten the disintegration time (6). Due to quick solubilization property, Maltodextrin was added to decrease the disintegration time of the film (10). Role of Maltodextrin DE 10 and Mannitol on disintegration time of HPMC film was checked during screening. Higher amount of polymer was required to load water insoluble active pharmaceutical ingredient (API) in film with satisfactory tensile strength. During screening, one variable at one time (OVAT) method was performed by considering polymer type as variable. The composition of various placebo films is shown in Tablet 2. Films were prepared by solvent casting method. In this method, sucralose, PEG 300, maltodextrin and mannitol were dissolved in half quantity of purified water and HPMC was soaked in

remaining half quantity of purified water for 4 h. Then both dispersions were mixed under gentle stirring for 1 h and resultant mixture was sonicated for 1h to remove bubbles. After complete removal of bubbles, 3 g dispersion was casted on glass petri plate. The glass petri plate was kept in controlled temperature oven (Vinayak Pharma Technology, Vatva, India) at 55°C for 8 hr. After drying, films were peeled and cut into 3.5 cm X 3 cm (10.5 cm^2) and store in aluminium foil. These films were further subjected to various evaluation tests. Selection of prototype placebo film composition was done on the basis of observations and results of all placebo trials given in Table 2 and quantity per unit film of 10.5 cm^2 was calculated as given in Table 3.

2.6 Preparation of fast dissolving films containing tadalafil and different surfactants

For BCS class II drugs like tadalafil, solubility is a critical factor for dissolution, absorption and ultimately quick onset of action of tadalafil. So, cremophor RH 40 (polyoxyl40 hydrogenated castor oil) and poloxamer 407 (copolymer of ethylene oxide and propylene oxide) were selected for solubility enhancement of tadalafil. Compositions of tadalafil loaded films were shown in Table 4. For better content uniformity, stirring for uniform dispersion of active in polymer-plasticizer blend was not sufficient. So, homogenization of micronized API (Particle Size D (0.9) = 4.290µ) was necessary. Without homogenization, agglomeration of active had been occurred at bottom of container. Briefly solubilizer, plasticizer, color, sweetener, flavor and film forming polymer were dispersed in 80 % total quantity of purified water under continuous stirring for 2 Hr. Resultant dispersion was sonicated for 1 hr to remove bubbles, meanwhile tadalafil was dispersed in remaining quantity of purified water under continuous homogenization with high speed homogenizer (Remielekronik, Vasai, India) at 1400 RPM for 1 Hr. Drug dispersion was slowly added to bubble free polymer plasticizer blend under gentle stirring. Then dispersion was casted and dried in a same way as placebo film trials.

2.7 Optimization of tadalafil film formulation using 32 full factorial design

From the results of preliminary screening and films with different ratio of drug and surfactant, the optimization was carried out using design of expert (DOE) approach. To study the effect of two independent variables on three different quantitative levels i.e. concentration of poloxamer 407 (X1) and concentration of propylene glycol on responses 32 full factorial design was used. In this design, concentration of poloxamer 407 and propylene glycol were used as independent variables while tensile strength, disintegration time and % drug release at 10 min were selected as response variables. Trials were taken at all possible combinations. The detailed layout of factorial batches is shown in Table 5. The equations relating independent variables and responses were obtained by subjecting the results to statistical evaluation. Design Expert 9.0.4.1 (Built date February 23, 2015) was used to perform multiple linear regressions to determine the control factors that significantly affect the responses.

Polynomial equation for 3^2 full factorial design: $Y = b_0 + b_1X_1 + b_2X_2 + b_{11}X_{12} + b_{22}X_{22} + b_{12}X_1X_2$ was used. In this equation, Y is the dependent variable, b0 is the arithmetic mean response of the 9 runs, and bi is the estimated coefficient for the factor Xi. The significant factors in the equations were selected using a stepwise forward and backward elimination for the calculation of regression analysis. The terms of full model having non-significant p value ($p > 0.05$) have negligible contribution hence they were neglected.

2.8 Evaluation of fast dissolving films

The prepared films were evaluated for tensile strength, folding endurance, surface pH, thickness, in vitro disintegration, solution time, assay and in vitro dissolution studies. The tensile strength of the film was

evaluated by using the push pull tensiometer instrument. It consists of two load cell grip, the lower one was fixed and upper one was movable. Film strips with dimensions of 3.5×3 cm^2 were fixed between these cell grips and force was gradually applied till the film break. The break force was taken directly from the dial reading in g. It is calculated by equation, tensile strength = break force/area of film in cm^2(11,12). Folding endurance of the film was measured by folding the film at the same point until it breaks. The number of folds before the film breaks is the folding endurance of the film. The surface pH of oral dissolving film was determined in order to investigate the possibility of any side effect in vivo. As an acidic or alkaline pH may cause irritation of the oral mucosa, it was decided to keep the surface pH as close to neutral as possible. A combined pH electrode was used for this purpose. Film was slightly wetted with the help of water. The pH was measured by bringing the electrode in contact with the surface of the oral film (13). A thickness of the film was measured by using micrometer screw gauge. Film was measured at three positions i.e. central and the two corners and the mean thickness was calculated (11). The in vitro disintegration time is the time at which the film starts to break. The disintegration time was measured in a beaker containing 20 mL demineralized water. The time film starts to break was measured as disintegration time of film. The time at which the film completely dissolves is considered as dissolution time or solution time (14,15). The assay was determined by dissolving one film of dimension 3.5 cm × 3 cm containing 10 mg of tadalafil in diluents mixture of Acetonitrile: Water (1:1) in 100 mL volumetric flask under continuous shaking for about 15 min. If any large undispersed fragments of film were seen, sonication was performed for 5 min to disperse film fragments residues. The above solution was further diluted to get concentration of tadalafil in range of 7.5-20 µg/mL and the absorbance was measured at 285nm using UV-visible spectrophotometer. The experiments were carried out in triplicate for the films of all formulations and average values were recorded. The in vitro dissolution study of tadalafil fast dissolving film was performed using USP apparatus (model TDT-08T, Electrolab, Mumbai, India) fitted with paddle (50 rpm) maintained at 37 ± 0.5 ^oC. Dissolution media was 1000 mL of deionized water and tested for drug release up to 20 mins. During the study, 10 mL of aliquots were withdrawn at 5, 10, 15 and 20 mins and were replaced by fresh deionized water. Dissolution method recommended by office of generic drug contains 0.5% sodium lauryl sulphate (SLS) in media but for studying the impact of solubility enhancers, SLS was not added in dissolution media (16). Collected aliquots were withdrawn, filtered through a 0.45µm membrane filter, diluted, and assayed at 285 nm using a Shimadzu UV 1800 double-beam spectrophotometer (Shimadzu, Kyoto, Japan). Cumulative percentage drug release was calculated using an equation obtained from a calibration curves.

3. RESULTS AND DISCUSSION

3.1 Drug- excipients compatibility study

DSC thermogram of tadalafil (Fig. 1) revealed a very small endothermic peak at 95.29°C. DSC thermogram of excipient mixture alone (Fig. 2) and drug–excipient mixture (Fig. 3) showed sharp endotherm at 289.63°C and 294.53°C respectively which was absent in thermogram of tadalafil but small characteristic endiothermic peak of tadalafil was retained indicating absence of any physical compatibility of drug with excipients used in final formulation.

3.2 Analytical method development

The drug exhibited λmax at 285 nm. The calibration curve was generated using different concentration (7.5-20 µg/mL) of drug solutions in the Beer-Lambert law. The data for calibration curve is shown in Table 6 and calibration curve is shown in Fig. 4.

3.3 Preliminary screening of components for placebo film formulations

HPMC was used as primary film former due to its excellent film forming ability. Initial studied indicated that amongst different grades, films of HPMC E 15 had excellent strength but during disintegration, very slight gelling property and low rate of hydration was found which ultimately caused increase disintegration time. So, Lower viscosity grade HPMC E6 was selected that showed decrease in disintegration time comparatively but still not up to targeted CQA. So, maltodextrin DE 10 and mannitol were selected to decrease disintegration time. Instant phase separation was found due to miscibility issue of PEG 300 and maltodextrin in trial PL-4 while opaque brittle and uneven films with white spots were formed in trial PL-5. So, Concentration of HPMC E6 was decreased in trial PL-6 but drop in tensile strength was seen. So, plasticizer concentration was increased in trial PL-7 which was good amongst all trials. Quantity per unit film (10.5 cm^2) was calculated from trial PL-7 as shown in Table 3 and final drug loading was done in next trials.

3.4 Preparation of fast dissolving films containing tadalafil and different surfactants

For solubility enhancement of tadalafil, cremophor RH 40 and poloxamer 407 were added to formulation in different ratio with tadalafil. Poor film formation was found with tadalafil: cremophor RH 40 (1:1) containing Trial F1. Films of trial F1 were very sticky brittle and also having very poor peelability. After 3 hrs drying, films contained same stickiness and brittleness. By decreasing concentration of cremophor RH40 (Trial F2 and F3) or by increasing plasticizer concentration (Trial F4), no significant change in stickiness and tensile strength was observed. From trial F1 to F4, it is concluded that cremophor RH 40, itself a viscous liquid prepared from castor oil, destroyed the film forming ability of polymer due to its sticky and adhesive property. So, cremophor RH 40 was replaced with poloxamer 407 in trial F5 and F6. Propylene glycol containing films (Trial F6)(0.177kg/cm^2 with 55-60 Folds) had better tensile strength and folding endurance compared to PEG 300 containing films (Trial F5) (0.120 kg/cm^2 with 30-40 Folds).

3.5 Evaluation of fast dissolving films

The factorial batches were evaluated for various parameters by the methods described in methodology section. The evaluation results are shown in Table 7.Thicknesswas found in the range of 0.18 to 0.29 mm, the different solid content per unit film of 10.5 cm^2 could be the reason for variable thickness of the films. Folding endurance gives an indication of brittleness of the film. A result showed as the concentration of plasticizer increases, folding endurance of film increases. Surface pH of all the films prepared was found to be in the range 6.61 to 6.95, which was close to the neutral pH. Thus films may have less potential to irritate the oral mucosa. Tensile strength was found in range of 0.177±0.007 to 0.230±0.004 kg/cm^2. A result showed as the concentration of plasticizer increases, tensile strength of film increases. Content uniformity of formulations OT1, OT2, OT3, OT6 and OT9 showed better drug content of above 98 %. No significant difference in the drug content among the films indicated good content uniformity. Also it can be concluded that tadalafil was uniformly dispersed by homogenization and continuous stirring throughout the process. So, agglomeration of suspended particles was not occurred. *In vitro* disintegration time and dissolution time for fast dissolving film was in the range from 14.78 ±0.96 to 24.87 ±0.96 and 1.65 ±0.59 to 2.57 ±0.59, respectively. Results showed that as the plasticizer concentration increases, disintegration time increases. *In vitro* dissolution study in demineralized water was conducted as per method described earlier. The data for *in vitro* release are shown in Table 8 and are compared in Fig. 5.

3.6 Statistical analysis of 3^2 full factorial batches

3.6.1 Full and reduced model for disintegration time

The summary of regression analysis and ANOVA for disintegration time is shown in Table 9. The contour plot and 3D surface plot are shown in Fig. 6 and Fig. 7, respectively. From the equation of full model, reduced model is drawn by rejecting insignificant factors on the basis of p value. From the model, it was found that concentration of poloxamer 407 showed negative effect on the disintegration time. As its concentration increases, disintegration time of film decreases. From the results in Table 9, it was concluded that poloxamer 407 improved wettability by decreasing surface tension of film. So, Disintegration time was reduced with increasing concentration of poloxamer 407. Concentration of PG showed positive effect on the disintegration time. It was concluded that both factors had significant effect on the disintegration time.

3.6.2 Full and reduced model for tensile strength

The summary of regression analysis and ANOVA for tensile strength is shown in Table 10. The contour plot and 3D surface plot are shown in Fig. 8 and Fig. 9, respectively. From the equation of full model, reduced model is drawn by rejecting insignificant factors on the basis of p value. From the reduced model, it was found that concentration of poloxamer 407 has no significant effect on tensile strength of film. Only concentration of PG showed significant and positive effect on the tensile strength. As its concentration increases, tensile strength of film increases.

3.6.3 Full and reduced model for in vitro drug release at 10 min

The summary of regression analysis and ANOVA for *in vitro* drug release at 10 min is shown in Table 11. The contour plot and 3D surface plot are shown in Fig. 10 and Fig. 11, respectively. From the equation of full model, reduced model is drawn by rejecting insignificant factors on the basis of p value. From the model, it was found that concentration of poloxamer 407 showed positive effect on the *in vitro* drug release. As its concentration increases, *in vitro* drug release increases. It was concluded that concentration of poloxamer 407 had the largest effect on the drug release at 10 min, which indicated that poloxamer 407 had improved the solubility of tadalafil and ultimately improves the dissolution. Also concentration of PG shows positive effect on the tensile strength.

3.6.4 Validation of Model by Check Point Batch

The overlay plot of all factorial batches is shown in Fig. 12. Check point batches C1 and C2 were selected from the overlay plot of responses. The amount of poloxamer 407 and PG were selected from overlay plot and predicted responses were calculated and are given in the Table 12. Actual response of C1 and C2 batch were measured and compared with the predicted response of check point batches. All the values of responses were within the upper and lower predicted interval. Hence, this model is valid and optimized batch can be selected from the overlay plot of this model.

3.6.5 Optimization of Batch from Overlay Plot

From the overlay plot it was seen that batches OT8 and OT9 fall under the optimized area. So, the batch with faster dissolution was considered optimized batch. Thus batch OT9 (92.11% released within 10 min) was selected as the optimized batch.

4. CONCLUSION

The physical properties like tensile strength, folding endurance etc. were affected by type and concentration of polymer and plasticizer while solubility of tadalafil was enhanced by increasing

concentration of poloxamer 407. The development of fast dissolving film of tadalafil is one of the alternative routes to provide quick onset of action. In addition, this formulation enhances patient compliance because patient can take it by simply putting it in mouth without need of water. Propylene glycol (34mg) in concentration of 45.95% of dry polymer weight was required to provide good mechanical strength to film. On the basis of data obtained from *in vitro* dissolution studies, it was concluded that formulation containing tadalafil: poloxamer 407 in 1:2 ratiowas promising formulation for intended use.

FIGURES

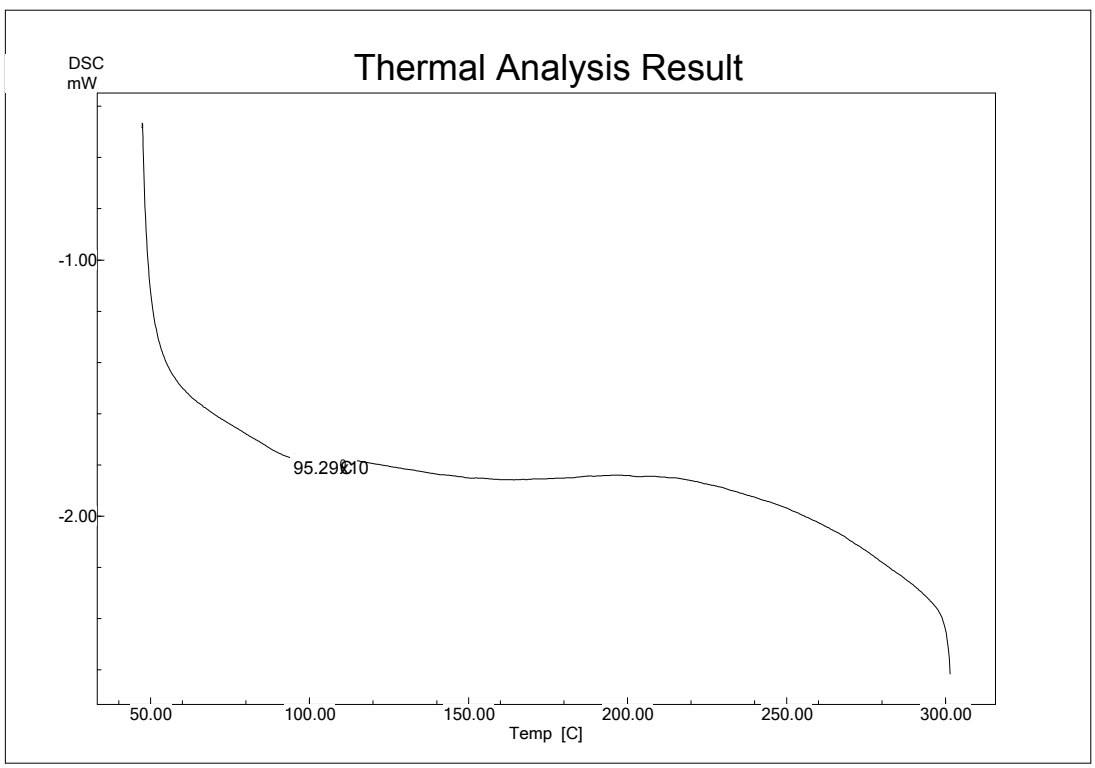


Fig. 1. DSC Thermogram of tadalafil

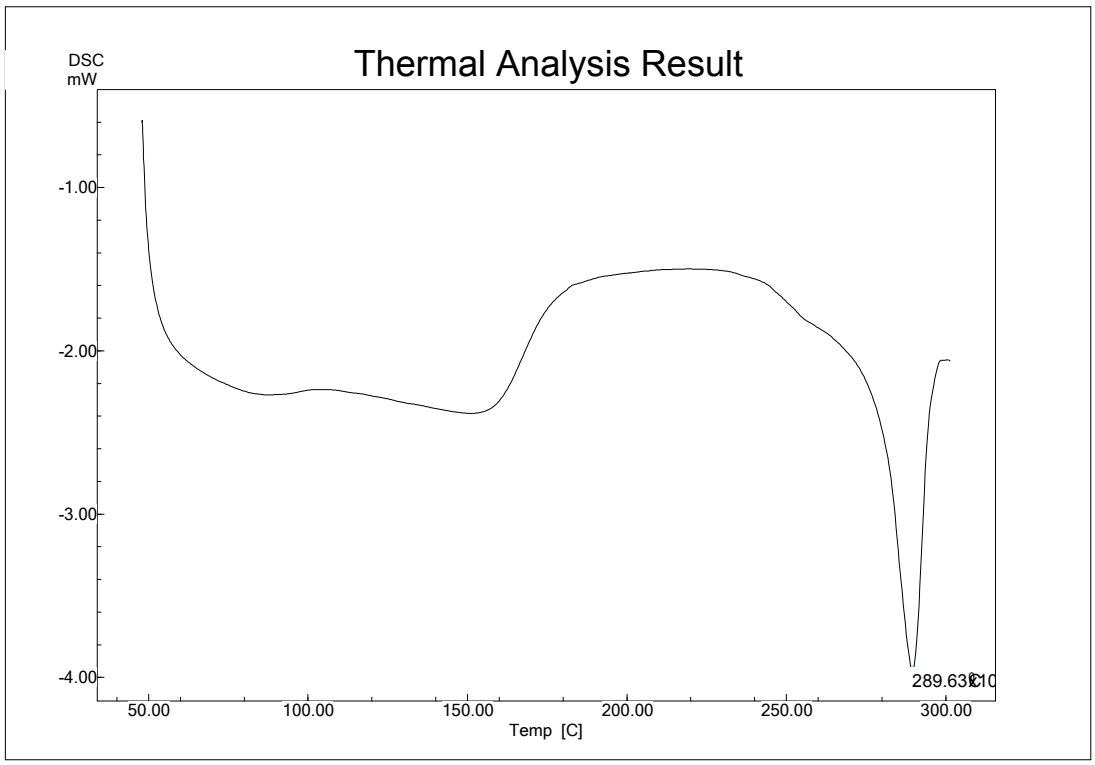


Fig. 2. DSC Thermogram of excipient combination

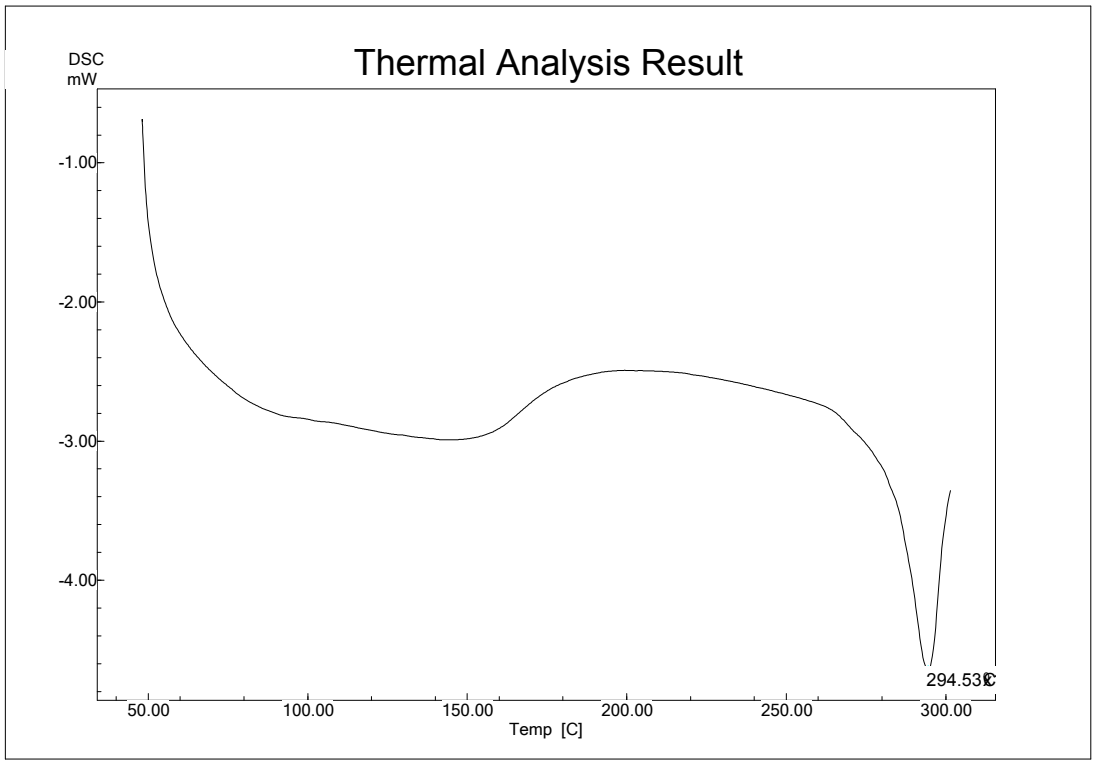


Fig. 3. DSC Thermogram of tadalafil and excipients mixture

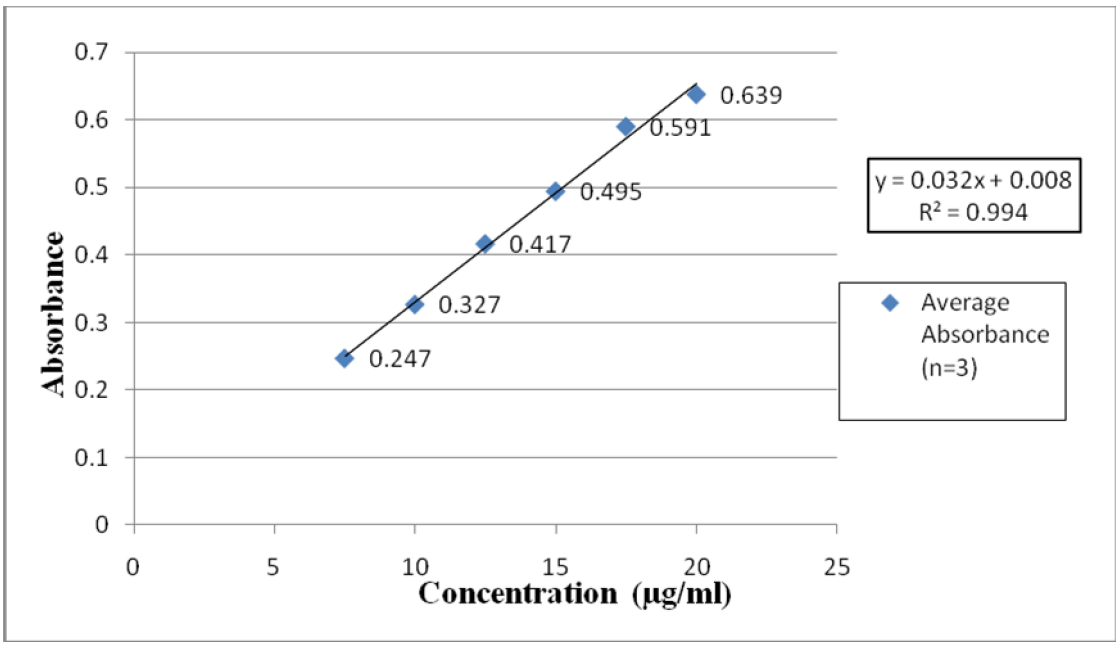


Fig. 4. Calibration curve of tadalafil in demineralized water

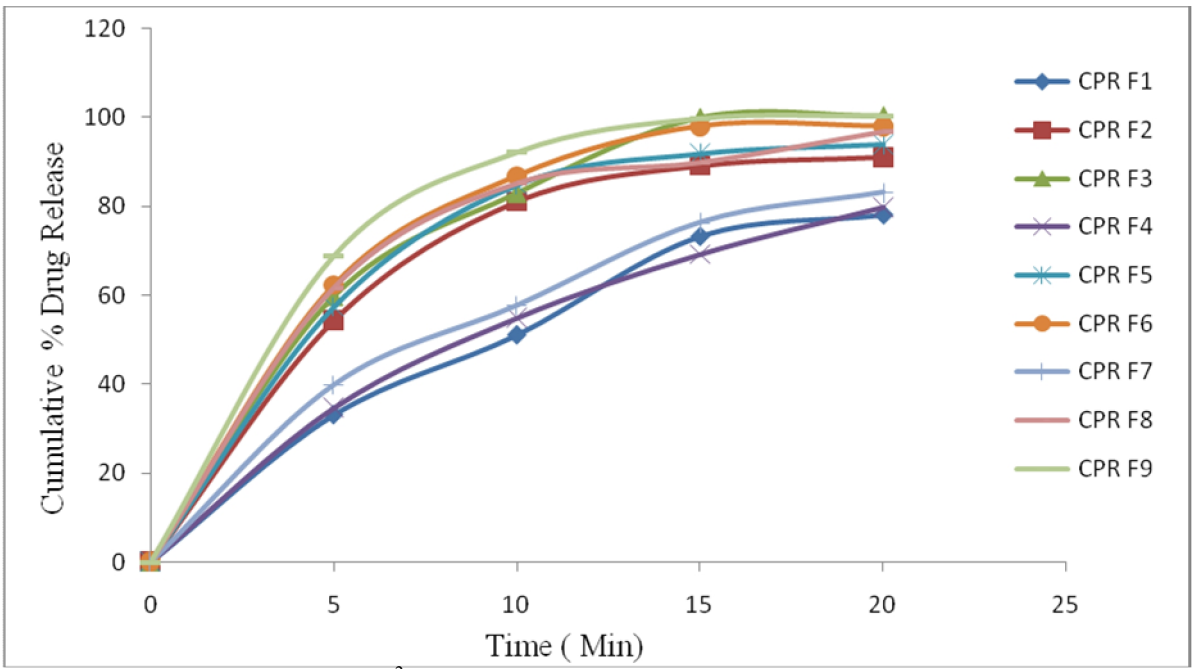


Fig. 5. Drug release comparison of 3^2 factorial trial batches

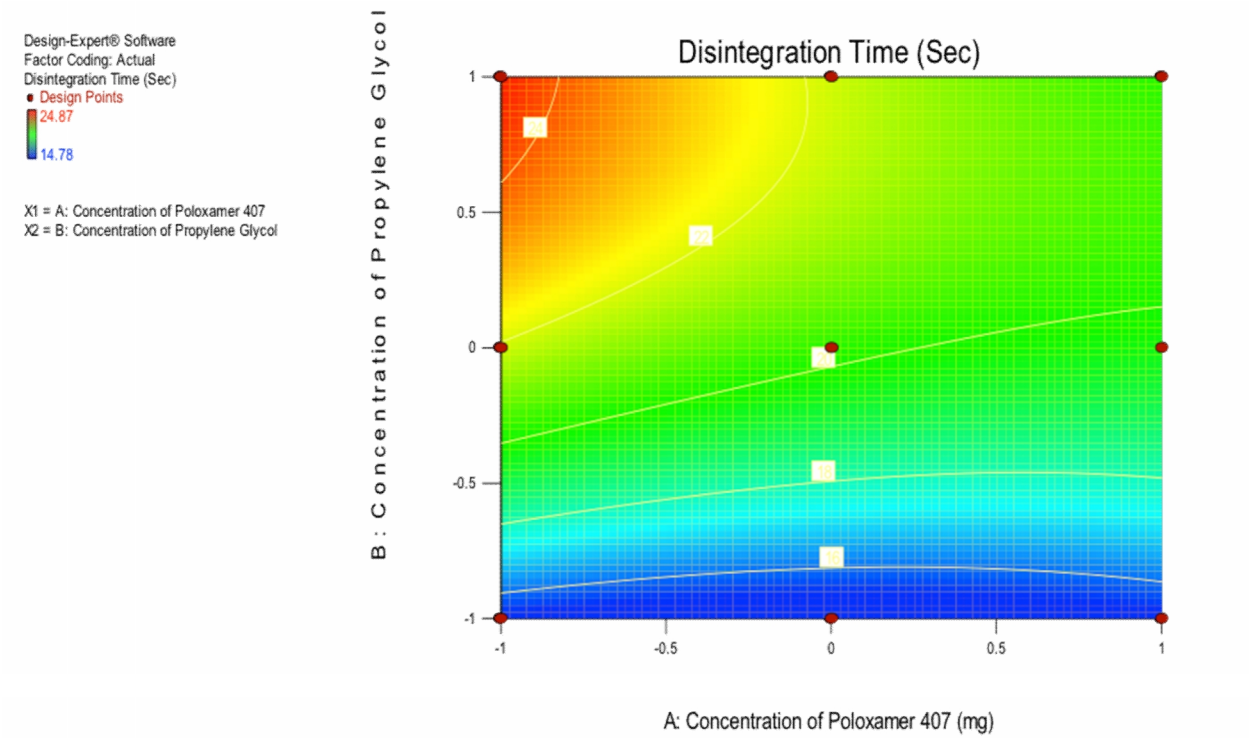


Fig. 6. Contour plot of disintegration time

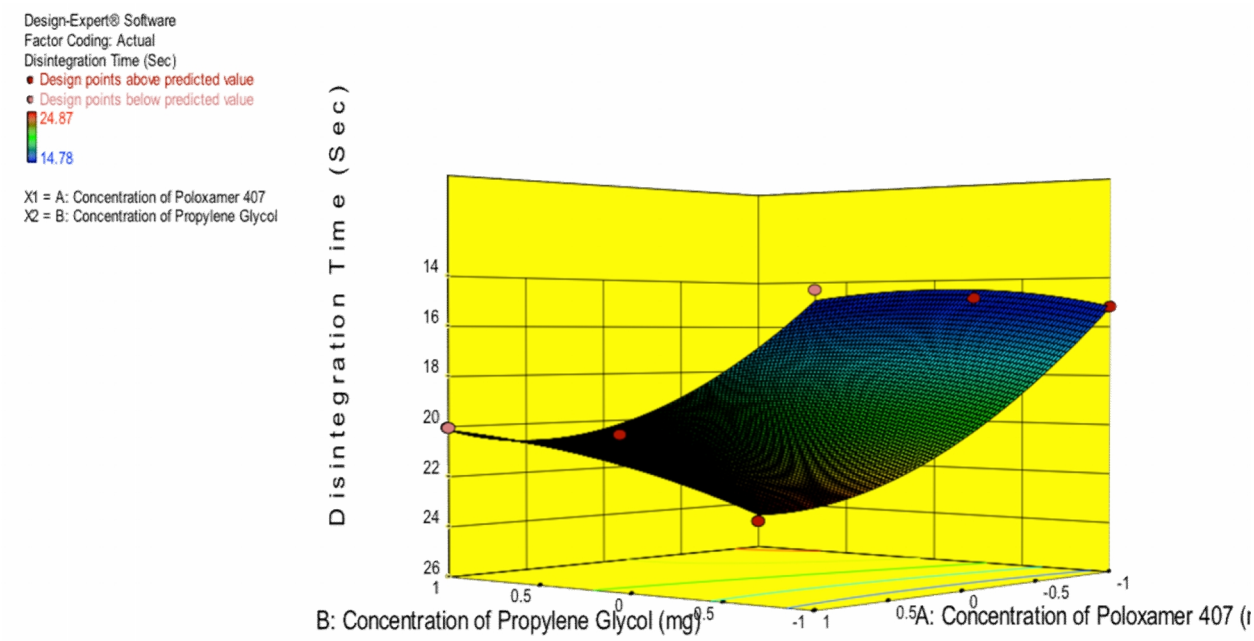


Fig. 7. 3D surface plot of disintegration time

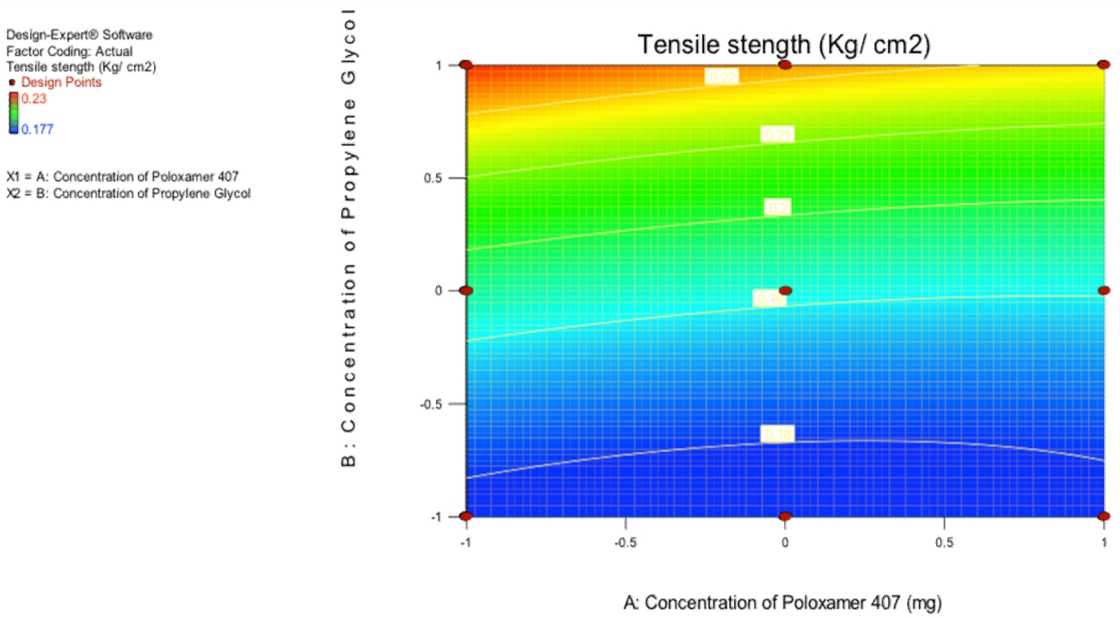


Fig. 8. Contour plot of Tensile Strength

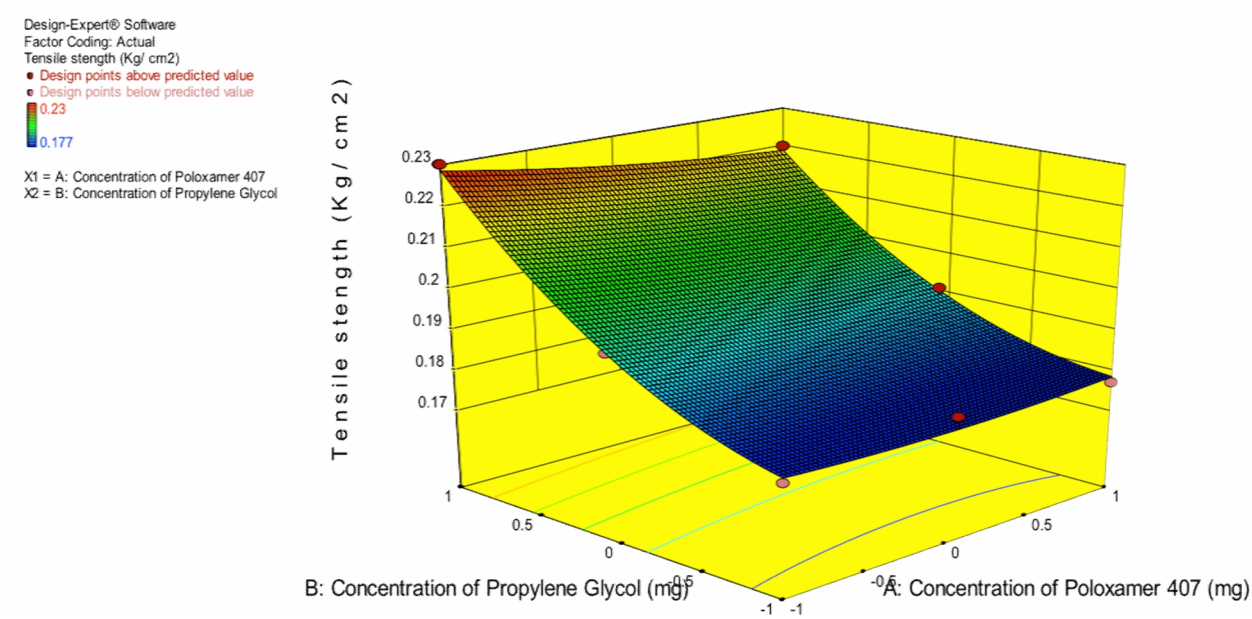


Fig. 9. 3D surface plot of Tensile strength

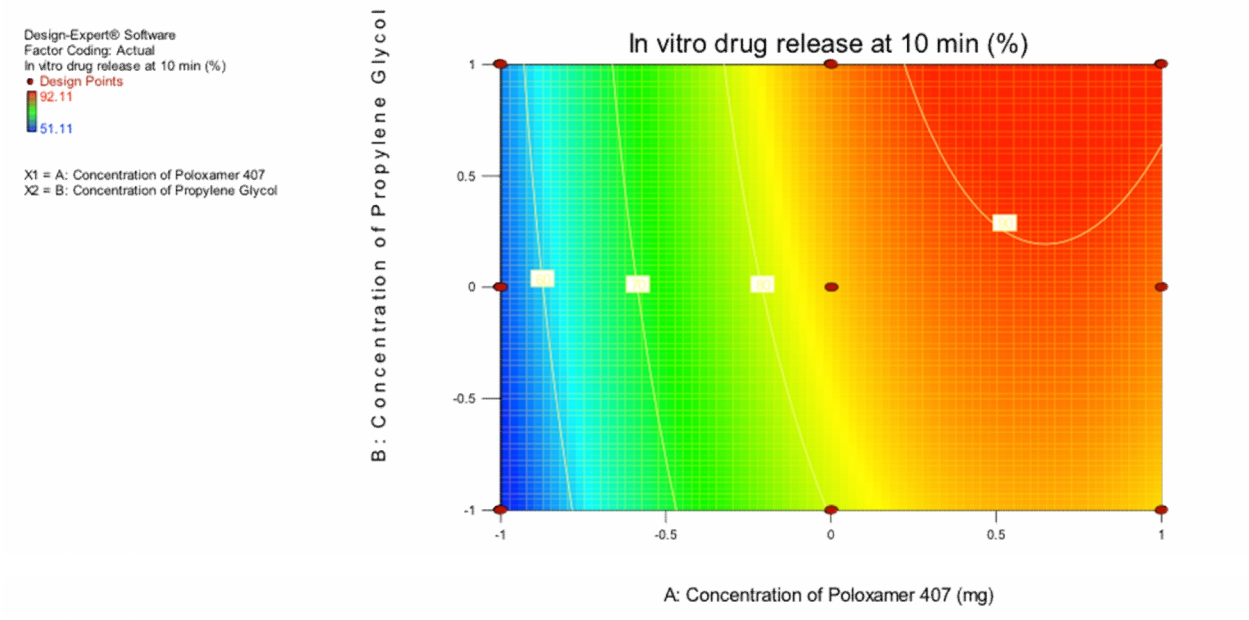


Fig. 10. Contour plot of in vitro drug release at 10 min

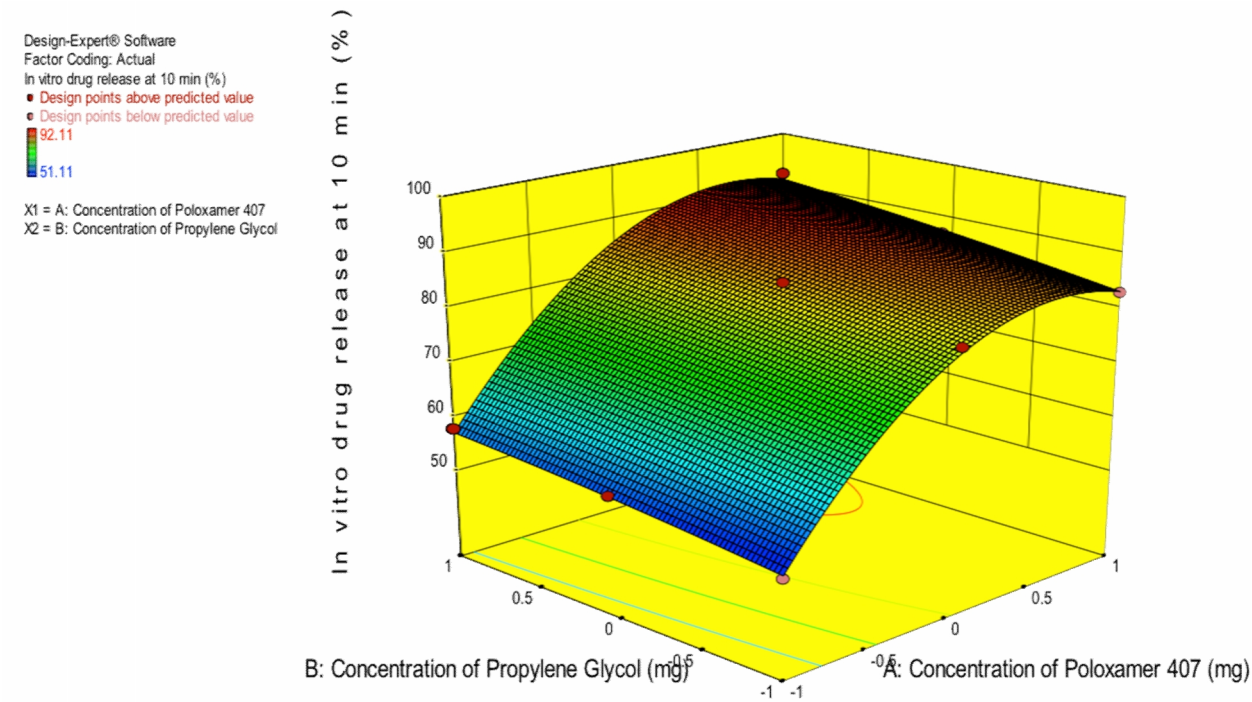


Fig. 11. 3D surface plot of in vitro drug release at 10 min

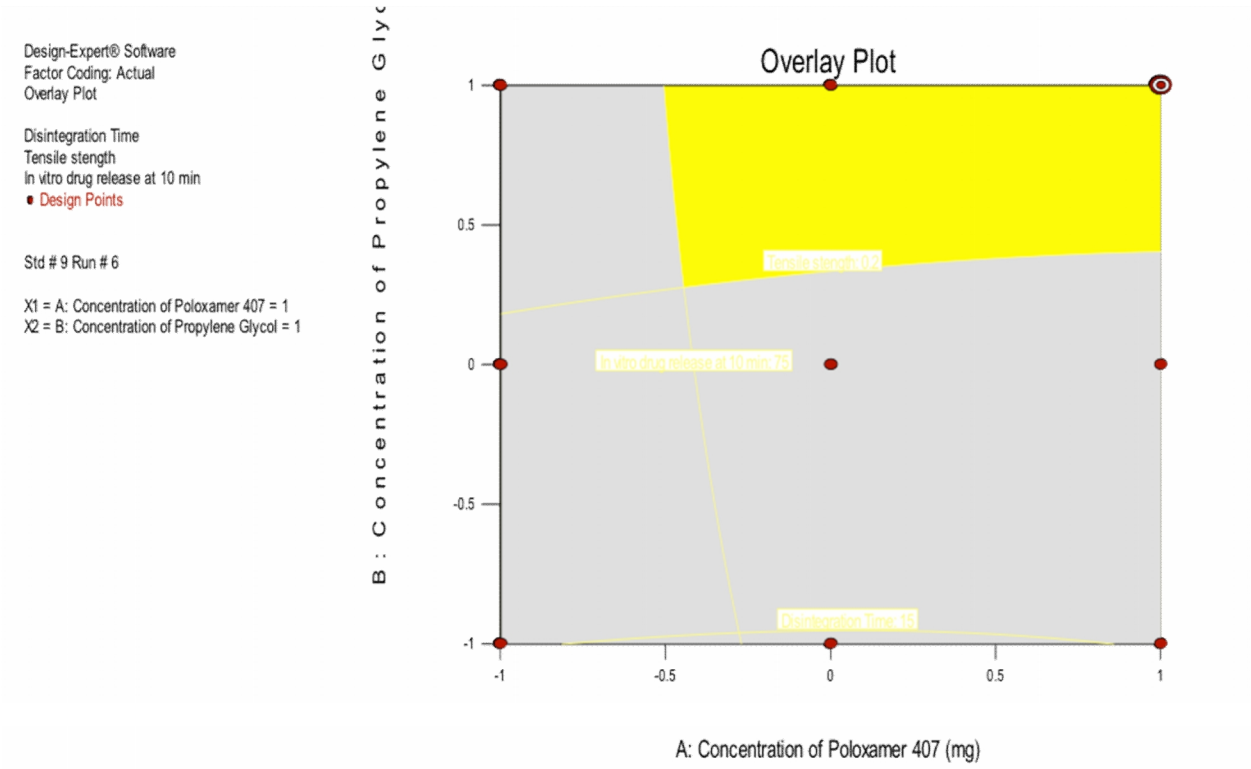


Fig. 12. Overlay plot of 3^2 full factorial batches

TABLES

Table 1 Identification of quality by design elements

Identification of quality target product profile elements		
QTPP elements	**Target**	**Justification**
1)Dosage form	Oral film	-
2)Dosage form design	Fast dissolving single layer oral film	For quick onset and product uniqueness - differentiation from other marketed product.
3)Dosage strength	10 mg	Same strength as marketed tablet formulations
4)Route of administration	Oral	For patient compliance aspect
5)Pharmacokinetics	Immediate release enabling maximum plasma concentration achieved between 30 mins to 6 hrs (2)	Needed to enhance solubility for poorly water soluble drug
6)Stability	At least 24-month shelf-life at room temperature	Equivalent to or better than marketed formulations
7)Container closure system	Aluminium- PET (Polyethylene) laminated pouches	Needed to achieve the target shelf-life
8)Administration	Administer before half an hour before starting activity	-
Identification of critical quality attributes (CQA) elements		
Quality attributes	**Target**	**Justification**
1) Physical		
1.1) Appearance	Color and shape acceptable to patient	Not critical. Not directly linked to safety and efficacy. Light orange color rectangular film is a target product.
1.2) Odor	No unpleasant odor	Not critical.
1.3) Size	10.5 cm^2 (3.5 cm X 3 cm)	Not critical. For comparable ease for administration to oral cavity.

1.4)Taste	No unpleasant bitter taste	Critical. Targeted product will disintegrate in oral cavity. Acceptable sweetness and pleasant flavors are necessary.
1.5)Disintegration time	30 Seconds or less	Critical because safety and efficacy will directly linked to quick disintegration and dissolution
2)Assay	100% W/W of label claim	Critical. Evaluation required throughout development, optimization and stability.
3) Uniformity of weight and content	As per pharmacopoeial requirements	Critical. Evaluation required throughout development, optimization and stability.
4)Dissolution	Confirms to OGD specifications	Critical. Evaluation required throughout development, optimization and stability.

Table 2 Composition of placebo film trials

Composition of placebo film trials							
Components	Formula on wet basis (% W/W)						
	PL-1	PL-2	PL-3	PL-4	PL-5	PL-6	PL-7
HPMC E 15	9	5.5	-	-	-	-	-
HPMC E6	-	-	13	13	13	12	11.8
Maltodextrin 10DE	-	-	-	5	-	-	-
Mannitol	-	-	-	-	2	-	-
PEG 300	2.2	2.2	2.2	2.2	2.2	2.2	3
Sucralose	0.1	0.1	0.1	0.1	0.1	0.1	0.15
Purified water	88.7	92.2	84.7	79.7	82.7	85.7	85.05
Evaluation parameters for placebo film trials							
Tensile strength (kg/cm^2)	0.550	0.400	0.340			0.207	0.300
Disintegration time[@](Sec.)	135	115	75	Not formed*	Uneven rough opaque film formed	25	20
Solution time[@] (Sec.)	210	165	150			120	120
Folding endurance	> 300	230-244	90-100			90-100	110-125
Surface texture	Smooth	Smooth	Smooth			Smooth	Smooth

[@] Disintegration time and solution time varies in range of ±4 Sec.

* Instant phase separation of blend found

Table 3 Calculation of quantity per unit film of 10.5 cm^2

Calculation of quantity per unit film (10.5 cm^2)		
1)Area of petridish = πr^2 ,where r is radius of petridish= $3.14*(4)^2$ =50.24 cm^2		
2)Area of film (Rectangular) = Length * Width =3.5 * 3= 10.5 cm^2		
3) For satisfactory casting, 3 g solution was casted per petridish (50.24 cm^2)		
So, 0.63 g solution was required per unit film.		
Components	Formula on wet basis	
	% W/W	mg/ film
HPMC E6	11.8	74.34
PEG 300	3	18.9
Sucralose	0.15	0.95
Purified water	85.05	535.81
Total weight	100	630

Table 4 Preparation of fast dissolving film containing tadalafil and different surfactants

Preparation of Fast dissolving film containing Tadalafil and different surfactants						
Components	**Formula (mg/ unit film of 10.5 cm^2)**					
	F1	**F2**	**F3**	**F4**	**F5**	**F6**
Tadalafil	10	10	10	10	10	10
Cremophor RH 40	10	7.5	5	5	-	-
Poloxamer 407	-	-	-	-	5	5
HPMC E6	74	74	74	74	74	74
PEG 300	19	19	19	34	19	-
Propylene Glycol	-	-	-	-	-	19
Sucrlose	1	1	1	1	1	1
Mixed Fruit Flavor	0.8	0.8	0.8	0.8	0.8	0.8
Color Sunset Yellow FCF	0.1	0.1	0.1	0.1	0.1	0.1
Purified Water*	q.s.					
Evaluation parameters						
Tensile strength (kg/cm^2)	Poor Film formation. Films were sticky and very brittle in nature.				0.120	0.177
Disintegration time[@](Sec.)					15-20	15-20
Solution time[@] (Sec.)					150 Sec	150 Sec
Folding endurance					30-40	55-60
Appearance & Surface texture					Light orange colored opaque film with small white spots	
Surface pH					6.91±0.03	6.97±0.02

[@] Disintegration time and solution time varies in range of ±4 Sec

* Up to 630 mg per unit film.3g solution was casted per each petriplate.

Table 5 Detailed layouts of Optimization trials using 3^2 full factorial design

Ingredients	**Formula (mg/ unit film of 10.5 cm^2)**								
	OT1	**OT2**	**OT3**	**OT4**	**OT5**	**OT6**	**OT7**	**OT8**	**OT9**
Tadalafil	10	10	10	10	10	10	10	10	10
Poloxamer 407	10	15	20	10	15	20	10	15	20
HPMC E6	74	74	74	74	74	74	74	74	74
Propylene Glycol	22	22	22	28	28	28	34	34	34
Sucralose	1	1	1	1	1	1	1	1	1
Mixed fruit flavor	0.8	0.8	0.8	0.8	0.8	0.8	0.8	0.8	0.8
Sunset Yellow FCF	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Distilled water q.s (ml)	q.s.	q.s.	q.s.	q.s.	q.s.	q.s.	q.s.	q.s.	q.s.

Independent Variable	**Coded Value**			**Actual Value**		
Poloxamer 407 (mg)	-1	0	+1	10	15	20
Propylene Glycol (mg)	-1	0	+1	22	28	34

Table 6 Calibration curve data of drug in demineralized water

Concentration (µg/ml)	Absorbance			Average Absorbance ± SD (n=3)
	I	II	III	
7.5	0.247	0.247	0.247	0.247±0.000
10	0.325	0.329	0.327	0.327±0.002
12.5	0.419	0.417	0.415	0.417±0.002
15	0.495	0.495	0.495	0.495±0.000
17.5	0.589	0.591	0.593	0.591±0.002
20	0.639	0.640	0.638	0.639±0.001

Table 7 Evaluation parameters of 3^2 factorial design batches

Batch Code	Tensile Strength	Folding Endurance	Surface pH	Thickness (mm)	C.U (%)	*In Vitro* D.T (sec.)	Solution Time (min.)
F1	0.177 ±0.004	60 ±2.00	6.95 ±0.02	0.18 ±0.02	99.47 ±0.45	15.21 ±1.00	2.32 ±0.59
F2	0.180 ±0.009	55 ±3.00	6.91 ±0.03	0.22 ±0.03	99.10 ±0.90	14.96 ±1.11	2.19 ±0.94
F3	0.177 ±0.007	57 ±4.00	6.84 ±0.01	0.24 ±0.02	98.87 ±0.36	14.78 ±0.96	1.65 ±0.59
F4	0.195 ±0.007	83 ±2.00	6.89 ±0.04	0.22 ±0.01	96.93 ±0.93	21.54 ±1.81	2.41 ±0.42
F5	0.191 ±0.009	79 ±3.00	6.83 ±0.03	0.23 ±0.03	97.85 ±0.12	20.18 ±1.00	2.38 ±0.57
F6	0.191 ±0.007	77 ±2.00	6.79 ±0.04	0.27 ±0.03	100.13 ±1.00	20.09 ±2.09	2.32 ±0.31
F7	0.230 ±0.004	115 ±3.00	6.69 ±0.03	0.25 ±0.01	97.11 ±0.96	24.87 ±0.96	2.57 ±0.59
F8	0.220 ±0.007	99 ±3.00	6.64 ±0.01	0.27 ±0.02	96.93 ±0.44	21.56 ±0.91	2.44 ±0.31
F9	0.220 ±0.009	103 ±2.00	6.61 ±0.05	0.29 ±0.03	99.47 ±0.45	20.07 ±0.96	2.41 ±0.39

C.U: content uniformity, D.T: disintegration time. Values are mean ± S.D for 3 determinations

Table 8 Results of *In vitro* drug release study of factorial batches

Time (min)	%CPR*								
	OT1	**OT2**	**OT3**	**OT4**	**OT5**	**OT6**	**OT7**	**OT8**	**OT9**
5	33.12 ±0.54	54.31 ±1.41	59.76 ±0.81	34.71 ±0.51	57.31 ±1.85	62.34 ±0.96	39.91 ±1.58	61.77 ±0.42	68.79 ±1.10
10	51.11 ±1.31	81.11 ±1.33	82.97 ±0.40	54.97 ±1.10	84.76 ±1.17	86.91 ±1.28	57.77 ±0.91	85.29 ±1.55	92.11 ±0.73
15	73.27 ±1.41	89.17 ±0.99	99.93 ±1.29	69.31 ±1.31	91.79 ±0.99	98.11 ±0.41	76.42 ±1.18	89.99 ±0.49	99.71 ±0.66
20	78.11 ±0.27	91.12 ±0.55	100.31 ±0.21	79.92 ±1.87	93.90 ±0.49	98.11 ±1.17	83.17 ±1.10	96.94 ±0.90	100.3 ±0.66

** Values are expressed as mean ± S.D for three determinations*

Table 9 Summary outputs of regression analysis and ANOVA for disintegration time

	DF	**SS**	**MS**	**F**	**P-value Prob> F**	
Regression	5	98.39	19.68	82.07	0.0021	
Residual	3	0.72	0.24			
Total	8	99.11			Significant	
Coefficient	**b_0**	**b_1**	**b_2**	**b_{11}**	**b_{22}**	**b_{12}**
Coefficient value	20.25	-1.11	3.59	0.53	-2.03	-1.09
P-value	1.289E-05	0.0114	0.0004	0.2256	0.0099	0.0210
Full Model: $Y_1 = 20.25 - 1.11\ X_1 + 3.59\ X_2 + 0.53\ X_1^2 - 2.03\ X_2^2 - 1.09\ X_1X_2$						
Reduced Model: $Y_1 = 20.25 - 1.11\ X_1 + 3.59\ X_2 - 2.03\ X_2^2 - 1.09\ X_1X_2$						

Table 10 Summary outputs of regression analysis and ANOVA for tensile strength

	DF	**SS**	**MS**	**F**	**P-value Prob> F**	
Regression	5	3.283E-03	6.566E-04	89.08	0.0018	
Residual	3	2.211E-05	7.370E-06			
Total	8	3.305E-03			Significant	
Coefficient	**b_0**	**b_1**	**b_2**	**b_{11}**	**b_{22}**	**b_{12}**
Coefficient value	0.1914	-0.0023	0.0227	0.0013	0.0083	-0.0025
P-value	2.603E-06	0.1259	0.0003	0.5373	0.0226	0.1628
Full Model: $Y_1 = 0.19 - 0.0023\ X_1 + 0.0227\ X_2 + 0.0013\ X_1^2 - 0.0083\ X_2^2 - 0.0025\ X_1X_2$						
Reduced Model: $Y_1 = 0.19 + 0.0227\ X_2 - 0.0083\ X_2^2$						

Table 11 Summary outputs of regression analysis and ANOVA for *In vitro* drug release at 10 mins

	DF	**SS**	**MS**	**F**		**P-value Prob> F**
Regression	5	1998.74	399.75	193.01		0.00058
Residual	3	6.21	2.07			
Total	8	2004.96				Significant
Coefficient	**b_0**	**b_1**	**b_2**	**b_{11}**	**b_{22}**	**b_{12}**
Coefficient value	84.04	16.36	3.33	-12.75	-0.49	0.62
P-value	4.582E-06	0.0001	0.0109	0.0011	0.6652	0.4523
Full Model: $Y_1 = 84.04 + 16.36\ X_1 + 3.33\ X_2 - 12.75X_1^2 - 0.49\ X_2^2 + 0.62\ X_1X_2$						
Reduced Model: $Y_1 = 84.04 + 16.36\ X_1 + 3.33\ X_2 - 12.75X_1^2$						

Table 12 Predicted and actual responses of check point batches

Batches	Values of independent factors from overlay plot	Calculated values of independent variables (mg/unit)	Predicted Response			Actual Response		
			D.T. (sec)	% CPR	Tensile Strength (kg/cm^2)	D.T. (sec)	% CPR	Tensile Strength (kg/cm^2)
C1	X1=0.8176 X2=0.9144	X1=19.09 X2=33.49	20.47	91.99	0.216	21.31	86.19	0.210
C2	X1=0.4971 X2=0.9548	X1=17.49 X2=28.73	20.89	92.05	0.218	19.47	84.77	0.191

ACKNOWLEDGEMENT

Authors thank Principal, Shri Sarvajanik Pharmacy College, Mehsana for extending laboratory and instrumental facilities to carry out the work. Authors are grateful to Dean, Faculty of doctoral studies and research and school of pharmacy, RK University, Rajkot for guidance and support to carry out research.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th& Jan. 6th, 2016)

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RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

Influence on drug dissolution, cellular toxicity and physical stability of methotrexate loaded mesoporous nanoparticles

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ABSTRACT

Purpose of the study is to determine the effect of drug loaded mesoporous nanoparticles, on drug dissolution, their physical stability within the mesopores and their nano-toxicological properties on different selected cell cultures. Methotrexate was loaded in MSU-H nanoparticles by direct impregnation method and drug loaded nanoparticles were characterized by different techniques. Nano-toxicological study was performed by MTT assay technique on two cell cultures, K-562 and L-132. Physical stability of the drug loaded nanoparticles was conducted by performing accelerated stability study, according to ICH guideline. Methotrexate loaded MSU-H nanoparticles revealed a remarkable dissolution rate improvement in comparison to the crystalline drug and marketed formulation in all tested conditions. Drug loaded MSNs does not produce significant toxicity to the selected cell culture when exposed at a concentration of 1000 µg/mL for 96 h of exposure time. Drug loaded nanoparticles were physically checked within the mesopores of MSU-H nanoparticles and indicated good stability.

SUMMARY

Methotrexate loaded MSU-H mesoporous nanoparticles were prepared with a aim of enhancement of drug dissolution and evaluated for their physical characteristics, stability and cytotoxicity.

Keywords: *Mesoporous nanoparticles, Methotrexate, In Vitro dissolution, Cytotoxicity study, Physical stability*

INTRODUCTION

Mesoporous material has explored as drug carrier and efficientlyemployed fordissolution enhancement ofpoorlywater soluble compound.Themesoporous structure especially pore size and pore channels play animportant rolein transformation of drug molecules from crystalline to an amorphous state. Theporechannelsprevent therecrystalization of drug and decreased particle sizeof the amorphousdrugresults in dissolution enhancement of poorlysoluble compound (*1-7*). MSU-H mesoporous materials obtained through an assembly mechanism between non-ionic poly- (ethylene oxide)-based surfactants and silica precursors. In present study MSU-H nanoparticles were used as drug carrier and poorly soluble drug i.e. methotrexate (MTX) was used as a model drug. Drug loaded MSNs were checked for its physical stability and cytotoxic behavior.

MATERIALS AND METHODS

Drug loading efficiency

Drug loading was performed by taking known amount of MTX and was dissolved in fixed volume of 0.1 M HCL solution and specified amount of mesoporous nanoparticles were added. The above mixture was stirred with magnetic stirrer for 24 h at room temperature and set aside to settle for 2 h and was collected by filtration. Thus collected solid was dried in vacuum oven for 24 h and finally stored in glass vial at room temperature. The loading efficiency was determined indirectly bycalculating the non-entrapped amount of drug, found in the loading solvent and in the washing liquid. The total amount of drug loading was analyzed by double beam spectrophotometer at λmax of 306 nm. Drug loading efficiency was calculated after application of appropriate dilution factor according to the following equation.

$$Wt\ \% = \frac{m1 - \frac{50}{v}CV}{m2 + \left(m1 - \frac{50}{v}CV\right)} \times 100$$

Where, Wt% is the total amount of drug loaded, m1 and m2 express the initial mass of drug and mesoporous nanoparticles added into to loading solvent (0.1 M HCL) respectively. C is the concentration of filtrates diluted in 50 ml volumetric flask, v is sampled volume from filtrates, and V is the volume of 0.1 M HCL solution for drug loading.

Physical stability study

Physical stability of drug loaded MSNs was evaluated by performing accelerated stability study: (a) 30 °C ± 2 °C and 65% ± 5% relative humidity. (b) 40 °C ± 2 °C and 75% ± 5% of relative humidity.

Cytotoxicity study by MTT assay

Cytotoxicities of MSNs were evaluated on K 562 and L 132 cells by 96-well plate method by MTT assay technique at predetermined time period.

In Vitro dissolution study

Drug release from the mesoporous nanoparticles was performed on rotating paddle method at rotating speed of 50 rpm (Veego dissolution test apparatus-basket type USP), at 37±0.5 oC. Dissolution was performed in the different dissolution media representing different pH conditions.

RESULTS AND DISCUSSION

Characterization of drug loaded MSU-H nanoparticles

Drug loaded MSU-H nanoparticles were evaluated by different techniques like transmission electron microscope, differential scanning calorimetry, FTIR analysis, powder X-ray diffraction and BETanalysis and BJH pore size distribution. The data of all the characterization techniques revealed that drug molecules are confined within the mesopores of MSU-H nanoparticles (Figure 1).

Physical stability study

The eventual presences of crystallineform of drugs within the mesopores weremonitored byXRD and DSC,physical stabilityof MSNs was checked byTEM. The data of the study indicate that drug molecules are successfully confined within the mesopores and same time the mesoporosity of MSU-H nanoparticles retained within the specified period of time (Figure 2).

Cell cytotoxicity study

The in vitro cytotoxicity of MTX loaded MSNs on K 562 and L 132 cells at specified concentration and incubation time, demonstrated that the decrease in cell viability of selected cells cultures was more prominent when MSNs were exposed to higher concentration for longer duration of incubation. Under the tested experimental conditions it was found that, the MSNs were non toxic when used at concentration of 1000 µg/ml and 96 h of incubation time (Figure 3).

***In Vitro* Drug dissolution study**

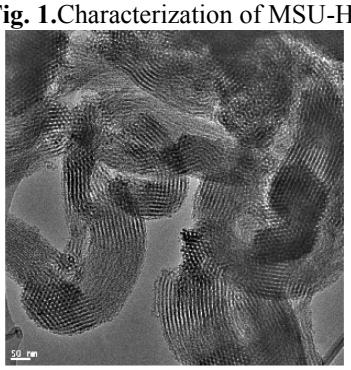
Drug dissolution was conducted at different pH environment to investigate the drug release profile. The dissolution of MTX from MSNs was compared with those from crystalline MTX, physical mixture and marketed formulation. Table 4 and 5 show the MTX release percentages at the tested pHs after 10 and 30 min respectively. The results of drug release data revealed that the release of drug was more prominent in gastric fluids. In fact, in pH 1.2 medium more than 60% and 95% of drug release was found from MTX loaded MSNs after 10 and 30 min respectively. It can be observed that the extend of MTX release from the crystalline powder was quite less, as low as 15% and less than 40% after 10 and 30 min respectively. The release of MTX from MTX loaded MSNs at pH 1.2 was compared to that of marketed formulation of MTX. The data of dissolution profiles revealed that after the 10 min more than 60% of drug was released from MTX loaded MSNs whereas only 28% was released from marketed formulation. After 30 min of drug release profile it was found that drug release from marketed formulation was 79% whereas more than 95% release was obtained from the developed formulation.However, the data of in vitro dissolution study revealed that in all selected and tested conditions the MTX release rate was enhanced as compare to crystalline drug, physical mixture and marketed formulation (Figure 4).

CONCLUSION

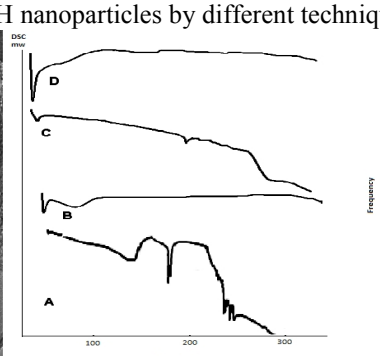
The result of the study demonstrated the potentiality of mesoporous MSU-H material as drug carrier. The drug loaded mesoporous MSNs system was successfully developed with poorly soluble anticancer drug and established that the rate of drug dissolution was considerably enhanced.

FIGURES

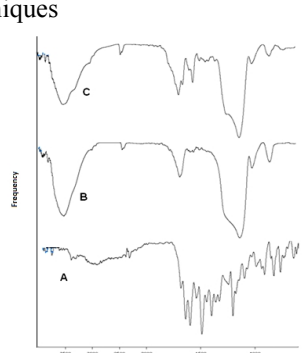
Fig. 1. Characterization of MSU-H nanoparticles by different techniques



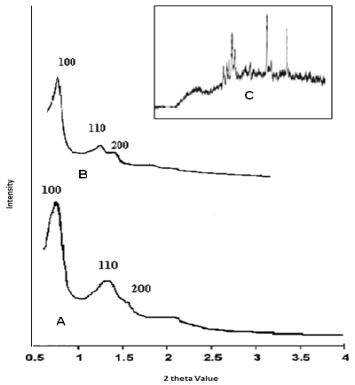
TEM images of MSU-H



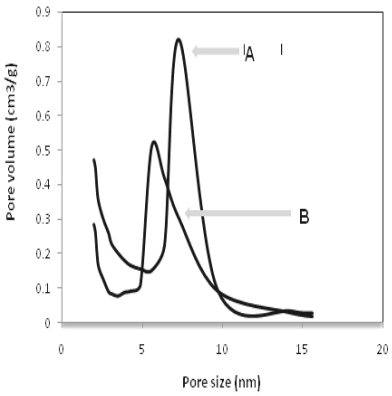
DSC thermogram – (A) MTX (B) MSU-H MSNs (C) MTX + MSU-H physical mixture (D) MTX loaded MSNs MSNs



FTIR spectra –(A) MTX (B) MSU-H MSNs (C) MTX loadedMSNs MSNs



XRD pattern –(A) MSU-H MSNs (before drug loading) (B) MSU-H MSNs (after drug loading) (C) Pure MTX MSNs



Pore size distribution and pore volume of MSU-H MSN (A) before drug loading (B) after drug loading

Fig. 2.(A) TEM images of MTX loaded MSU-H MSNs at 30 ◦C ± 2 ◦C and 65% ± 5% relative humidity after 6 months, (B) TEM images of MTX loaded MSU-H MSNs at 40 ◦C ± 2 ◦C and 75% ± 5% relative humidity after 6 months

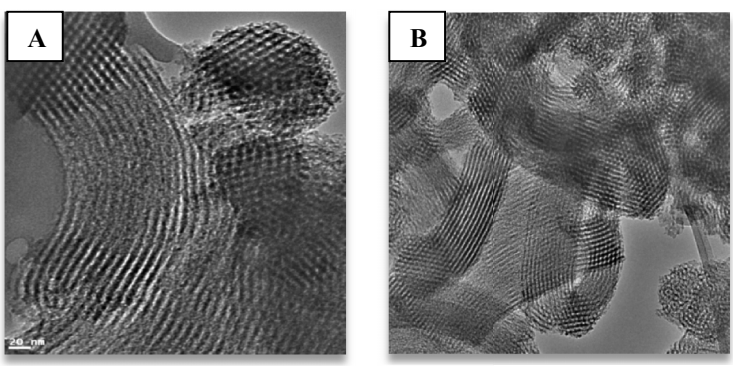
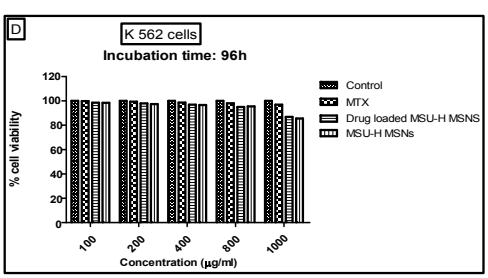


Fig. 3. Effect of MTX + MSU-H MSNs on cell viability of K-562 cells and L- 132 cells at different concentrations and incubation time of 96 h



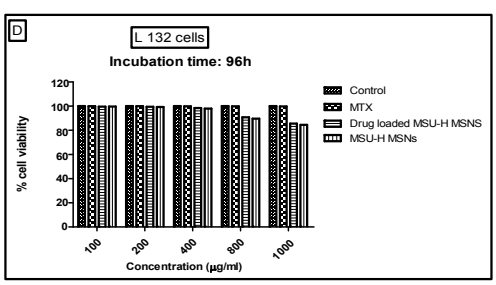
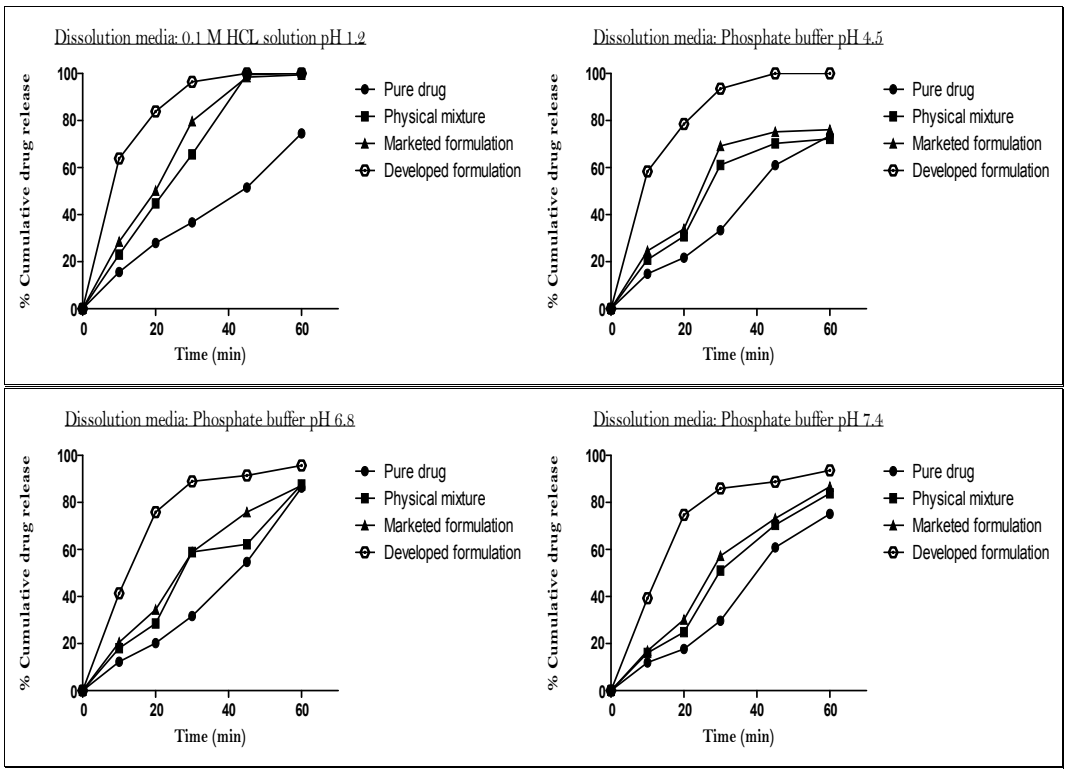


Fig. 4. Drug release profile of MTX from crystalline MTX, physical mixture, marketed formulation, and MTX loaded MSNs from different dissolution media



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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th& Jan. 6th, 2016)

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RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

Effectiveness of *Withaniacoagulans*in Treatment ofType 2 Diabetes

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ABSTRACT

We conducted Diabetes screening camps and interviewed 1001 participants (499 Non-diabetics and 502 Diabetics) using pre-validated questionnaire. Anthropometrical measurements like height, weight, waist, hips were noted. Body Mass Index (BMI), Waist-to-Hips Ratio (WHR) and fasting blood sugars were significantly higher among diabetics. Polyuria, polyphagia, polydipsia were prevailing among diabetics. Diabetics were categorized on the basis of the antidiabetic therapy they were taking. Few patients were not taking any antidiabetics. Ghanvatis prepared from the aqueous extract of *Withaniacoagulans* (known as Paneerful/Paneerdodi) were given to all these patients for nine months. Fasting blood sugars were measured initially and afterwards at the end of first, third, sixth and ninth month. Strong follow up was taken for the compliance of medication. *Withaniacoagulans* significantly reduced fasting blood sugar of Diabetics. Traditional use of *Withaniacoagulans*was previously established by pre-clinical studies in our laboratory which is further substantiated by the present clinical study.

SUMMARY

Ghanvatis prepared from the aqueous extract of the fruits of*Withaniacoagulans*significantly reduces the fasting blood sugar of the patients having Type 2 Diabetes Mellitus.

Keywords: *Ghanvatis, Withania coagulans, Paneerful/Paneerdodi,Type 2 Diabetes, Fasting Blood Sugar, WHR, BMI*

INTRODUCTION

Diabetes mellitus is one of the most common chronic endocrine disorders affecting millions of people worldwide (*1, 2*). Diabetes is characterized by an increase in fasting and post-prandial blood glucose levels, insulin deficiency and/or decreased insulin actions. Because of the sustained high glucose levels, glycosylated haemoglobin increases in diabetics (*3,4*), which in turn causes thickening of the capillary basement membrane throughout the body leading to microangiopathy, macroangiopathy, nephropathy and retinopathy(*5,6*). Thus, in diabetes mellitus there occurs derangement in carbohydrate, lipid and protein metabolism (*7*) and the development of long-term complications (*8*), such as microangiopathy, macroangiopathy, atherosclerosis, retinopathy (*9*), nephropathy (*10-12*), cardiomyopathy (*13*), autonomic neuropathy (*14*), etc. Diabetes mellitus is also associated with increased incidence of morbidity and mortality due to cardiac and renal complications.We studied various Risk Factors which may lead to Diabetes and also observed the Prevalence of few Cardinal Symptoms ofDiabetes Mellitus among big population of Type 2 Diabetics (*15*).*Withaniacoagulans*(known as Paneerful or Paneerdodi) belongs to Family Leguminosae. In addition to being used as "Indian cheesemaker" or "vegetable rennet" it is one of the important medicinal plants. Traditionally the natives have been using *Withaniacoagulans* for various ailments including diabetes mellitus (*16, 17*). Pre-clinical studies performed in our laboratory gave very much encouraging results and significant reduction in the blood sugar of Streptozotocin induced diabetic rats was observed (*18*). This has mimicked the present clinical study.

MATERIALS AND METHODS

Collection and Authentication of *Withaniacoagulans*

Fruits of*Withaniacoagulans*werecollected from G.Y.Hakim, Raopura, Vadodara. Herbarium sheet was prepared and Dr. P. S. Nagar (Taxonomist) authenticated it by comparing with the specimen, database and other documents available at the Botany dept., M.S University, Vadodara.

Preparation of Ghanvatis for *Withaniacoagulans*

Coarse power of the fruits was subjected to extraction with water at 70-80°C. Water was evaporated from the aqueous extract of *Withaniacoagulans*till it was reduced to a semisolid mass. Water was evaporated

from the aqueous extract (3.75 mg/ml) of *Withaniacoagulans*till it was reduced to a semisolid mass. Ghanvatis were prepared from that mass using Pill making machine. From one Kg of dry powder of *Withaniacoagulans*taken initially for the preparation of aqueous extract 4000 (four thousand) ghanvatis were prepared.The weight of each ghanvati was 250 mg. No other excipients were added while preparing the ghanvatis except dry powder of *W. coagulans*to help formation of pills from the semisolid aqueous extract. Thus, each ghanvati represented 250 mg of the dry weight of *Withaniacoagulans*powder.

Clinical Study of *Withaniacoagulans*Ghanvatis for the treatment of diabetes

An open, multicentric clinical study based on parallel group design was conducted. The local ethical committee approved the protocol and proforma. Diabetes screening camps were organized at three different places in Baroda (Sigma Institute of Pharmacy, Different outlets of Apollo Pharmacy and Exhibition of VCCI held at Navlakhi ground) and participants were enrolled for the Clinical study.

Inclusion criteria - Patients of either sex with mild to moderate diabetes mellitus were selected for the study. The patients having 12 hours fasting blood sugar above 140 mg/dl were considered diabetic and were selected for the study. Patients already on some anti-diabetic treatment having well-controlled 12 hours fasting blood sugar (<120 mg/dl) were also included in the present study. We also included NIDDM patients having hypertension and taking some antihypertensive treatment along with antidiabetic treatment. All the patients were within plus or minus 25% of the ideal body weight.

Exclusion criteria - Patients were excluded from the study if their age was above 70 years, had complicated hypertension, severe diabetes (>450 mg/dl), recent myocardial infarction (less than 3 months from the study), bradycardia (heart rate less than 50 beats/minute), second or third degree atrioventricular (AV) block, congestive cardiac failure (CCF), recent cerebrovascular events (less than 3 months), Severe dyslipidaemia (total cholesterol more than 450 mg/dl), renal or hepatic failure, severe concomitant diseases. Patients with insulin-dependent diabetes mellitus (IDDM) were also not included in the study.

Detailed interviews were performed with the help of a pre-tested and validated carefully planned questionnaire i.e. Clinical history sheet (Appendix–1) and Participant consent forms (Appendix-2) were filled separately for every patient and every non-diabetic subject. Approval letter for Clinical Study of *Withaniacoagulans*Ghanvatis was obtained from the ethics committee (Appendix–3). Height, weight, waist and hips girths were measured with the subject standing wearing thin clothes. WHR and BMI were calculated. Sedentary lifestyle was considered if the person walks less than 1.45 km/week, climbs fewer than 20 flights of stairs per week or performs no moderate physical activity (300 Kcal/day) on 5 days per

week. We categorized users of any form of tobacco together with former smokers, as smokers only. Blood pressure and pulse rates were measured. Glucostix reagent strips of Glucometer were used for measuring the blood sugar.

Particular day of the month was fixed for the OPD of diabetic patients. No specific dietary prescription was provided to avoid any diet fluctuation. Patients were required to continue their usual diet habits throughout the study. Patients were asked not to make changes in physical exercise or smoking habits during the course of study. Patients were asked to continue their existing therapy. All these patients were given 4 ghanvatis of *Withaniacoagulans*(250 mg *Withaniacoagulans*per ghanvati) two times a day (b.i.d.) for a period of nine months. Those diabetics who were not taking any other antidiabetics (untreated diabetics UN-DM) were also asked to take same dose of *Withaniacoagulans*. Drug compliance was assessed by pill counts. All the patients were asked to report immediately if at all they had any side effects of *Withaniacoagulans*like gastric pain, headache, diarrhoea, uneasiness etc. The patients were asked to come for check up at the regular interval of one month. The blood sample collection and monitoring of blood pressure and pulse rates were made before beginning the treatment of *Withaniacoagulans*and afterwards at the interval of 1st, 3rd, 6th and 9th month of active treatment with *Withaniacoagulans*. Non-diabetic control subjects were not given the treatment of the pills of *Withaniacoagulans.*

Groups of Patients

Non–diabetic Group (Group A, n = 499): Non-diabetic healthy subjects were selected during random screening in the clinics and medical camps. They served as control (non-diabetic). They were having normal blood pressure. They were not taking any antidiabetics or antihypertensives.

Diabetic Patients (n = 502): During the period of 21 months out of about 1001 cases examined, for diabetes, hypertension and associated complications and after considering the inclusion and exclusion criteria 502 cases of diabetes were registered for the study. The diabetic patients were already on the therapy of combination of sulphonyl urea, biguanide and thiazolidinedione group of oral antidiabetic drugs.

Patients taking Sulphonylurea and/or Biguanide and/or Thiazolidinedione and/or Acarbose etc. (Group G, n = 07): These diabetic patients were already on the therapy of combination of sulphonyl urea and/or biguanide and/or thiazolidinedione and/or acarboseetc. group of oral antidiabetic drugs.

Patients on Insulin Injection Therapy (Group H, n = 13): These diabetic patients were already on the therapy of insulin injections with or without any other oral antidiabetic therapy.

Untreated Diabetics (Group I, n = 84): In addition to above patients [taking only antidiabetics and/or taking combination of antidiabetic+antihypertensive], there were another 84 patients who were not taking any antidiabetic or antihypertensive treatment. Most of these patients were diagnosed as hyperglycemic for the first time during our random screening and were totally unaware about their harbouring the diabetes. Most of these patients were having normal blood pressure. Few patients knew that they had diabetes but they were trying to control it by good exercise as well as by diet control.

Various parameters, among the diabetic and non-diabetic groups, were represented as mean ±SEM and were analysed by analysis of variance (ANOVA single factor) followed by Tuckey's 't' test to find out the significant difference due to treatment. The value of probability less than 5% ($p<0.05$) was considered as statistically significant.

RESULTS AND DISCUSSION

During the period of 21 months (March 2013 to Nov. 2014), out of about 1001 cases examined for the inclusion in the study and after considering inclusion and exclusion criteria 502 cases of diabetes (344 males and 158 females) were registered. 499 non-diabetic healthy subjects (317 males and 182 females) were selected during random screening in the clinics and medical camps. They served as control (non-diabetic). They were having normal blood pressure. They were not taking any antidiabetic or antihypertensive drugs.The initial systolic blood pressure, diastolic blood pressure and pulse rate were found to be significantly higher in diabetics as compared to non- diabetics (Table 1).

Comparison of BMI & WHR and other factors which may predispose Type 2 Diabetes

It was found that average BMI of diabetics was 31 (indicating to be obese) while that of non-diabetics was 24. Both diabetic males and females had WHR >0.94 (indicating to be having Apple-shaped fat distribution) and non-diabetic males and females have <0.79 ((indicating to be having Pear-shaped fat distribution). While comparing the occupational and spare time physical activities it was found that diabetics were having very less physical activities. On the other hand non-diabetics were involved with regular exercises including Yoga and had tendency to perform regular physical work. Diabetics were found to have more mental stress, prevalence of constipation (70%) and 30-40% of them are taking tobacco in different forms.

Comparisonof symptoms of Diabetes among Non-Diabetics & Patients with Type 2 Diabetes

All the cardinal symptoms of diabetes like higher fasting blood sugar (>140 mg/dl), polyuria > 6-7 micturation/day and > 3-4 micturation /night), polydipsia as well as polyphagia are significantly more

among diabetics as compared to non-diabetics. Diabetics had higher prevalence of polyuria. The frequency of night micturation among diabetics was significantly higher as compared to non-diabetics. Similar was the case with the frequency of day micturation among diabetics and non-diabetics. The frequency of day micturation was found to be higher among diabetics as compared to non-diabetics but it was not statistically significant. Polyphagia and polydipsia in diabetics was found to be higher than non-diabetics. The frequency of night micturation was found to be correlated with the disturbed night sleep. The diabetics were found to be having significantly higher percentage of disturbed night sleep as compared to non-diabetics. The feeling of pain in upper and lower arms, polyphagia and polydipsia in diabetics was found to be higher than non-diabetics.

Effect of Ghanvatis prepared from the aqueous extract of *Withaniacoagulans*on Blood Sugar of Type 2 Diabetic Patients

After considering all the inclusion and exclusion criteria 1001 cases were registered during the period of 21 months (March 2013 to Nov. 2014). 502 cases of diabetes (344 males and 158 females) and 499 non-diabetic healthy subjects (317 males and 182 females) were selected during screening in the medical camps. Gradually the no. of patients decreased for varied reasons as discussed below and at the end of nine months there remained 94 patients who were continuing the treatment of the ghanvatis of *Withaniacoagulans.*

Initial blood glucose levels of diabetics were significantly higher than non-diabetics. Treatment, of these diabetic patients, with the ghanvatis of *Withaniacoagulans*for a continuous period of nine months showed significant reduction of blood glucose levels. Decrease in blood glucose in the patients started from the first month and then continued onwards until the end of the nine months. Initial value of blood glucose of diabetic patients (208.8±68.3 mg/dl) was reduced to 129±27.53 mg/dl at the end of nine months (Table 3, Figure 1).

Apparently, it appears that treatment with *Withaniacoagulans*could not bring the blood glucose level to normal level comparable to those of non-diabetics (Figure 1). However, it is important to note at this stage that, this set of observation represents collectively the patients with varied drug history. Before starting of the therapy with *Withaniacoagulans*the diabetic patients were taking antidiabetic and/or antihypertensive drugs.

Withdrawal of Patients during the Course of Study

Total 502 patients participated in the present study. Though the attempts were made to communicate with the patients by varied media like personal counselling by visiting their residences or office premises or by writing the letters or by telephone, there were drop outs from the study due to medical and non-medical reasons (Table 3). Total 408 patients dropped out during the course of study due to varied reasons. Only 94 patients continued for a period more than 12 months. 380 patients left due to non-medical reasons mainly non-accessibility. Thus it appeared that the remaining 28 patients might have some of the genuine difficulties. On inquiring following information was received. 8 patients complained of flatulence and headache at different stages of study and they withdrew themselves from the study. Two patients suffered from diarrhoea and one patient got gastric pain after ingestion of ghanvatis and all these three patients opted to remain away from the study. One patient was very much afraid of vein-puncture to give blood for the estimation and we could not convince him to continue the present study. Three patients took lower dose (2 ghanvatis b.i.d.) instead of recommended dose of (4 ghanvatis b.i.d.) and their blood glucose could not come down as per their expectation and they left the study. Nine patients could not get any reduction in the blood glucose and they left the study. One female patient was really fed up by insulin injections for her treatment of diabetes. She got good reduction in her blood glucose at the end of 1st, 2nd and 3rd months of study period. Looking at the good reduction in blood glucose, she stopped taking insulin injections without informing us but that resulted into severe hyperglycaemia. On thorough interrogation by taking her in confidence, she confessed about stopping the insulin injections. We immediately consulted her family members and family physician. We convinced her to continue the insulin injections along with ghanvatis. She started again but again her habit of stopping the insulin injections started and we were forced to drop her from the present study based on non-compliance. On the contrary there were three patients (surprisingly one of them was an Ayurvedic physician) who did not have any faith in herbal medications and they left the study.

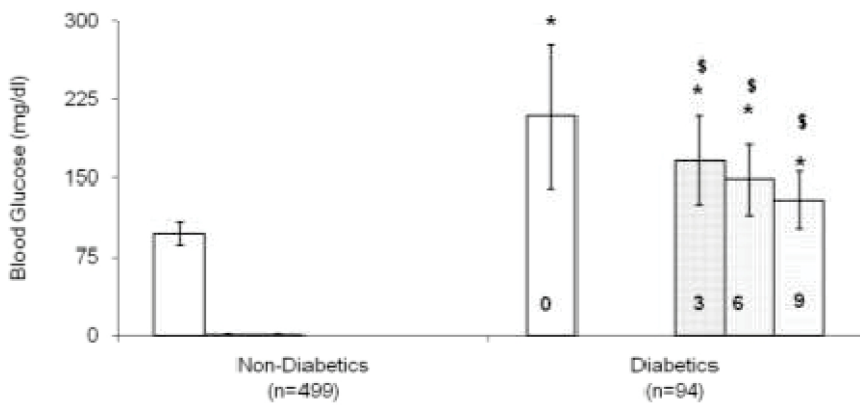
It appears that ghanvatis prepared from the aqueous extract of *Withania coagulans* (representing 250 mg of the dry weight of *Withania coagulans* powder) given to the diabetic patients (4 ghanvatis two times a day, b.i.d.) continuously for a period of nine months is well tolerable and does not produce any severe side effects.

CONCLUSION

The target of healthy life should be to maintain BMI < 25, WHR < 0.7, perform regular exercise including Yoga, and try to pacify mental stress, should have less constipation and avoid smoking. Our laboratory is performing pioneering work on *Withania coagulans.* The fruits of *Withania coagulans* have been traditionally used for the treatment of Type 2 Diabetes and this is now confirmed in our pre-clinical and clinical studies. The preparation of Ghanvatis from Paneerful present a good dosage form due to which the patient compliance has also been increased in a big way. Type 2 Diabetes can be treated effectively with the Ghanvatis prepared from the aqueous extract of Withania *coagulans* (Paneerful).

FIGURES

"Figure 1: Effect of *Withaniacoagulans*on blood sugar of Type 2 diabetic patients"



(*) Significantly different from Nondiabetic Control (P<0.05)

($) Significantly different from Initial value (P<0.05)

TABLES

"Table 1: Initial Fasting Blood Sugar and Blood Pressure of Diabetics with Non-diabetics"

Parameter	Non-Diabetic (Group A, n = 499)	Diabetics (Group B, n = 502)
Blood Glucose (mg/dl)	97±11.2	167.1±54.6*
Systolic Blood Pressure (mmHg)	123±4.1	153±2.4*
Diastolic Blood Pressure (mmHg)	80±1.6	86.44±0.88*
Pulse Rate (Beats/Min)	81±1.68	84.6±1.1*

(*) Significantly Different from Non-Diabetic Control (P<0.05)

"Table No. 2: Study of Risk factors and Cardinal symptoms of Diabetes"

Parameter	Non-Diabetics		Diabetics	
	Male	Female	Male	Female
Subjects	317 (64%)	182 (36%)	344 (69%)	158 (31%)
Blood Sugar (mg/dl)	99.2 ± 10.3	94.8 ± 12.1	167.6 ± 51.5*	163.8 ± 60.2*
Systolic B.P. (mmHg)	118 ± 2.8	128 ± 7	150 ± 2.3*	156 ± 3*
Diastolic B.P.(mmHg)	82.1 ± 4.4	78 ± 3.1	87 ± 1.1*	83.5 ± 1.5*
Pulse (beats/min)	79.9 ± 2.5	83.3 ± 2.1	83.2 ± 1.2	87.3 ± 1.4
Heavy Work (%)	197 (62%)	51 (28%)	28 (8%)	7 (4.5%)
Light Work (%)	120 (38%)	131 (72%)	316 (92%)	151 (95.5%)
Constipation (%)	60 (19%)	47 (26%)	162 (47%)	104 (66%)
Smoking (%)	73 (23%)	0 (0%)	158 (46%)	21 (13%)
BMI (Kg/sq.mt.)	23.2 ± 2.8	24.5 ± 1.9	28.4 ± 3.5	30.2 ± 4.0
BMI ≤ 25 (Normal)	275 (87%)	101 (55%)	55 (16%)	9 (6%)
BMI ≥ 25 and ≤ 30 (Overweight)	32 (10%)	72 (40%)	248 (72%)	24 (15%)
BMI ≥ 30 (Obese)	10 (3%)	9 (5%)	41 (12%)	125 (79%)
WHR	0.87 ± 0.01	0.79 ± 0.02	0.98 ± 0.01	0.94 ± 0.02*
Day Micturation	4.4 ± 0.5	4.8 ± 0.5	7.2 ± 0.4	7.4 ± 0.4
Night Micturation	0.5 ± 0.3	0.6 ± 0.5	3.4 ± 0.2*	4.1 ± 0.2
Polyphagia (%)	82 (26%)	35 (19%)	237 (69%)	90 (57%)
Polydipsia (%)	146 (46%)	25 (14%)	282 (82%)	115 (73%)
Disturbed Night Sleep (%)	6 (2%)	7 (3.6%)	138 (40%)	83 (52.7%)

(*) Significantly Different from Non-Diabetic Control (P<0.05)

"Table 3: Effect of *Withaniacoagulans*on Blood Sugar of Diabetic Patients"

Month	No. of Patients	Blood Sugar
Initial	94	208.8 ± 68.3*
Third Month	94	167 ± 42.3*, $
Sixth Month	94	148.3 ± 34.1*, $
Ninth Month	94	129 ± 27.53*, $

(*) Significantly different from Nondiabetic Control (P<0.05)

($) Significantly different from Initial value (P<0.05)

"Table No. 4: Trends of Antidiabetics among Diabetic Patients"

Group	Description	Number of Patients	Initial Fasting Blood Sugar
C	Patients taking only Sulphonyl urea	86	145.8±45.1*
D	Patients taking only Biguanide	21	192.2±63.7*
E	Patients taking Sulphonyl urea and Biguanide	175	159.51±51.7*
F	Patients taking Sulphonyl urea, Biguanide and Thiazolidinedione	116	180.8±55.5*
G	Patients taking Other Oral Antidiabetic Combinations	07	212.6±79.01*
H	Patients taking Insulin Injection with or without Other Oral Antidiabetics	13	228.6±73.9*
I	Patients not taking any Antidiabetics	84	165.9±46*
Total		502	

(*) Significantly different from Nondiabetic Control (P<0.05)

"Table 5: Effect of *Withaniacoagulans* on the blood sugar of the diabetic patients simultaneously taking different Antidiabetics"

Group	**Description of Medications which the diabetic patients are taking**	**No. of Patients (Initial)**	No. of Patients **(At the end of 9 Months)**	**Their Fasting Blood Sugar (Initial)**	**Their Fasting Blood Sugar (At the end of Nine Months)**
C	Patients taking only Sulphonyl Urea	86	14	185.4±68.3	120.7±17.6*
D	Patients taking only Biguanides	21	13	221.3 ± 59.3	142.9±29.1*
E	Patients taking Sulphonyl Urea and Biguanides	175	21	214.9 ± 70.9	126.7±22*
F	Patients taking Sulphonyl Urea, Biguanides and Thiazolidinedione	116	9	215.3 ± 80.3	114.4±24.3*
G	Other oral antidiabetic combinations	7	5	203±91.3	119.8±25.7*
H	Insulin Injection with or without other oral antidiabetics	13	12	231.9±76.2	147.5±45.7*
I	Patients not taking any antidiabetics	84	20	195.7 ± 56.4	126±18*
	Total	502	94		

(*) Significantly different from Initial value ($P<0.05$)

"Table 6: Reasons for Withdrawal of Patients during the Course of Study"

Reason for Withdrawal of Patient from the Clinical Study	**1st Month**	**2nd Month**	**3rd Month**	**6th Month**	**Total**
Flatulence and Headache	4	2	1	1	8
Gastric Pain	1				1
Diarrhoea	2				2
Afraid of venipuncture		1			1
Ingestion of lower dose (2-0-2)		3			3
Real non-responders		4	1	4	9
Non-Compliance				1	1
No faith in Herbal medication	3				3
Not accessible due to non-medical reasons like transfer to other city, marriage etc.	278	60	39	3	380
Total	288	70	41	9	408

ACKNOWLEDGEMENT

The corresponding author is very much pleased to acknowledge Shri Shaileshbhai Shah, Chairman, Sigma Group of Institutes for providing the infrastructure and resources to carry out the present work.

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APPENDICES

Appendix–1: Clinical history sheet for *Withaniacoagulans*

[01] Name of the Participant:-

[02] Participant's No.

[03] Area:-

[04] Sex: - Male /Female

[05] Age:-

[06] Address:-

[07] Telephone No:-

[08] Income (Rs. /month):- LIG / MIG / HIG

(<6000) / (6001-14000) / (>14001)

[09] Chief Complaints: - Polyuria

Frequency – Day micturation

– Night micturation

Polydipsia

Polyphagia

Pain in Hands

Pain in Legs

Pain in Whole body

[10] Past History : - Diabetic since _____years

Hypertensive since _____years

[11] Anthropometry: - Waist (meter)

Hips (meter) WHR

Height (meter)

Weight (kg)

B.M.I. (kg/sq.mt)

[12] Profession:-

[13] Life Style: - Heavy Work / Medium work / Sedentary life

[14] Presence of Stress: - Yes / No

[15] Food Habits: - Direct or Indirect Sweets / Non sugar diet

Vegetarian/ Non-Vegetarian

Occasional Non-Vegetarian

[16] Tobacco Consumption: - Yes / No

[17] Pattern of sleep: - Regular / Irregular

[18] Presence of Constipation: - Yes / No

[19] Menstrual History: - Regular Menstruation / Irregular Menstruation

Onset of Menopause since_____years back

[20] Obstetric History: - Age of marriage

No. of Children Abortions/Still birth

[21] Past Reports:-

Date	**Blood glucose (mg/dl)**		**Urine sugar (gm/dl)**		**Serum Cholesterol (mg/dl)**
	Fasting	**PPBS**	**Fasting**	**PPBS**	

[22] Present Medications:-

Name of the medicine taken by the patient	Contents of the medicine	Dose per day

[23] Investigations carried out in the present clinical study:-

Sr. No.	Parameter Name	Time Duration (Months)									
		First Day	1	2	3	4	5	6	7	8	9
1	*Fasting Blood Sugar*	√	√		√			√			√
2	Ghanvatis *of Withaniacoagulans*	√	√	√	√	√	√	√	√	√	√
3	Counselling	√	√	√	√	√	√	√	√	√	√

[24] Any other information

Place:-

Date of Admission:-

Signature of Participant **Signature of Principal Investigator**

Appendix -2: Participant consent form for *Withaniacoagulans*

I freely and voluntarily consent to be a participant in the research project on the topic of Clinical investigations of *Withaniacoagulans*to be conducted by Prof.SiddhibenUpadhyay as Principal investigator, who is a Ph.D. student at R. K. University, Rajkot. The broad goal of this research study is to explore Effect of *Withaniacoagulans*in the treatment of Diabetes mellitus. Specifically, I have been asked to take PaneerfulGhanvati prepared from water extract of fruits of *Withaniacoagulans*, which should take no longer than Nine months to complete. I have been told that my responses will be kept strictly confidential. I also understand that if at any time during the period of Nine months I feel unable or unwilling to continue, I am free to leave. That is, my participation in this study is completely voluntary, and I may withdraw from it at any time without negative consequences. [In addition, should I not wish to answer any particular question or questions, I am free to decline.] My name will not be linked with the research materials, and I will not be identified or identifiable in any report subsequently produced by the researcher. I have been given the opportunity to ask questions regarding the /interview/survey/procedure, and my questions have been answered to my satisfaction. I have been informed that if I have any general questions about this project, I should feel free to contact Prof.SiddhibenUpadhyay at M 09909976788, siupa.pharma@gmail.com. If I have any comments or concerns about the ethics procedures employed in this study, I can contact Dr. U. M. Upadhyay. I have read and understand the above and consent to participate in this study. My signature is not a waiver of any legal rights. Furthermore, I understand that I will be able to keep a copy of the informed consent form for my records.

Signature of Participant **Date**

I have explained and defined in detail the research procedure in which the respondent has consented to participate. Furthermore, I will retain one copy of the informed consent form for my records.

Signature of Principal Investigator **Date**

Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th& Jan. 6th, 2016)

(Proceedings available for download at rku.ac.in/icre)

RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

Development and Validation of Simultaneous estimation of Pregabalin, Methylcobalamin and Alpha lipoic acid in multicomponent dosage form by rp-hplc method

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ABSTRACT

A quick, precise, accurate, linear, and sensitive RP-HPLC method has been developed and validated for the simultaneous determination of Pregabalin (PRG), Methylcobalamin (MCA) and Alpha lipoic acid (ALA) in combined dosage form. The chromatographic separation was performed on Hibar® 250-4.6mm, Lichrosher® 100, RP- 18e (5 µm), Merck ltd., India; using mobile phase: Water : Methanol : Acetonitrile (75:15:10 %v/v/v), at a flow rate of 1.2 ml/min and 20 ± 5°C column temperature with the detection wavelength at 215nm. The retention times of PRG, MCA and ALA were 1.9 min, 5.7 min and 4.0 min respectively. The linearity was performed in the concentration range of 150 – 750 µg/ml (PRG), 8 – 40 µg/ml (MCA), and 200 - 1000µg/ml (ALA) with a squared correlation coefficient of 0.9973, 0.9965 and 0.9998 for PRG, MCA and ALA respectively. The Proposed method has been validated according to ICH guidelines.

SUMMARY

This analytical method provides quick ,sensitive and cost-effective method for simultaneous estimation of multicomponent dosage form.

Keywords: Pregabalin, Methylcobalamin, Alpha lipoic acid, RP-HPLC, Validation

INTRODUCTION

Pregabalin (PRG), (S)-3-(aminomethyl)-5-methylhexanoic acid (Fig.1) is an antiepileptic used in the treatment of peripheral neuropathy. Its molecular weight is 159.2 g/mol with empirical formula $C_8H_{17}NO_2$ (1,2). Methylcobalamin (MCA), (1R,2R,4S,7S)-7-{[(2S)-3-hydroxy-2-phenylpropanol]oxy}-9,9-

dimethyl-3-oxa-9-azoniatricyclo[3.3.1.02,4]nonane (Fig.2) is vitamin supplement used in inadequacy of Vitamin-B_{12} (3). Its molecular weight is 1344.38 g/mol with empirical formula $C_{65}H_{91}CoN_{13}O_{14}P$ (4). Alpha lipoic acid (ALA), (R)-5-(1,2-dithiolan-3-yl)pentanoic acid (Fig. 3)(5).It is an universal reducing agent which prevents oxidative damage of brain cells(6). Its molecular weight is 1344.38 g/mol with empirical formula $C_{65}H_{91}CoN_{13}O_{14}P$(5). Combination of PRG, MCA and ALA treats the problems related to all types of neuropathy and epilepsy(7). PRG is manifested to be very efficacious and well permitted in the treatment of neurogenic dysaesthesia. MCA increases myelin sheath formation thereby reforms brain cells(6). ALA is also used as universal antioxidant which disallows oxidative damage ofbrain cells(7).The marketed formulation is NERVUP PG which contains pregabalin75 mg, methylcobalamin 750 mcg and alpha lipoic acid 100 mg. Literature survey revealed that only few HPLC methods for the analysis of simultaneous estimation of Pregabalin, Methylcobalamin and Alpha lipoic acid have been reported. In the present work we are focused to acomplish the optimum chromatographic conditions for the simultaneous determination of PRG, MCA and ALA in the combined dosage form. The developed method can be implemented successfully asquality control tool. The method was validated as per ICH guideline to access the replicability and extensive practicability of the developed method(8,9).

MATERIALS AND METHODS

Instrumentation

Chemicals and Reagents

Pregabalin, Methylcobalamin and Alpha lipoic acid was procured as a gift sample from Alembic Pharmaceuticals, Vadodara, Influx Pharmaceuticals, Gandhinagar, Sunvij drugs ltd., Vadodara. All chemicals used of HPLC grade: Water, Methanol and Acetonitrile from Merck ltd. Mumbai, India.

Equipment

Young – Linn clarity 9100 HPLC system, vacuum degasser YL-9101,Photo diode array detector YL-9160.

Chromatographic Conditions

The mm Ø in size column hibar® 250-4.6mm, Lichrospher® 100, RP-18e (5 µm), Merck ltd., India.The mobile phase consists of Water: Methanol: Acetonitrile (75:15:10 % v/v/v). The rate of flow was kept to 1.2 ml/min. The instrument was operated at 20 ± 5 ºC and 1500 – 3000 psi. The UV detection was achieved at 215 nm and purity analysis was performed over a wavelength range 200-400 nm. The injected volume was 20 µL.

Preparation of Analytical Solutions

Preparation of Mobile Phase

The HPLC grade Methanol, Acetonitrile and Water were filtered through 0.20 µm membranefilter paper separately. Filtered solutions were ultrasonicated for 20 min. Solutions were allowed to stand for a while to come at room temperature if they were warmed due to sonication.

Preparation of Standard Solutions

MCA stock solution:10mg of MCA was accurately weighed and then it was transferred to 10 ml volumetric flask, dissolved and diluted up to mark with methanol.So the final concentration of MCA was 1000 µg/ml. Filteration was done by using 0.20 µm membrane filter paper and then sonicated for 5 min.

Preparation of standard working solution of mixture of PRG, MCA and ALA: Standard mixture solution were prepared by accurately weighed 75mg of PRG, 4 ml of MCA from stock solution and 100mg of ALA were transferred to 10ml volumetric flask.The solutions were dissolved and diluted up to mark with

methanol to obtain final concentration of 7500 µg/ml PRG, 240 µg/ml MCA and 10000 µg/ml ALA. This solutions were filtered by using 0.20µ membrane filter paper, sonicated for 5min and used as working standard solution. By appropriate dilution using mobile phase, from the working standard solution of PRG, MCA and ALA, standard mixture containing 150 + 8 + 200, 300 + 16 + 400, 450 + 24 + 600, 600 + 32 + 800, 750 + 40 + 1000µg/ml PRG + MCA + ALA were prepared and sonicated for 5min. Replication of standard mixture were prepared six times.

Preparation of Sample Solution: Weigh accurately content equivalent to 75 mg, 0.75 mg and 100 mg of ALA.Theywere moved to 10 ml volumetric flask and dissolved perfectly by using methanol and ultimately diluted up to mark with methanol to obtain final concentration of 7500 µg/ml of PRG, 750 µg/ml of MCA and 10000 µg/ml of ALA. From this solution sufficient ml was taken to obtain 100 % test solution. As concentration of MCA is less to be analysed in capsule formulation so, 3.25 mg of standard MCA was added in the above solution as per standard addition method. Pass the solution through a filter of 0.20 µm filter.

RESULTS AND DISCUSSION

HPLC Method development and validation

As per ICH guideline,the developed analytical method was validated with regard to certain parameters such as specificity, linearity, precision, accuracy, and system suitability.

Linearity

It is used to indicate ability to obtain test results which directly proportional to the concentration of analyte in the sample. The linearity of the method was engrafted by a spiking a series of sample mixtures of PRG, MCA and ALA, the solutions of five different concentration levels 150 – 750 µg/ml (PRG), 8 - 40µg/ml (MCA) and 200 - 1000µg/ml (ALA) are injected into the HPLC system.The same analysis was carried out for six times .from that mean area with its standard deviation and % relative standard deviation peak area were calculated. Calibration curve was obtained by putting mean AUC against concentration and regression equations, co-relation coefficients were derived from calibration curves.

Precision

It informs about the nearness of agreement between the series of measurement obtained from multiple sampling of same homogeneous sample under the prescribed conditions. Method precision was carried out in terms of repeatability (injection and analysis) and intermediate precision (It shows the degree of reproducibility of test results obtained by analyzing the sample under variety of normal test conditions such as analyst, instruments). To determine precision, six independent sample solution preparations from a single lot of formulation 450 µg/ml for PRG, 24µg/ml for MCA and 600µg/ml for ALA were injected in to HPLC system, the retention time and peak area was determined and determined as mean and %RSDcalculated from the data obtained which are found to be within the specified limits.

Accuracy

Accuracy was intended in terms of percentage recovery.It was carried out for 80%, 100% and 120 % for PRG, MCA and ALA. Standard and sample solutions are injected into HPLC system in triplicate and percentage recoveries of PRG, MCA and ALA were calculated.At each level the area was used for finding out of % recovery.

Specificity

It is nothing but judging its ability to measure accurately and specifically the analyte of interest without interferences from blank. Solution containing 450 µg/ml PRG, 24 µg/ml MCA and 600 µg/ml ALA mixture were prepared from their respective stock – working standard solution prepared as described in section 5.2.7. The analysis of prepared solutions was carried out as per the proposed method and interferences from blank were accurately measured.

Robustness

By using robustness phenomena, the developed method evaluating the influence of small deliberate variations in procedure variables like flow rate, change in mobile phase composition and change in water manufacturer. The robustness was performed for the flow rate variations from 0.8ml/min, 1ml/min and 1.2ml/min, for variation in mobile phase Water : Methanol : Acetonitrile :: 73 : 13 : 7 ; 75 : 15 : 10 ; 77 : 17 : 12 and change in water manufacturer Finar, Rankem. At each level the mean peak area with its standard deviation and %relative standard deviation was calculated and found within specified limts.

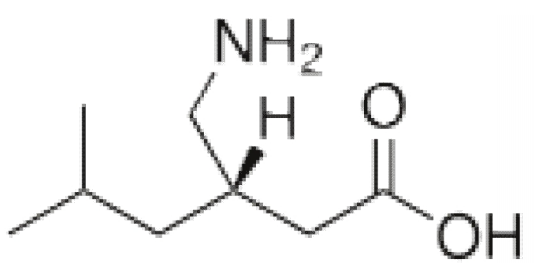
System Suitability Parameters

Solution containing mixture of 450 µg/ml PRG, 24 µg/ml MCA and 600 µg/ml ALA were analysed.Data related to peak like area, height, width, retention time, resolution, tailing (symmetry) factor, column efficiency (theoretical plates) etc. was recorded. All system suitability parameters were computed using these recorded data.

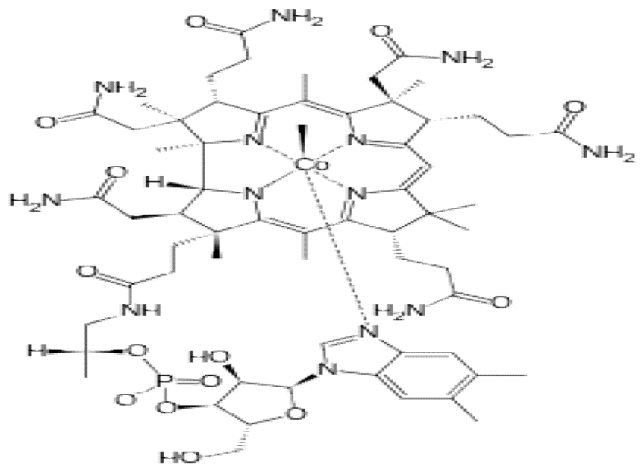
CONCLUSION

The developed method was established to be precise accurate, linear ,robust and specific for determination of PRG, MCA and ALA. The method was developed and validated as per ICH guideline and all parameters tested were found to be within limits.

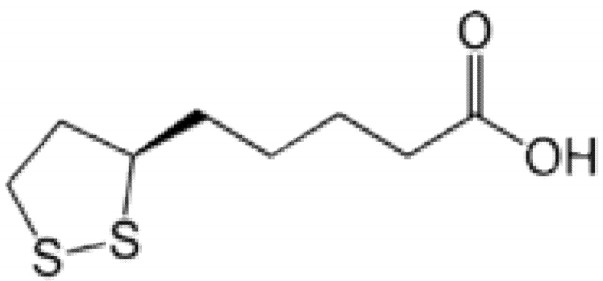
FIGURES



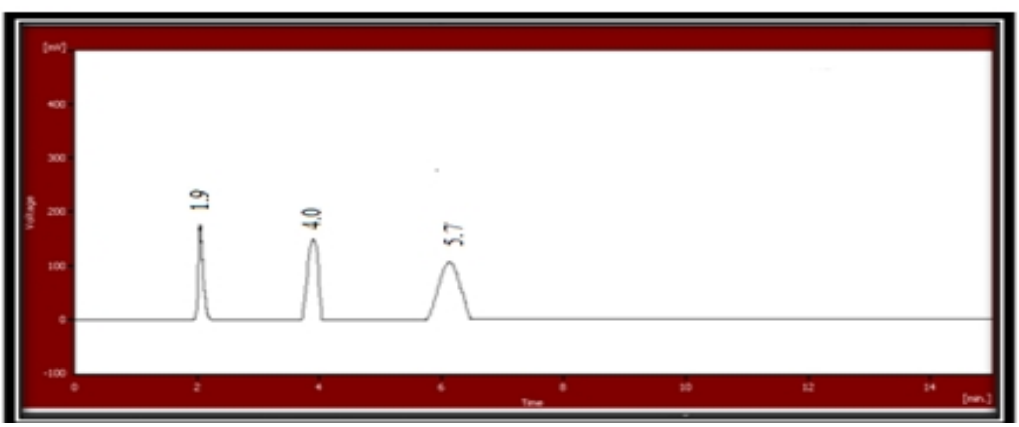
“Fig. 1 Chemical structure for Pregabalin”



"Fig. 2 Chemical structure for Methylcobalamin"



"Fig. 3 Chemical structure for Alpha lipoic acid"



"Fig.4 Chromatogram of PRG (450 µg/ml), MCA (24 µg/ml) and ALA (600 µg/ml) in Water: Methanol: ACN (75: 15: 10 % v/v/v), Flow Rate 1.2 ml/min Detection Wavelength 215 nm"

TABLES

“Table 1: Linearity results for PRG, MCA and ALA”

PRG		MCA		ALA	
Concentration(µg/ml)	AUC(mV.s)	Concentration(µg/ml)	AUC(mV.s)	Concentration(µg/ml)	AUC(mV.s)
150	227.4578	8	2968.7651	200	1018.4041
300	287.0160	16	3944.4234	400	1495.2584
450	322.2825	24	4580.9190	600	1971.0290
600	378.1570	32	5373.0551	800	2465.0011
750	426.1398	40	6239.2754	1000	2975.7742

"Table 2 System Precision of PRG, MCA and ALA"

Sr. No.	Concentration(µg/ml)			AUC (mV.s)		
	PRG	MCA	ALA	PRG	MCA	ALA
1	450	24	600	331.921	4584.653	1967.281
2	450	24	600	336.136	4501.279	1943.921
3	450	24	600	334.192	4593.173	1974.378
4	450	24	600	329.026	4641.728	1962.388
5	450	24	600	329.156	4557.293	1986.329
6	450	24	600	337.128	4607.387	1996.482
Mean AUC				332.9265	4581.639	1971.029
SD				3.4640	47.8813	18.5104
% RSD				1.0404	1.1. 1.0452	0.9387

"Table 3 Method Precision of PRG, MCA and ALA"

Sr. No.	Concentration(µg/ml)			AUC (mV.s)		
	PRG	MCA	ALA	PRG	MCA	ALA
1	450	24	600	330.567	4584.653	1968.359
2	450	24	600	335.615	4501.279	1945.231
3	450	24	600	332.816	4593.173	1973.218
4	450	24	600	328.413	4641.728	1957.214
5	450	24	600	329.156	4557.293	1982.157
6	450	24	600	337.128	4607.387	1999.994
Mean AUC				332.2825	4581.639	1971.029
SD				3.5372	48.795	19.1369
% RSD				1.0645	1.0650	0.9709

"Table 4 Recovery for PRG"

Level	Amount Recovered	% Recovery	SD	% RSD
80 %	60.476	100.794	0.8525	1.4097
100 %	75.001	100.244	0.0068	1.2847
120 %	90.229	100.568	0.8246	1.0873

“Table 5 Recovery for MCA”

Level	Amount Recovered	% Recovery	SD	% RSD
80 %	3.201	100.061	0.0080	1.0458
100 %	4.009	100.244	0.0068	1.1282
120 %	4.819	100.568	0.8246	1.1265

“Table 6 Recovery for ALA”

Level 1.2.	Amount Recovered	% Recovery	SD	% RSD
80 %	80.179	100.223	0.8385	1.4844
100 %	99.725	100.244	1.1318	0.9985
120 %	120.743	100.568	1.3602	1.2386

"Table 7 System suitability parameters"

Parameters		Value obtained	Standard Value
Capacityfactor (K')	PRG-ALA	2.438	1.3.
	ALA-MCA	4.591	1.4.
Resolution(R_S)	PRG-ALA	4.3125	>2
	ALA-MCA	1.0117	
Tailing factor orSymmetry factor(T)	PRG	1.466	<2
	ALA	1.045	
	MCA	1.215	
Column efficiencyor Theoretical plates(N)	PRG	25703	>2000
	ALA	16106	
	MCA	16768	

ACKNOWLEDGEMENT

I am thankful to Alembic Pharmaceuticals, Vadodara and Influx Pharmaceuticals, Gandhinagar, Sunvij drugs ltd., Vadodara for providing gift samples of Pregabalin, Methylcobalamin and Alpha lipoic acid for research.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th& Jan. 6th, 2016)

(Proceedings available for download at rku.ac.in/icre)

RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

Overcoming the Difficulty in the Detection of the Pesticide Chlorantraniliprole

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ABSTRACT

Chlorantraniliprole is widely used as pesticide on vegetables, fruits and major crop for larva control. Excessive use of such Chlorantraniliprole on vegetables and fruits produced very toxic effect on children and also adults. Major toxic effect was reported as aberration and polyploidy. Number of studies was reported on detection of chlorantraniliprole by HPLC and UV analytical methods. Chlorantraniliprole is only soluble in DMF as per theoretical and practical solubility data. But most of reported method was used methanol and other solvent except than Dimethyl formamide. So it was our interest to focus on solubility of chlorantraniliprole for method development and discover the suitable sample preparation condition. We had developed rapid, accurate, precise and sensitive UV method for chlorantraniprole.

SUMMARY

Spectroscopic method development for detection of chlorantraniliprole residues.
Keywords: chlorantraniliprole ,uv spectroscopy,pesticide, DMF,larvicide,

INTRODUCTION

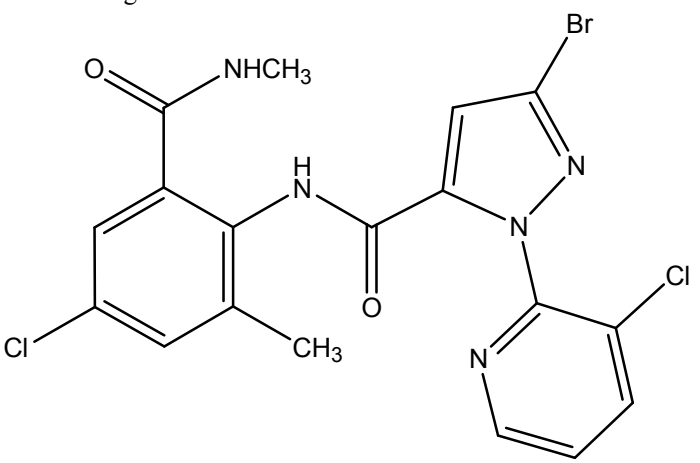
Pesticide can be defined as substance or may be mixture of compounds intended for destroying, preventing, or controlling any pest, including human or animal disease, or insect attack on plants or animals, causing harm during production, related processing, storage of that, transportation of food, or marketing of any food, or substances and related agricultural commodities, or animal feed stuffs, that may be administered to animals for the control of insects or other pests in their bodies. The term includes substances intended for use as a plant growth promoting agent for retreating fruit and vegetables or stopping the premature reduction of produce. Also used as elements applied to crops either before or after harvest to protect the commodity from deterioration during packing and transference.*(1, 2)*

Type of pesticide:*(3)*

Algaecides AntimicrobialsBio pesticides

DesiccantsDefoliants Disinfectants Fungicides Herbicides Illegal and Counterfeit PesticidesInsecticidesInsect Growth RegulatorsMinimum Risk PesticidesMiticides MolluscicidesPlant Growth Regulators Ovicides Pheromones Natural and Biological PesticidesRodenticidesMothballsRepellentsWood Preservatives

CHLORANTRANILIPROLE DRUG PROFILE(*4, 5*)

parameter	**Specification**
Chemical Name*(4)*	5-Bromo-2-(3-chloro-2-pyridine-2-yl)-2H-pyrazole-3-carboxylic acid (4-chloro-2 methyl-phenyl)-amide
Molecular Formulary*(4)*	$C_{18}H_{14}N_5O_2BrCl_2$
Molecular Weight*(4)*	483.14606 g/mol
Structure*(4)*	
CAS Number*(4)*	500008-45-7
pKa*(5)*	10.88 ± 0.71
Melting Point*(5)*	200-210 °C
Solubility:	Dimethyl formamide

Guideline Followed In India:

- Food safety and standards authority of India (FSSAI):
- SANCO GUIDELINE [SANCO/12571/2013]:

Reported methods:

1. Pengjun X., et al, (2010) developed method for the determination of chlorantraniliprole in 6 types of grains, fruits and Vegetables. Chlorantraniliprole residues from foodstuff was extracted with Acetonitrile and cleanup of raw extracts done with same solvent. Detection and quantification of chlorantraniliprole was performed by LC with an UV detector. Detection Up to 200 pg was LOD of the LC-UV system. The LOQ of the entire method was <0.01 mg kg−1.*(6)*
2. Qian MR., et al, (2010) was developed a HPLC- tandem mass spectrometry analytical method for determine chlorantraniliprole and flubendiamide residues from vegetables. They used acetonitrile to extract chlorantraniliprole and flubendiamide residues by humanised the mixture with some magnesium Sulphate and sodium chloride at high speed. After that shacking thoroughly and centrifugation of mixture, the upper layer was collected and directly analysed after serial dilution. LOQ for Chlorantraniliprole and flubendiamide was 0.01 and 0.006 mg/kg respectively.*(7)*
3. Qin D., et al. (2010) developed an analytical method of liquid chromatography for determining chlorantraniliprole residue in soil and tomato. Chlorantraniliprole residue was extracted by methanol, then extracted by dichloromethane, and determined by HPLC equipped with a DAD detector. Limit

of detection was 1.0×10^{-7}g and the lowest concentrations detected were 0.005 mg·kg^{-1}In soil and tomato.*(8)*

4. Dugald MM. (2010) reported an additional LC/MS/MS for the analysis of Residues of chlorantraniliprole in diverse crops including citrus (fruit and juice) by liquid chromatography with mass selective detection using a triple quadrupole mass spectrometer (LC/MS/MS). In that Chlorantraniliprole was extracted from the samples in two stages: the first by soaking the samples in water for 20 minutes, followed by the addition of acetonitrile. The first extract was decanted and further acetonitrile was added for a second extraction. The two extractions were combined. An aliquot was taken, evaporated to dryness and redissolved in acetonitrile andwater. The resulting solution was filtered by using a 0.22 µm filter. Quantification was performed through LC-MS/MS. Recoveries for samples of citrus fruit and juice fortified at 0.01–0.1 mg/kg were acceptable. Zhan X., et al (2011) developed HPLC method for detection of Residues of pesticides Chlorantraniliprole and Methoxyfenozide in vegetables like cowpea, greengrocery, cabbage, cucumber and tomato and also in soil. For that they were used acetonitrile for extraction of residues from vegetables and soil samples. Further cleaned up vegetables sample were done with NH2 solid phase extraction and condensed to dry. Detection was done at 230 nm for determination of analytes content. The LOD of drug was 1 ng.*(9)*
5. Balwinder S., et al. (2012) was validated and standardized analytical method for estimation of Chlorantraniliprole residues present in different vegetables (brinjal, cabbage, capsicum, cauliflower, okra and tomato).the residues were extracted using ethyl acetate and then finally reconstituted with HPLC grade acetonitrile, and estimated using HPLC equipped with PDA detector system, C18 column and confirmed by liquid chromatograph mass spectrometer (LCMS/MS), and high performance thin layer chromatograph (HPTLC). Acetonitrile: water (80:20, v/v) of HPLC grade used as mobile phase, flow rate is 0.4mL/min. where retention time was 9.82 min .the limit of quantification of this method was 0.10 mg/kg.*(10)*
6. Abhijit K., et al., (2012) was conducted field trial for study of residues of chlorantraniliprole in cabbage and cauliflower. He was suggested that the washing samples with tap water to removed 17-40 % of the chlorantraniliprole while by boiling removed 100% of chlorantraniliprole residues.*(11)*
7. Farag MM., et al (2012) was developed a novel analytical method for residue analysis of a novel insecticide chlorantraniliprole and its dissipation in grape was studied. Ethyl acetate used for extraction of pesticide and the extract was cleaned up with QuEChERS method and determined by high-performance liquid chromatography with PAD. The LOD and LOQ were found to be 0.02 and 0.06 mgkg^{-1}, respectively.*(12)*
8. Jin MZ., et al, (2012) developed an analytical method for detection of chlorantraniliprole in brown rice, paddy water, rice straw, and soil. The residues of chlorantraniliprole pesticide was extracted with acetonitrile and determined by high performance Liquid chromatography electrospray ionization tandem mass spectrometry (LC–ESI–MS/MS). The limits of detection (LODs) of chlorantraniliprole 0.012 µg L^{-1} for paddy water and 0.15 µg kg^{-1}for soil, brown rice and rice straw.*(13)*
9. California Department of Food and Agriculture, (2013) developed stepwise procedure for chlorantraniliprole analysis in surface water. Methylene chloride was used for extraction of chlorantraniliprole residue from the surface water. The extract was passed through sodium Sulphate to remove residual water. Than analyzed by Liquid Chromatography coupled to a Linear Ion Trap Quadrupole LC/MS/MS).*(14)*

According to review of literature chlorantraniliprole drug is soluble in solvent like Hexane, chloroform, ethyl acetate, methanol, acetone, acetonitrile and water

But practically drug is only soluble in DMF, so suggested methods may not be work in all concentration range.

Proposed method for increasing solubility of chlorantraniprole

1. Drug + methanol
2. Drug + methanol + sonication
3. Drug + ethyl acetate
4. Drug + methanol + Dimethyl formamide
5. Drug + Dimethyl formamide

MATERIALS AND METHODS

Chemicals and Reagent

Chlorantraniliprole was procured from Dupont-Mumbai. Methanol, Acetonitrile, Dimethyl formamide was purchased from Merck laboratories.

Instrumentation

A double beam instrument of Labtronic-LT2900, UV/Visible spectrophotometer was working with wavelength accuracy of ± 0.3 nm and spectral bandwidth of 1 nm with automatic wavelength correction with a pair of 10 mm quartz cells. For weighing of the sample an electronic analytical balance from Shimadzu (BL – 220H) was used.

Preparation of stock solution and Calibration Curve:

100 milligram of chlorantraniliprole was transferred in volumetric flask of 10 ml. Drugs was dissolved in 2 ml of Dimethyl formamide by vigorous shaking and make volume up to mark which gives 10mg/ml concentrated stock solution. from that Take 1ml solution and transfer in another 10 ml volumetric flask and then make up to mark with methanol to obtain 1000µg/ml. Again take 1ml from above solution and transfer in to 10 ml volumetric flask and then make up to mark with methanol to obtain 100µg/ml. for final dilution take 1ml from above solution and transfer in to 10 ml volumetric flask and then make up to mark with methanol to obtain 10µg/ml.

Selection of Analytical Wavelength

For selection of analytical wave length the appropriate diluted stock solution was used and, scanned sin the wavelength region of 300 – 190 nanometre. The obtained absorbance spectra were reprivatized to remove the interference of absorbing species. And from that the suitable wavelength was selected. The examination of the chlorantraniliprole spectra 270 nm was selected as working wavelength.

Analytical Concentration Range Selection:*(15)*

From the 10 µg/ml diluted stock solutions of chlorantraniliprole aliquots series of concentrated solution was pipetted into 10 different 10 ml volumetric flasks to get appropriate drug amount in to final dilution. By makeup volume up to mark with methanol to get the set of 1, 2, 3, 4, 5, 10, 20,30,40,50 µg/ml concentrated solution. At 270 nm wavelength absorbance of these solutions were measured and then absorbance vs concentration graph was plotted. The working curve equation was obtained to be $y = 0.1618x + 0.03$ with an r^2 value of 0.9901.

Study of Validation

Validation study was performed as per guideline. Parameter was evaluated like precision, linearity and recovery. For calculations of recoveries linear regression slope were used. The repeating of the same assay method three times of five replicates in same day gives intraday precision and repeating on consecutive three day of five replicates gives Interday precision.

Accuracy and Precision

For the performance of accuracy study, in known concentration of chlorantraniliprole known amount of same was added. The assessment by intra and inter-day validation were gives Precision of the method. The determination concentrations of chlorantraniliprole in five replicates of different concentration levels obtained by repeating the assay method three times on the same day gives intraday precision and

determination concentrations of chlorantraniliprole in five replicates of different concentration levels obtained by repeating the assay method three times on the consecutive day gives inter day precision.

Calibration curve

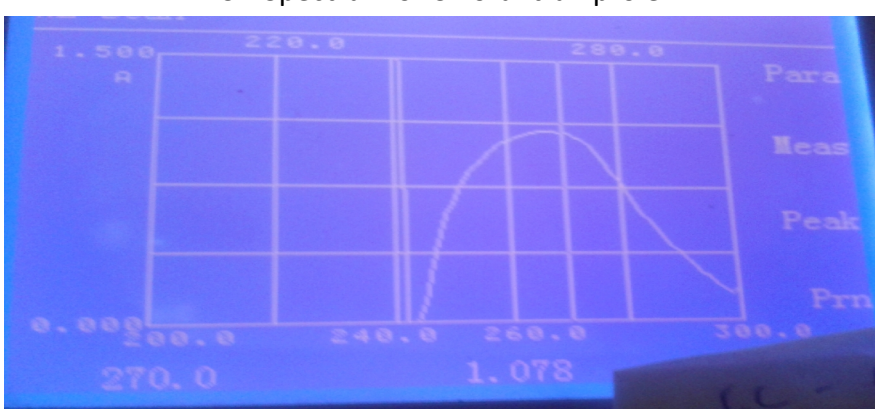
The calibration curves was obtained in the concentration range of 1-5 mg/mL with significant r^2>0.9901. The Limit of Detection (LOD) and Limit of Quantification (LOQ) values were determined as per ICH guideline.

Procedure for Precision:

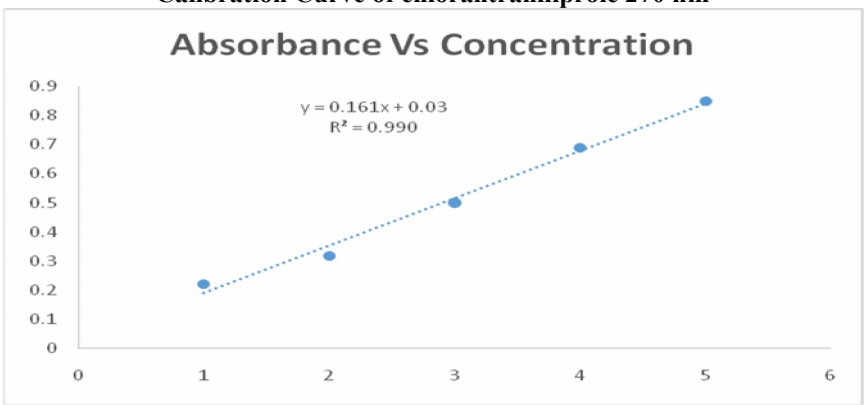
3 µg/ml concentrated chlorantraniliprole sample were scanned for intra and inter-day precision six times at different time interval in the same day and different day respectively

RESULTS AND DISCUSSION

UV Spectrum of Chlorantraniprole



Calibration Curve of chlorantraniliprole 270 nm



Standard Calibration Curve Data for chlorantraniliprole at 270nm

Sr. No.	Concentration (µg /ml)	Absorbance at 270 nm
1	1	0.223
2	2	0.318
3	3	0.501
4	4	0.688
5	5	0.847

Regression and Optical Characteristics of chlorantraniliprole

Parameter	Chlorantraniliprole
Working λ	270
Beer's Law range	1 - 5 µg/ml
Regression Parameter	
i. Slope of curve	0.0042
ii. Intercept of curve	0.003
iii. Regression coefficient (r^2)	0.990

Data for LOD and LOQ of chlorantraniliprole

Drug	Limit Of Detection (µg/ml)	Limit Of Quantitation (µg/ml)
Chlorantraniprole	0.0580	0.150

Statistical Validation for Recovery Studies

Level of % Recovery	% Mean Recovery*	Standard Deviation*	Co-efficient of Variation* (% R.S.D.)	S.E.*

80	98.15	0.893	0.867	0.456
100	99.46	0.765	0.766	0.481
120	99.22	0.548	0.561	0.254

Intra – day Precision

Drug	% Mean*	S.D*	% R.S.D.*	S.E.*
Chlorantraniprole	99.08	0.767	0.767	0.326

Inter – day precision

Drug	% Mean*	S.D.*	% R.S.D.*	S.E.*
Chlorantraniprole	98.25	0.560	0.560	0.356

*** n=6**

DISCUSSION

Chlorantraniprole has less solubility in methanol, ethanol Isopropyl alcohol acetone, acetonitrile, ether, water and other most suitable solvent while good solubility in Dimethyformamide and dimethyl suphoxide, so we have develop method for estimation of chlorantraniprole using combination of solvent DMF and methanol. As per our experiment, Chlorantraniprole dissolved in DMF than further 80 % volume made by methanol in first dilution and rest of all dilution was made by methanol. Any changing in order or using other solvents were not gave appropriate result. Finally the develop method has good precision, sensitivity, accuracy and reproducibility.

CONCLUSION

The method was successfully used to estimate the amount of chlorantraniprole in bulk and formulation. This method can be applicable for detection of low amount of chlorantraniprole in bulk and formulation. As compare to previous literature method, we have overcome of issue regarding solubility of chlorantraniprole and develop methods was produced significant validation parameter.

FIGURES

Figure 1: UV Spectrum of Chlorantraniprole

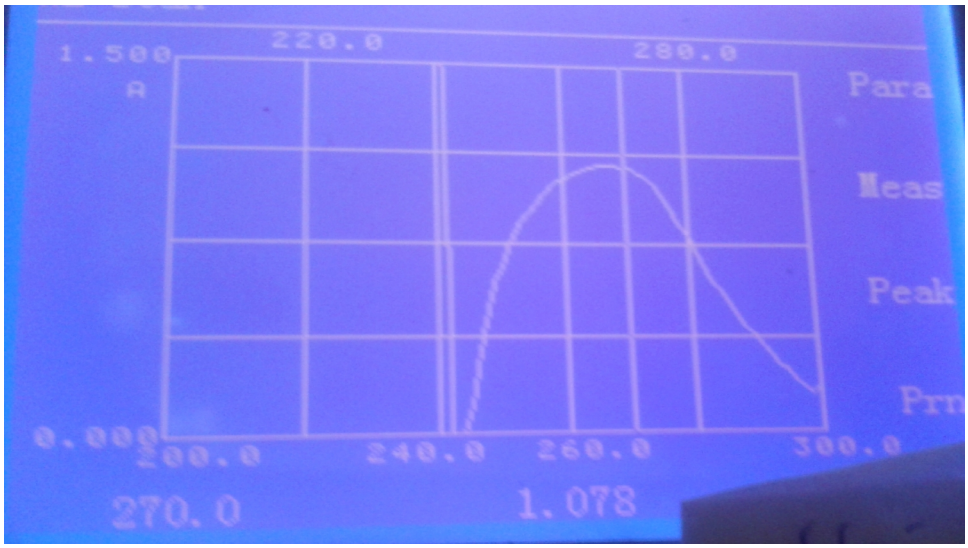
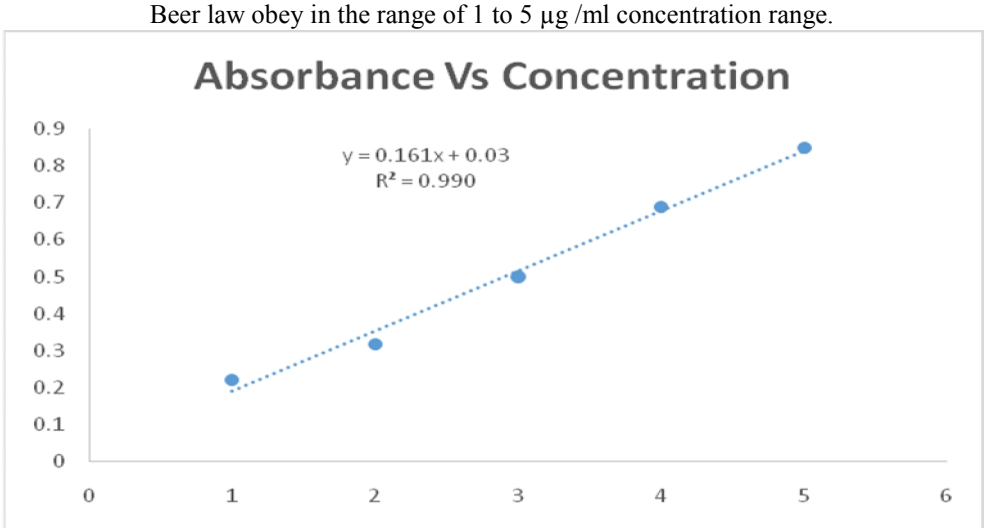


Figure 2: Calibration Curve of chlorantraniliprole 270 nm
Beer law obey in the range of 1 to 5 µg /ml concentration range.



TABLES

Table 1: Standard Calibration Curve Data for chlorantraniliprole at 270nm

Sr. No.	Concentration (µg /ml)	Absorbance at 270 nm
1	1	0.223
2	2	0.318
3	3	0.501

4	4	0.688
5	5	0.847

Table 2: Regression and Optical Characteristics of chlorantraniliprole

Parameter	Chlorantraniliprole
Working λ	270
Beer's Law range	1 - 5 µg/ml
Regression Parameter	
i. Slope of curve	0.0042
ii. Intercept of curve	0.003
iii. Regression coefficient (r^2)	0.990

Table 3: Data for LOD and LOQ of chlorantraniliprole

Drug	Limit Of Detection (µg/ml)	Limit Of Quantitation (µg/ml)
Chlorantraniprole	0.0580	0.150

Table 4. Statistical Validation for Recovery Studies

Level of % Recovery	% Mean Recovery*	Standard Deviation*	Co-efficient of Variation* (% R.S.D.)	S.E.*
80	98.15	0.893	0.867	0.456
100	99.46	0.765	0.766	0.481
120	99.22	0.548	0.561	0.254

Table 5: Intra – day Precision

Drug	% Mean*	S.D*	% R.S.D.*	S.E.*
Chlorantraniprole	99.08	0.767	0.767	0.326

Table 6: Inter – day precision

Drug	% Mean*	S.D.*	% R.S.D.*	S.E.*

Chlorantraniprole	98.25	0.560	0.560	0.356

*** n=6**

ACKNOWLEDGEMENT

Author would like to thanks Dupont-Mumbaifor providing gift sample of Chlorantraniliprole pesticide and also express sincere thanks to ShamjibhaiHarjibhaiTalaviya Charitable Trust and Management of RK University for providing us all the necessary facilities for carrying out our research work.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

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RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

Formulation and Evaluation of Colon Targeted Osmotic Tablets of Albuterol Sulphate for Nocturnal Asthma

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ABSTRACT

Time and pH dependent colon targeted osmotic tablets of Albuterol Sulphate were formulated for nocturnal asthma. From the experimental work, it was concluded that 3 % and 7 % of Eudragit S-100 could not produce tablets with 5 hrs lag time because Eudragit S-100 (3 %) coating gave release in intestine after 3 hrs while Eudragit S-100 (7 %) coating gave release after 7-8 hrs. According to diseased condition, the formulation should give lag time of 5 hrs, so when the patient take tablet at around 10-10:30 pm, the drug should give release at 3:00 am - 3.30 am where chances of asthmatic attack are higher. Here the formulation C1-C3 with 5 % coating showed lag time of 5 hrs which was best suited to prevent nocturnal asthma.

SUMMARY

Albuterol Sulphate colon targeted osmotic tablets were formulated & evaluated for Noctural Asthmatic patients.

Keywords: Osmotic Tablet, Colon Targeted Tablet, Nocturnal Asthma

INTRODUCTION

Oral drug delivery system has been known for decades among all the routes that have been discovered for the systemic delivery of drugs via various pharmaceutical products of differentdosage forms. Various controlled drug delivery system like microspheres, liposomes, nanoparticles etc. Among these, osmotic tablet for oral administration offers several advantages, such as ease of administration and improved patient compliance. Colon is a reliable site for those drugs where a delay in drug absorption is required from therapeutic point of view e.g. nocturnal asthma, cardiac arrhythmias, arthritis which are affected by circadian biorhythms (*1,2*).

Circadian rhythms are self-sustaining, endogenous oscillations that occur with a period of about 24 hours. Diseases, such as cardiovascular, asthma, peptic ulcer, arthritis, etc follow the body's circadian rhythm (*3*). There are different approaches to delivered drug to colon like Coating with pH Sensitive Polymers, Time Release System, Bio adhesive System, pulsing cap system, Coating with Micro particles, Osmotic Controlled Drug Delivery, etc.

Osmotic drug delivery systems utilize osmotic pressure as energy source and driving force for delivery of drugs. Osmotic tablet consists of a core including the drug and osmotic agent, other excipients and semi permeable membrane coat (*4*).

MATERIALS AND METHODS

Albuterol sulphate obtained as a gift sample from the Jayco Chemical Industries. Mannitol , lactose, PEG-4000, sodium CMC, sodium alginate, HPMC, cellulose acetate, Eudrgit s 100 are obtained from S.D. Fine Chem. Ltd, Mumbai. All ingredients are of analytical grade.

Methods of Prepration

I.Calibration Curve of Albuterol Sulphate

Stock solution of pure drug was prepared by dissolving accurately weighed drug in solution of 0.1N HCl pH 1.2, phosphate buffer pH 6.8 and phosphate buffer pH-7.4. Suitable dilution ware prepared and absorbance was measured in UV- visible spectroscopy at 276 nm wavelength.

1. Preparation of Calibration Curve in 0.1N HCl and Phosphate Buffer pH6.8

100 mg Albuterol powder was accurately weighed and transferred in to 1000 ml volumetric flask and dissolved in 0.1N HCl (1.2 pH) and 6.8 phosphate buffer. That solution (100µg/ml) was further diluted by taking 2 ml, 4 ml, 6 ml, 8 ml, 10 ml, 15 ml, 20 ml, 25 ml, 30 ml of the stock solution and the volume was made up to 100 ml which gave the solutions concentrations, 2 µg/ml, 4 µg/ml, 6 µg/ml, 8 µg/ml, 10 µg/ml, 15 µg/ml , 20 µg/ml, 25 µg/ml, 30 µg/ml. The standard calibration curve was prepared by plotting the graph of absorbance vs. concentration at 276 nm.

2. Preparation of Calibration Curve in Phosphate Buffer pH 7.4

100 mg Albuterol powder and transferred in to 1000 volumetric flask and dissolved in 7.4 phosphate buffer. That solution (100µg/ml) was further diluted by taking 2 ml, 4 ml, 6 ml, 8 ml, 10 ml, 12 ml, 14 ml, 16 ml, 18 ml, 20 ml of the stock solution and the volume was made upto 100 ml which gave the solutions of concentrations, 2 µg/ml, 4 µg/ml, 6 µg/ml, 8 µg/ml, 10 µg/ml, 12 µg /ml, 14 µg/ml, 16 µg/ml, 18 µg/ml, 20 µg/ml. The standard calibration was prepared by plotting the graph of absorbance vs. concentration at 276 nm.

II. Osmotic tablet are prepare by two different approach

1. Press coated tablet

a. Formulation of core tablet

Fig. 1. Formulation of core tablet
Table 1. Formula of core tablets

b. Coating of Tablet (Semi-Permeable Coating)
Fig. 2. Semi-Permeable Coating

c. Press coating
Fig. 3. Press coating
Table 2. Formulation of press coated tablet

2. Dip coated tablet
Fig. 4. Dip coating
Table 3. Formulation of dip coated tablet

III. Evaluation Parameter (*5*)

1. Press coated

a. In vitro dissolution study

2. Dip coated tablet

a. Tablet Thickness.
b. Tablet Hardness
c. Friability
d. In Vitro dissolution study
e. Weight uniformity
f. Drug content

RESULTS AND DISCUSSION

I. Calibration curve of Albuterol sulphate

Stock solution of pure drug was prepared by dissolving accurately weighed drug in solution of 0.1N HCL pH 1.2, phosphate buffer having pH 6.8, phosphate buffer having pH 7.4. Suitable dilution was prepared and absorbance was measured in UV-visible spectroscopy at 276 nm wavelength.

Table 4. Absorbance of Standard Solution of Albuterol Sulphate in0.1NHCl
Fig. 5. Standard calibration plot of Albuterol sulphate in 0.1N HCl
Table 5. Absorbance of Standard Solution of Albuterol Sulphate in pH 6.8
Fig. 6. Standard calibration curve of Albuterol sulphate in phosphate buffer pH 6.8
Table 6. Absorbance of Standard Solution of Albuterol Sulphate in pH 7.4
Fig. 7. Standard calibration curve of Albuterol sulphate in phosphate buffer pH 7

***II. In vitro* Dissolution study**

Table 7. *In-vitro* dissolution of press coated tablets [F1 – F6]
Fig. 8. *In vitro* dissolution of press coated tablets
Table 8. *In-Vitro* Dissolution of Dip Coated Tablets [F7 –F15]
Fig. 9. *In vitro* dissolution of dip coated tablets

III. Tablet thickness

Table 9. Thickness of Core Tablets
Table 10. Thickness of dip coated tablets

IV. Tablet hardness

Table 11. Hardness of core tablets
Table 12. Hardness of dip coated tablets

V. Tablet friability

Table 13. %Friability of core tablets

Table 14. %Friability of dip coated tablets

VI. Weight variation

Table 15. Weight of core tablets

Table 16. Weight of dip coated tablets

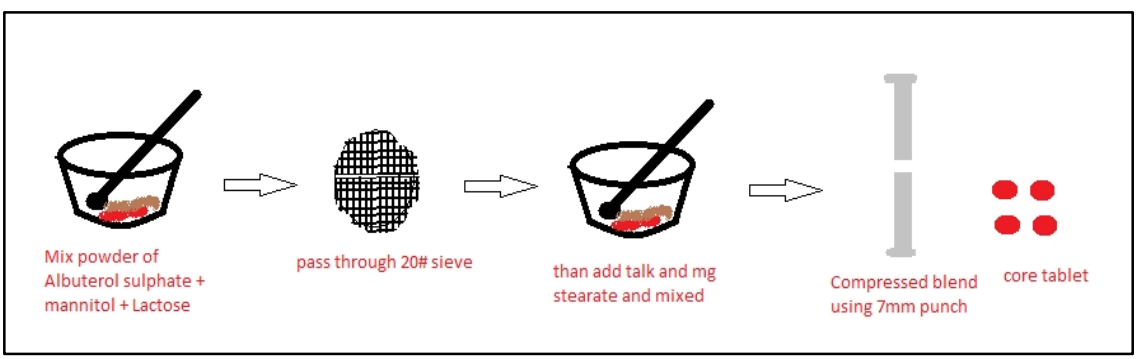
VII. Drug content

Table 17. Drug Content of core tablets

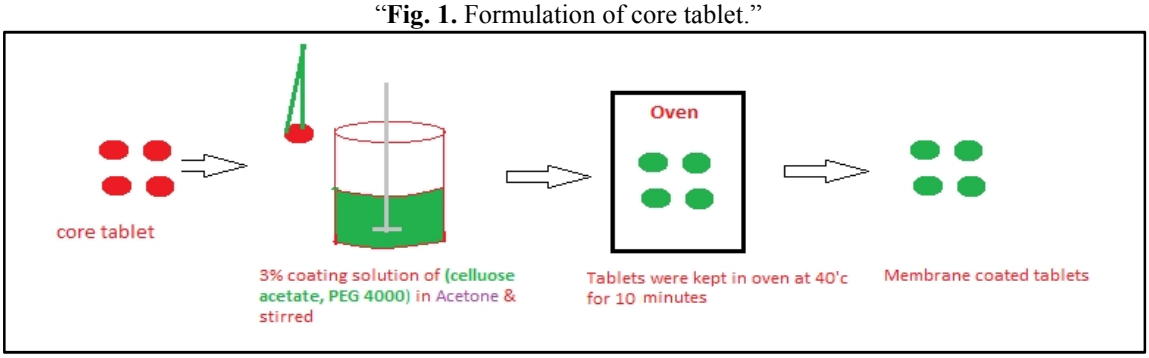
CONCLUSION

From the above research outcomes it can be concluded that batch F7 (Eudragit S-100) can be used to protect the drug in the hostile environment of upper GIT. The *in vitro* studies showed that when Albuterol Sulphate was administered as Enteric coated tablet, the tablet remained intact in the stomach and small intestine and it successfully delivered the maximum amount of drug to the colon. This Enteric Coated Osmotic Tablets showed excellent zero order drug release. Enteric Coated F10 – F12 (5 % Eudragit S-100) Osmotic Tablets of Albuterol Sulphate targeted to colon modulate the drug level in synchrony with the circadian rhythm of nocturnal asthma. Osmotic tablet dosage form, taken at bed time with a programmed start of drug release early in morning hours can prevent sharp increase in the incidence of asthmatic attacks during the early morning hours (nocturnal asthma), a time when the risk of asthmatic attack is most prominent

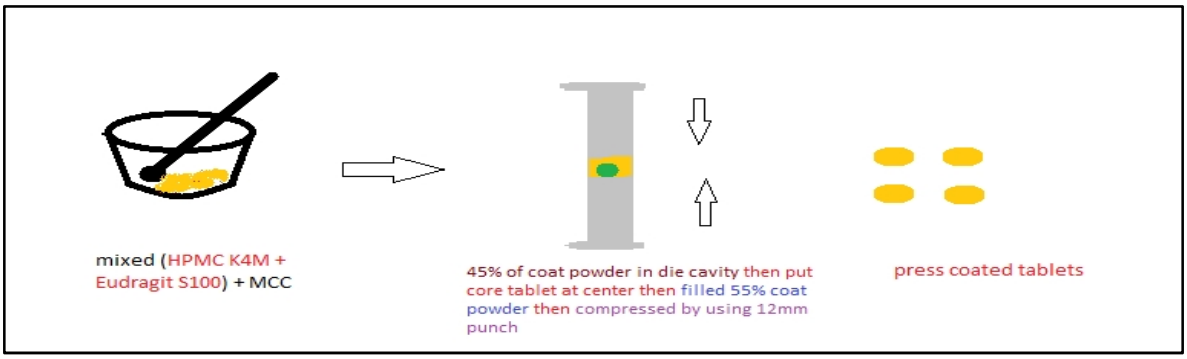
FIGURES



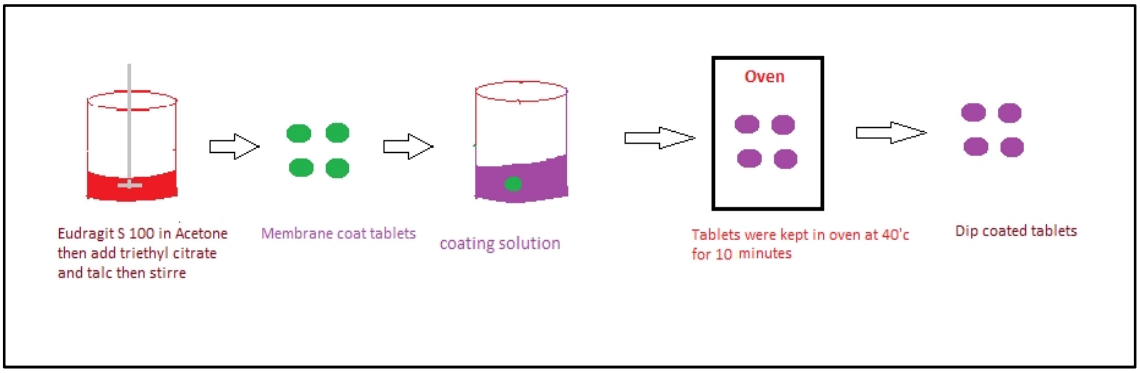
"**Fig. 1.** Formulation of core tablet."



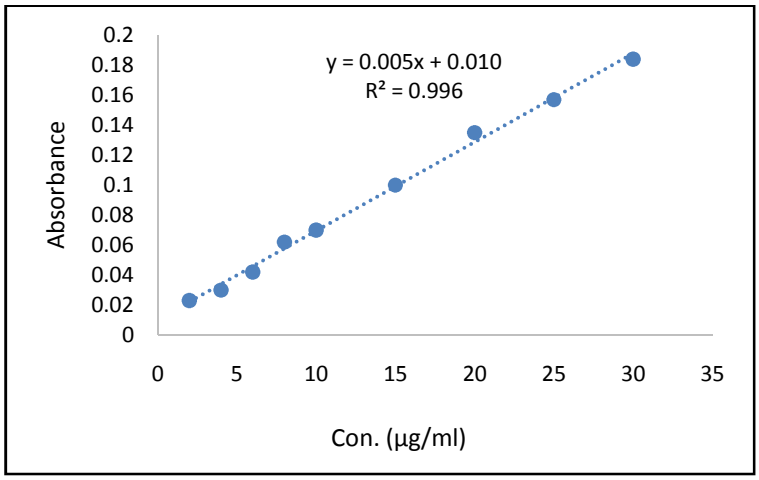
"**Fig. 2.**Semi-Permeable Coating."



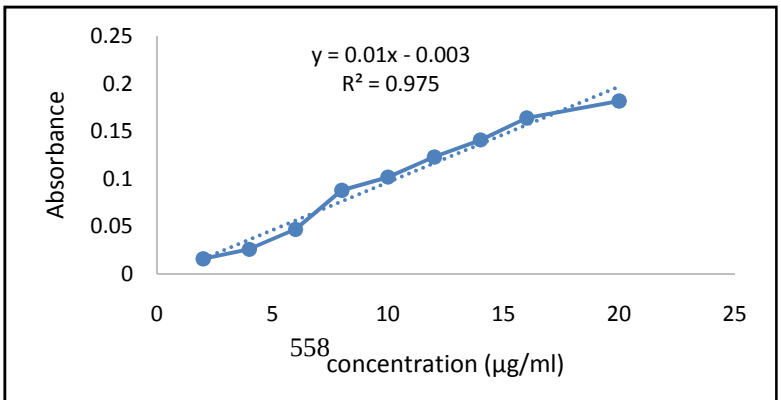
"Fig. 3. Press coating**"**



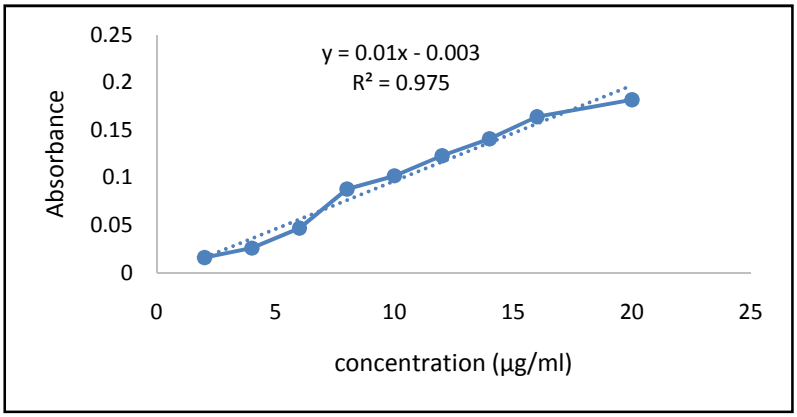
"Fig. 4. Dip coating"



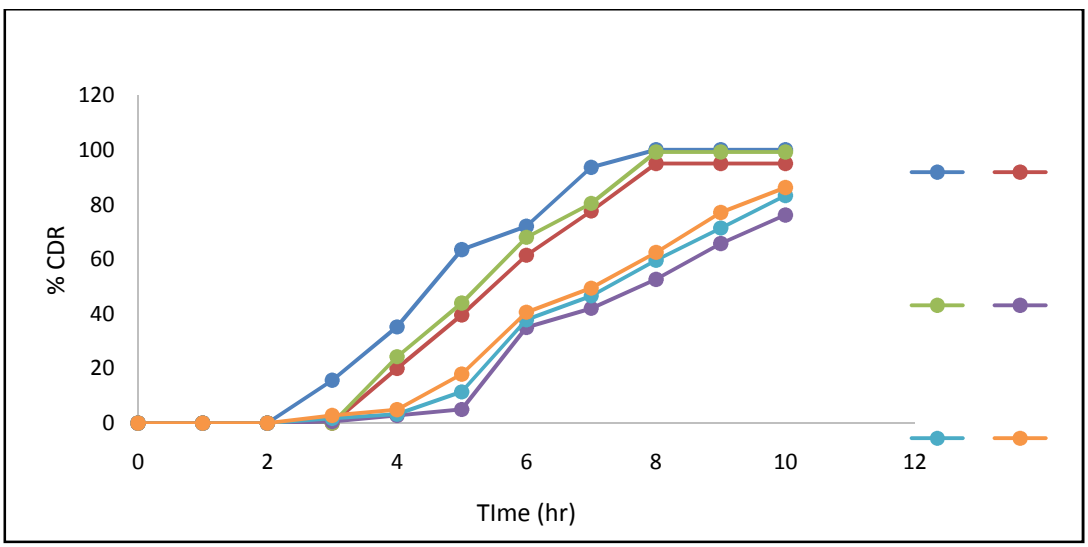
"Fig. 5. Standard calibration plot of Albuterol sulphate in 0.1N HCl**"**



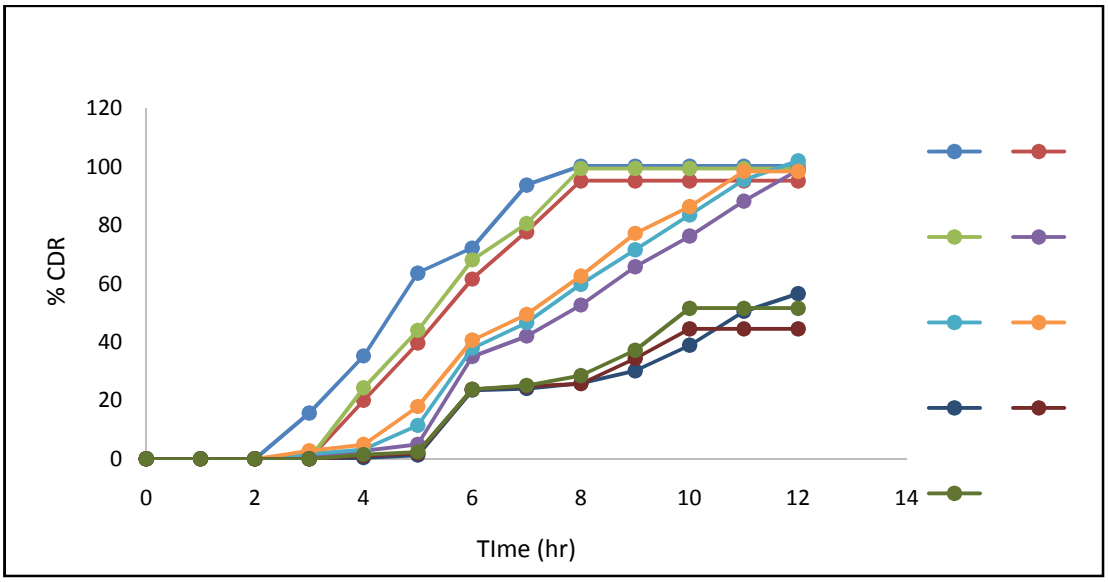
"**Fig. 6.** Standard calibration curve of Albuterol sulphate in phosphate buffer pH 6."



"Fig. 7. Standard calibration curve of Albuterol sulphate in phosphate buffer pH 7**"**



"**Fig. 8.***In vitro* dissolution of press coated tablets"



"**Fig. 9.** *In vitro* dissolution of dip coated tablets"

TABLES

"**Table 1.** Formula of core tablets"

Ingredients	C1(mg)	C2(mg)	C3(mg)
Albuterol sulphate	8	8	8
Mannitol	10	15	20
Lactose	90	85	80
Talc	1	1	1
Mg. stearate	1	1	1
Total weight	**110**	**110**	**110**

"**Table 2.** Formulation of press coated tablet"

Ingredients	F1	F2	F3	F4	F5	F6
Core tablet (mg)						
Albuterol sulphate	8	8	8	8	8	8
Mannitol	10	15	20	10	15	20
Lactose	90	85	80	90	85	80
Talc	1	1	1	1	1	1

Mg. stearate	1	1	1	1	1	1
Coating by semipermeable membrane (ml)						
Cellulose Acetate: PEG-4000	75:25	75:25	75:25	75:25	75:25	75:25
Press coat (mg)						
Eudragit : HPMC K4M (6:4)	120:80	120:80	120:80	-	-	-
Eudragit : HPMC K4M (7:3)	-	-	-	140:60	140:60	140:60
MCC	19.5	19.5	19.5	19.5	19.5	19.5
Mg stearate	7	7	7	7	7	7
Talc	3.5	3.5	3.5	3.5	3.5	3.5

"**Table 3.** Formulation of dip coated tablet"

Ingredients	**F7**	**F8**	**F9**	**F10**	**F11**	**F12**	**F13**	**F14**	**F15**
Core tablet (mg)									
Albuterol sulphate	8	8	8	8	8	8	8	8	8
Mannitol	10	15	20	10	15	20	10	15	20
Lactose	90	85	80	90	85	80	90	85	80
Talc	1	1	1	1	1	1	1	1	1
Mg stearate	1	1	1	1	1	1	1	1	1
Coating by semipermeable membrane (ml)									
Cellulose Acetate: PEG-4000	75:25	75:25	75:25	75:25	75:25	75:25	75:25	75:25	75:25
Dip coating (%)									
Eudragit s100	3	3	3	5	5	5	7	7	7

"**Table 4.** Absorbance of Standard Solution of Albuterol Sulphate in0.1NHCl"

Sr.no.	Concentration	Absorbance*
1	2	0.023±0.0005
2	4	0.030±0.0010
3	6	0.042±0.0005
4	8	0.062±0.0005
5	10	0.070±0.0000
6	15	0.100±0.0050
7	20	0.1350±0.005
8	25	0.1570±0.005
9	30	0.184±0.005

***The values represent mean ± S.D.

"**Table 5.** Absorbance of Standard Solution of Albuterol Sulphate in pH 6.8"

Sr.No.	Concentration (µg/ml)	Absorbance*
1	2	0.020±0.002
2	4	0.031±0.002
3	6	0.044±0.002
4	8	0.056±0.002
5	10	0.065±0.002
6	15	0.089±0.002
7	20	0.123±0.003
8	25	0.153±0.010
9	30	0.186±0.006

*The values represent mean ± S.D.

"**Table 6.** Absorbance of Standard Solution of Albuterol Sulphate in pH 7.4"

Sr.No.	Concentration (µg/ml)	Absorbance*
1	2	0.016±0.0010
2	4	0.026±0.0005
3	6	0.047±0.0005
4	8	0.088±0.0010
5	10	0.102±0.0010

6	12	0.123±0.0005
7	14	0.141±0.0005
8	16	0.164±0.010
9	20	0.182± 0.000

"**Table 7.** *In-vitro* dissolution of press coated tablets [F1 – F6]"

Time	**% cumulative Albuterol sulphate release (CDR)**					
(hr)	**F1**	**F2**	**F3**	**F4**	**F5**	**F6**
1	0	0	0	0	0	0
2	0	0	0	0	0	0
3	0	0	0	0	0	0
4	0	0	0	0	0	0
5	0	0	0	0	0	0
6	20.76	20.09	0	20.34	20.62	20.76
7	21.8	22.09	21.04	21.34	21.52	21.80
8	23.28	23.14	22.37	22.29	22.71	23.42
9	25.89	28.7	23.57	28.68	28.69	31.50
10	48.49	45.81	32.97	40.18	42.99	47.24
11	60.28	58.88	50.63	51.79	54.63	58.92

"Table 8.*In-Vitro* Dissolution of Dip Coated Tablets [F7 –F15]**"**

Time	**%Cumulative drug release (CDR)**								
(hr)	**F7**	**F8**	**F9**	**F10**	**F11**	**F12**	**F13**	**F14**	**F15**
0	0	0	0	0	0	0	0	0	0
1	0	0	0	0	0	0	0	0	0
2	0	0	0	0	0	0	0	0	0
3	15.73	0	0	0.64	1.72	2.80	O	0	0
4	35.28	20.04	24.35	2.8	3.25	4.98	0.43	1.07	1.50
5	63.65	39.64	43.99	4.99	11.47	17.96	1.29	1.95	2.38
6	72.22	61.58	68.13	35.06	37.87	40.68	23.56	23.84	23.84
7	93.77	77.77	80.55	42.06	46.66	49.5	24.08	24.92	25.20

8	100.26	95.22	99.41	52.66	59.75	62.61	25.90	25.73	28.53
9	-	-	-	65.80	71.55	77.25	30.17	34.39	37.23
10	-	-	-	76.26	83.47	86.41	38.88	44.55	51.62
11	-	-	-	88.22	95.5	98.47	50.47	-	-
12	-	-	-	98.88	102.02	-	56.57	-	-

"**Table 9.** Thickness of Core Tablets**"**

Formulation	Thickness*(mm)
C1	1.89±0.01
C2	1.87±0.02
C3	1.88±0.02

*The values represent mean ± S.D

"**Table 10.** Thickness of dip coated tablets"

Formulation	Thickness*(mm)
F10	1.71±0.44
F11	1.98±0.01
F12	1.96±0.01

*The values represent mean ± S.D.

"**Table 11.** Hardness of core tablets"

Formulation	Hardness*(Kg/Cm^2)
C1	3.16±0.25
C2	2.96±0.05
C3	3.26±0.11

*The values represent mean ± S.D

"**Table 12.** Hardness of dip coated tablets"

Formulation	Hardness*(Kg/Cm^2)
F10	4.26±0.05
F11	4.13±0.05
F12	3.93±0.05

*The values represent mean ± S.D.

"**Table 13.** %Friability of core tablets"

Formulation	% Friability*
C1	0.97±0.05
C2	0.71±04
C3	0.65±0.16

*The values represent mean ± S.D

"**Table 14.** %Friability of dip coated tablets**"**

Formulation	% Friability
F10	0.64±0.22
F11	0.73±0.25
F12	0.67±0.22

*The values represent mean ± S.D.

"**Table 15.** Weight of core tablets"

Formulation	Avg. weight (mg)
C1	112
C2	112.5
C3	111.5

"**Table 16.** Weight of dip coated tablets"

Formulation	Avg. weight (mg)
F10	119.5
F11	119
F12	121.5

"**Table 17.** Drug Content of core tablets"

Formulation	%Drug content
C1	97.01± 1.49
C2	96.51± 2.28
C3	96.51± 0.86

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th& Jan. 6th, 2016)

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Simultaneous Determination of Sertaconazole Nitrate and BeclomethasoneDipropionate in cream by Reverse-Phase HPLC method

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ABSTRACT

RP-HPLC method was developed, optimized and validated for simultaneous determination of BeclomethasoneDipropionate (BD) and Sertaconazole Nitrate (SN) in Cream. Method was found as simple, specific, precise, rapid, and reproducible. Isocratic chromatographic separation was carried out on an YMC Pack Pro ODS (C-18) Column (250 × 4.60 mm) 5 µm particle size at column temperature 45°C ± 0.5°C. The composition of mobile phase used for separation was Acetonitrile–Phosphate buffer (70:30 v/v) at a flow rate of 1.5 ml/min. Using UV-PDA detector at 240 nm detection was carried out. Validation Parameters like linearity, specificity, precision, recovery study, and ruggedness were performed as specified in ICH guidelines. The retention times for BD and SN were 2.80 ± 0.02 min and 4.82 ± 0.05 min, respectively. The linearity range and % recoveries for BD and SN were 1–6 µg/ml, 80–480 µg/ml and 97.47-98.38%, 97.15-100.22% respectively. The correlation coefficients for BD and SN were 0.9917 and 0.9987 respectively and also relative standard deviations were found satisfactory.

SUMMARY

Developed RP-HPLC method is rapid, specific, accurate and precise so it can be used in routine analysis.

Keywords: BeclomethasoneDipropionate, Sertaconazole Nitrate, Simultaneous estimation, RP-HPLC.

INTRODUCTION

BD is a synthetic halogenated glucocorticoid with anti-inflammatory and vasoconstrictive effects and also being investigated for mild-to-moderate Crohn's disease (oral treatment). BeclomethasoneDipropionate (BD) (9a-chloro-11b–hydroxy-16b–methyl-3, 20-dioxopregna 1, 4-diene-17, 21-diyldipropionate) as shown in Figure 1.

Sertaconazole Nitrate is (RS)-1-[2-[(7-chloro-1-benzothiophen-3-yl) methoxy]-2-(2, 4-dichlorophenyl) ethyl]-1H-imidazole nitrate, structure as shown in Figure 2. It is indicated for treatment of superficial skin mycoses and also has broad-spectrum antifungal activity [1-6].Cream containing 0.025% BeclomethasoneDipropoionate and 2% Sertaconazole Nitrate is available in market. BD has been reported to be determined by HPLC from formulation. Determination of SN alone or in combination with other drugs has been reported by HPLC [7], spectrofluorometric [8] and capillary zone electrophoresis [9] methods in pharmaceutical dosage form. Estimation of BD alone or in combination has been reported by spectrophotometry [10-12], RP-HPLC [13-15] and stability indicating RP-HPLC [16] methods in Marketed formulation. So far estimation of BD and SN was not reported. Here in present study developed method was validated according to ICH guidelines. [17]

MATERIALS AND METHODS

Instrumentation

The Agilent Technologies LC system used was equipped with a 1260 Quaternary Pump VL and automated injector system. Detection was accomplished using photodiode array (DAD) detector. It was connected to software for controlling instrumentation and also for processing data generated. BD and SN were obtained as working standards from Advance analytical research and training laboratory, Ahmedabad by means of a gift sample. Merck Ltd supplied HPLC grade: ACN, MeOH India. Cream Onabet-B of Glenmark Pharmaceutical Limited procured from a local market containing BD and SN in ratio of 1:80 respectively.

Chromatographic condition

Optimizes chromatographic experimental conditions are as follows

Sr. No	Parameters	Chromatographic condition
1	Mode of elution	Isocratic
2	Mobile Phase	ACN: 0.01 M $NaH2PO_4$ (70: 30)
3	Column	YMC®-Pack pro C-18, (250mm×4.6mm), 5µm
4	Flow rate	1.5 ml/min.
5	Temperature	45° C
6	Run time	10 min
7	Injection volume	20 µl
8	Detection wavelength	240 nm (UV–DAD)

Standard preparation

BD and SN standard API were appropriately weighed and dissolved in Acetonitrile (1000 µg/ml of BD and SN each). Working standard solutions were prepared to have 1–5 µg/ml for BD and 80–480 µg/ml for SN.

Sample preparation

Transfer 1.6 g cream in to 25 ml beaker. 10 ml of Acetonitrile was added, dispersed by spatula and transferred to 20 ml flask. Beaker was washed with ACN (triplicate) then washings were added to flask.

The flask was sonicated for 30 min; meanwhile every 5 min interval flask was vigorously shaken. After solubilization required amount of Acetonitrile was added. Then, aliquot was taken and kept aside for 10 min, 1 ml of supernatant was transferred to a 10 ml VF and volume was make up with Acetonitrile. An aliquot of this latter solution was filtered and transferred to a vial. 20µL were injected in to column.

RESULTS AND DISCUSSION

Chromatography

According Literature review, Initially Acetonitrile: Buffer was tried for reversed Phase LC separation of BD & SN both the compound was separated but tailing of Beclomethasone appear. Less polar part of mobile phase was also investigated to optimize separation of BD and SN and 70:30 Acetonitrile: Buffer was optimized for desirable peak. To improve separation, pH of mobile phase becomes an important factor. pH effect were also checked by adjusting acidic & basic pH but at higher pH both the compound were eluted faster with poor signal-to-noise ratio and at lower pH compounds were eluted at higher retention time more than 10. However response of low concentration drug (BD) was higher and also response of high concentration drug (SN) were good at 240 nm hence it was selected as wavelength for simultaneous determination of both the compound, as shown in Figure 3. Thereafter, Acetonitrile–Phosphate buffer in ratio of 70:30 v/v at 1.5 ml/min flow rate and 45^{o}C column temperature was found optimum as shown in Figure 4.

SYSTEM SUITABILITY PARAMETERS

The results obtained are satisfactorily, shown in Table 1.

LINEARITY

BD presented linearity in range of 1–5µg/ml,and for SN 80–480 µg/ml.

Linearity equation for BD and SN are as follows.

Y (Area) =53972X (Conc.) + 2272.6 (R^2 = 0.9917)

Y (Area) = 29193X (Conc.) + 273733 (R^2 = 0.9987).

Linearity data are summarized in Table-2 and Calibration Curve for BD and SN are shown in Figure 5 and Figure 6 respectively.

LIMIT OF DETECTION (LOD) AND LIMIT OF QUANTIFICATION (LOQ)

It can be calculated using following equation;

$$LOD = 3.3 \times \sigma/s \quad LOQ = 10 \times \sigma/s$$

Where, σ = standard deviation of response
s = mean slope of the calibration curve

LOD and LOQ data are summarized in Table-3

ACCURACY

The accuracy of proposed method were evaluated by analyzing three synthetic mixtures containing pre-analyzed sample solution spiked with (50 %, 100 %, 150 % of target concentration, where **(n=3)**). These results of recovery study of BD and SN are summarized in Table 4 and Table 5 respectively.

PRECISION

Repeatability

Repeatability studies were carried out by six replicate injections of standard preparation in instrument. Results fall within acceptable limits (RSD < 2), as shown in Table 6.

Intermediate precision

Between days precision (Inter-day precision) and within day precision (Intraday precision) by the proposed method are presented in Table 7 and Table 8. All results fall within acceptable limits (RSD < 2), as shown in Table 7 and Table 8.

ROBUSTNESS

According to ICH, deliberate discrepancies were done to check method's ability to remain unaffected (method stability), by varying composition of mobile phase & flow rate. The upsurge of implementation of Quality by Design (QbD) has put emphasis on role of robustness study in analytical method development. Alteration was done in ratio of mobile phase: instead of ACN–Phosphate buffer (70:30 v/v); ACN–Phosphate buffer (68:32 v/v & 72:28 v/v) was used as mobile phase. The change was also made in flow rate: instead of 1.5 ml/min, 1.3 ml/min &1.7ml/min were set as flow rate. Results of analysis were show % RSD, acceptable as less than 2. It proves the robust nature of method.

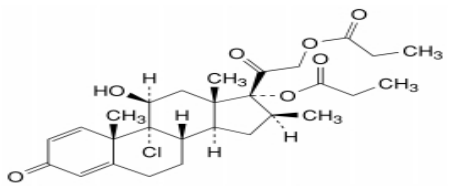
Assay of Marketed Formulation

% Assay of BD and SN in cream by proposed method was found acceptable as shown in Table 9.

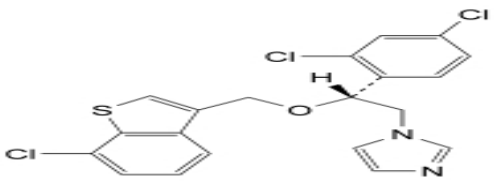
CONCLUSION

The current work helps in meeting Day to day analytical requirement for Pharmaceuticals dealing with cream containing combination of BD and SN in ratio (1:80). The developed extraction procedure completely extracts the desired components. The run time being less than 10 min, will significantly reduce the total time required for Batch analysis, and hence more number of samples could be processed, and instrument utility could be improved. Thus, developed method can be employed for routine quality control of cream containing these two drugs in industries.

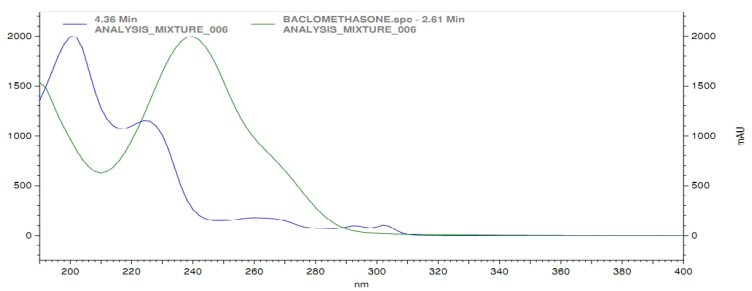
FIGURES



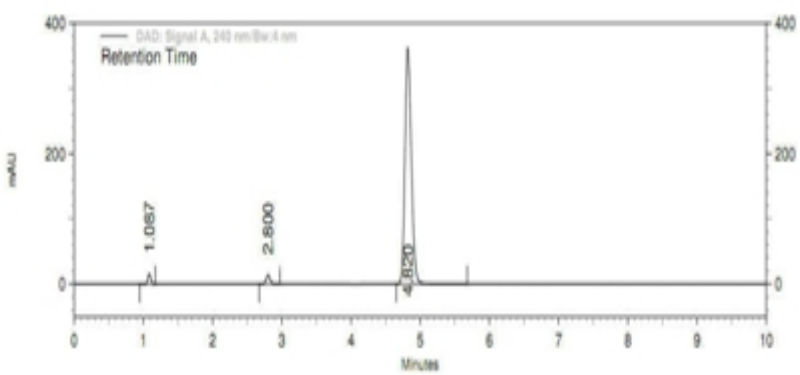
"Fig. 1: Structure of BeclomethasoneDipropionate**"**



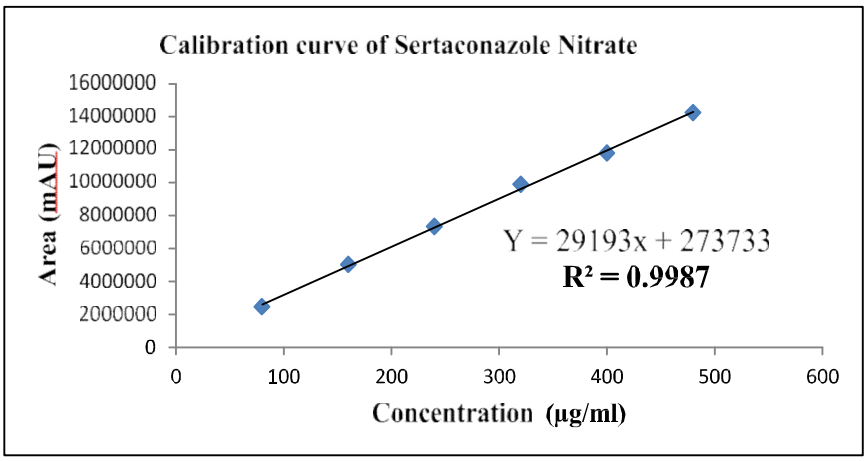
"Fig. 2: Structure of Sertaconazole Nitrate**"**



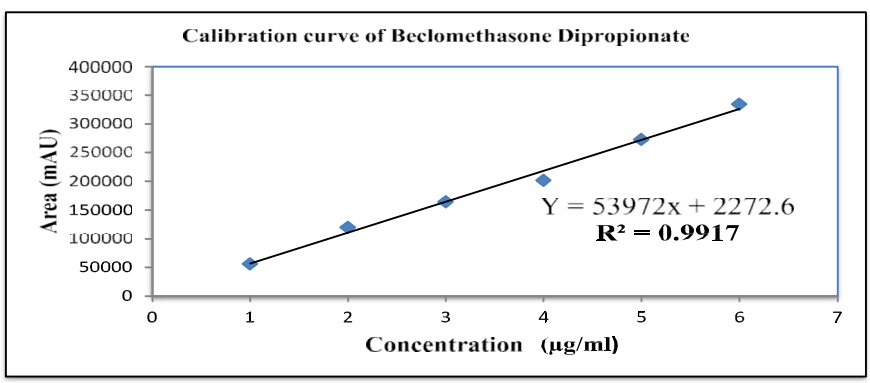
"Fig. 3: Wavelength Selection of BD and SN**"**



"Fig.4: Optimized Chromatogram**"**



"Fig.5: Calibration Curve of BD**"**



"Fig.6: Calibration Curve of SN**"**

TABLES

"Table 1: System Suitability Parameters**"**

Parameters	Developed Method		Standard Values
	BD	**SN**	
Retention times (t_R)	2.80 ± 0.02	4.82 ± 0.05	Up to 10 %
Theoretical plates (N)	4878.3± 60.76	6312.67 ± 61.93	Greater than 2000
Resolution (R_S)	10.45 ± 0.06		Greater than 2
Tailing factor	1.57 ± 0.013	1.79 ± 0.012	Not more than 2.0

"Table 2: Linearity Study**"**

BeclomethasoneDipropionate			**Sertaconazole Nitrate**		
Conc. (µg/ml)	**Mean Area ± S.D. (n=6)**	**% RSD**	**Conc. (µg/ml)**	**Mean Area ± S.D. (n=6)**	**% RSD**
1	55883.83±66.2	0.12	80	2452284 ±6166.6	0.25
2	119361.5±138.3	0.12	160	5021407 ±821.9	0.17
3	163615.2±169.5	0.10	240	7323038 ±8823.9	0.12
4	201542±187.9	0.09	320	9871944 ±19169.5	0.19
5	272299.7±252.9	0.09	400	11787603 ±11875.8	0.10

"Table 3:LOD-LOQ Study**"**

Drug Name	BD	SN
LOD (µg/ml)	0.1	0.45
LOQ (µg/ml)	0.3	1.36

"Table 4: Recovery Study of BD**"**

Amount Taken (µg/ml)	Amount Added (µg/ml)	Total Amount (µg/ml)	Amount Recovered (µg/ml)	%Recovery (Mean ± S.D.) (n=3)	% RSD
2	1	3	2.87	97.47± 1.05	1.08
2	2	4	3.94	98.38 ± 1.64	1.67
2	3	5	4.87	98.33 ± 1.33	1.35

"Table 5: Recovery Study of SN**"**

Amount Taken (µg/ml)	Amount Added (µg/ml)	Total Amount (µg/ml)	Amount Recovered (µg/ml)	% Recovery (Mean ± S.D.) (n=3)	% RSD
160	80	240	233.47	97.15±0.12	0.12
160	160	320	318.00	100.22±0.80	0.80
160	240	400	392.97	97.39±1.26	1.29

"Table 6: Repeatability Data**"**

Sr. No.	Area (mAU)	
	BD	SN
1	115938	4864147
2	116641	4892566
3	116443	4901241
4	117619	4969219
5	114982	4953202
6	114212	4926731
MEAN (n=6)	115972.5	4917851
SD	1115.113	35978.15
%RSD	0.961532	0.731583

"Table 7: Inter-day precision data**"**

BeclomethasoneDipropionate			Sertaconazole Nitrate		
Conc. (µg/ml)	**Mean Area (mAU) ± S.D. (n=3)**	**%RSD**	**Conc. (µg/ml)**	**Mean Area (mAU) ± S.D. (n=3)**	**%RSD**
2	118722±583.96	0.49	160	4966097±1147.6	0.03
3	162606.3±499.83	0.30	240	7203670.7±1451.7	0.02
4	220514.3±719.0	0.32	320	9803298±2189.1	0.02

"Table 8: Intra-day precision data**"**

Beclomethasonedipropionate			Sertaconazole Nitrate		
Conc. (µg/ml)	**Mean Area (mAU) ± S.D. (n=3)**	**% RSD**	**Conc. (µg/ml)**	**Mean Area (mAU) ± S.D. (n=3)**	**% RSD**
2	118634.3±347.17	0.29	160	498714 ±5122.9	0.10
3	164594±436.17	0.26	240	7252379 ±5455.59	0.09
4	220454.3±491.41	0.22	320	9829549±5506.39	0.06

"Table 9: Assay of marketed Formulation**"**

Pharmaceutical Formulation ONABET-B	Label Claim %w/w	Amount Taken (µg/ml)	Amount Found (µg/ml)	% Assay (Mean± S.D) (n=3)
BD	0.025 %	2	1.97	98.83 ± 0.56
SN	2 %	160	159.55	99.72 ± 0.51

ACKNOWLEDGEMENT

Authors are thankful to Advance Analytical Research & Training Institute for providing standard API.

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Improvement of Physicomechanical and Pharmacotechnical parameters of Ketoprofen crystals

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ABSTRACT

Present research work emphasized on the improvement in the mechanical and processing parameters of Ketoprofen, BSC Class-II drug, having poor micromeritic properties which belongs to the NSAID category, by way of novel crystallization technique. Various method of preparations and additives/excipients are subjected for the crystal formulation which ultimately improves the micromeritic properties like flowability, compressibility, pressure tensile strength relationship and compactibility by way of improvement in Kawakita's constant, Kuno's constant and Heckel Plot analysis of Ketoprofen loaded crystals compared to the plain drug i.e., Ketoprofen. Therefore Heckel plot analysis of treated crystals depicted the greater plastic deformation (K= 0.230) and yield strength (σ_0= 1.452) with negligible elastic recovery (% ER= 0.628) compared to plain drug and also its control batch formulation. Present research work revealed that crystal engineering technique can be used to modify the mechanical and processing parameters of Ketoprofen which is of BCS Class-II drug having poor flowabiltiy.

SUMMARY

Crystal engineering approach can be useful to improve physicomechanical and pharmacotechnical parameters Ketoprofen

Keywords: Ketoprofen, Mechanical property, Excipient, Kawakita, Heckel plot, Pharmacotechnical

INTRODUCTION

Due to poor biopharmaceutical properties, hardly 1% of active pharmaceutical ingredients (APIs) are getting entry in to the market place (*1*). Improvement in aqueous solubility and *in-vitro* dissolution study is considered as the most challenging issues for pharmaceutical industries (*2*). Many techniques can be used for the improvement in the aqueous solubility and dissolution of selected drugs includes cosolvency, emulsification (*3*), salt formation, complexation with β-cyclodextrin (*4*), high-pressure homogenization, micronisation, co-precipitation, jet milling, cocrystallization (*5*) and use of structured vehicle for the release of poorly water soluble drugs (*6*). Though these techniques are reported to improve the pharmacokinetic parameters, success is again dependent on the physicomechanical and pharmacotechnical properties of the drugs under investigation (*2*).

Majority of APIs have poor flow property, cohesive nature, poor packability and compressibility (*7*). Thus, high amount of directly compressible excipients are always required for their tableting by direct compression (*8*). But this theory might be invalid for APIs having higher dosage and poor packability as it might result into increment in the final weight of the tablet which is not acceptable by patients (*9*). Hence, wet granulation for this type of APIs is the only choice. Again, wet granulation is a tedious, laborious, time consuming and uneconomical process due to involvement of several processing steps like blending, wetting, granulation, sieving, drying, etc (*10*). Therefore, the major advantage of preparing tablets by direct compression technique in the pharmaceutical industry is it requires less manufacturing steps, greater stability against moisture and heat, higher dissolution rate, time saving and economic process and simple process of validation (*11*). Direct compression process, in spite of being a simple process, is highly affected by powder characteristics such as particle shape, size, flow property, compressibility, packability, compactibility, plastic behavior and dilution potential (*12*).

Pharmaceutical drug loaded crystals are the crystalline components comprised of an API and one or more excipient/s, which are solids at room temperature. Excipient interacts with the drug under investigation through non covalent bonding which includes hydrogen bonding, π-π stacking bond and vander waals forces. Among all these connections, treated crystals frequently depends on hydrogen bonding with the drug under investigation. Excipients should be pharmaceutically acceptable (GRAS) compounds which

should not alter therapeutic efficacy of selected API but can help to improve its mechanical and pharmacotechnical parameters (*13-15*). Very few authors have reported crystallization approach for simultaneous improvement in the physicochemical, mechanical, processing and manufacturing properties without alternating its pharmacological behavior (*16, 17*).

Ketoprofen is a potent non steroidal anti inflammatory drug (NSAID), is a proprionic acid derivative which is used for symptomatic treatment of osteoarthritis, anti-inflammatory, spondylitis, rheumatoid arthritis, antipyretic properties and is widely used clinically. On the basis of powder rheology, it is classified as a powder with poor flow property and compressibility (*18-22*) which is not processed by directly compressible technique. Moreover, Ketoprofen is of BCS Class II drug which has poor aqueous solubility and high permeability with analgesic activity (*23, 24*).

In the present work, the aim was to improve physicomechanical and processing parameters of poorly water soluble and poorly flowable drug Ketoprofen (KETO) by its recrystallization in presence of different excipient/s for direct compression technique (*18-22*). Various excipients used for the preparation of treated crystals were Lactose, Benzoic acid (BA), Oxalic acid (OA), Maleic acid (MA) and Saccharin sodium dihydrate (SAC-Na). The solid phase of the prepared treated crystals was also examined to study the generation of new crystal phase in Ketoprofen during the process of crystallization.

MATERIALS AND METHODS

1 Materials

Ketoprofen (KETO) was gifted by Emcure Pharmaceuticals Limited, Pune. Lactose, Benzoic acid (BA), Oxalic acid (OA) and Maleic acid (MA) were purchased from Sisco Research Lab., Mumbai, India. Potassium dihydrogen phosphate (KH_2PO_4) and Disodium hydrogen orthophsophate dihydrate (Na_2HPO_4) were purchased from SDFCL, Mumbai. Sodium Hydroxide (NaOH) and Sodium acetate trihydrate were procured from Rankem, New Delhi, India. Saccharin sodium dihydrate (SAC-Na) was gifted by Pure Chem Pvt. Ltd., Ankleshwar, Gujarat. All other solvents, excipients and chemicals used were of analytical and HPLC grade (Merck Pvt. Ltd., Mumbai, India).

2 Preparation of Treated crystals by conventional solvent evaporation process

Ketoprofen treated crystals were prepared using Saccharin sodium dihydrate (SAC-Na) as an excipient in various molar proportions of drug (2.54 g ≅ 0.01 mol) and excipient (2.41 g ≅ 0.01 mol) (1:1, 1:2, 1:3, 2:1 and 3:1) by dissolving both in sufficient volume of Ethanol (95%) to get a saturated solution. The content

was stirred properly at Room Temperature (*25*). The stirring was continued till the complete removal of the solvent at Room Temperature and the treated crystals were dried at Room Temperature for complete removal of the surface moisture. Solvent residue was further removed from the dried treated crystals by vacuum oven (Nova Instruments, Ahmedabad) at 30 °C for 48 h. The treated crystals were gently triturated in a mortar pestle and passed through a sieve 60 ASTM before characterization. A batch of control crystals was also prepared without adding excipient i.e., Saccharin sodium dihydrate by maintaining the same experimental conditions.

3 Micromeretic properties

3.1 Optical Microscopy

Particle size measurement was determined by optical microscopy (MLX-DX, Olympus (India) Private Limited, New Delhi, India) using pre-calibrated eye piece. Sample was put on the slide and particle size (minimum of 300 particles) was measured by considering longest dimension of the particle. Mean aspect ratio (AR) is defined as the ratio of horizontal maximum (longest dimension from edge to edge of a particle oriented parallel to the ocular scale) to the vertical maximum (the longest dimension of the particle measured at right angles of the length) distance of the particle, was measured (*26*). Randomly selected 300 particles were assessed for their particle size of the respective sample under investigation.

3.2 Angle of repose

Angle of repose was measured by fixed funnel (height) method. The powder was flow through the funnel which is placed at a fixed height till it vertically approaches a maximum heap height (h) (*27*). Radius of the heap (r) was determined and angle of repose was measured using the following equation :-

$$\theta = \tan^{-1}\left(\frac{h}{r}\right) \quad (1)$$

Where, θ = angle of repose, h = height of the heap, r = radius of the heap

3.3 Carr's compressibility index

Flowability of pure drug, its control batch and treated crystals was evaluated from Carr's compressibility index (CI) (*28-29*). The Carr's compressibility index was assessed from the bulk and tapped densities. Tapped density machine (ETD-1020, Electrolab, Mumbai, India) was used for the determination of tapped density which was measured by tapping the samples (5 gm) into 10 mL measuring cylinder. The CI was calculated as per the following equation :-

$$\text{Carr's Compressibility Index} = \left[\frac{t_i \quad d \quad (\rho_t) - b \quad d \quad (\rho_0)}{t_i \quad d \quad (\rho_t)}\right] \times 100 \quad (2)$$

3.4 Hausner's ratio

Hausner's ratio is an indirect indicator of how powder flows easily. It was measured by the following equation (*27*) :-

$$\text{Hausner's ratio} = \frac{t_i}{b} \frac{d}{d} \frac{(\rho_t)}{(\rho_0)} \quad (3)$$

4 Measurement of packability, compactibility and compressibility

4.1 Kawakita constants

The packability of each sample was determined by tapping them into a 10 mL graduated cylinder using a USP Tap density tester (*30*). Accurately weighed powder (5 gm) was taken into a graduated cylinder. Volume encompass by powder in the graduated measuring cylinder was recorded. Bulk density (lowest density) was determined from the mass/weight (5 gm) of the powder and its volume. Then the graduated cylinder was tapped and reduction in volume of powder mass was noted after every 100 tapping until the volume remains unchanged. The packability was estimated by determining the tapped density as per the following Kawakita equation :-

$$\frac{n}{C} = \frac{n}{a} + \frac{1}{a} \quad (4)$$

Where, a and b are the constants, n = number of tapping, C = reduction in volume which is calculated using following equation :-

$$C = \frac{V_0 - V_n}{V_0} \quad (5)$$

Where, V_0 is the volume of powder mass at original condition and V_n is the volume of powder mass at n^{th} tapped condition. The packability and compactibility parameters include 'a' which denotes the value of original porosity of the powder mass, degree of reduction in volume for the powder mass at infinite applied pressure while '1/b' represents the pressure necessary to reduce the powder mass to one half of the total volume. From the Kawakita plot of n/C vs. n (*31*), 'a' value obtained from the slop while '1/b' value obtained from the plot intercept.

4.2 Kuno's constant

The data obtained during Kawakita analysis was also analyzed by the Kuno's equation. The packability was investigated by comparison between the constants a, 1/b and K.

$$\ln(\rho_t - \rho_n) = -K_n + \ln(\rho_t - \rho_0) \quad (6)$$

Where, ρ_t is an apparent density at equilibrium, ρ_n is an apparent density at n^{th} tapped and ρ_0 is an apparent density at an original condition, 'K' is Kuno's constant represents the rate of packing process.

4.3 Heckel plot analysis

Heckel plot analysis implies a method of converting force in tons and displacement signals to a linear correlation for the powder bed experiencing compaction. The equation assumes that densification depends on pressure is of first order, with the inter particulate pores as a reactant and the material densification as a product. The punch and die were lubricated with the dispersion of 1% w/v magnesium stearate in acetone before the materials are compressed. Accurately weighed 500 ± 5 mg of plain drug, its control batch and treated crystals were compressed using 8 mm flat-faced punch at various forces (1, 3, 5, 7 and 9 tons) using KBr press (Techno-search Instruments, Mumbai, India) (*32*). True/Actual density was measured as mass/weight to volume of the pellet at a maximum applied force in tons i.e., 9 tons.

$$\ln\left(\frac{1}{1-D}\right) = KP + A \quad (7)$$

Where, 'D' is the solid fraction which is the ratio of density of the tablet to the true/actual density of powder materials at applied force in tons, (1-D) denotes the % porosity (Є) of the powder material, 'P' indicates applied pressure, 'K' is the material dependent constant which was obtained from the slop value of the straight line of the Heckel plot and the mean yield pressure (Py) is inversely proportional to the value of 'K', intercept is the value of 'A' gives the densification of the powder material under investigation because of the initial particle rearrangement. Yield strength (σ_0) suggested the behavior of the powder materials i.e., either plastic flow or fragmentation (*33*). 'K' is the inverse relationship to the yield strength, where $K = 1/3\sigma_0$. 'D' value was investigated by the determination of diameter and thickness of the compacts after each applied pressure in tons. Compression behavior of the respective powder materials was denoted as parameters of Heckel equation (*34*). Heckel analysis was carried out by plotting the graph of $\ln\left(\frac{1}{1-D}\right)$ vs. P.

4.4 Elastic recovery

After determination of diameter and thickness, pellets used in the Heckel plot analysis were subjected to relaxation for 24 hours to judge the elastic recovery. The width of pellets was determined immediately after ejection (H_c) from the die and also after relaxation period of 24 hours (H_e). Elastic recovery was determined from the following equation (*35*) :-

$$\%ER = \left[\frac{(H_e - H_c)}{H_c}\right] \times 100 \quad (8)$$

4.5 Pressure-Tensile strength relationship

The prepared pellets in the Heckel plot analysis were subjected to tensile strength measurement in which the force required to break the pellets (F) was measured (*36*). Tensile strength of the pellets was assessed by using the following equation (*37*) :-

$$T = \frac{2F}{\pi} \quad (9)$$

Where, D is the diameter of the pellets in cm while t is the thickness of the pellets in cm.

*5*Saturation solubility study (*49*)

An excess quantity of API, its control batch and API loaded crystals were added separately into 10 mLof Distilled water, Acidic buffer pH 1.2, Acetate buffer pH 4.5, Phosphate buffer pH 6.8, Phosphate buffer pH 7.2 (For Ibuprofen) and Phosphate buffer pH 7.5 (For Ketoprofen). The samples were placed in Cryostatic constant temperature reciprocating shaker bath at the temperature 37± 1°C with constant shaking at 120 RPM for 48 hours to allow saturation.

The solutions were then centrifuged and if necessary, the supernatant was filtered through whatman filter paper No. 41 and the filtrate was suitably diluted with respective buffer solutions and analyzed spectrophotometrically against blank.Concentration of API in each solution was calculated from absorbance obtained using regression line equation of calibration curve. The procedure was repeated for three times.

*6*Melting point determination

The melting point was evaluated using a Melting point apparatus (Veego®, Model: VMP-DS) in order to find themelting point of pure drug, control batch and API loaded crystals. The procedure was repeated for three times.

*7*Characterization parameter of API loaded crystals

7.1 Fourier Transform Infra Red spectroscopy(FT-IR) study

Compatibility study and characterization of pure drug, excipient, physical mixture of drug and excipient, control batch and the final formulation (API loaded crystals) were done by interpreting the IR spectra of the respective samples analyzedby Nicolet IS 10, FT-IR spectrometer (Thermo scientific, Japan).

Each sample were milled separately with KBr to form a very fine powder. The thin KBr pellets of each powder sample were prepared by compressing them in KBr press separately for analysis and their IR spectra were recorded in the fingerprint region of 400-4000 cm^{-1}.

RESULTS AND DISCUSSION

1 Preliminary trials for the preparation of Ketoprofen treated crystals

Preliminary trial batches of Ketoprofen treated crystals were prepared using different excipients like Lactose, Oxalic acid, Maleic acid, Benzoic acid and Saccharin sodium dihydrate (SAC-Na) by changing various experimental conditions and techniques like antisolvent addition technique, fusion technique, solvent evaporation technique and deep freezing at 4 to 5 °C. All the techniques were processed with various molar ratios of drug and excipients (1:1, 1:2, 1:3, 2:1 and 3:1). Among various organic solvents used to obtain Ketoprofen crystals, one solvent i.e., Ethanol (95%) gave expected results. Because of the very poor solubility of either drug or excipient or both, solvents like methanol, hexane, chloroform and other low polarity solvent could not formulate the desired crystals. In each trials, treated crystals were obtained either with poor yield, poor drug loading, low aqueous solubility and dissolution rate or poor flow property. Hence, those batches were screened from the present work. One excipient i.e., SAC-Na dihydrate gave desired result with solvent evaporation process to develop treated crystals of Ketoprofen where Ethanol (95%) was used as a solvent. Among different molar ratios of KETO: SAC-Na, only 1:1 molar ratio was found to generate treated crystals with improved flow property compared to pure Ketoprofen. Hence, those batches were subjected for further evaluations. A control batch of recrystallized Ketoprofen was also prepared to compare the effect of excipient/s and its concentrations on the crystal morphology and other properties of treated crystals.

2 Micromeritic properties

The mean particle size of control batch as well as treated crystals with molar ratio of 1:1 of KETO: SAC-Na was greatly improved, which could be responsible for its good flow property. The aspect ratio (AR) for control batch and treated crystals was found to be 1.23 ± 0.63 and 1.83 ± 0.87 (1:1), respectively. This changed in the pattern of treated crystals with small AR imparted better flow property as compared to pure Ketoprofen and its control formulation (*26*). As per the data depicted in Table 1, it was found that the flowability of pure drug was very poor because of very small and irregular shaped particles with its strong tendency to aggregate due to electrostatic charge formed on their surface (*38-39*). Control batch crystals were improved in their flow property.

A great improvement in flow property of treated crystals was observed, which might be because of the increment in its particle size and nearly equidimentional shape (*40-41*). Measurement of flow property (Table 1) of control batch crystals as well as treated crystals showed marked improvement in the flow compared to pure Ketoprofen. Treated crystals showed even more improved flow characteristics compared to pure drug as well as control batch crystals. Because of equidimentional shape of treated crystals better flowability was observed in comparison with pure Ketoprofen (*26*). Moreover, small agglomerated particles of pure Ketoprofen implies its poor flowability and stickiness due to electrostatic

charged particles. The distinctive crystal structure also suggested the development of a new solid phase (*42*).

3 Packability study

The packability parameters are shown in Table 2 and Fig. 1. It was illustrated that the decreased value of 'a' and increased value of '1/b' compared to pure Ketoprofen suggested the improvement in packability of drug loaded crystals (treated crystals) (*43*). Increased values of 'K' (Kuno's constant) compared to pure Ketoprofen showed marked improvement in compressibility and packability of treated crystals (*43-44*).

4 Compressibility study

The true density of Saccharin sodium dihydrate treated crystals with molar ratio 1:1 is (2.08 mg/cc), control batch (1.76 mg/cc), pure drug (1.39 mg/cc) indicated that there was an improvement in the packability and compactibility of drug loaded crystals in comparison with the pure drug (Fig. 2 and Fig. 3). The heckel plot constants of pure drug, control batch and drug loaded crystals were investigated are shown in Table 3.

Heckel plot constant i.e., slop value 'K' suggested the plastic flow of the material. Larger the value of 'K', greater was the plastic flow of the material under investigation (*45*). Linearity in the graph (Fig. 2) also suggested the plastic deformation. Parameters of heckel plot is depicted in Table 3. Lower "P_y" value in comparison with the pure drug implies the less compression pressure was required to acquire closest packing in treated crystals which also suggested the plastic behavior of the treated crystals. Fractured texture and densifying the particles was observed in drug loaded crystals.

Lower yield strength (σ_0) and yield pressure (Py) of treated crystals indicated the lower resistance to applied pressure owing to its plastic flow and fragmentation which indicated the better densification and easy compaction behavior of the powder material under investigation (*33, 46*). Thus, heckel plot data analysis suggested the easy fractured and new surface of treated crystals formed in order to encourage the plastic deformation under applied compression pressure (*45, 47*).

5 Pressure-tensile strength relationship

Tensile strength of pellets made for Heckel plot analysis was measured and plotted in Table 4 and Fig. 4. It was revealed that as the compression pressure increased, there was an increment in tensile strength of all the samples as compression pressure was raised from 1 to 9 tons. A much higher tensile strength was observed for the compacts of treated crystals at 9 tons force with molar ratio 1:1 of KETO:SAC-Na

because of its higher packability, compactibility and compressibility compared to pure Ketoprofen (Table 2 and Table 3). It was an indication of plastic nature of the resultant treated crystals (*48*).

*6*Saturation solubility study

Saturation solubility (mg/mL) study was performed for pure drug, its control batch andAPI loaded crystals in buffer solutions of pH 1.2, pH 4.5, pH 6.8 and pH 7.5 (dissolution media of API under study) at 37°C ± 0.5°C.

The results of Table 5 suggested that Ketoprofen was practically insoluble in water, HCl buffer pH 1.2 and acetate buffer pH 4.5 while slight solubility was observed with Phosphate buffer pH 6.8 and pH 7.5. The comparison of solubility data of KETO pure drug in water and different buffer solutions with its control batch and treated crystal formulations was found to be increased as per the following Table 6.

From the results of Table 6, it was depicted that treated crystals of KETO:SAC-Na (Molar proportion of 1:1) showed 16.26 fold increment in aqueous solubility compared to Ketoprofen pure drug. Moreover, there was a remarkable increment of solubility in HCl buffer pH 1.2, Acetate buffer pH 4.5, Phosphate buffer pH 6.8 and dissolution media of Phosphate buffer pH 7.5 compared to Ketoprofen pure drug. Saturation solubility results of Table 5 and Table 6 clearly suggested that treated crystals of KETO:SAC-Na prepared with molar ratio of [1:1] was the optimized ratio.

*7*Melting point determination

The melting point of Ketoprofen pure drug, excipient, control batch and treated crystal formulations was determined and the results are depicted in Table 7.

As shown in Table 7, the depression in melting point of control batch compared to pure drug was found which suggested that, there might be an increase in solubility compared to pure drug. There was further remarkable reduction in melting point of treated crystals which clearly suggested that there was a drastic increase in solubility of drug. It was also found that the melting point of treated crystal was less than the melting point of pure drug (KETO) and excipient (SAC-Na) which again suggested that there might be a formation of new treated crystal phase (*50*).

*8*Characterization parameter of API loaded crystals

8.1 Fourier Transform Infra Red spectroscopy(FT-IR) study

FT-IR spectra as shown in the above Figures(5 to 8)suggested that there was no any possible intermolecular interactions found between the drug and excipient as there was no change in the characteristic peaks of the drug as revealed in the following Table 8 (*51*).

By interpreting and comparing the absorption bands of treated crystal formulation with pure Ketoprofen, it clearly depicted that the excipient Sachharin sodium was able to form hydrogen bond with the drug as new band formed which shown in the row no. 1, 2, 4, 5, 6, 9 and 10 of the above table in the treated crystal formulation. Hence, formation of hydrogen bond was responsible to enhance the solubility of the Ketoprofen.

CONCLUSION

Ketoprofen is a class II drug with poor manufacturability because of poor physicalmechanical properties. In the present study, crystallization approach with solvent evaporation technique at room temperature has been utilized in order to enhance the physicomechanical properties of Ketoprofen drug. Out of various excipients, Saccharin sodium dihydrate gave encouraging results. Pharmacotechnical parameters of Ketoprofen treated crystals was enhanced to a greater extent compared to pure Ketoprofen drug. The mechanical property and compressibility results also suggested the plastic nature of treated crystals, which enabled to formulate its directly compressible tablets without using any directly compressible excipients. The use of treated crystals could be a viable and effective approach for improving the physicomechanical and manufacturing properties of Ketoprofen drug.

FIGURES

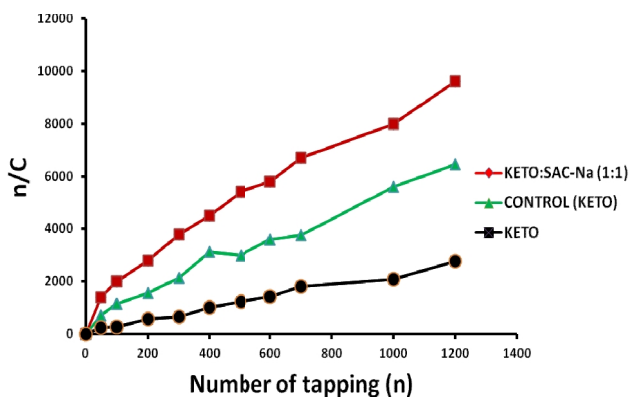


Fig. 1 Packability and flow parameters

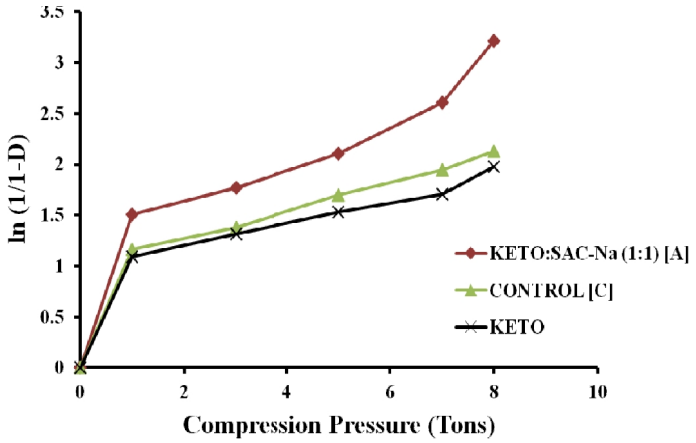


Fig. 2 Comparison of compression behavior by Heckel parameters

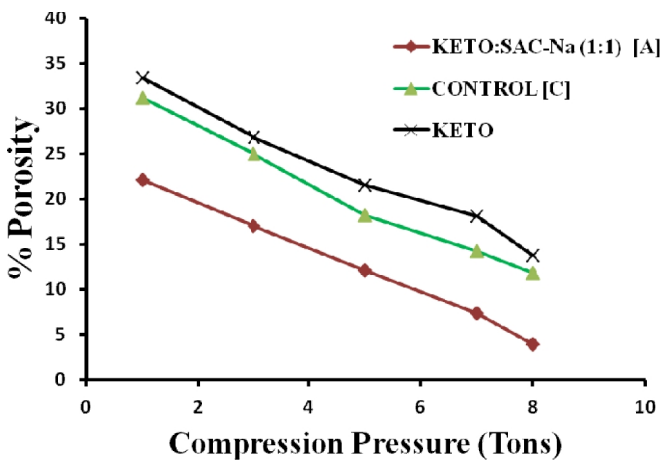


Fig. 3 Comparison of % porosity of samples

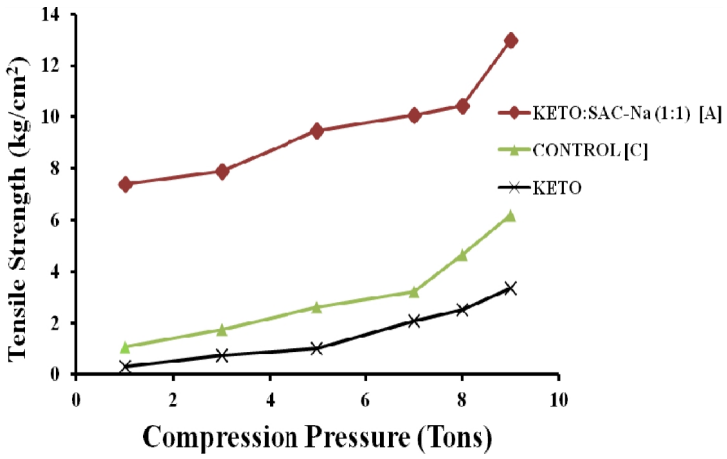


Fig. 4 Comparison of pressure-tensile strength relationship

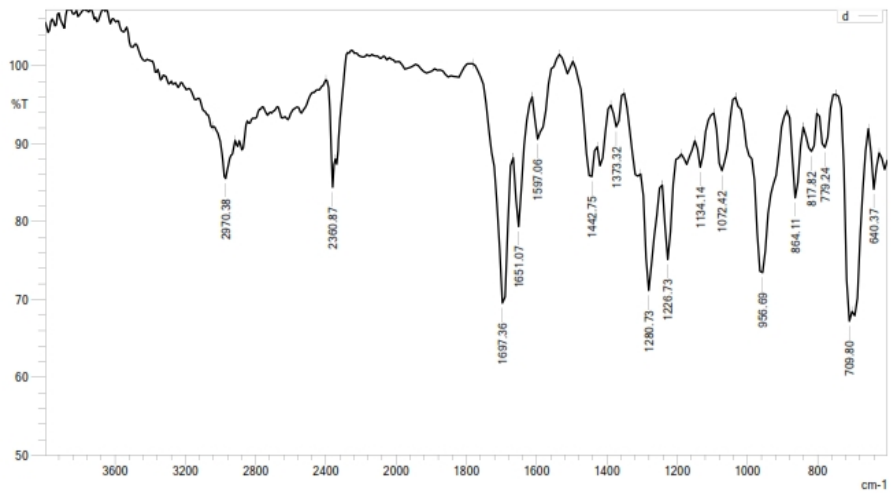


Fig. 5 FT-IR spectra of Ketoprofen pure drug

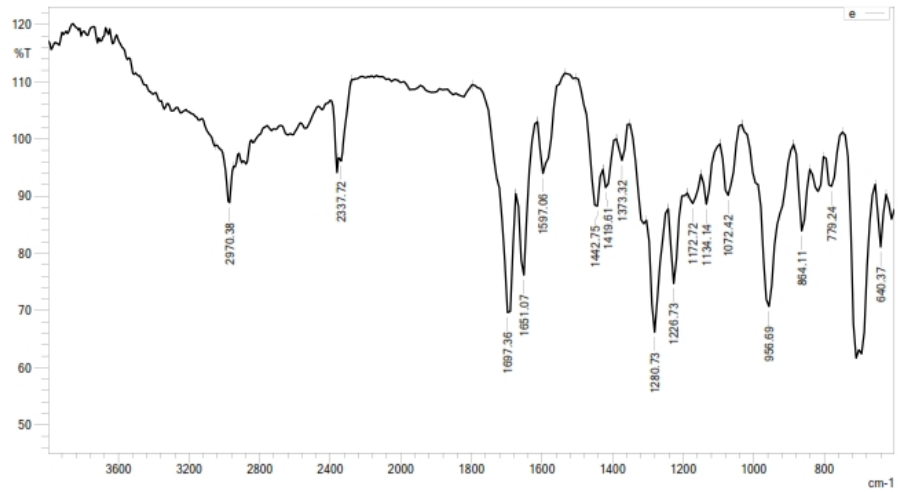


Fig. 6 FT-IR spectra of Ketoprofen control batch

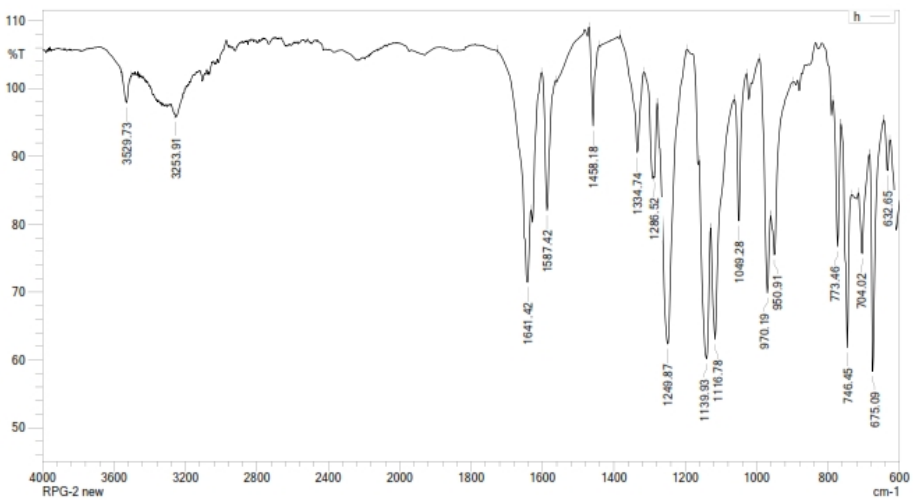


Fig. 7 FT-IR spectra of Saccharin Sodium [Excipient]

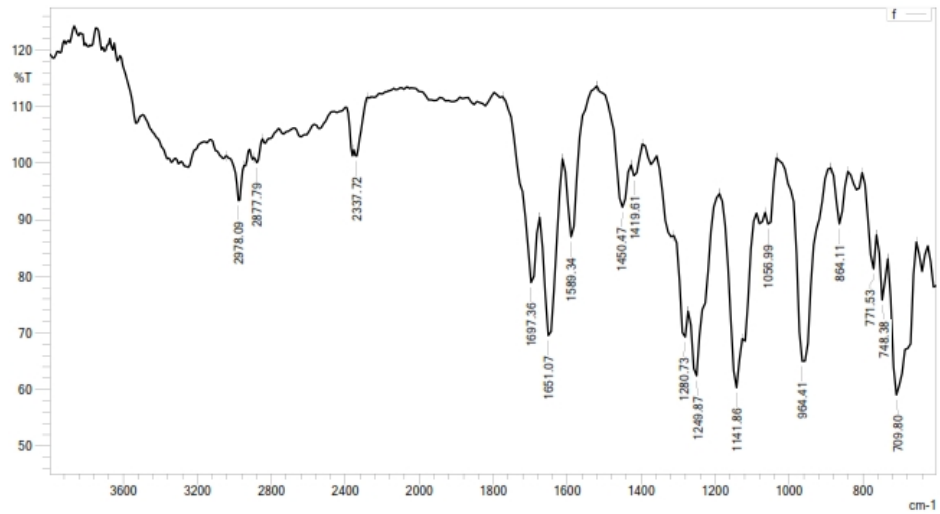


Fig. 8 FT-IR spectra of KETO:SAC-Na [1M:1M] treated crystal

TABLES

Table 1 Comparison of flow properties of Ketoprofen pure drug, its control batch and treated crystals

Sr. No.	Parameters	Pure Ketoprofen [KETO]	CONTROL [C]	KETO:SAC-Na (0.1M:0.1M) [A]
1	Mean Particle Size (μm) ± SD*	Agglomerated form	25.243 ± 0.734[a]	27.407 ± 0.452[a]
2	Angle of Repose (θ) ± SD*	41.621° ± 0.628	33.517° ± 0.344[a]	27.933° ± 0.281[a]
3	Loose Bulk Density (ρo) (gm/ml) ± SD*	0.450 ± 0.063	0.251 ± 0.058[a]	0.260 ± 0.047[a]
4	Tapped Bulk Density (ρt) (gm/ml) ± SD*	0.653 ± 0.029	0.311 ± 0.062[a]	0.297 ± 0.041[a]
5	% Carr's Compressibility Index (CI) ± SD*	31.0873 ± 0.632	19.2926 ± 0.586[a]	12.4579 ± 0.437[a]
6	Hausner's Ratio ± SD*	1.4511 ± 0.065	1.2390 ± 0.047[a]	1.1423 ± 0.038[a]

* Results are mean ± SD of three observations

[a] Significantly different from the pure drug, $P < 0.05$

Table 2 Comparison of packability and compactibility parameters of Ketoprofen pure drug, its control batch and treated crystals

Samples	Kawakita's Constants		Kuno's constant
	$a = \frac{1}{m}$	$\frac{1}{b} = \frac{C}{m}$	K
Pure Ketoprofen [KETO]	0.447587503	37.88112076	0.000733924
CONTROL [C]	0.202032446	117.8697699	0.00172821
KETO:SAC-Na (1:1) [A]	0.125	150	0.003035366

Table 3 Comparison of Compressibility parameters and Elastic recovery of Ketoprofen pure drug, its control batch and treated crystal formulation

Samples	Heckel Plot Constants		Mean yield pressure (P_y)	Yield Strength (σ_0 OR S)	% Elastic Recovery ± SD*
	K (Slop)	A (Intercept)	$Py = \frac{1}{K}$	$S = \frac{1}{3K}$	$\%ER = \left[\frac{(H_e - H_c)}{H_c}\right] \times 100$
Pure Ketoprofen [KETO]	**0.1181**	**0.9605**	**8.467400508**	**2.822466836**	**4.761905 ± 0.872**
CONTROL [C]	0.1378	1.0040	7.256894049	2.418964683	3.191489 ± 0.563
KETO:SAC-Na (1:1) [A]	0.2296	1.1400	4.355400697	1.451800232	0.627615 ± 0.456

Table 4 Comparison of Pressure-Tensile strength relationship of Ketoprofen pure drug, its control batch and treated crystal formulation

Compression Pressure 'P' (Tons)	Tensile Strength (kg/cm^2)		
	Pure Ketoprofen [KETO] ± SD*	CONTROL [C] ± SD*	KETO:SAC-Na (1:1) [A] ± SD*
1	0.322 ± 0.72	1.076 ± 0.64	7.417 ± 0.42
3	0.754 ± 0.73	1.743 ± 0.93	7.901 ± 0.68
5	1.018 ± 0.69	2.610 ± 0.70	9.484 ± 0.99
7	2.082 ± 0.97	3.219 ± 0.96	10.091 ± 0.52
8	2.519 ± 1.67	4.678 ± 0.47	10.456 ± 0.74
9	3.368 ± 1.51	6.211 ± 0.84	12.992 ± 0.63

Table 5: Solubility (mg/mL) study of pure drug, control batch and treated crystals of Ketoprofen

Sr. No.	Sample	Solubility (mg/ml) at 37 ± 0.5°C				
		Distilled Water ± SD*	HCl buffer pH 1.2 ± SD*	Acetate buffer pH 4.5 ± SD*	Phosphate buffer pH 6.8 ± SD*	Phosphate buffer pH 7.5 ± SD*
1	**Ketoprofen pure drug [KETO]**	**0.0492 ± 0.54**	**0.0498 ± 0.94**	**0.0687 ± 0.52**	**2.1118 ± 0.63**	**4.6584 ± 0.39**
2	**CONTROL [C]**	0.2817 ± 0.78	0.2161 ± 0.48	0.3032 ± 0.74	11.1553 ± 0.75	22.0807 ± 0.58
3	**KETO:SAC-Na (1:1) [A]**	**0.7997 ± 0.49**	**0.4640 ± 0.62**	**0.6848 ± 0.48**	**24.2236 ± 0.94**	**84.5411 ± 0.77**

* Results are mean of three observations ± SD

Table 6: Comparison of Solubility (mg/mL) in pH 1.2, pH 4.5, pH 6.8 and pH 7.5

Sr. No.	Sample	Number of fold increase in solubility at 37 ± 0.5°C as compared to Pure KETOPROFEN				
		Distilled Water	HCl buffer pH 1.2	Acetate buffer pH 4.5	Phosphate buffer pH 6.8	Phosphate buffer pH 7.5
1	**CONTROL [C]**	5.73	4.34	4.42	5.28	4.74
2	**KETO:SAC-Na (1:1) [A]**	16.26	9.31	9.97	11.47	18.15

Table 7: Melting point study

Sr. No.	Samples	Melting Point ± SD*
1	Pure Ketoprofen [KETO]	97.3°C ± 1.45
2	Saccharin Sodium [SAC-Na]	126.8°C ± 1.63
3	CONTROL [C]	96.2°C ± 1.28
4	KETO:SAC-Na (1M:1M) [A]	89.2°C ± 2.69

* Results are mean of three observations ± SD

Table 8 FT-IR interpretation of pure Ketoprofen and its Treated crystal formulation

Sr. No.	Pure Ketoprofen PEAK AT (cm^{-1})	PEAK INTERPRETATION	Treated Crystal Formulation PEAK AT (cm^{-1})	OBSERVATION
1	**3400 cm^{-1} to 2400 cm^{-1}**	O-H group of Carboxylic acid	**3600 cm^{-1} to 2300 cm^{-1}**	**Broad peak may be due to Hydrogen bond formation**
2	---	---	**3300 cm^{-1} to 3500 cm^{-1}**	**Intermolecular O…H-N (H-bond)**
3	**2970.38 cm^{-1}**	Aromatic (C-H stretching) group may be present	**2978.09 cm^{-1}**	Aromatic (C-H stretching) group may be present
4	---	---	**2400 cm^{-1} to 2600 cm^{-1}**	**Intermolecular O-H…N (H-bond)**
5	**1697.36 cm^{-1}**	C=O of Carboxyl carbonyl stretching group may be present	**1697.36 cm^{-1}**	**Markedly decreased the peak of C=O of Carboxyl carbonyl stretching group**
6	**1651.07 cm^{-1}**	C=O of Ketonic carbonyl stretching group may be present	**1651.07 cm^{-1}**	**Markedly increased the peak of C=O of Ketonic carbonyl stretching group**
7	**1442.75 cm^{-1}**	-CH of Alkane (-CH_2 bend) group may be present	**1450.47 cm^{-1}**	Peak shift is observed for -CH of Alkane (-CH_2 bend) group
8	**1373.32 cm^{-1}**	-CH of Alkane (-CH_3 bend) group may be present	**1373.32 cm^{-1}**	-CH of Alkane (-CH_3 bend) group may be present
9	**1072.42, 1226.73 & 1280.73 cm^{-1}**	C-O stretching of Carboxylic acid group may be present	**1056.99 cm^{-1}, 1249.87 cm^{-1} & 1280.73 cm^{-1}**	**Peak shift with broadness of peak suggest the formation of H-bonding with C-O stretching of Carboxylic acid group may be present**
10	---	---	**748.38 cm^{-1} & 771.53 cm^{-1}**	**Fingerprint region (For cocrystals)**

ACKNOWLEDGEMENT

The authors greatly acknowledge Emcure Pharmaceuticals Ltd., Pune for the gift sample of Ketoprofen. Authors are also grateful to Gujarat Council on Science and Technology (GUJCOST), Gandhinagar for their kind financial support in the study.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

(Proceedings available for download at rku.ac.in/icre)

RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

Preparation and characterization of Olanzapine microemulsion

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ABSTRACT

Present study deals with preparation, optimization and characterization of microemulsion based drug delivery system of Olanzapine. Olanzapine is an atypical antipsychotic agent and BCS class-II drug. It was formulated as microemulsion using capryol 90, Tween 20, Acconon CC6 as Oil, surfactant and cosurfactant respectively. Formulation was optimized on basis of percentage transmittance (99.87±0.23 nm), Globule size (123.4 ± 1.3) and zeta potential (-8.34 ± 0.85). Optimized formulation was analyzed for dilutability, dye solubility, assay, pH, viscosity and conductivity. Prepared formulation was subjected to tissue (nasal) toxicity study for its intended nasal delivery. Optimized formulations were found to be stable.

SUMMARY

Microemulsion based delivery system of Olanzapine was successfully formulated and characterized which can be effectively used for desire action

Keywords: Olanzapine, Microemulsion, Tissue toxicity study

INTRODUCTION

Olanzapine {2-methyl-4-(4-methyl-1-piperazinyl)-10H-thieno[2,3-b] benzodiazepine} is a neuroleptic medicine used in many CNS disorders (*1*). Olanzapine is highly lipophilic drug, very less soluble in water. Hepatic first pass metabolism reduces the bioavailability of drug to a great extent and there are many dose related side effects such as Parkinsonism. Microemulsion based drug delivery through oral or preferentially nasal route will help to overcome this limitations.

Microemulsion as dosage form have some useful properties like thermodynamic stability which provides long shelf life, Easy to formulate as the interfacial tension is negligible and forms quickly, Optical isotropy, Increased surface area improves solubility, small globule size and lipophilic nature improves bioavailability. Microemulsion can be formulated using various Excipients. Chief ingredients are oil and surfactants. All components should be Biocompatible and clinically safe. (*2-5*).

Current experiment involve development of Microemulsion based drug delivery system of Olanzapine which can be used by suitable route to get symptomatic relief in conditions like Schizophrenia and other psychotic disorders that carries high risk of suicide and life threatening behavior. Olanzapine microemulsion was formulated using Capryol90 as oil, Tween20 as a surfactant and AccononCC6 as cosurfactant. Experimental work involves solubility study of drug in various components, preparation of microemulsion using phase titration method and optimization by constructing pseudo ternary phase diagram and characterization of optimized formulation for various parameters. Optimized microemulsion was used for tissue toxicity study and was found to be safe. Stability study of formulations reveals that the formulations were stable.

MATERIALS AND METHODS

Olanzapine was kindly gifted by Cadila Healthcare Ltd. Ahmedabad. Capryol 90, labrafil M were procured from Gattefosse. Captex was purchased from Abitec corp. Olive oil, Isopropyl Myristate, PEG 200, PEG 400, Tween 20 and Tween 40 was procured from Himedia Ltd. Ethyle oleate from Fine chemi. and Soyabean oil form A.B.Enterprise

Solubility study:

The solubility of olanzapine in different Excipients like oil, surfactant and cosurfactant was carried out using Shaking flask (gradient addition) solubility Study method (*6*).

Methods of Preparation

Microemulsion was formulated by Phase Titration Method which is also known as

Spontaneous emulsification method. In this method fix amount of oil was mixed with the mixture of surfactant and cosurfactant (Smix) followed by drop wise addition of water. Emulsion, micelles and different associative structure forms based on composition and concentration of different Excipients, leads to formation of microemulsion (*7*). Construction of phase diagram is a useful approach to study the complex series of interactions that can occur when different components are mixed.

Optimization of Surfactant/Co-Surfactant and Oil Ratio by Phase Diagram Preparation:

In microemulsion system surfactant and co-surfactant get preferentially adsorbed at the interface, reducing the interfacial energy as well as providing a mechanical barrier tocoalescence. The decrease in the free energy required for the emulsion formation consequently improves the thermodynamic stability of the microemulsion formulation. Therefore, the selection of oil and surfactant, and the mixing ratio of oil to S/CoS, play an

important role in the formation of the microemulsion. This can be ascertained by pseudoternary phase diagram as it differentiates the microemulsion region from that of macroemulsion region (*8*).
Pseudoternary phase diagram was plotted using water titration method; this method is most suitable as it is easy & scalable. Microemulsions were prepared to find the area of particular component system. In this method surfactant and co-surfactant were mixed in fixed weight ratios (1:1, 2:1, 3:1, and 4:1). Aliquots of each surfactant and cosurfactant mixture (Smix) were then mixed with oil at ambient temperature. For each phase diagram, the ratio of oil to the Smix was varied as 9:1, 8:2, 7:3, 6:4, 5:5, 4:6, 3:7, 2:8, 1:9(v/v). Water was added drop wise to each oil-Smix mixture under vigorous stirring. After equilibrium, the samples were visually checked and determined as being clear microemulsions or emulsions or gels. No heating is conducted during the preparation. Phase diagrams were prepared using chemix software.

Optimization of formulation

Based on preliminary studies, various batches of microemulsion were prepared using surfactant to co-surfactant ratio of 1:1. One important consideration when formulating a microemulsion formulation is avoiding precipitation of the drug on dilution. Optimization of system was done using dilutability and transparency.

Characterization of optimized formulation

Any developed formulation must be evaluated to conform that it meets the predetermined requirements or not. Microemulsions are the optically transparent systems which should remain clear on dilution. Moreover, they should have optimum drug content, globule size and zeta potential and flow characteristics. Olanzapine loaded microemulsions were characterized for parameters such as Clarity, Dilutability and its effect on globule size and zeta potential, Assay, Globule size determination, Zeta-potential, pH, Viscosity and Conductivity.

Instruments:

The instruments used for the preparation of formulations and estimation of drugs include UV-visible spectrophotometer (UV, 1601,220x Shimadzu, Japan) and pH Meter (Systonics 335, Japan), Bath Sonicator(Labindia, Ambala), Analytical Balance (Precisa 205 Ascs, Switzerland), Remi Magnetic Stirrer 1mlh (Remi Equipments, Mumbai, India), Vortex Mixer (Remi Equipments, Mumbai, India), Rheometer (Cone and Plate Brookfield Programmable DV-III+ rheometer, brookfield engineering laboratories, inc., ma, usa.), Remi Centrifuge(Remi Equipments, Mumbai, India) and Malvern ZetaSizer NanoZS series, Malvern Instruments, Germany.

Tissue (nasal) toxicity study

Optimized formulation was evaluated for nasal toxicity using Goat nasal mucosa
Tissue was treated with Isopropyl Alcohol (IPA) as positive control, Normal saline as negative control and olanzapine microemulsion

Stability Study:

The formulations were subjected to stability study for a period of Six months at Refrigerated temp (2^0C- 8^0C), room temperature and Elevated temp (50^0C$\pm 2^0$C). After three months of storage the microemulsions were evaluated for Physical Stability, Percentage Transmittance, Assay and Globule Size.

RESULTS AND DISCUSSION

Solubility: The solubility data are shown in table 1.

Phase diagram outcome: For Capryol 90: Tween 20: Acconon CC6 system highest region was found in 1:1 ratio and hence selected for further preparation. Results of phase diagram system are shown in Fig.1

Optimization of formulation: Few batches of Microemulsion were prepared and as shown in Table: 2, Batch no 10 has been selected based on transmittance, globule size, zeta potential, concentration of oil and solubility of Olanzapine in oil. At lower concentration of oil the drug was precipitated out after 10-15 days. Oil concentration above 25% consumes more surfactant and cosurfactant concentration which is not advisable. Microemulsion with 20% oil concentration was considered as optimized batch because in this formulation drug did not precipitate out. Optimizations of experimental parameter in microemulsion formulation are shown in Table 3. which indicate, there was negligible effect on globule size because of stirring time and stirring speed. Based on result obtained, optimized formula for microemulsion system is shown Table 4.

Characterization: The results of characterization tests are shown in Table.5.

Tissue (nasal) toxicity study: From the Histopathological investigation it was revealed that there was no apparent damage to nasal mucosa by formulation. Histopathological image are shown in Fig.2

Stability study: Formulations were found to be stable for a period of Six months as shown in Table.6.

CONCLUSION

Characterization results of developed formulations meet the predetermined requirements of microemulsion as a dosage form. Microemulsions were optically transparent and remain clear on dilution. They have optimum drug content, globule size, zeta potential and flow characteristics. Microemulsions were stable and Histopathological investigation suggests safety for its intended nasal applications.

FIGURES

Fig.1: Pseudoternary phase diagrams of Capryol 90 (oil), Tween 20/ Acconon CC6 (s/cos) and water system

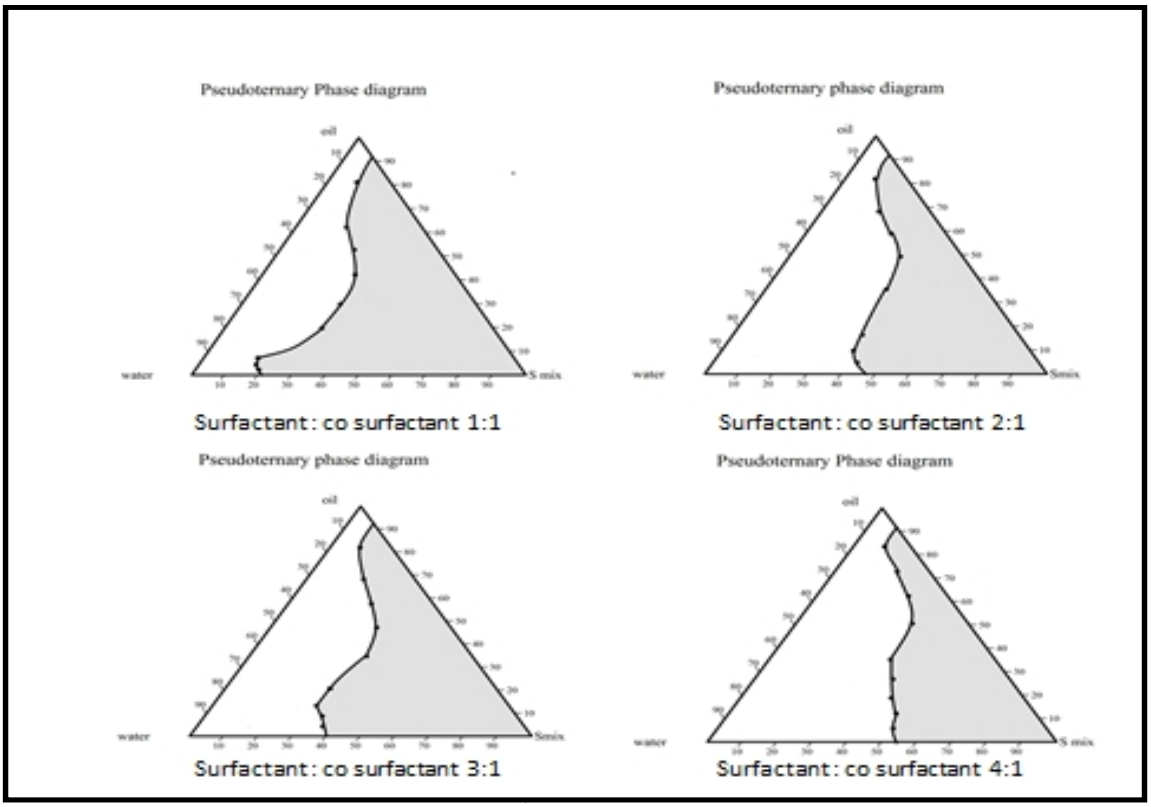


Fig. 2(a): Histopathological images of nasal mucosa treated with Positive control: (IPA)

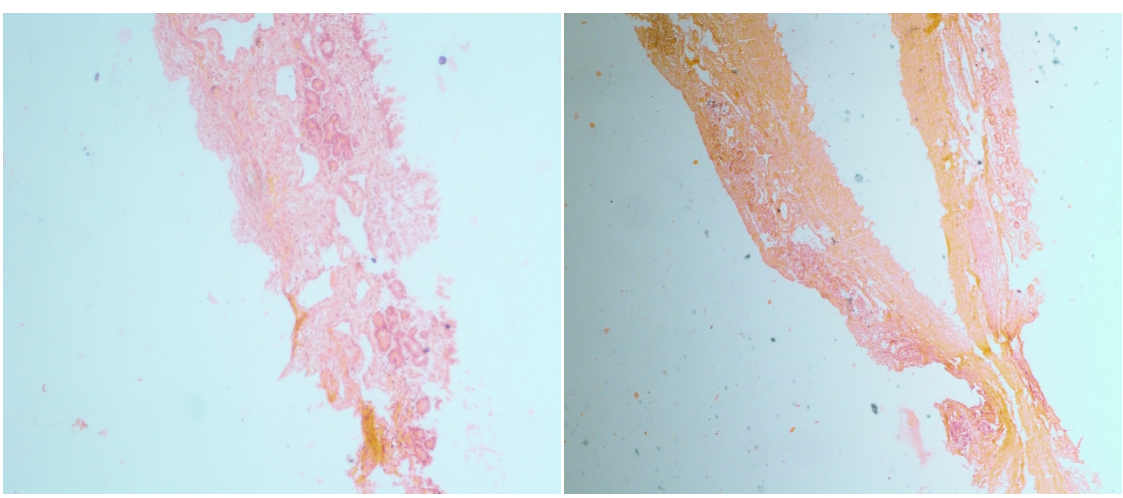


Fig. 2(b): Histopathological images of nasal mucosa treated with Negative control (Normal Saline)

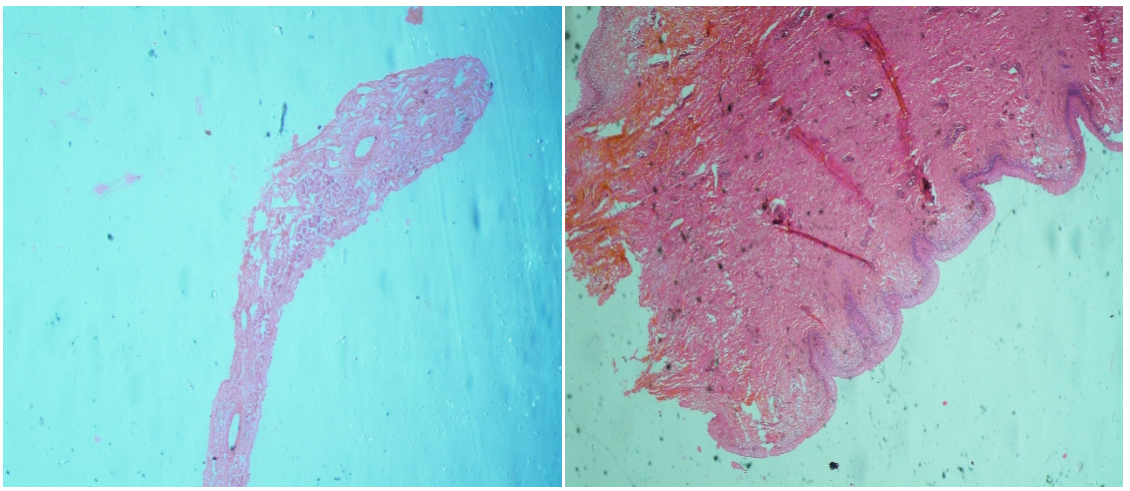
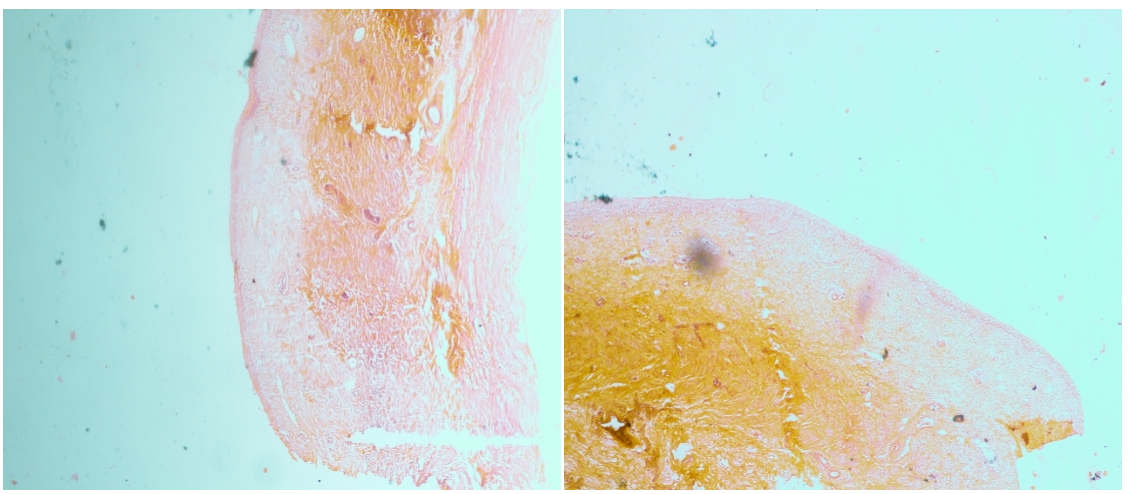


Fig.2(c): Histopathological images of nasal mucosa treated with Formulations:



TABLES

Table 1: Solubility data of Olanzapine in various Excipients:

Oil/surfactant/cosurfactant	Solubility mg/ml	Oil/surfactant/cosurfactant	Solubility mg/ml
Captex/miglyol	<10 mg/ml	Labrafil M	80 mg/ml
Capryol 90	120 mg/ml	Acconon CC6	100 mg/ml
Soyabean oil	<10 mg/ml	PEG 200	100 mg/ml
Olive oil	<10 mg/ml	PEG 400	100 mg/ml
Isopropyl Myristate	<10 mg/ml	Kolliphor HS (Solutol HS)	110 mg/ml
Ethyle oleate	<10 mg/ml	Tween 20	120 mg/ml

Table 2: Size & Zeta potential of selected batches

Batch No.	%Oil	%S/Cos	%Water	Size (nm)	Zeta Potential(mV)	%Transmittance
1	5	30	65	125.3	-2.42	99.3%
2	5	35	60	141.0	-2.30	98.0%
3	10	35	55	135.6	-4.59	99.7%
5	10	40	50	146.3	-5.36	98%
6	15	30	55	135.6	-6.36	97.8%
7	15	40	45	141.3	-5.59	99.9%
8	15	45	40	136.8	-5.21	99.7%
9	20	40	40	125.3	-6.32	100.0%
10	20	50	30	121.5	-8.12	100.0%
11	20	60	20	123.6	-8.06	99.4%
12	25	60	15	142.6	-7.25	75.8%

Table 3: Optimization of experimental parameter for Olanzapine microemulsion

Parameter	**rpm**	**Size(nm)**
Stirring speed	Low	141.3 ± 4.5
	Medium	125.3 ± 4.1
	High	146.3 ± 3.9
Parameter	**Time(Min)**	**Size(nm)**
Stirring time	10	121.3 ± 3.9
	20	136.8 ± 4.8
	30	147.9 ± 3.3

Table 4: Optimized formula for microemulsion

Ingredient	**Olanzapine Microemulsion (%w/w)**
Drug	15mg/ml
Oil	20 %w/w
Surfactant	25 %w/w
Co surfactant	25 %w/w
aq. phase	30 %w/w

Table 5: Characterization of optimized drug loaded microemulsion

Tests	Olanzapine Microemulsion
% Transmittance (256 nm)	99.87±0.23
% Assay	99.39±0.68
Globule Size (nm)	123.4 ± 1.3
Zeta Potential (mV)	-8.34 ± 0.85
pH	6.17 ± 0.8
Viscosity(cps)	45.56 ± 1.0
Conductivity(µs/cm)	0.3±0.09

Table 6: Stability study of Olanzapine microemulsion

Temperature	Globule Size(nm)		%Transmittance		Phase Separation		% of Assay	
	After 1 Month	After 6 months	After 1 Month	After 6 months	After 1 Month	After 6 months	After 1 Month	After 6 months
2^0C- 8^0C	119.4	HAZZY	99.25	HAZZY	No	yes	99.12	HAZZY
Room temp	116.9	123.5	99.15	99.05	No	No	99.35	99.41
Elevated temp. (50^0C$\pm 2^0$C)	164.0	188.3	99.00	99.75	No	No	98.42	98.66

Values are expressed as mean ±SEM of three estimation.

ACKNOWLEDGEMENT

Authors are thankful to Cadila Healthcare Ltd. (India) for providing Olanzapine pure powder, Physics department, Life sciences department (M.K.Bhavnagar University, Bhavnagar) for kind support and providing facility to carry out analytical work.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th& Jan. 6th, 2016)

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Formulation and Evaluation of Bilayer Tablets of Repaglinide

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ABSTRACT

The aim of formulation of bilayer tablets of repaglinide was to provide immediate action after oral administration of dosage form and sustaine release of repaglinide for prolong period of time. Here 7.5% SSG was give fast release of immediate release layer while 43.21mg of HPMC K15M and 39.23mg of HPMC K100M was give sustaine release till 480 min at zero order release rate.

SUMMARY

Repaglinide bilayer tablets were formulated & evaluated for type 2 diabetic patients.

Keywords:Repaglinide, Bilayer Tablets, Sustaine release tablets, modified release tablets

INTRODUCTION

Bilayer tablet is suitable for sequential release of two drugs in combination, separate two incompatible substances an also for sustained release tablet in which one layer is immediate release as initial dose and second layer is maintenance dose(*1, 2*).
The main objective of bilayer tablet is to improve effectiveness of drug as well as patient compliance.

MATERIALS AND METHODS

Materials:

Rapaglinide was received as a gift sample from Torrent Research Centre, Microcrystalline cellulose and Mg- stearate purchased from Seva Fine Chemicals, Sodium starch glycolate purchased from Thrine Enterprise, Hydroxyl propyl methyl cellulose(HPMC- K15M & K100M) purchased from Astron Chemicals Pvt. Ltd, Dibasic calcium phosphate Dehydrate purchased from Loba chemical Pvt. Ltd.

Methodology:

1. Determination of λ_{max} for Repaglinide:

A 100 µg/ml solution of repaglinide in 0.1 N HCl (pH 1.2) and phosphate buffer (pH 6.8) were scanned in UV range between 200 to 400 nm and λ_{max} of repaglinide was measured.

2. Procedure of Calibration Curve:

Weighed quantity of repaglinide (100 mg) was place in 1000 ml of standard volumetric flask and made up the volume with 0.1 N HCl up to 1000 ml. The stock solution gave 100 µg/ml concentration of repaglinide. Aliquots of 2, 4, 6, 8 up to 30 ml of stock solution pipette into 100 ml standard volumetric flasks and final volume were adjusted up to 100 ml with 0.1N HCl gave the concentration of 2, 4, 6, 8, up to 30µg/ml. The absorbance was measured at 243 nm in UV spectrophotometer against 0.1 N HCl as blank.

Fig. 1Calibration Curve of Repaglinide in 0.1N HCl

Table 1. Calibration Curve of Repaglinide

Then same procedure was conducted for phosphate buffer absorbance was measured at 243 nm.

Fig.2Calibration Curve of Repaglinide in Phosphate Buffer pH 6.8

Table 1. Calibration Curve of Repaglinide

3. Fabrication of Immediate Release Layer(*3*).

Dispensing and shifting: Repaglinide, Microcrystalline Cellulose and sodium starch glycolate were accurately weighted and shifted through 40 # sieve. Magnesium Stearate was accurately weighted and sifted through 60 # sieve.

Dry mixing of drug, polymer and excipients: Repaglinide was mixed geometrically with Microcrystalline Cellulose and sodium starch glycolate for 15minutes in double lined polythene bag.

Lubrication: The above blend was lubricated by mixing with Magnesium Stearate in double lined polythene bag for 5 minutes.

Table 2. Formulation of Bilayer Tablets by Factorial Design (3^2)

4. Fabrication of Sustained Release Layer(*3*).

Dispensing and shifting: Repaglinide, HPMC K15M, HPMC K100M and calcium phosphate dibasic were accurately weighted and shifted through 40 # sieve. Magnesium Stearate and talc were accurately weighted and sifted through 60 # sieve.

Dry mixing of drug, polymer and excipients:Repaglinide was mixed geometrically with HPMC K15M, HPMC K100M and calcium phosphate dibasic for 15 minutes in double lined polythene bag.

Lubrication: The above blend was lubricated by mixing with Magnesium Stearate and talc in double lined polythene bag for 5 minutes.

Table 2. Formulation of Bilayer Tablets by Factorial Design (3^2)

Steps For Compression:
Filling powder blend of control release matrix layer in to dies.
Slightly compressed control release powder blend using 7 mm flat punch.
Ejection of upper punch.
Add immediate release layer powder blend above control release layer.
Compression of both layers.
Ejection of bilayer tablet.

5. Dose Calculation of Repaglinide for Sustaine Release Layer:
The total dose of repaglinide for a CR layer was calculated by the following equation using available pharmacokinetic data(*2, 4, 5*).
D Total = D_{IR} (1+0.693 × t /$t_{1/2}$)
Where, D Total = Total dose of drug in bilayer tablet (IR dose + CR dose);
D_{IR} = dose of the immediate release layer (IR dose = 0.5 mg);
t = time in hours during which the control release is desired (8 hours)
$t_{1/2}$= half-life of the drug in hours. (1 hour)
From above equation total dose of drug was found to be 3.272 mg. So dose of CR layer was found using following equation ,
Dose of sustaine release layer = D Total - D_{IR}
From above equation dose of control release layer was found to be ≈ 3.0 mg (2.772 mg).
So 3 mg dose of repaglinide was selected for control release layer.

6. Evaluation Parameters:
6.1 Tablet Diameter(*6*).
Five tablets were taken and their diameter was recorded using digital verniercalipers.

6.2 Tablet Thickness(*6*).
Five tablets were taken and their thickness was recorded using digital verniercalipers.

6.3 Tablet Hardness(*6*).
Hardness of tablet is defined as the force applied across the diameter of the tablet in the order to break the tablet. Five tablets were randomly selected from each formulations and the hardness was noted using Pfizer hardness tester.

6.4 %Friability(*6*).
Friability is measured of mechanical strength of tablets. Roche friabilator was used to determine the friability by following procedure. A preweighed tablet was placed in the friabilator. Friabilator consist of a plastic-chamber that revolves at 25 rpm, dropping those tablets at a distance of 6 inches with each revolution. The tablets were rotated in the friabilator for at least 4 minutes (For total 100 rotation). At the end of test tablets was dusted and reweighed, the loss in the weight of tablet is the measure of friability and is expressed in percentage as below equation.

Percentage Friability= [(Initial Weight – Final Weight) × 100]/Initial Weight

6.5 Uniformity of weight(*6*).
I.P. procedure for uniformity of weight was followed, Maximum % different of weight of tablet allowed was given in below table according to I.P.

6.6 Drug Content(*6*).

Accurately weighed quantity of the powder (Powder of crushed tablets) equivalent to 4 mg of the repaglinide was transferred to 100 ml volumetric flask. 50 ml of 0.1N HCl was added. Mix with the aid of ultrasound for 10 min, and then the volume was made up to 100 ml with the same solution, mixed solution was filtered and then examined under U.V Spectrophotometer at 243nm.

6.7 Swelling Index(*6.*)

The extent of swelling was measured in terms of % weight gain by the tablet. Three tablets from each formulation was kept in a individual petridish containing 0.1N HCl for initial 2 hr and then phosphate buffer till end of the study. Tablets were withdrawn, dried with tissue paper, and weighed at interval of one hour. Then weights of the tablets were noted, and the process was continued at one hour interval till the end of 8 hr. Percentage weight gain by the tablets was calculated by following formula.

$$\% \text{ S.I.} = \{(Mt-Mo) / Mo\} \text{ X } 100$$

Where, % S.I. = % swelling index, Mt = weight of tablet at time t (hr) and Mo = weight of tablet at zero time.

6.8*In vitro* Dissolution(*6*).

Drug release studies were carried out using USP dissolution test apparatus type 2 (paddle). Initial studies was carried out in 700 ml of 0.1N HCl (pH 1.2) for 2 h. After 2 hour 0.2M tri-sodium phosphate was added to the dissolution media and the pH adjusted to 6.8 then make up volume up to 900 ml. The study at a pH 6.8 was continued till end of study.

RESULTS AND DISCUSSION

1. Evaluation of F1-F9

1.1 Diameter

Tablet diameter of all formulation batches were found in 6.86-6.89 mm range
Table. 3 Tablet Diameter, Thickness, Hardness, %Friability

1.2 Thickness

Tablet thickness of all formulation batches were found in 3.94-3.99 mm range.
Table. 3 Tablet Diameter, Thickness, Hardness, %Friability

1.3 Hardness

Tablet hardness of all formulation batches were found in 4.12-60 kg/cm^{2}range.
Table. 3 Tablet Diameter, Thickness, Hardness, %Friability

1.4 % Friability

Tablet % friability of all formulation batches were found in 0.32-0.65% range.
Table. 3 Tablet Diameter, Thickness, Hardness, %Friability

1.5 Uniformity of Weight

Tablet weight uniformity of all formulation batches were found in 199.85-200.25mg range
Table 4. Uniformity of Weight & Drug Content

1.6 Drug content

Tablet drug content of all formulation of all batches were found in 97.53-98.96% range.
Table 4. Uniformity of Weight & Drug Content

1.7 % Swelling Index

Tablet % swelling index of all formulation for 8 hr. were given in table no.5.
Table 5. % Swelling Index

1.8 % Cumulative Drug Release(%CDR)

Tablet % cumulative drug release of all formulation batches were sustained for 8 hr.
Table 6 % Cumulative Drug Release

2. Optimization of Bilayer Tablet

Optimized batch of bilayer tablets was derived by use of design expert software, independent and dependent variables are selected by 3 level 2 factor factorial design.
Table 7. Selection of Independent & Dependent variable

2.1 Data Input of Software:

Details of data inputted in software were given in following table.
Table 8.Data Input of Software

2.2 Data Output of Software:

Output data of software include polynomial equation for all dependent variables, effect independent variables on dependent variables, desirability of dependent variables and overlay contour plot for optimized region of independent variables.

2.2.1 Polynomial Equation:

Polynomial equation for % cumulative drug release at 15 min (Y1) for coded factor was as below

Y1 = 14.01-0.47(X1)-0.71(X2) +0.075(X1) (X2)-0.032(X1) 2+0.078(X2) 2

Y2 = 345.67+77.33(X1) +102.67(X2) -4.25(X1) (X2) +19.00(X1) 2+9.00(X2) 2

Y_3 = 180.33+49.50(X1) +63.33 (X2) +4.25 (X1) (X2) -2.50(X1) 2+3.00(X2) 2

Where X1 = coded value of HPMC K15M
X2 = coded value of HPMC K100M

Standard error and R2 value of above equations were tabulated in table 9
Table 9.Statistical Evaluation of Polynomial Equations.
R^2 value of all equations were found in range of 0.9876-0.9956 which is nearest to 1 indicating that values of variables were exactly fitted into all polynomial equations.

Significance p-value for all main effects, interaction effects and quadratic effects were mentioned in below table.
Table 10. p-Value of Coefficient
Above table (Table 10) indicates that p-value of X1 and X2 was less than 0.005. So, main effect of X1 factor and main effect of X2 factor were significant terms in polynomial equations.

2.2.2 Effect of Independent Variables on Dependent Variables:
Effect of independent variables on dependent variables was observed by contour plot and 3-D surface response.
Effect on Percentage Cumulative Drug Release at 15 Min (Y1):
Effect of independent variables on %cumulative drug release at 15 min (Y1) was observed on contour plot and 3-D surface response.
Fig 3. Contour Plot of Effect on % CDR at 15 Min
Fig 4. 3-D Surface Response on % CDR at 15 Min
From the figure (Fig. 3 & 4) it was concluded that as amounts of HPMC K15M and HPMC K100M increased % cumulative drug release at 15 min was decreased.
Effect on Time Requires to 90% Cumulative Drug Release (Y2):
Effect of independent variables on time requires to 90% cumulative drug release (Y2) was observed on contour plot and 3-D surface response.
Fig. 5. Contour Plot of Effect on Time Requires To 90% CDR
Fig. 6 3-D Surface Response of Effect on Time Requires to 90% CDR
From the figure (Fig. 5 & 6) it was concluded that as amounts of HPMC K15M and HPMC K100M increased time require to 90 % cumulative drug release was also increased.
Effect on Time Requires to 50% Cumulative Drug Release (Y3):
Effect of independent variables on time requires to 50% cumulative drug release (Y3) was observed on contour plot and 3-D surface response.
Fig. 7. Contour Plot of Effect on Time Requires to 50% CDR
Fig. 8. 3-D Surface Response of Effect on Time Requires to 50% CDR
From the figure, (Fig. 7 & 8) it was concluded that as amounts of HPMC K15M and HPMC K100M increased time require to 50 % cumulative drug release was also increased.

2.2.3 Desirability:
Desirability for optimized level of independent variables was observed on contour plot and 3-D surface response.
Fig. 9. Contour Plot of Desirability of Dependent Variables
Fig. 10. 3-D Surface Response of Desirability of Dependent Variables
From the figure (Fig. 9 & 10) it was concluded that desirability was depends on amountsof both independent variables (HPMC K15M and HPMC K100M).

2.2.4Overlay Contour Plot for Optimization:

Overlay contour plot obtained by software was important tool for optimization of formulation. Overlay contour plot was described in following figure.

Fig. 11. Overlay Contour Plot

In overlay plot area covered by yellow color indicate optimized region of independent variables which give optimum response. (Fig. 11)

2.3 Optimization from Overlay Contour Plot:

In overlay plot area covered by yellow color indicates optimized region of independent variables which give optimum response.

Fig. 12. Overlay Contour Plot with Check Point

One point in area covered by yellow color of overlay plot was selected and flag was added for obtain detail about that point like level of independent variables and predict response for dependent variables. (Fig. 12)

In overlay plot flag of selected point was give optimize level for independent variables to achieve optimum response of dependent variables. From the detail of flag 43.21 mg HPMC K15M and 39.23 mg HPMC K100M was selected for formulation of optimized batch of bilayer tablet (Check point batch).

3. Evaluation of Optimized Batch:

3.1 Diameter

Tablet diameter of optimized batch was found to be6.90±0.01 mm.

Table. 11.Tablet Diameter, Thickness, Hardness, %Friability, Weight Uniformity, %Drug Content

3.2 Thickness

Tablet thickness of optimized batch was found to be 3.99 ±0.02mm.

Table. 11.Tablet Diameter, Thickness, Hardness, %Friability, Weight Uniformity, %Drug Content

3.3 Hardness

Tablet hardness of optimized batch was found to be 4.08± 0.084 kg/cm^2.

Table. 11.Tablet Diameter, Thickness, Hardness, %Friability, Weight Uniformity, %Drug Content

3.4 % Friability

Tablet % friability of optimized batch was found to be 0.63±0.03%.

Table. 11.Tablet Diameter, Thickness, Hardness, %Friability, Weight Uniformity, %Drug Content

3.5 Uniformity of Weight

Tablet weight uniformity of optimized batch was found to be200.00±0.97mg.

Table 11.Tablet Diameter, Thickness, Hardness, %Friability, Weight Uniformity, %Drug Content

3.6 Drug content

Tablet drug content of optimized batch was found to be97.92± 0.10%.

Table 11.Tablet Diameter, Thickness, Hardness, %Friability, Weight Uniformity, %Drug Content

3.7 % Swelling Index

Tablet % swelling index of optimized batch for 8 hr. were given in table no.12.

Fig. 13. % Swelling Index of Optimized Batch

Table 12. % Swelling Index

3.8 % Cumulative Drug Release(%CDR)

Tablet % cumulative drug release of optimized batch were sustained for 8 hr.

Fig. 14. % Cumulative Drug Release of Optimized Batch

Table 13. % Cumulative Drug Release

CONCLUSION

The present research work was carried out to develop bilayer tablets of Repaglinide. Immediate release layer was developed by use of sodium starch glycolate as super disintegrating agent and control release layer was prepared by use of HPMC K15M and HPMC K100M polymers.

The research demonstrated that initially burst release was observed due to presence of sodium starch glycolate in immediate release layer and Sustained release action was observed due to presence of HPMC K15M and HPMC K100M in control release layer.

Hence it was concluded that bilayer tablet showed an immediate release effect to provide loading dose of drug followed by Sustained release for 8 hours indicating promising potential for Repaglinide drug administration. In short, success of research work on bilayer tablet of Repaglinide beneficial to improve the patient compliance.

FIGURES

Fig. 1. Calibration Curve of Repaglinide in 0.1N HCl

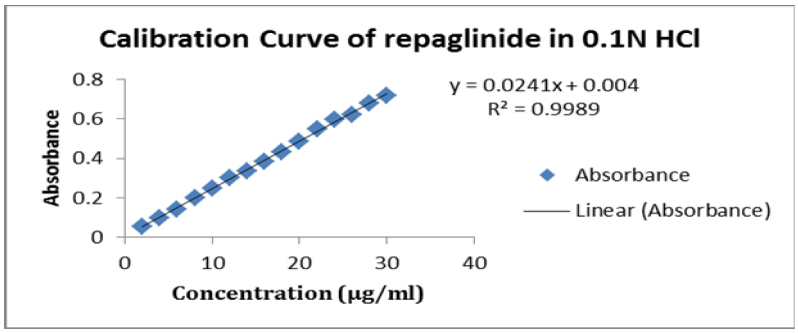


Fig. 2. Calibration Curve of Repaglinide in Phosphate Buffer pH 6.8

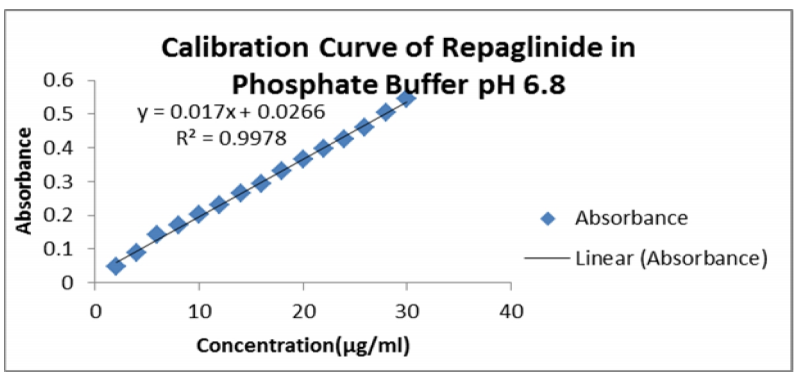


Fig. 3. Contour Plot of Effect on % CDR at 15 Min

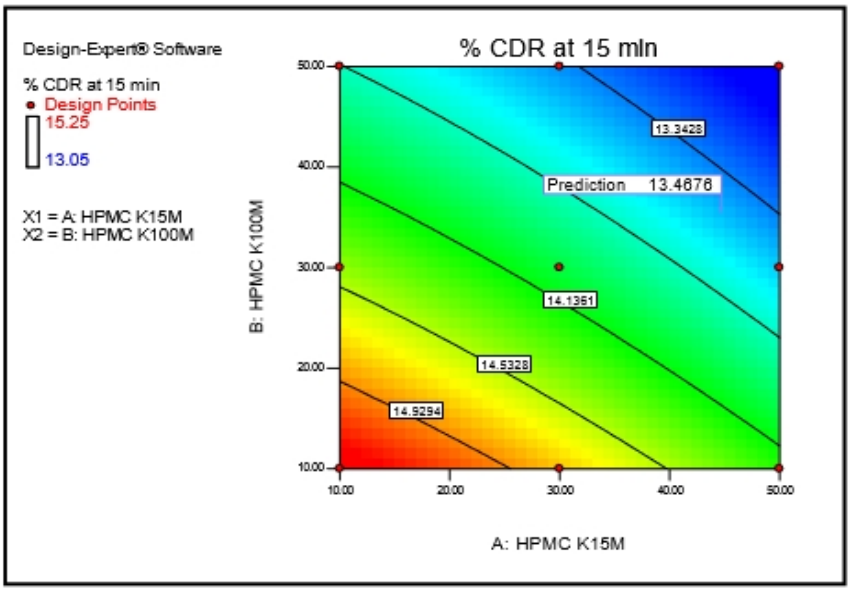


Fig. 4. 3-D Surface Response on % CDR at 15 Min

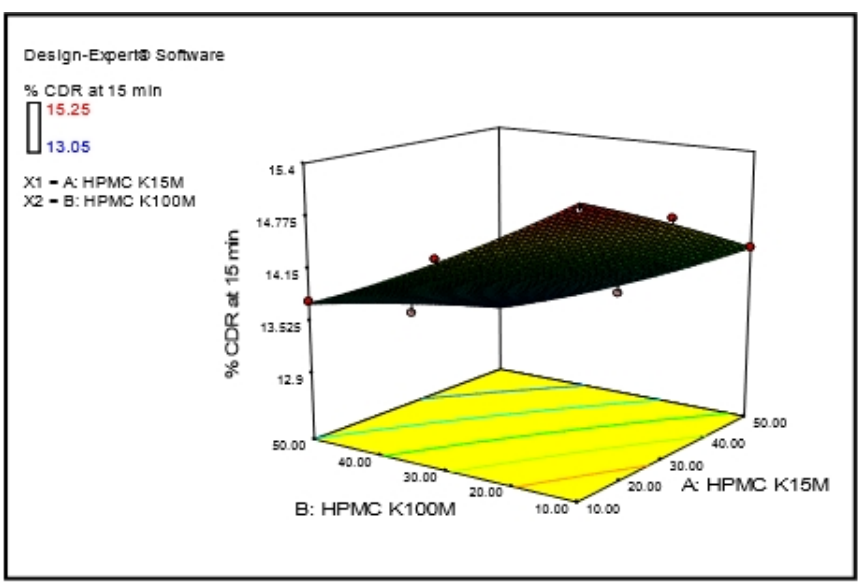


Fig. 5. Contour Plot of Effect on Time Requires To 90% CDR

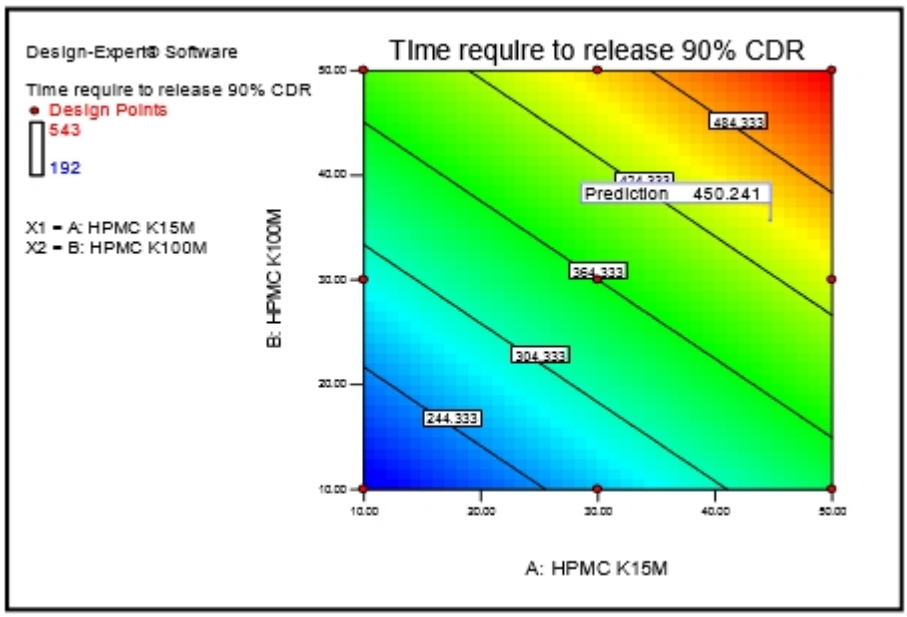


Fig. 6. 3-D Surface Response of Effect on Time Requires to 90% CDR

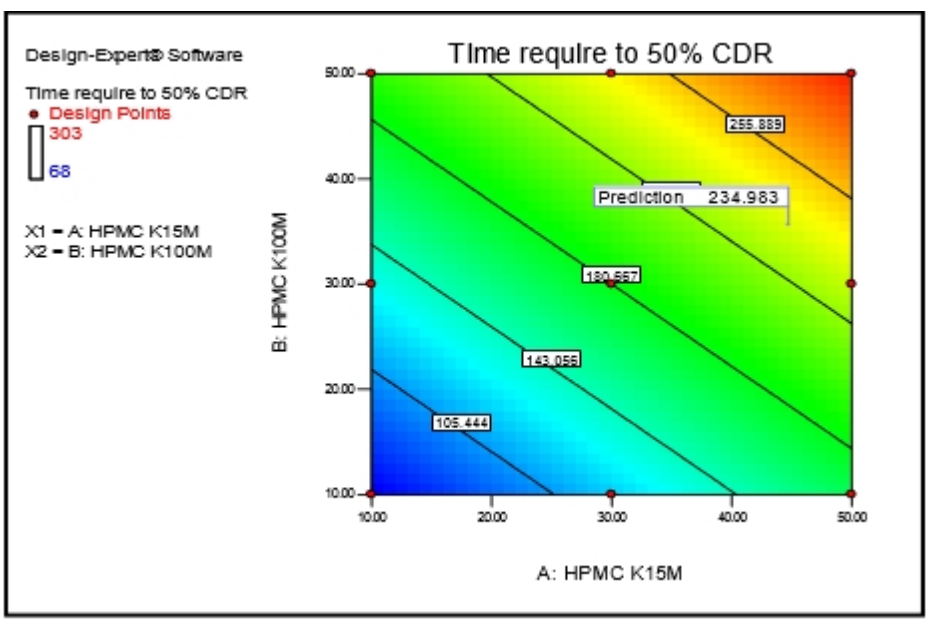


Fig. 7. Contour Plot of Effect on Time Requires to 50% CDR

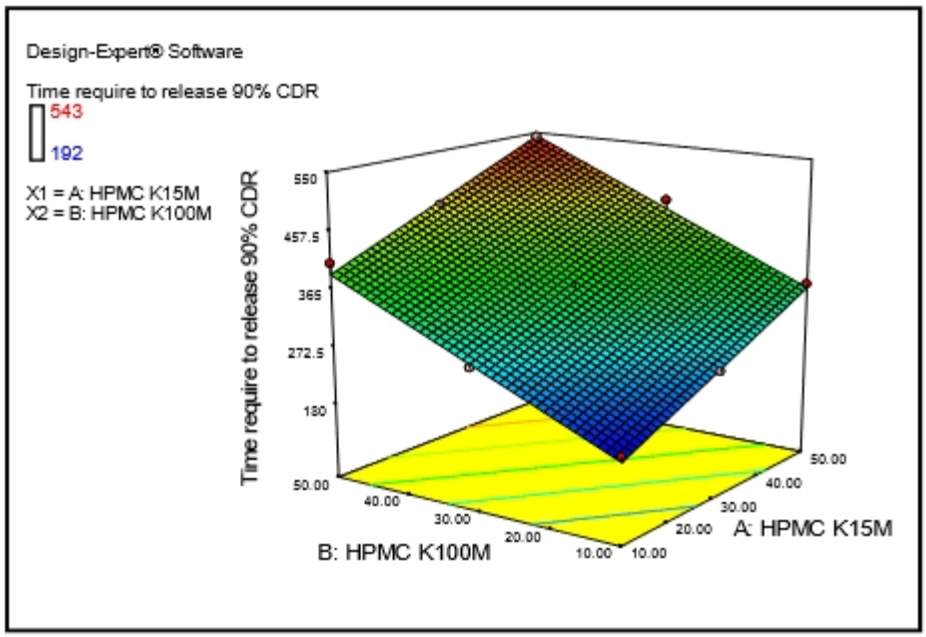


Fig. 8. 3-D Surface Response of Effect on Time Requires to 50% CDR

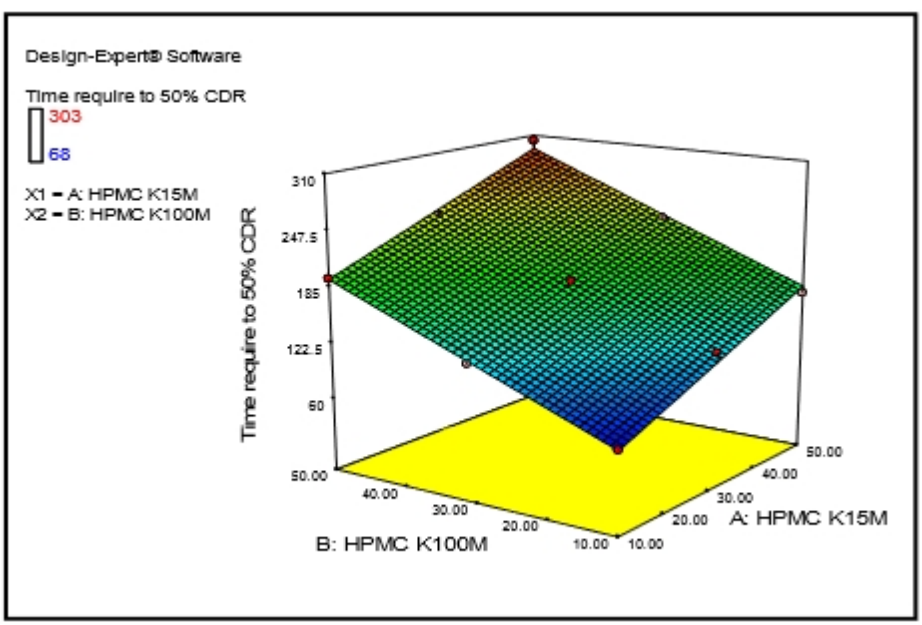


Fig. 9. Contour Plot of Desirability of Dependent Variables

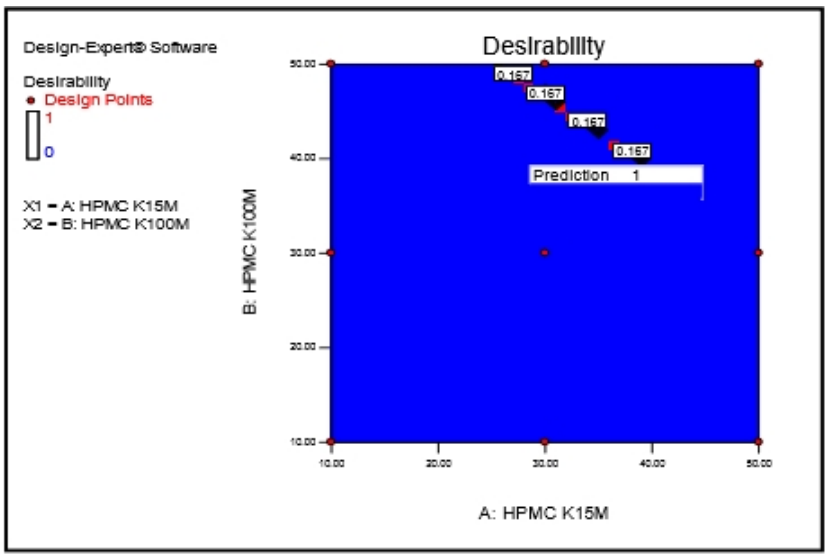


Fig. 10. 3-D Surface Response of Desirability of Dependent Variables

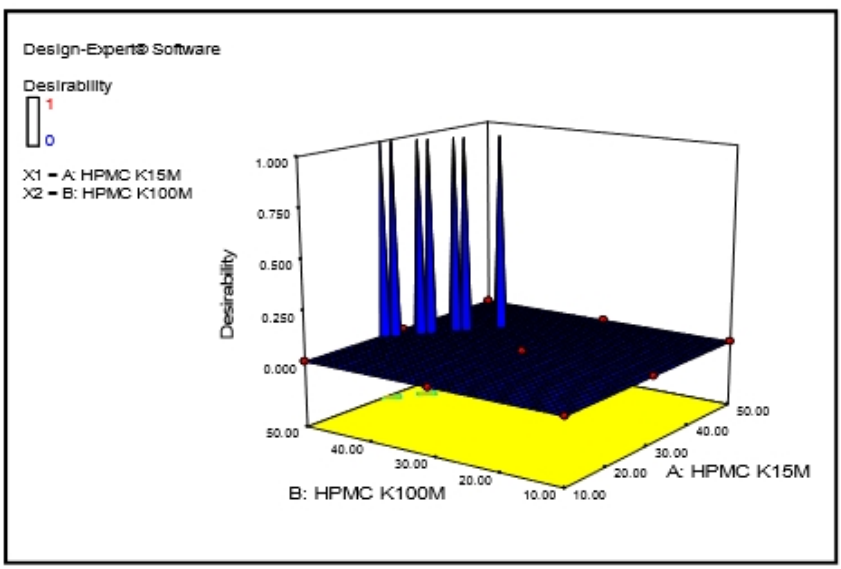


Fig. 11. Overlay Contour Plot

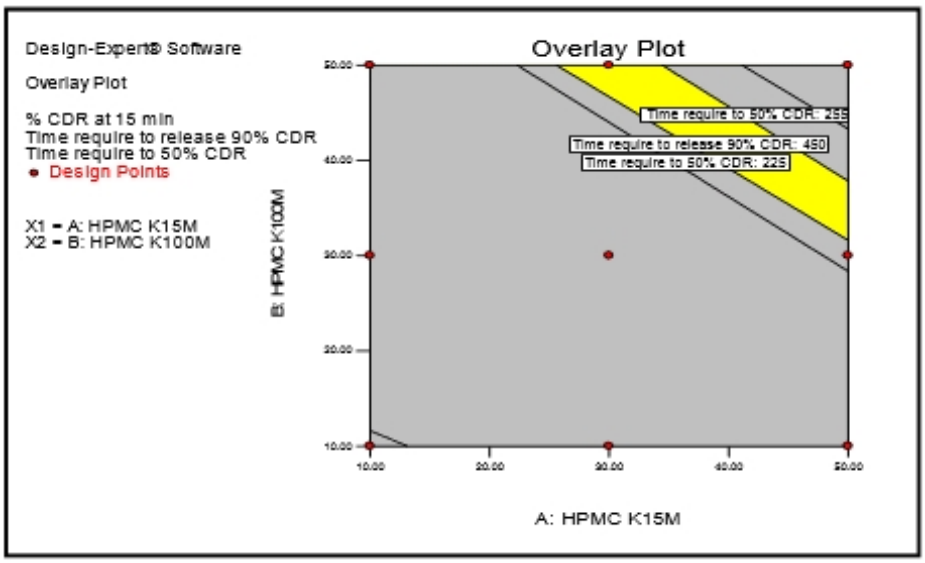


Fig. 12. Overlay Contour Plot with Check Point

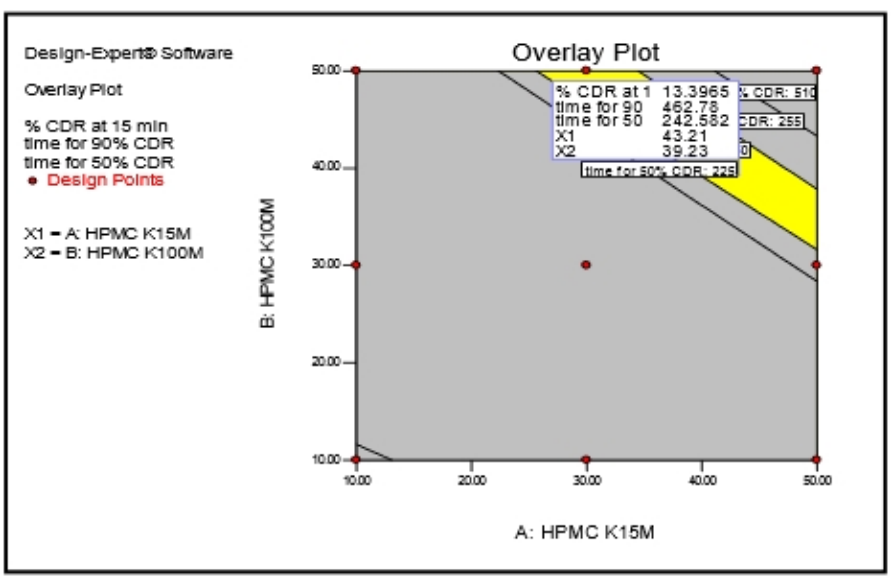


Fig. 13. Percentage Swelling Index of Optimized Batch

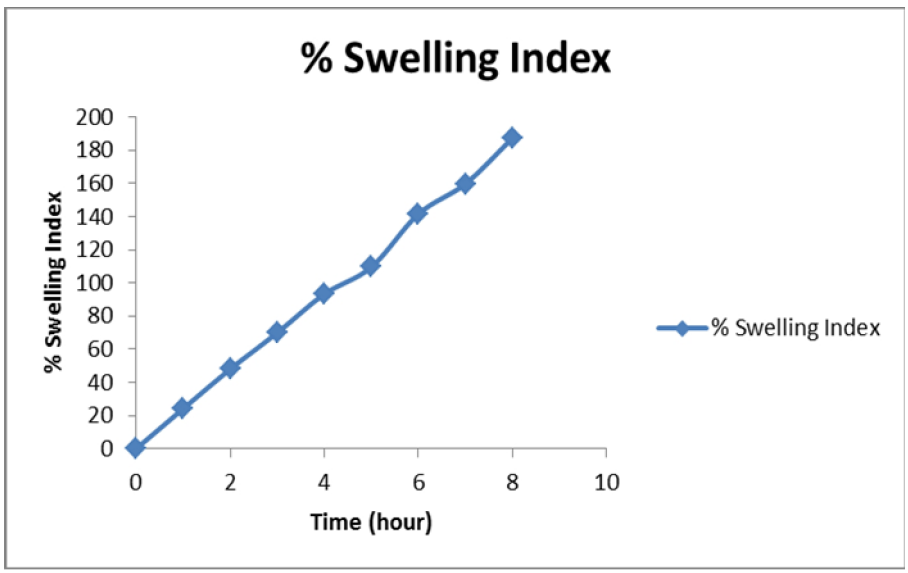
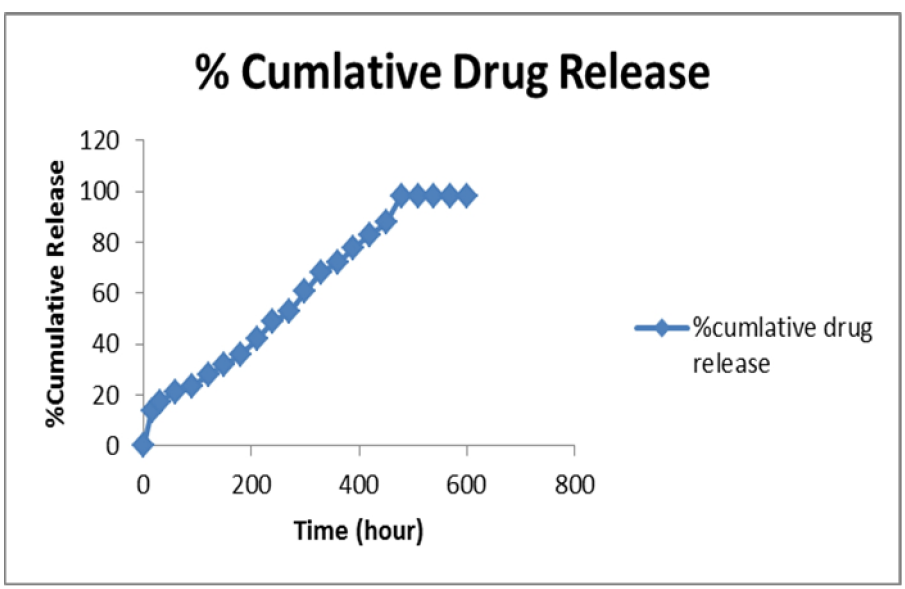


Fig. 14. % Cumulative Drug Release of Optimized Batch



TABLES

Table 1. Calibration Curve of Repaglinide

Concentration(µg/ml)	Absorbance* in 0.1 N HCl	Absorbance* in Phosphate Buffer pH 6.8
2	0.053±0.005	0.047±0.005
4	0.096±0.008	0.090±0.004
6	0.144±0006	0.142±0.004
8	0.199±0.009	0.171±0.007
10	0.250±0.005	0.202±0.007
12	0.299±0.007	0.232±0.005
14	0.336±0.004	0.266±0.006
16	0.382±0.008	0.293±0.005
18	0.434±0.006	0.333±0.006
20	0.486±0.006	0.365±0.006
22	0.607±0.009	0.399±0.007
24	0.727±0.006	0.427±0.006
26	0.619±0.001	0.461±0.007
28	0.680±0.001	0.505±0.007
30	0.718±0.007	0.545±0.007

*The values represent mean ± S.D.

Table 2. Formulation of Bilayer Tablets by Factorial Design (3^2)

Ingredients	Quantity(mg)								
	F-1	**F-2**	**F-3**	**F-4**	**F-5**	**F-6**	**F-7**	**F-8**	**F-9**
Immediate Release(IR)-LAYER									
Repaglinide	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
SSG	6	6	6	6	6	6	6	6	6
MCC	72	72	72	72	72	72	72	72	72
Mg. Stearate	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
Sustained Release(SR)-LAYER									
Repaglinide	3	3	3	3	3	3	3	3	3
HPMC K15M	10	30	50	10	30	50	10	30	50
HPMC K100M	10	10	10	30	30	30	50	50	50
Calcium Phosphate Dibasic	95	75	55	75	55	35	55	35	15
Talc	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
Mg. Stearate	05	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Total Weight	200	200	200	200	200	200	200	200	200

Table 3. Tablet Diameter, Thickness, Hardness, %Friability

Formulation	Diameter*(mm)	Thickness*(mm)	Hardness*(kg/cm^2)	%Friability*
F-1	6.89±0.01	3.96±0.01	4.48±0.130	0.51±0051
F-2	6.87±0.02	3.98±0.01	4.52±0.084	0.54±0.029
F-3	6.89±0.03	3.99±0.01	4.34±0.114	0.39±0.03
F-4	6.88±0.01	3.99±0.01	4.12±0.084	0.42±0.029
F-5	6.89±0.01	3.97±0.01	4.48±0.130	0.51±0.051
F-6	6.88±0.02	3.98±0.02	4.50±0.071	0.51±0.051
F-7	6.89±0.01	3.94±0.01	4.12±0.084	0.39±0.029
F-8	6.87±0.02	3.97±0.01	4.60±0.122	0.51±0.051
F-9	6.86±0.03	3.97±0.02	4.20±0.071	0.36±0.051

*The values represent mean ± S.D

Table 4. Uniformity of Weight & Drug Content

Formulation	Uniformity of Weight* (mg)	Drug Content*(%)
F-1	200.25±1.33	97.53±0.16
F-2	200.10±1.17	97.92±0.10
F-3	200.20±1.76	98.72±0.10
F-4	199.55±1.67	97.85±0.06
F-5	199.75±1.71	98.40±0.06
F-6	200.15±0.88	98.96±0.10
F-7	199.80±1.67	97.67±0.06
F-8	199.85±1.36	96.56±0.10
F-9	200.00±0.97	97.01±0.06

Table 5. % Swelling Index

Time (Hour)	% Swelling Index								
	F-1*	**F-2***	**F-3***	**F-4***	**F-5***	**F-6***	**F-7***	**F-8***	**F-9***
0	0	0	0	0	0	0	0	0	0
1	13.05± 0.48	16.15± 0.44	18.61± 0.62	16.66± 0.83	21.17± 1.01	22.23± 1.45	25.90± 0.94	25.56± 1.15	26.18± 1.03
2	26.90± 1.90	31.75± 0.97	38.27± 1.07	34.46± 1.97	39.83± 1.16	46.93± 1.03	45.40± 1.09	48.88± 0.63	52.09± 1.51
3	39.89± 1.47	47.90± 1.82	47.06± 0.39	49.15± 0.42	49.49± 1.69	69.19± 1.86	55.46± 1.15	77.55± 1.06	77.04± 0.91
4	50.41± 0.41	59.45± 1.76	70.83± 0.59	60.94± 1.24	72.23± 1.86	90.03± 1.38	75.56± 1.89	94.18± 1.40	102.7± 1.44
5	60.38± 0.41	76.39± 1.07	83.00± 0.44	78.11± 0.37	86.39± 1.98	113.9± 1.80	90.84± 1.59	118.9± 1.83	121.6± 1.00
6	75.35± 1.55	89.64± 1.67	108.3± 0.87	91.04± 1.21	111.7± 1.54	137.6± 0.99	116.8± 1.58	141.7± 1.01	147.0± 1.48
7	85.08± 0.82	101.6± 1.45	124.5± 1.39	99.72± 0.47	127.0± 1.87	155.0± 0.83	132.5± 0.97	160.2± 0.48	172.7± 1.76
8	101.0± 1.73	118.1± 0.53	144.0± 1.44	119.6± 1.35	149.0± 0.63	182.1± 1.66	156.8± 1.08	188.5± 1.22	206.6± 1.63

*The values represent mean ± S.D.

Table 6. Cumulative Drug Release (CDR)

Time (Min.)	% CDR								
	F-1*	**F-2***	**F-3***	**F-4***	**F-5***	**F-6***	**F-7***	**F-8***	**F-9***
0	0	0	0	0	0	0	0	0	0
15	15.25± 0.42	14.88± 0.10	14.23± 0.77	14.51± 0.37	14.07± 0.09	13.40± 0.14	13.77± 0.35	13.25± 0.33	13.05± 0.45
30	33.48± 0.42	19.73± 0.10	17.85± 0.78	18.38± 0.37	18.26± 0.09	17.50± 0.14	17.54± 0.35	17.49± 0.34	16.04± 0.45
60	46.78± 0.43	25.58± 0.10	25.37± 0.69	25.72± 0.39	23.05± 0.09	21.00± 0.14	21.61± 0.36	22.22± 0.34	17.35± 0.46
90	58.33± 0.44	38.54± 0.10	32.46± 0.80	38.68± 0.38	29.26± 0.10	26.70± 0.14	26.83± 0.36	26.69± 0.35	21.18± 0.47
120	70.06± 0.44	47.26± 0.11	41.57± 0.82	47.41± 0.39	37.94± 0.10	29.40± 0.14	32.01± 0.37	30.44± 0.45	24.64± 0.47
150	81.45± 0.57	56.94± 0.14	48.14± 1.05	59.91± 0.50	43.62± 0.12	35.00± 0.18	38.33± 0.47	34.26± 0.45	29.67± 0.61
180	88.10± 0.58	63.99± 0.14	54.65± 1.06	63.97± 0.50	49.55± 0.13	41.50± 0.19	43.83± 0.48	39.45± 0.45	33.79± 0.61
210	93.03± 0.58	72.96± 0.14	62.16± 1.07	70.91± 0.51	56.57± 0.13	45.60± 0.19	51.20± 0.48	45.43± 0.46	37.62± 0.62
240	93.03± 0.59	83.42± 0.14	68.28± 1.08	78.41± 0.51	62.34± 0.13	51.00± 0.19	57.95± 0.49	50.95± 0.46	42.41± 0.63
270	93.04± 0.59	95.24± 0.14	75.28± 1.09	86.46± 0.52	68.80± 0.13	56.30± 0.19	64.14± 0.49	55.35± 0.47	45.04± 0.63
300	93.04± 0.60	95.30± 0.14	80.43± 1.10	96.24± 0.52	72.81± 0.13	61.20± 0.19	70.24± 0.50	60.03± 0.47	49.54± 0.64
330	93.05± 0.60	95.36± 0.14	85.73± 1.11	96.29± 0.53	87.89± 0.13	68.10± 0.20	75.08± 0.50	64.71± 0.48	53.54± 0.65
360	93.07± 0.61	95.38± 0.15	94.23± 1.12	96.33± 0.53	96.31± 0.13	72.80± 0.20	80.86± 0.51	69.91± 0.48	57.19± 0.65
390	93.07± 0.62	95.41± 0.15	94.24± 1.13	96.36± 0.54	96.35± 0.13	78.20± 0.20	86.76± 0.51	74.57± 0.49	61.90± 0.66
420	93.07± 0.62	95.42± 0.15	94.24± 1.14	96.38± 0.54	96.38± 0.14	83.40± 0.20	92.35± 0.52	80.55± 0.49	66.85± 0.67
450	93.09± 0.63	95.48± 0.15	94.23± 1.15	96.41± 0.55	96.41± 0.14	88.30± 0.20	93.39± 0.52	86.75± 0.50	71.81± 0.67
480	93.10± 0.63	95.49± 0.15	94.26± 1.16	96.43± 0.55	96.42± 0.14	98.00± 0.20	92.42± 0.53	96.30± 0.50	78.00± 0.68
510	93.11± 0.64	95.63± 0.15	94.26± 1.17	96.49± 0.56	96.43± 0.14	98.00± 0.21	92.49± 0.53	96.52± 0.51	82.01± 0.68
540	93.18± 0.65	95.57± 0.16	94.30± 1.18	96.41± 0.56	96.43± 0.14	98.10± 0.21	95.77± 0.15	96.78± 0.51	89.14± 0.69
570	93.10± 0.65	95.80± 0.16	94.30± 1.19	96.66± 0.57	96.53± 0.14	98.10± 0.21	95.80± 0.16	96.82± 0.52	96.40± 0.70
600	93.18± 0.66	95.80± 0.16	94.38± 1.20	96.72± 0.58	96.64± 0.14	98.10± 0.21	95.80± 0.16	96.87± 0.52	96.57± 0.70

*The value represent mean ±S.D

Table 7. Selection of Independent & Dependent variable

<table>
<tr><th colspan="4">3 Level 2 Factor Factorial Design</th></tr>
<tr><th colspan="2">Independent Variable</th><th colspan="2">Dependent Variable</th></tr>
<tr><th>Name</th><th>Quantity</th><th>Name</th><th>Indication</th></tr>
<tr><td rowspan="3">HPMC K15m</td><td>10</td><td rowspan="2">Y1</td><td rowspan="2">% Cumulative drug release at 15 min.</td></tr>
<tr><td>30</td></tr>
<tr><td>50</td><td rowspan="2">Y2</td><td rowspan="2">Time require to release 90% CDR</td></tr>
<tr><td rowspan="3">HPMC K100M</td><td>10</td></tr>
<tr><td>30</td><td rowspan="2">Y3</td><td rowspan="2">Time require to release 50%CDR</td></tr>
<tr><td>50</td></tr>
</table>

Table 8. Data Input of Software

Batch	Independent variables				Dependent variables		
	Coded value		Actual value (mg)		Y1 (%CDR)	Y2 (%CDR)	Y3 (%CDR)
	HPMC K15M	HPMC K100M	HPMC K15M	HPMC K100M			
F-1	-1	-1	10	10	15.25	192	68
F-2	0	-1	30	10	14.88	257	128
F-3	+1	-1	50	10	14.23	345	159
F-4	-1	0	10	30	14.51	281	128
F-5	0	0	30	30	14.07	338	182
F-6	+1	0	50	30	13.40	456	226
F-7	-1	+1	10	50	13.77	407	195
F-8	0	+1	30	50	13.25	460	237
F-9	+1	+1	50	50	13.05	543	303

Table 9. Statistical Evaluation of Polynomial Equations

Statistical parameter	Equation of Y1	Equation of Y2	Equation of Y3
Standard error	0.14	12.20	8.96
R^2 Value	0.9876	0.9959	0.9938

Table 10. p-Value of Coefficient

Coefficient	p- value		
	Equation of Y1	Equation of Y2	Equation of Y3
X1	0.0034	0.0026	0.0006
X2	0.0010	0.0002	0.0002
X1*X2	0.3530	0.5361	0.5361
$(X1)^2$	0.7649	0.1149	0.1149
$(X2)^2$	0.4773	0.3734	0.3734

Table 11. Evaluation of Diameter, Thickness, Hardness, %Friability, Weight Uniformity, %Drug Content.

Formulation	Diameter* (mm)	Thickness* (mm)	Hardness* (kg/cm^2)	% Friability*	Weight Uniformity*	% Drug Content*
F-10	6.90± 0.01	3.99 ± 0.02	4.08± 0.084	0.63± 0.03	200.00± 0.97	97.92± 0.10

*The values represent mean ± S.D.

Table 12. Swelling Index

Time (hours)	% swelling index*
0	0
1	24.16±0.55
2	48.04±1.05
3	69.94±0.91
4	93.26±1.44
5	109.60±1.00
6	141.60±1.48
7	159.80±1.76
8	187.40±1.63

*The values represent mean ± S.D.

Table 13. % Cumulative drug release

Time (hour)	% Cumulative Drug Release*
	F-10
0	0
15	13.48±0.29
30	17.33±0.29
60	21.08±0.30
90	23.51±0.30
120	27.97±0.31
150	31.80±0.39
180	36.02±0.40
210	41.91±0.40
240	48.59±0.41
270	52.65±0.41
300	60.55±0.41
330	67.98±0.42
360	71.90±0.42
390	77.71±0.43
420	82.81±0.43
450	87.93±0.44
480	97.92±0.44
510	97.96±0.44
540	98.02±0.45
570	98.03±0.45
600	98.05±0.46

*The values represent mean ± S.D.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

(Proceedings available for download at rku.ac.in/icre)

RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

Formulation and Evaluation of Colon Target Beads of Azathioprine

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ABSTRACT

The present study is planned to provide colon targeted delivery of azathioprine beads using pectin as polysaccharide. The beads were prepared by iontropic gelation method using $CaCl_2$ and pectin in different proportions. The core bead is coated withEudragit S100 to retard the drug release in stomach. Acetone as solvent and triethyl citrates as plasticizer were used as coating media. Beads were evaluated for physical appearance, micromeritics property, and encapsulation efficiency, coating efficiency, dissolution study and compatability studies. The results of compatability study assure that drug is compatible with all ingredients. The results of dissolution study indicate that optimized batch showing prominent drug release in colon region.

SUMMARY

These formulation was made to treat disease in colon.

Keywords: Azathioprine, Colon target drug delivery , Coating.

INTRODUCTION

Inflammatory bowel disease consists of inflammatory disorders of gastrointestinal tract like asUlcerative colitis, Crohn's disease, microscopic colitis, indeterminate colitis, and collagenous colitis. Crohn's disease and ulcerative colitis are the most common disease.[1]Crohn's disease affects the last part of the small intestine and parts of large intestine. Patient may exhibit colitis and perianal disease, gastroduodenal disease,ileocolitis.[2] Ulcerative colitis affects large intestine. Patient may exhibit abdominal pain and bloody diarrhea. [3] Azathioprine is a prodrug which is converted in the active drug by non-enzymatic reaction to 6-mercaptopurine, a purine analog that acts as an antimetabolite interfering with the synthesis of nucleotides, thereby inhibiting T-cell proliferation. Azatihoprine is a non-specific immunosuppresant whose main targets are the bone marrow stem cells. As a immunosuppressant azathioprine is useful for treatment of rheumatoid arthritis, lupus nephritis, Crohn's disease, and other autoimmune diseases[4].

MATERIALS AND METHODS

2.1. Method for Preparation of pectin beads

Pectin beads were prepared by Ionotropic gelation method.

1. Different amounts of pectin were dissolved into distilled water with constant agitation.
2. Drug was added to the pectin solution with constant agitation to prepare uniform dispersion.
3. The dispersions were allowed to stand overnight for removal of air bobbles and socking.
4. Varying concentrations of $CaCl_2$ solution were prepared in distilled water.
5. The different dispersions of pectin and drug were added in different solutions of $CaCl_2$ using innovative assembly.
6. The beads of calcium pactinate formed were allowed to stand for sufficient time into the $CaCl_2$ solution for proper hardening.
7. The formed beads were separated and washed with distilled water and dried. All the batches were prepared at least three times.

2.2. Composition of Colon targeted beads of Azathioprine

In table 2

2.3. Preparation of coating solution

Coating solution was prepared by dissolving different concentrations of Eudragit S100(5%, 10% and 15%) in acetone. 1ml of tri ethyl citrate was used as plasticizer.

2.3.1. Coating method

The coating operation was performed in pan coater. 20gm of beads were taken in coating pan. Hot air flows was started and spray coating solutionwas done on beads at constant rate. During the process of coating.Sample of the coated materials was taken at fixed interval to determine the coating efficiency. Three batches were formulated using different concentration of Eudragit S100 (5%, 10% and 15%).In table 3

2.4. Evaluation Parameters

2.4.1. Physical appearance

Physical appearance of prepared beads was observed visually.

2.4.2. Micromeritics Properties

Bulk Density:

The bulk density was determined by below given equation. Value of all batches is in table 4.

$$\rho_b = \frac{W}{V_b}$$

Bulk density of A1 to A8 batches was found in range from 0.643 to 0.725g/ml.

Tapped Density:

It was determined by the below given equation. Value of all batches is in table 4.

$$\rho_t = \frac{W}{V_t}$$

Tapped density of A1 to A8 batches was found in range from 0.890 to 1.03g/ml.

Hausner's Ratio:

Hausner's ratio is an indirect index of ease of flow of beads .

It is calculated by following formula. Value of all batches is in table 4.

$$H = \frac{\rho_t}{\rho_b}$$

Hausner's ratio of A1 to A8 batches was observed <1.25, which indicate flow property of beads was good.

Compressibility Index:

Compressibility index is determined bythe below given equation. Value of all batches is in table 4.

$$\text{Compressibility index} = 1 - \frac{V}{V_0} \times 100$$

2.4.3. Drug Entrapment Efficiency

Actual drug content of the beads and the theoretical drug content were used to calculate the encapsulation efficiency of beads. The azathioprine loaded beads (50 mg) were crushed in a glass mortar-pestle and extracted with 100 ml of Phosphate buffer of pH-7.4 for 1 hr by stirring on magnetic stirrer and filtered it. The filtrate was analyzed using UV visible double beam spectrophotometer at the wavelength of 279nm. Result of batches A1 to A8 is in table 5.

$$\%\text{Entrapment Efficiency} = \frac{\text{Actual drug content}}{\text{Theoritical drug content}} \times 100$$

2.4.4. Scanning Electron Microscopy

The external morphology of the beads was studied by scanning electron microscopy. The sample batches were prepared by mounting bead particles on a double adhesive tape stuck to an aluminum stub. The stubs are then coated with gold palladium under an argon atmosphere using a gold sputter module in a high-vacuum evaporator.

2.4.5. Coating Efficiency

Coating efficiency can be determined by calculating beads weight at some interval of time during coating. Observed results were in table 8.

$$\%\text{Coating efficiency} = \frac{W_t}{W_p} \times 100$$

Where, W_t= Theoritical weight gain of beads

W_p= Practical weight gain of beads

2.4.6. In Vitro Drug Release Study:

Dissolution study was first carried out in HCl buffer pH-1.2 for 2 hours. Then HCl was replaced with phosphate buffer pH-6.8 and tested for 3 hours. Then again dissolution medium was replaced with phosphate buffer pH-7.4 and tested. Volume of dissolution medium is taken 900ml for every pH.

2.4.7. Compatibility studies

Differential Scanning Calorimetry (DSC)

The molecular state of the drug evaluated by performing DSC analysis of pure drug azathioprine and mixture of azathioprine-pectin using a differential scanning calorimeter (DSC-60, Shimadzu).

The samples were heated in sealed aluminum pans at temperature range of 10^0C–350^0C and constant rate of 10.0^0C/min under nitrogen purge of 20 ml/min. [5]

Fourier Transform Infrared Spectroscopy (FTIR)

The compatibility study between pure drug azathioprine and pectin was carried out using FTIR spectrophotometer. The scanning was performed in the range of 400 to 4000 cm^{-1} with 1 cm^{-1} resolution.

RESULTS AND DISCUSSION

3.1. Physical appearance

Physical appearance of prepared beads was observed visually. The figure show the physical appearance of beads prepared with different concentrations (5%, 7.5%,) of pectin. Both batches observed spherical shape beads but as the viscosity increase size of beads was increase. 7.5% pectin solution beads have slight larger size than 5% pectin solution beads. Therefore it can be concluded that viscosity of pectin solution affects the size and shape of azathioprine beads. See figure 1 and 2 for physical appearance of pectin beads of 5% and 7.5% respectively.

3.2. Scanning Electron Microscopy

The external morphology of the beads was studied by scanning electron microscopy. The samples batch B2 were prepared by mounting bead particles onto a double adhesive tape stuck to an aluminum stub. The stubs are then coated with gold palladium under an argon atmosphere using a gold sputter module in a high-vacuum evaporator. The coated samples were randomly scanned and photomicrographs were taken at different magnification with a scanning electron microscope.

From the photomicrograph of SEM it showed uniform coating in figure 4,figure 5 and figure 6.Photomicrograph was captured at 407X and 203X magnifications. That shows uniform coating of Eudragit S100 at whole surface of the beads.

3.3. In Vitro Drug Release Profile

%Drug release was observed wide in range from 66.64% to 83.95%. From the %drug release profile observed that almost 20% drug was release at stomach pH 1.2. That indicates our aim was not fulfilled, but from the %drug release of all batches it was observed that A5 batch having very low drug release (18.29%) at pH 1.2 and its show good release (81.45%) at pH 7.4 and this A5 batch having good % entrapment efficiency 87.06%.

From the above evaluated result it can be concluded that beads give 18.29% to 23.05% drug release at stomach pH-1.2. But objective of present was to retard the drug release in the

physiological environment of stomach and small intestine and deliver the drug to the colon. In order to fulfil this another approach another is to be used.Optimized batch A5 was taken for the further study.

The % drug release of Eudragit S100 coated azathioprine beads of different batches B1, B2 and B3 observed 75.05%, 79.77% and 74.62% respectively drug release in phosphate buffer pH-7.4. But in HCl buffer pH-1.2 %drug release of these batches were observed 11.76%, 6.98% and 8.51%. From the above result it was observed that 10% Eudragit S100 coated beads show minimum drug release (6.98%) in the physiological environment of stomach and small intestine and it delivered the drug to the colon. Therefore object ofpresent work was succeeding.

3.4. Contour plots

From the counter plot of $CaCl_2$, drug (Figure 9) observed that $CaCl_2$ concentration was decrease then %CDR decrease, and if drug concentration increase then %CDR increase.

From the counter plot of $CaCl_2$, Pectin (Figure 10) observed that $CaCl_2$ concentration decrease then %CDR decrease, but if pectin concentration was decrease then increases in %CDR.

From the counter plot of drug, Pectin (Figure 11) observed that drug concentration decrease then %CDR decrease, but if pectin concentration was decrease then increases in %CDR.

3.5. Compatibility studies

Differential Scanning Calorimetry (DSC)

The molecular state of the drug evaluated by performing DSC analysis of pure drug azathioprine and mixture of azathioprine-pectin using a differential scanning calorimeter (DSC-60, Shimadzu). The samples were heated in sealed aluminum pans at temperature range of 10^0C–350^0C and constant rate of 10.0^0C/min under nitrogen purge of 20 ml/min. [5]The pick of azathioprine was observed at 245^0C it can be seen in both figure, and pick of pectin observed at 160^0C. From the pick of azathioprine shown in both figure it indicates that there is no interaction between azathioprine and pectin.

Fourier Transform Infrared Spectroscopy (FTIR)

The compatibility study between pure drug azathioprine and pectin was carried out using FTIR spectrophotometer. The scanning was performed in the range of 400 to 4000 cm^{-1} with 1 cm^{-1}

resolution. From the results it was observed that there is no interaction between azathioprine and pectin

CONCLUSION

It was concluded that colon targeted delivery of azathioprine beads possible using pectin as polymer. The beads prepared by iontropic gelation method using $CaCl_2$ and pectin in different proportions were found satisfactory as per researcher demand. Research work also concluded that Eudragit S100 was able to retard the drug release in stomach. Acetone as solvent and triethyl citrates as plasticizer were found useful for the present study. It was also established that optimized batch showed prominent drug release in colon region. The optimized bead formulation were further added into hard gelatin capsules and then can be administered.

FIGURES

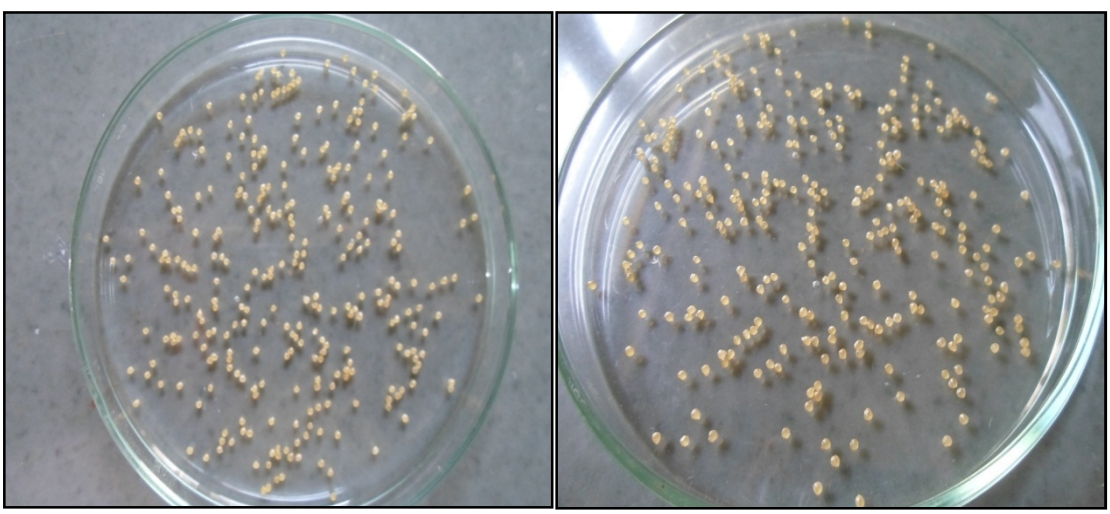


Fig. 01. Appearance of Pectin beads 5% **Fig. 02.** AppearancePectin beads 7.5%

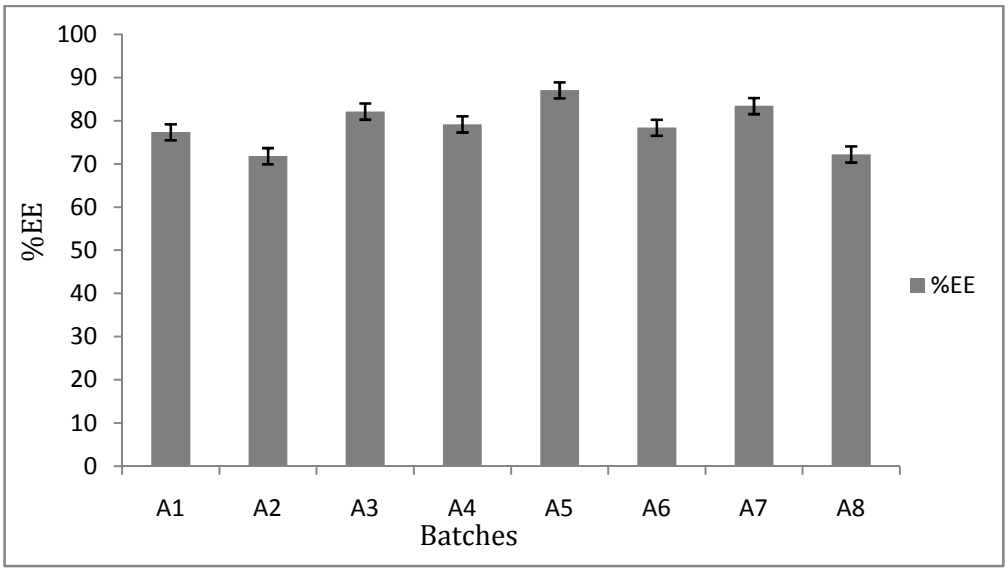


Fig. 03. Chart of %Entrapment Efficiency

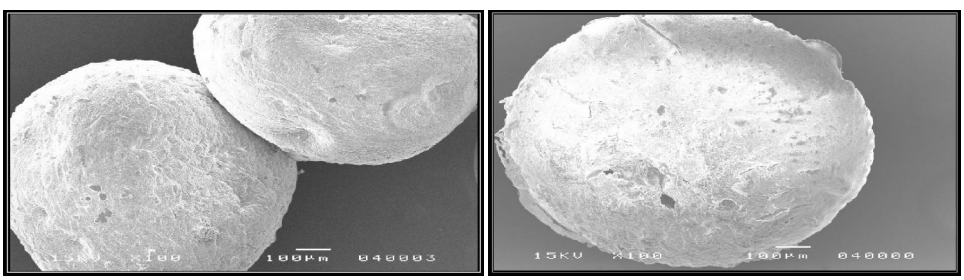


Fig. 04. SEM of uncoated bead

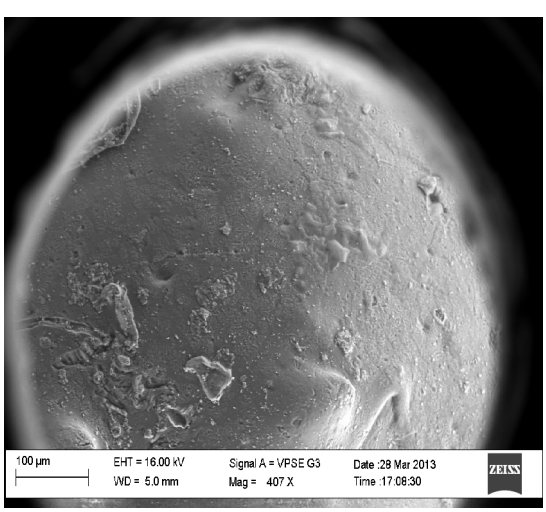


Fig. 05. Magnification 407X

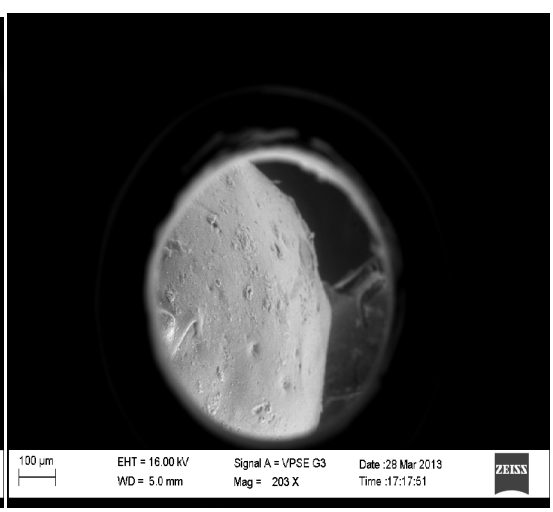


Fig. 06. Magnification 207X

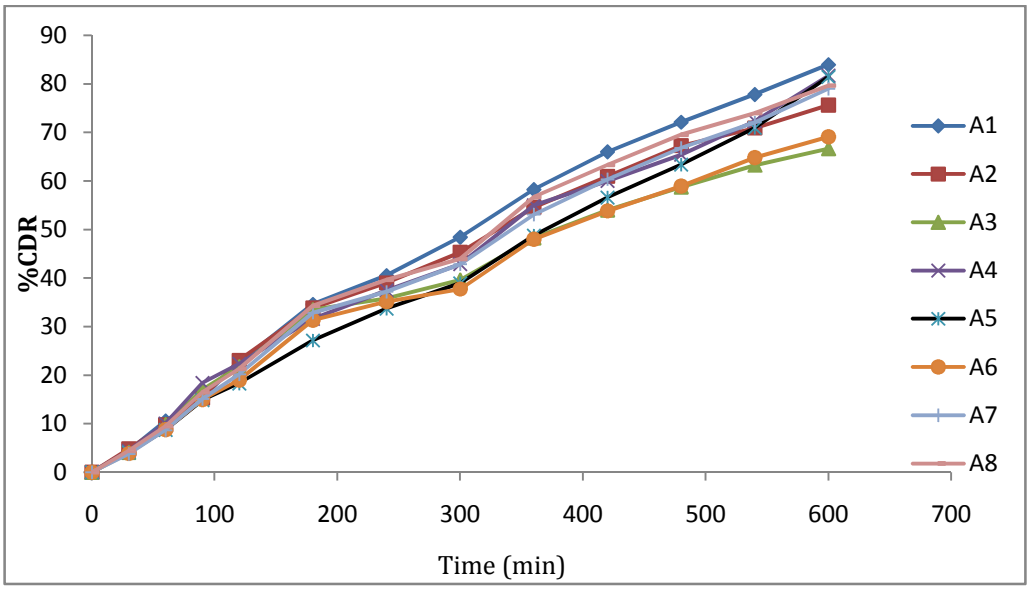


Fig. 07. %Cumulative Drug Release

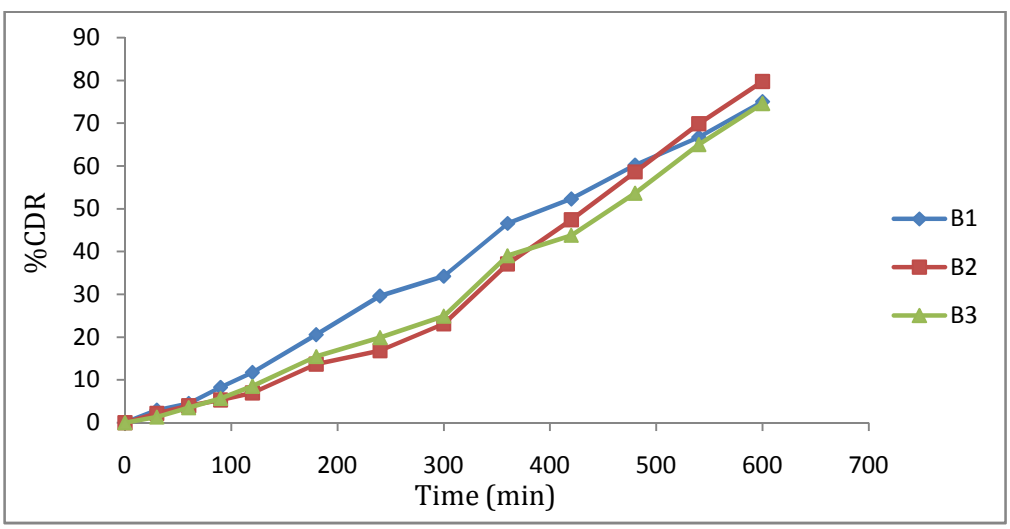
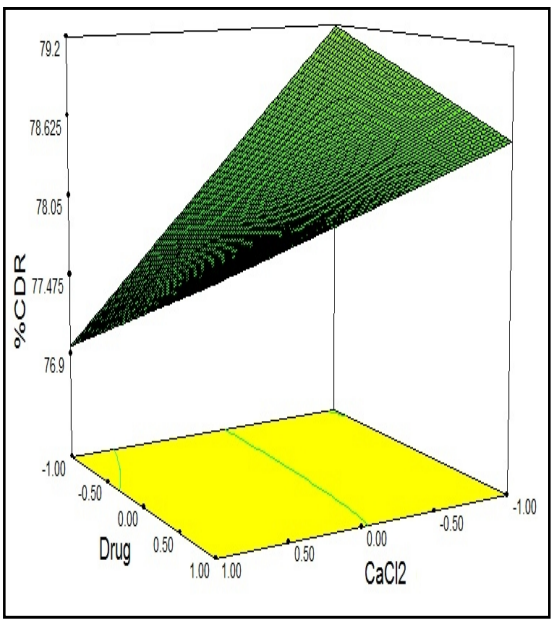


Fig. 08. %Cumulative Drug Release of Coated Beads



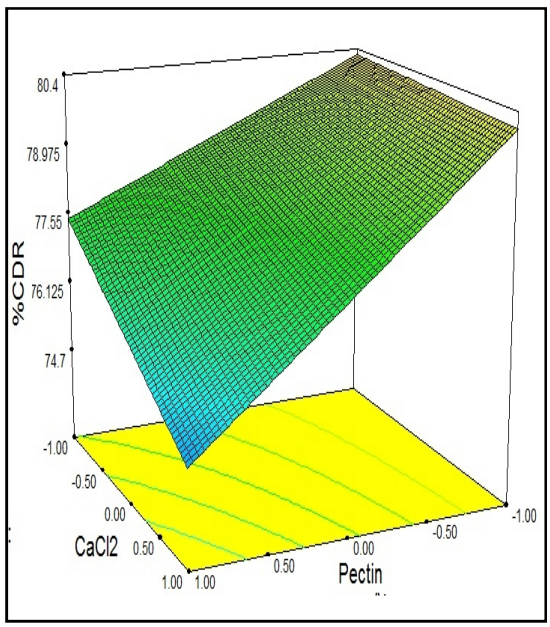


Fig. 09. Counter plots of Drug, $CaCl_2$ Fig. 10. $CaCl_2$, Pectin

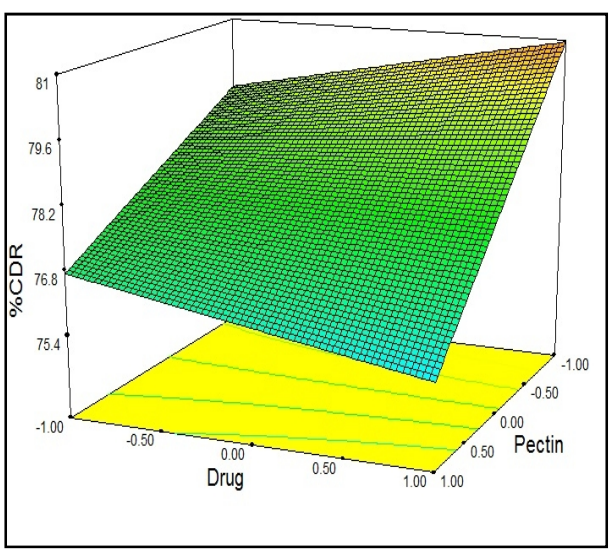


Fig. 11. Counter plot of Drug, Pectin

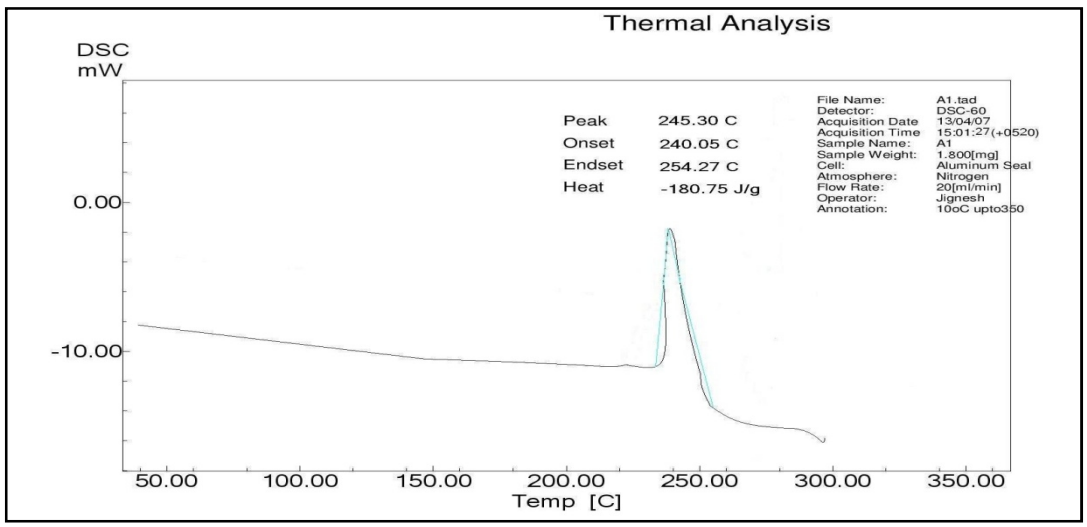


Fig. 12. DSC of Azathioprine

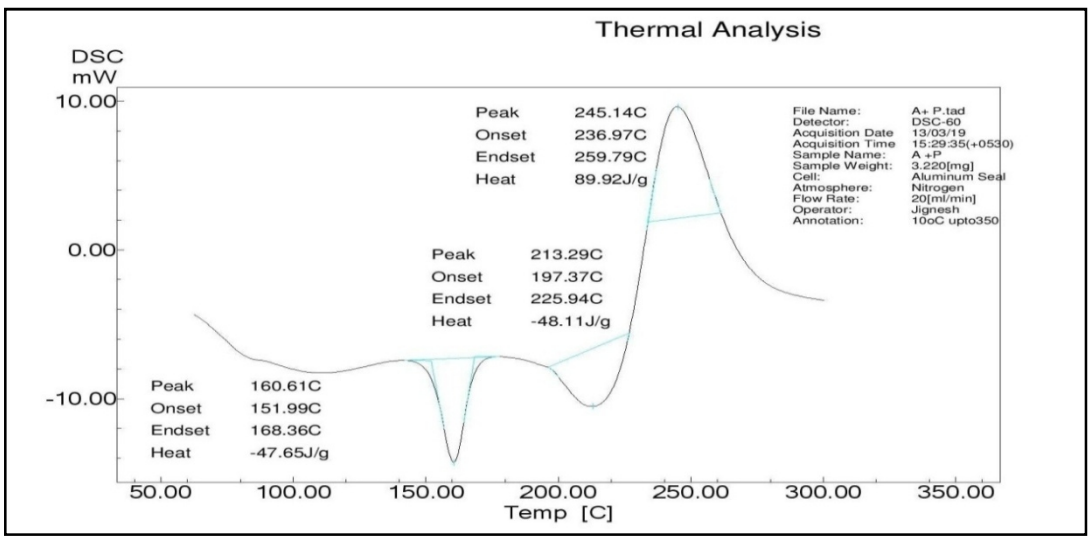


Fig. 13. DSC of Azathioprine + Pectin

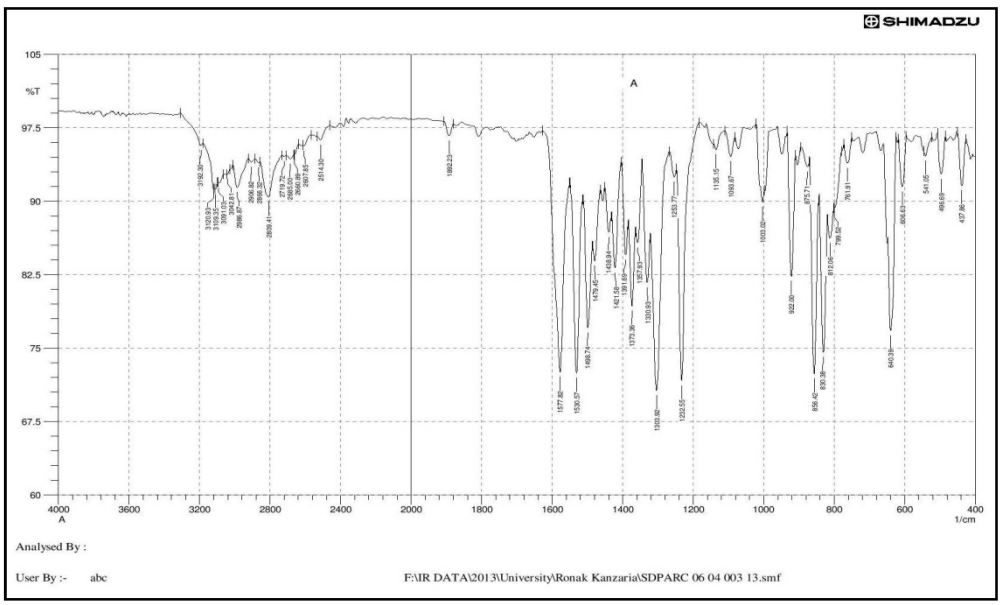


Fig. 14. FTIR of Azathioprine

TABLES

Table 01. Material and Reagents used

Ingredient	Category	Supplier
Azathioprine	API	Troika pharma
Pectin	Polymer	Oxford lab.
$CaCl_2$	Cross linking agent	Chem. Lab.
Eudrajit S 100	Coating material	Oxford lab.
Tri rthylcitarte	Plasticizer	Unichem..

Table 02. Formulation composition of colon targeted beads of Azathioprine

Batch no	Pectin conc. (%) X1	$CaCl_2$ conc. (%) X2	Amt of Azathioprine (mg)
A1	5	5	600
A2	7.5	5	600
A3	5	7.5	600
A4	7.5	7.5	600
A5	5	5	800
A6	7.5	5	800
A7	5	7.5	800
A8	7.5	7.5	800

Table 03. Formulation of Coating Solution

Batch	Conc. Eudrajit S100
B1	5%
B2	10%
B3	15%

Table 04.Result of Micromeritics Properties

Batch no	Bulk Density (g/ml)	Tapped Density (g/ml)	Hausner's Ratio	Compressibility Index
A1	0.69	0.89	1.20	18.99
A2	0.721	0.926	1.22	18.43
A3	0.643	0.954	1.21	28.37
A4	0.715	1.03	1.23	24.06

A5	0.713	0.976	1.20	16.89
A6	0.685	0.898	1.21	21.09
A7	0.725	0.965	1.24	23.10
A8	0.715	0.985	1.22	27.76

Table 05. %Entrapment Efficiency

Batch no.	Pectin conc.(%)	CaCl2 conc.	Amt of azathioprine (mg)	% Entrapment efficiency
A1	5	5	600	77.36±3.49
A2	7.5	5	600	71.83±3.42
A3	5	75	600	82.16±3.49
A4	7.5	7.5	600	79.2±2.95
A5	5	5	800	87.06±0.41
A6	7.5	5	800	78.4±3.55
A7	5	7.5	800	83.43±1.10
A8	7.5	7.5	800	72.23±2.62

Table 06. %Coating Efficiency

Batch	Theoretical Weight	Practical Weight	%Coating Efficiency
B1	8gm	5.3gm	66.25
B2	16gm	9.6gm	61.87
B3	24gm	15.6gm	65

Table 07. %Cumulative Drug Release

Time (min)	%Cumulative Drug Release							
	A1	A2	A3	A4	A5	A6	A7	A8
0	0	0	0	0	0	0	0	0
30	4.48	4.84	4.09	4.44	4.09	3.85	3.77	4.44

60	10.53	9.75	9.82	10.06	8.71	8.75	8.67	9.47
90	15.27	15.30	17.03	18.42	14.85	14.96	15.12	16.48
120	22.58	23.05	21.76	22.33	18.29	19.00	20.18	21.28
180	34.59	33.82	33.48	31.57	27.15	31.29	32.86	34.29
240	4.50	39.00	35.76	37.45	33.69	35.11	37.15	39.64
300	48.43	45.28	39.59	42.87	38.92	37.72	42.83	43.95
360	58.24	54.57	48.26	54.97	48.73	47.96	53.07	56.72
420	65.99	60.93	53.99	59.98	56.58	53.80	60.35	63.31
480	72.11	67.26	58.73	65.42	63.36	58.90	66.80	69.54
540	77.81	70.89	63.24	72.30	70.93	64.76	72.02	73.97
600	83.95	75.61	66.64	81.70	81.45	69.07	78.98	79.64

Table 08. %Cumulative Drug Release of Coated Beads

Time (min)	%Cumulative Drug Release		
	B1 (5%)	B2 (10%)	B3 (15%)
0	0	0	0
30	2.90	2.19	1.33
60	4.48	3.99	3.51
90	8.28	5.34	5.68
120	11.76	6.98	8.51
180	20.57	13.69	15.45
240	29.60	16.82	19.92
300	34.23	23.13	24.92
360	46.58	37.10	39.03
420	52.30	47.42	43.82
480	60.23	58.62	53.67
540	66.73	69.90	65.01
600	75.05	79.77	74.62

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th& Jan. 6th, 2016)

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RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

Antioxidant and anti-inflammatory properties of *Bombax ceiba* stem bark

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ABSTRACT

Bombax ceiba (Bombacaceae) stem bark is described in The Ayurvedic Pharmacopoeia of India and it recommends its in treatment of burning, acne and leprosy. Ethnobotanical reports suggest use of *Bombax ceiba* stem bark in inflammation. To evaluate anti-inflammatory and antioxidant effects stem bark of *Bombax ceiba,* Carrageenan induced paw edema in rat and DPPH free radical scavenging ability methods were used respectively. Analysis revealed that methanolic extract of stem bark of *Bombax ceiba* has been observed with dose dependent and significant inhibition of edema at 75 and 150 mg/kg body weight. The results of antioxidant activity showed that methanolic extract of stem bark of *Bombax ceiba* strongly scavenge DPPH free radical with IC_{50} value of 35.30 ± 1.11 µg/ml. This study, first time scientifically documents anti-inflammatory potential of *Bombax ceiba* stem bark powder correlate it with its antioxidant effect, which has been used by indigenous communities since many years.

SUMMARY

Methanolic extract of *Bombax cieba* stem bark has been found with anti-inflammatory potential evidently supported by anti- oxidant activity.

Keywords: Bombax ceiba, DPPH, Anti-iflammatory, Antioxidant

INTRODUCTION

Inflammation is a part of defense mechanism of body that protect against infection, allergens, burn or other noxious stimuli. But persistent and uncontrolled inflammation may act as a causative factor for many chronic diseases (*1*). One more study suggests that the complex events and mediators involved in

the inflammatory reaction can induce, maintain or aggravate several diseases (*2*). Most of currently used non steroidal anti-inflammatory drugs are associated with several side effects. Hence the need has been arise to develop safer anti-inflammatory drug.

Bombax ceiba Linn. istall deciduous tree, widely distributed throughout tropical parts of India. Bark of *Bombax ceiba* is official in The Ayurvedic Pharmacopoeia of India and it recommends its use in the treatment of acne, leprosy and burning. (*3*) An ethnobotanical survey in Lohit district of eastern Arunachal Pradesh suggested use of *Bombax ceiba* stem bark paste in inflammation and hotness. (*4*)

More over free radicals (ROS) can contribute to number of ailments including atherosclerosis, ischemia, liver diseases, gastritis, arthritis, ageing, respiratory diseases, cancer, AIDS and inflammation. (*5-7*)

But scientific evidence has not been established for anti-inflammatory potential of stem bark and its correlation with its antioxidant effect. Considering the use of *Bombax ceiba* in traditional ayurvedic as well as ethnobotanical reports, as anti-inflammatory stem bark, the present study was an attempt: 1) To study the anti-inflammatory activity of methanol extract of stem bark of *Bombax ceiba* by Carrageenan induced hind paw edema in rat, and 2) To evaluate antioxidant effect of methanol extract of stem bark of *Bombax ceiba* by DPPH free radical scavenging activity.

MATERIALS AND METHODS

Plant Material

Plant material was collected and extracted as per **Fig. 1.**

Drugs and Chemicals

Carrageenan (Sigma Aldrich USA), Aspirin (Merck Pvt. Ltd., India) α, α–Diphenyl–β–Picryl Hydrazyl (DPPH) and Ascorbic acid were purchased from Himedia Pvt. Ltd., Mumbai and S. D. Fine Chem. Pvt. Ltd. Respectively. Methanol was acquired from Merck Pvt. Ltd. India.

Animal

Information related to Animals used in protocol is described in **Fig.2.**

Assessment Of Anti-Inflammatory Activity

Carrageenan induced hind paw edema in rat model used for evaluation of anti-inflammatory activity. (*8*) The procedure followed is described in **Fig.3.**

DPPH Free Radical Scavenging Activity

To evaluate antioxidant potential of MSBC, DPPH free radical scavenging activity method was studied.(*9,10*) Details of activity is described as per **Fig. 4.** IC_{50} was graphically determined by a linear regression method and results expressed as IC50 ± S.E.M.

RESULTS AND DISCUSSION

Carrageenan induced hind paw edema

The anti-inflammatory activity of Methanol extract of stem bark of *Bombax ceiba* was evaluated by using Carrageenan induced hind paw edema in rat. The results of the animal studies are described in **Table 1.** Paw edema was measured as a difference in paw volume (mm) ± S.E.M..

The mean increase in paw edema volume in the vehicle treated control rat was about 0.326 ± 0.009 ml. MSBC (75, and 150 mg/kg, *p.o.*) significantly ($p < 0.001$) reduced the mean paw edema volume at 3h after Carrageenan injection. MSBC (75, 150 mg/kg, p.o.) exhibited anti-inflammatory effect with % inhibition of paw edema of 69.32% and 75.46%, respectively, as compared with the control group in dose dependent manner. Reference standard drug Aspirin showed significant ($p < 0.001$) 62.26% of inhibitions

of paw edema. Effect of MSBC on carrageenan-induced paw edema in rat after 3h, are expressed graphically in **Fig. 5.**

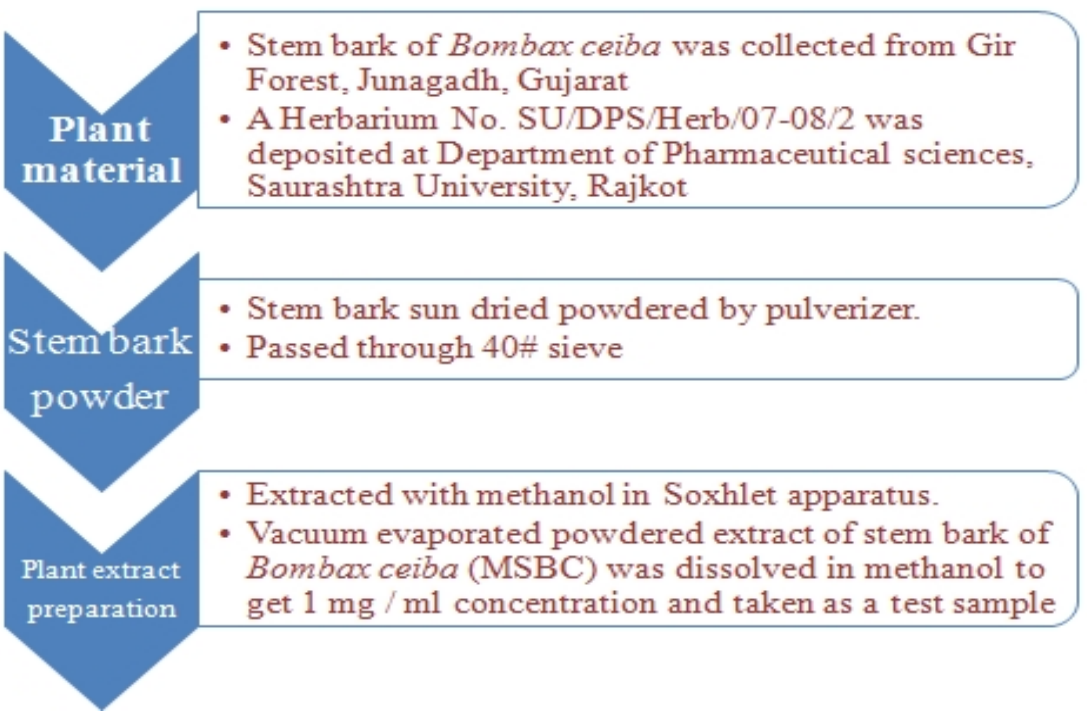
Figure5. Clearly indicates significant inhibition by MSBC extract at 75 and 150 mg/kg in dose dependent manner. Thus Stem bark methanol extract upon challenge by phlogistic stimuli in acute model of inflammation shows significant anti-inflammatory activity.

The antioxidant effect of MSBC was evaluated by DPPH free radical scavenging activity. DPPH is a stable free radical which accepts hydrogen or electron radical to become a stable molecule. (*11*) The absorbance of DPPH radical decreased as a result of reaction between antioxidant and DPPH free radical, which is the result of scavenging of the free radical by hydrogen donation. The reduction indicated visually by color change purple to yellow which can be measured at 517 nm. Therefore DPPH is used as a substrate to measure the antioxidant effect of the extracts and other chemicals. (*12*). As shown in **Fig.6**, MSBC strongly scavenged DPPH free radical in a dose dependent manner, with an IC_{50} value of 35.30 ± 1.11 µg/ml. On the other side the reference compound of Ascorbic acid was found to be IC_{50} value 31.30 ± 1.67 µg/ml.

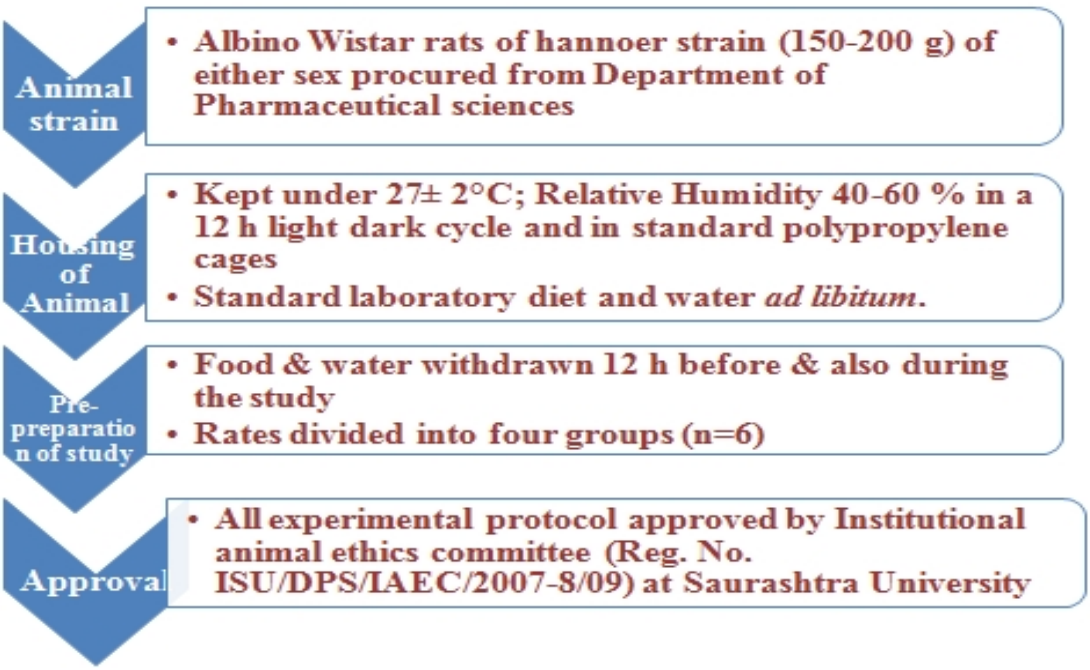
CONCLUSION

The results of present study observed with dose dependent and significant anti-inflammatory activities of methanol extract of *Bombax ceiba* stem bark at 75 and 150 mg/kg dose in rat. Moreover study also revealed antioxidant effect of methanol extract of *Bombax ceiba* stem bark at IC50 value of 35.30 ± 1.11µg/ml while Ascorbic acid was found withIC_{50} value 31.30 ± 1.67µg/ml. Therefore, We can speculate that *Bombax ceiba* stem bark methanol extract might reduce the risk of inflammation related illness.

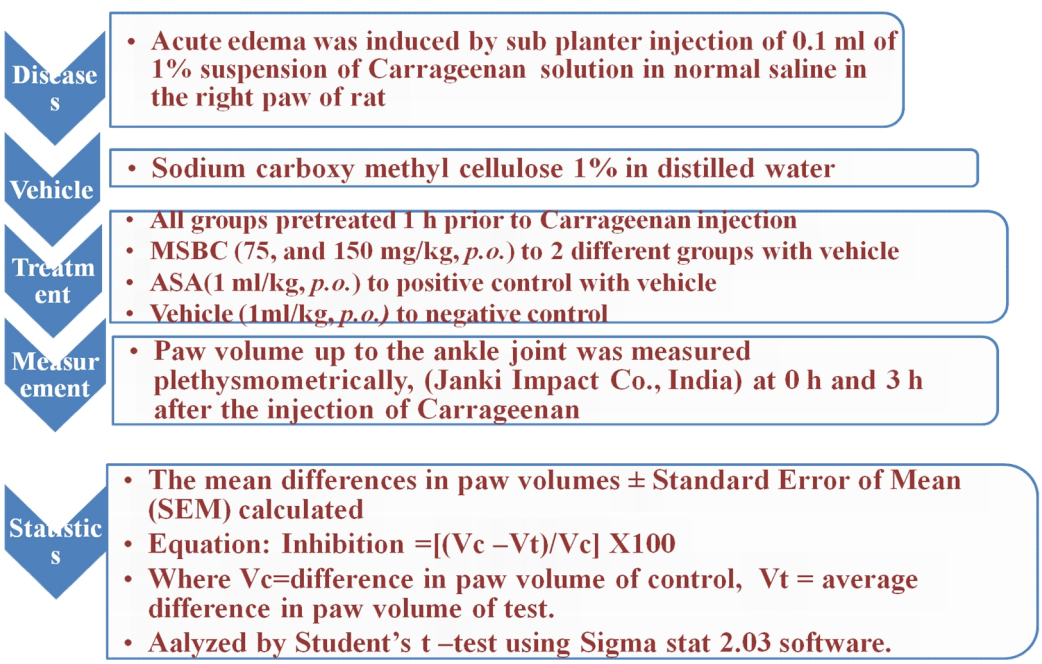
FIGURES



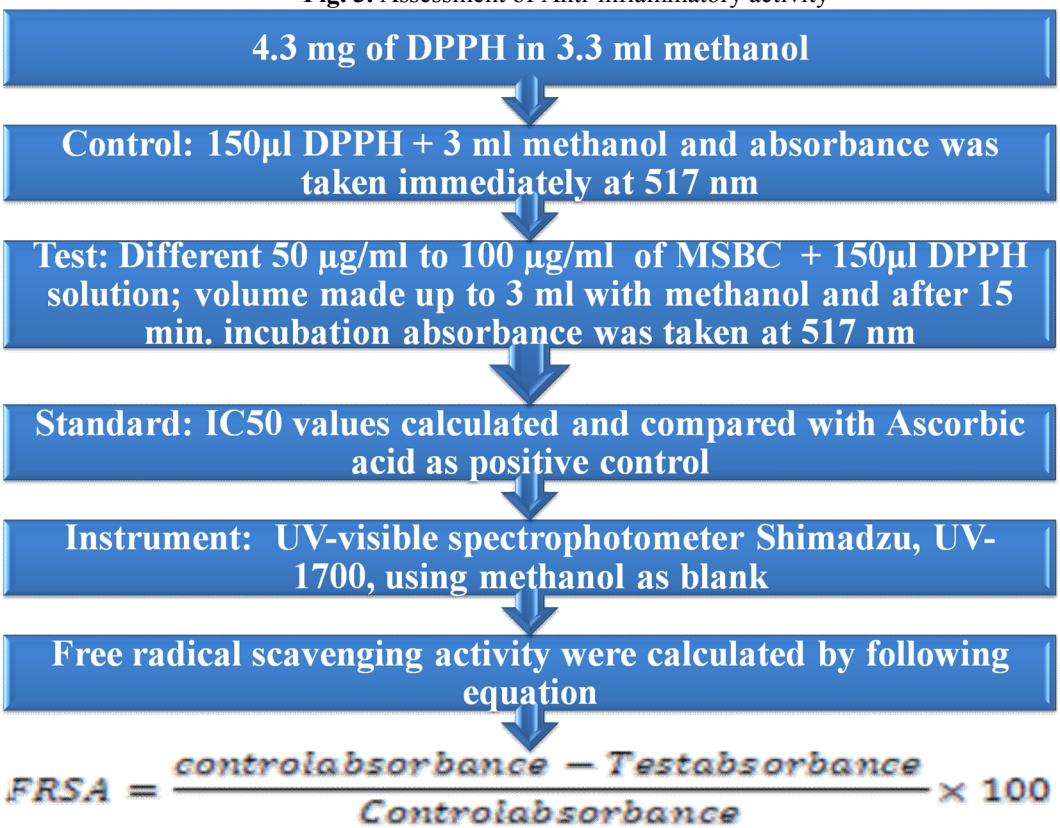
"Fig. 1. Plant material collection and extraction**"**



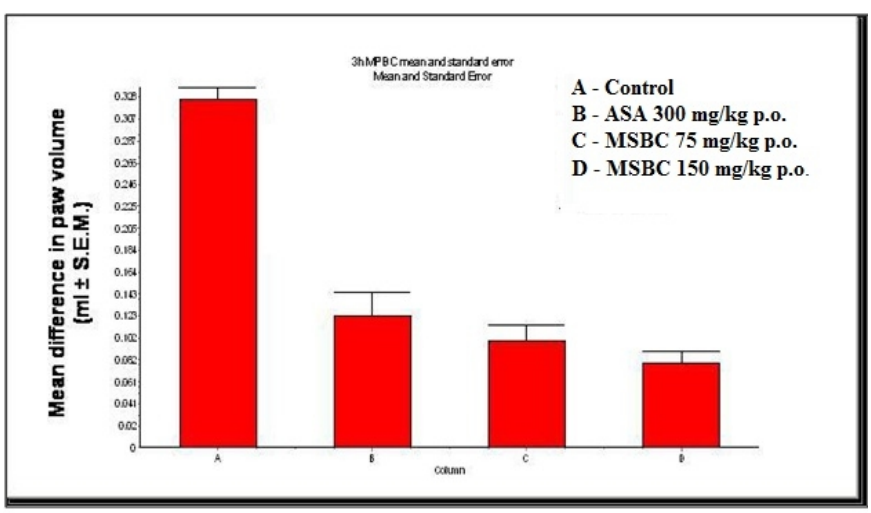
"**Fig. 2.** Animals used in protocol"



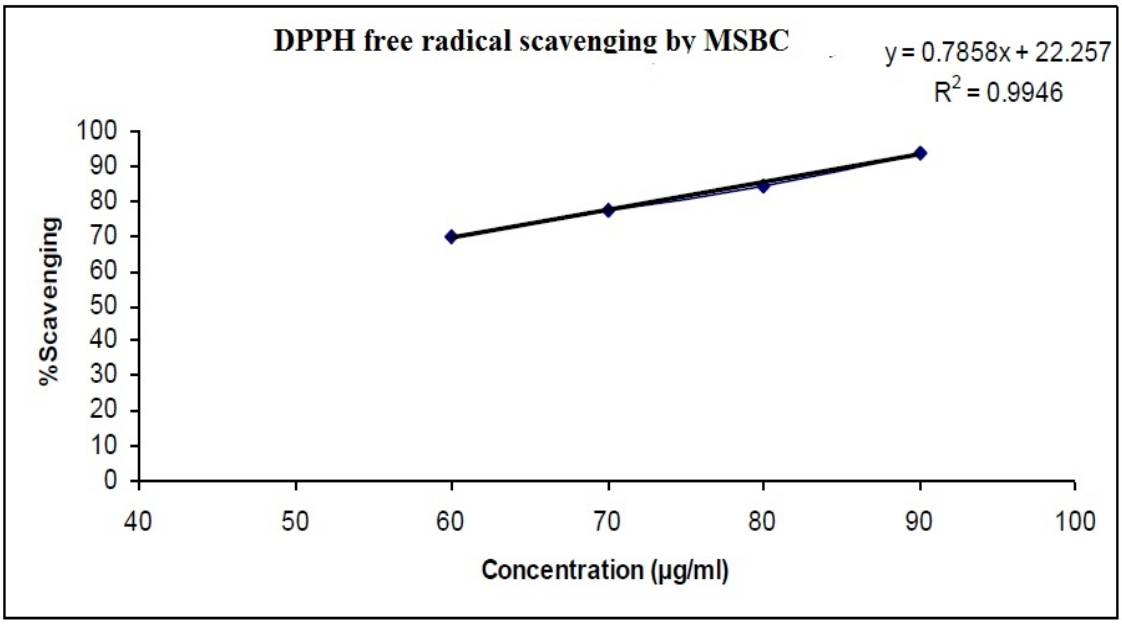
"Fig. 3. Assessment of Anti-inflammatory activity"



"**Fig.4.** DPPH Free Radical Scavenging Activity"



"**Fig. 5.**Effect of MSBC on Carrageenan-induced rat paw edema after 3h, Results are mean paw edema ±S.E.M.(n=6)"



"**Fig.6.** Percentage scavenging of DPPH free radical by MSBC at different concentrations"

TABLES

"**Table 1.** Effect of MSBC on Carrageenan induced paw edema"

Treatment	**Dose (mg/kg,p.o.)**	**Mean difference in paw volume (ml ± S.E.M.) at 3 h**	**% Inhibition**
Control	**-**	**0.326 ± 0.009**	**-**
ASA	**300**	**0.123 ± 0.022 *****	**62.26**
MSBC	**75**	**0.1 ± 0.014 *****	**69.32**
MSBC	**150**	**0.08 ± 0.010 *****	**75.46**

Values are mean ± S.E.M.; n=6, *** p< 0.001 compared to control, student's *t*-test.

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Quality by Design enabled fabrication and optimization of self-micro emulsifying drug delivery system for Efavirenz

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ABSTRACT

The research envisioned accentuations on quality by design (QbD) methodology for systemic development of Efavirenz SMEDDS. To portray effect of particular failure modes cognate to concrete formulation variable, Risk assessment employing failure mode and effects analysis was carried out. A pseudo ternary phase diagram was prepared to figure out optimum proportions of oil (Capmul MCM C8), surfactant (Cremophore-EL) and co-surfactant (PEG 400) and evaluating its effect on Critical Quality Attributes like globule size, zeta potential, % transmittance and dissolution. The optimised dosage form revealed quantity of oil 7%; surfactant 37.5%; co-surfactant 12.5% and water 43%. The results of same revealed globule size of 15.75 ± 0.55nm, zeta potential -22.90mV, % transmittance 99.29 ± 0.04 and in vitro dissolution of more than 90% in 5 minutes. The developed SMEDDS showed significant enhancement in dissolution as compared to marketed preparation and plain active substance thus revealing a promising carrier for Efavirenz.

SUMMARY

The goal of this investigation was to augment oral bioavailability for the delivery of Efavirenz by self-micro emulsifying drug delivery system using quality by design approach.

Keywords: SMEDDS, Quality by Design, Pseudo ternary phase diagram, Solubility enhancement, Failure mode and effects analysis

INTRODUCTION

1. Efavirenz belongs to non-nucleoside reverse transcriptase inhibitor (NNRTI) category of anti-HIV medicine approved for the treatment of HIV disease. Efavirenz comes under Class-II drug as per BCS relegation, has least water solvency, higher permeability upon delivery by oral route and prone to hepatic metabolism (*1*). Recently, much contemplation has been engrossed on lipoid based dosage forms, with precise eminence on self-micro emulsifying drug delivery systems (SMEDDS) to augment solubility and bioavailability of ailing aqueous soluble medicines via lymphatic pathways (*2,3*). Self-micro emulsifying drug delivery systems are isotropous blends of active substance, oils and surfactants, frequently with one or more hydrophilic co-surfactant/solubilizer that facilitates fine oil in water microemulsions upon mild stirring in an aqueous medium (*4,5*). SMEDDS have risen as an efficient drug delivery system owing to their inherent aspects like minute globule dimension, comfort of production, ameliorated biocompatibility and better stability on storage (*6*). On succinct amount of literature survey; no results have been found for in the area of SMEDDS for solubility enhancement of Efavirenz. This type of system would exemplify the objectives like solubility and bioavailability enhancement which would be of prodigious concern for combating the battle against management of infection.

The major goals of this research were: (i) Step sapient screening, understanding and optimization of formulation development utilizing QbD approach (ii) Coherent formulation development for achieving solubility enhancement by applying risk assessment and FMEA approach (iii) to enforce pseudoternary phase diagram for ascertaining optimum ratios for culled formulation variables.

MATERIALS AND METHODS

Materials

Efavirenz received as gift sample from Mylan Laboratories (Hyderabad), Capmul MCM C8 and Capmul MCM L8 (Abitech Corporation, USA), Cremophor EL (Sigma Aldrich, India), Labrasol, Labrafac Lipophilic WL and Transcutol P (Gattefosse, Saint-Priest, France), Brij 35, Tween-80 and PEG-400 (SDFCL, Mumbai, India), Soyabean oil, Olive oil (Himedia laboratories Pvt. Ltd., Mumbai, India), Methanol (Merck), Trifluroacetic acid (sigma Aldrich), and Sodium lauryl sulphate (Merck).

HPLC analysis

The HPLC apparatus of Agilent HPLC (1260 Infinity series) equipped with quaternary pump system and PDA/UV detector (Santa Clara, United states) and an on-line degasser. A reverse phase cyano column (Zorbax SB-CN, 150x4.6 mm, 3.5µm) was used at 40°C column temperature. Methanol: Water: Trifluoro acetic acid in a proportion 60:40:0.1 v/v used as mobile phase. The chromatograph set at 1.2 mL/minute flow; 30µL injection volume and λmax at 250nm.

Quality Target Product Profile (QTPP) of Efavirenz SMEDDS

Developed formulation must have the quality traits in order to finish the goals set in target product profile as quantifiable characteristics are enrolled as target product quality profile and ICH Q8 (R2) summarizes them as QTPP (*7,8*). To meet the required of clinical efficacy and safety of the finished product some quality characteristics are required in the QTPP. After QTPP distinguishing proof, the following step is to

recognize applicable critical quality attributes (CQAs), procedure variable which are critical and constrain stratagem. Intended QTPP for Efavirenz SMEDDS is delineated in Table 1.

Risk assessment of Efavirenz SMEDDS by Failure Mode and Effects Analysis

In ICH Q9, 2005 guidance the initial concepts of quality risk management were introduced (*9*). The Critical quality attributes (CQAs) linked with the drug substance, dosage form and production method, and so on employed and is chosen amongst numerous likely alternatives. Subsequently after preliminary studies, Dosage form design and process for manufacturing proposed by us have been specified in separate segments. FMEA technique is utilized for associate in attention overall risk assessment execution of the formulation variables. The failure modes were recognized by this technique which could have most prominent impact on dosage form recital and greatest likelihood of inciting dosage form. To organizing failure mode for risk management resolutions as showed by the importance of their outcome (result) FMEA methodology utilized to check how as frequently as would be prudent they happen and how successfully they can be distinguished. According to risk priority number (RPN) the comparative risk that belongs to every product variable was rated (*10*). Those qualities that could significantly influence the medication item traits ought to have been considered in purpose of interest while for those attributes no further examination required which had minor influence on the medication item characteristics. The CQAs designated in Table2 and Table 3 delineates FMEA of Efavirenz SMEDDS with corresponding RPN for each failure mode. Calculation of RPN done by Eq. 1.

$$\text{RPN} = \begin{bmatrix}5\\4\\3\\2\\1\end{bmatrix} \text{O x} \begin{bmatrix}5\\4\\3\\2\\1\end{bmatrix} \text{S x} \begin{bmatrix}1\\2\\3\\4\\5\end{bmatrix} \text{D} \qquad (1)$$

Where, O is the frequency likelihood or the possibility of an occasion to happen; which were categorized these as [5] continuous; [4] plausible; [3] incidental; [2] extreme and [1] implausible to transpire. The consequent parameter S is the seriousness, which is an assessment of how great of an outcome a specified failure mode would realize; which were classified as [5] calamitous; [4] precarious; [3] severe; [2] trivial and [1] minor (no impact). D is the last parameter, is detectable quality which suggests that a failure mode can be seen. Along these lines the more evident a failure mode is, the less hazard it presents to product quality. For D, it was ranked as [1] outright clear or effortlessly discernible; [2] high perceivable; [3] modestly discernible [4] little or remote measurable and [5] as hard to perceive or add up to vague.

Solubility studies

Equilibrium solubility study assessment on Efavirenz was done with several excipients like Tween-80, Cremophore EL, Soyabean oil, Olive oil, Campul MCM C8, Campul MCM L8, Transcutol P, PEG 400, Brij35, Labrafaclipophile and Labrasol. Overabundance amount of the Efavirenz was added to a 2mL eppendorf tube containing each excipient and stirred for 30s (Cyclomixer/Vortex mixer CM-101 plus, Remi, Mumbai, India). The tubes were placed into thermomixture C (Eppendorf AG, Hamburg, Germany) and shaken with 500RPM for 24 hours at 37±0.5°C. Centrifuge the resulting solution at 5000 RPM at the end of cycle for 15 min. Take appropriate volume of supernatant of the sample treated for solubility study and dilute with methanol, and analyzed by HPLC.

Pseudoternary phase diagram

The introductory stride toward the definition improvement was to look at the credibility of the micro emulsion advancement. The fringes of the microemulsion areas were set by construction of pseudoternary phase diagram considering the constituents shortlisted after solubility test. The pseudoternary phase diagram of lipid, surfactant co-surfactant blend and water was plotted by method for water titration technique. Different pseudoternary phase diagrams were developed keeping in mind the end goal to acquire the concentration assortment of constituents for the existing region of micro emulsions. The quantity of surfactant to co-surfactant was mixed in 1:1, 2:1 and 3:1 proportion. On the premise of pseudoternary phase diagrams, surfactant and co-surfactant ratio of was chosen. Presently by keeping that proportion steady, microemulsion formulations were formulated by utilizing the distinctive grouping of oil (Capmul MCM C8) 4%, 5%, 6%, 7%, 8%, 9%, and 10%.

Preparation of SMEDDS

After careful evaluation, oil, surfactant and co-surfactant were selected as a Self-emulsifying micro emulsion mixture for drug delivery. Quickly; oil, surfactant, and co-surfactant were precisely weighed into vials according to their ratio. Then, the components were mixed by mild stirring at 37°C until Efavirenz was completely dispersed. The blend was checked for indications of turbidity or phase detachment up to 48 hours.

Characterization of Efavirenz SMEDDS formulations

Droplet size measurement

To determine droplet size of SMEDDS, 100 times diluted with HPLC grade water, was resolved utilizing a Zeta Sizer S90 (Malvern, UK). Before the estimation, the mixtures were diluted with HPLC grade water to a suitable scattering intensity. All measurements were performed in triplicate.

Zeta Potential measurement

The zeta potential plays important role in the stability of colloidal dispersions. The degree of the zeta potential demonstrates the level of electrostatic repugnance between adjoining, comparably charged particles in scattering. Zeta Potential was measured utilizing a Zeta Sizer S90 (Malvern, UK). Each sample was suitably diluted with HPLC grade water and put in a dispensable zeta cell. The Zeta Potential

values were assessed by measuring the molecule electrophoretic mobility. All measurements were performed in triplicate.

Conductance

By estimation of conductance kind of microemulsion (oil in water or water in oil) can be resolved. The electric conductance of the resultant colloidal system was dictated by a conductivity meter (Labindia, Mumbai, India). For the estimations, the tried microemulsions were prepared through a 0.01N sodium chloride solution rather than HPLC grade water.

% Transmittance Measurement

For measurement 1mL of SMEDDS preparation diluted to 100 times with HPLC grade water. Estimation of the %T of diluted SMEDDS against blank as a water determined at 650nm by utilizing UV spectrophotometer (Shimadzu Tokyo Japan).

Morphology of SMEDDS

The microstructure of microemulsions from SMEDDS was investigated by TEM (Tecnai 20 Philips). For TEM analysis, SMEDDS was diluted with HPLC grade water and a droplet of it was mounted on a carbon coated copper grid (300 mesh, 3mm) and air dried.

In vitro dissolution

In vitro dissolution studies of SMEDDS and Marketed Formulation (Sustiva) containing 200mg of Efavirenz and 200 mg of standard Efavirenz were performed in 1% SLS in water using media volume 900mL, Paddle apparatus, 37 ± 0.5 °C bath temperature,50 RPM (FDA-Recommended Dissolution Methods) (*11*) and in water (without surfactant) to check influence of surfactant.

At preselected time interims, 10mL samples were pulled back and supplanted with 10mL of pre-thermostated fresh dissolution medium. Samples were filtered through suitable pore size syringe filter discarding first few mL of filtrate, the resulting filtrate was injected into the HPLC for analysis.

Stability studies

Robustness to dilution

Robustness of SMEDDS to dilution was determined as per Date et al., strategy with slight adjustment (*12*). SMEDDS were diluted to 10, 100 and 1000 times with different media. water, 0.1N hydrochloric acid and pH 7.4 phosphate buffers. The diluted microemulsions were preserved up to 12 hours and observed for indications of phase detachment or drug precipitation.

Thermodynamic stability studies

Thermodynamic stability study was executed to determine the phase separation and influence of temperature difference on SMEDDS formulations under specified storage conditions. Efavirenz SMEDDS were diluted to 100 times with HPLC grade water and centrifuged at 10,000 RPM up to 20

minutes and formulations were visually checked under dark background for phase separation. To evaluate the effect of temperature, the formulations were subjected to freeze–thaw cycles (-20°C for 2 days followed by 25°C for 2 days) (*13*). At the end of the cycle, the formulations were diluted and centrifuged as described above and phase separation and the change in droplet size were determined.

Physical and chemical stability

Physical and chemical stability was assessed by storing the SMEDDS samples at 2-8°C (refrigerator), 25°C/60%RH (Real time stability condition) and 40°C/75%RH (Accelerated condition) up to 6 months. Samples were pulled back at foreordained time interims following 1, 3, and 6 months. Various parameters like clarity, phase separation, globule size and zeta potential after dilution with HPLC grade water at 1:100 were measured for physical stability of Efavirenz in SMEDDS. Moreover, assay and in vitro dissolution were also performed.

RESULTS AND DISCUSSION

QTPP of Efavirenz SMEDDS

Selection of correct QTPP depends on type of dosage form and strategy chosen (*7*). After careful assessment of primary experiments, the specifications that will be accentuated in study were picked and enlisted as QTPP for Efavirenz SMEDDS (Table 1). Consequently, except for recitation of chosen QTPP, the QTPP are not discussed to depict the further strides. The said QTPP will set out the establishment for deciding CQAs.

Risk assessment by FMEA

For development of Efavirenz SMEDDS, critical factors that were set out and evaluated by FMEA (Refer Table 3.) Now the research envisioned for development, the factors that demonstrated RPN greater than 40 was regarded as potential risk, equivalent to greater than 20 to less than 40 was measured as moderate risk and less than equal to 20 was considered as little risk (*10*). It is clearly stipulated from Table 3, surfactant concentration, co-surfactant concentration and oil concentration under high hazard category and henceforth, their optimisation was done thoroughly.

Solubility studies

Solubility of Efavirenz in various oils, surfactants and co surfactants is given in Fig. 1. The SMEDDS formulated with oil, surfactant/co-surfactant and drug ought to be clear and monophasic liquid at 25°C temperature when added to the water medium with incredible dissolvable properties to permit show of active substance in solution. The Capmul MCM C8 indicated more prominent drug solubility contrasted with alternate oils. Furthermore, Capmul MCM C8 mixture of C8/C10 mono-/diglycerides which favors complete solubilization of Efavirenz in the vicinity of triglyceride chains due to shorter chain length. As a result of great solubility Capmul MCM C8 was chosen as the oil vehicle as it can possibly improve the release and availability of drug that are impermeable to tissue barriers and ineffectively assimilated because of either low partition coefficient or low diffusibility. Surfactants known to cause gastric irritation hence careful selection of surfactant concentration in formulation is critical factor associated with safety. Non-ionic surfactants are least noxious than ionic surfactants. Non-ionic surfactant based Oil in water nanoemulsion dosage forms offer more prominent in vivo stability for oral or parenteral

administration (*14*). Chremophore EL demonstrated the most astounding drug solubility among surfactant tried in experiment. This surfactant gave great solvency; enhance drug stacking with favorable SMEDDS formulation bringing about unconstrained fine emulsion formation.

A lipophilic, co-surfactant which is non-volatile in nature is less inclined to move to the capsule shell than solvent, for example, ethanol furthermore more inclined to be saved by the oil phase upon dilution with water media, accordingly keeping away from precipitation (*15,16*). Polyethyleneglycol-400 used as co-surfactant which helps surfactant to stabilize the system. Efavirenz showed significant solubility in this co-surfactant. Taking into account the solubility data, Capmul MCM C8 as oil, Chremophore EL as surfactant and Polyethyleneglycol-400 as co-surfactant were chosen as a SME mixture for drug delivery. The results are highlighted in Fig. 1.

Construction of Pseudoternary phase diagram

To categorize the self-emulsifying regions and to optimise the concentrations of oil, surfactant and co-surfactant in the SMEDDS dosage forms, Pseudo-ternary phase diagrams were constructed in absence of Efavirenz. Microemulsion formation was done when SMEDDS titrated with water under mild stirring. For the most part, the resulting particle size is under 100 nm and as the power important to create a microemulsion is practically nothing, and the process is thermodynamically unconstrained (*15*). By the region of surfactant this method is encouraged. Layer around oil globule is shaped by surfactant as the polar head lies towards fluid and non-polar tail pull out oil which in the long run diminishes surface strain between oil phase and a watery phase. (*17*). Additionally, proportion of surfactant and co-surfactant likewise influences development of microemulsion. The blends with diverse surfactant/cosurfactant and oil proportions lead to the development of SMEDDS with distinctive characteristics (*18*). Since surfactant and co-surfactant adsorb at interface and give mechanical obstruction to coalescence; decision of oil, surfactant, cosurfactant and blending extent of surfactant to co-surfactant [S/CoS (km)], demonstrates critical feature in microemulsion formulation (*19*). The psuedoternary phase diagrams were constructed at the ratios of S/CoS (km) 1:1, 2:1 and 3:1. At initially, compelling extent of oil, i.e. 90%, and least, i.e., 10% extent of S/CoS was taken and serially oil extent was reduced and that of S/CoS was increased. It was examined amid these trials that higher amount of oil forms poor emulsion with entrapment of too little amount of water on dilution. One more observation was that as amount of S/CoS increases, the time assessed to form microemulsion reduces. It is without a doubt comprehended that to fulfil the interfacial film to maintain and condense inclusion of surfactants in the microemulsion framework is required, while co-surfactant is in charge of development of the film in this way the relative degree of surfactant to co-surfactant successfully influences droplet size. Diverse extent of oil and S/CoS attempted to frame series of microemulsions, however degree of oil was seen to be a rate confining component and in all cases, high oil concentration brought about poor emulsion locale. The black boundary covers microemulsion region. At any point beyond this boundary, microemulsion if formed initially, become turbid on further dilution of solution. This indicates formation of emulsion with higher particle size.

Comparing pseudoternary phase diagram of every blend are exhibited in Fig.2. On the reason of pseudoternary phase diagram, surfactant and co-surfactant in proportion of 3:1 was picked. Now proportion of surfactant to co-surfactant (3:1) kept consistent, microemulsionformulations were prepared by utilizing distinctive the convergence of oil (Capmul MCM C8) 4%, 5%, 6%, 7%, 8%, 9%, and 10%. In view of mean globule size, Zeta potential and percentage transmittance stable microemulsion was chosen consisting oil concentration at 7% as mentioned in Table 4. The optimized formulation composition has been depicted in Table 5.

Characterization of Efavirenz SMEDDS

ZP is related to surface charge of microemulsion droplet. It is highly dependent on surfactant used. The theory states that system remains stable due to deflocculation of microemulsion particles and for identical system ZP charge should be between amplitudes of -10 to -30 mV (*20*). The charge of the oil droplets in traditional SMEDDS is negative because of the vicinity of free fatty acids. Non-ionic surfactants can be extensively used and have minimal toxicity (*21*). The droplet size manages the speed and amount of drug discharge from dosage form, likewise absorption is an essential variable in self-emulsification performance. The diagrams of droplet size measurement and zeta potential are depicted in Fig. 3 and Fig.4. The optimum composition exhibited mean droplet size of 15.75 ± 0.55nm and zeta potential of -22.90mV respectively. Conductivity estimations give a method for figuring out if a microemulsion is oil-continuous or water-continuous other than contribute a method for observing percolation or phase inversion phenomena (*22*). The excipients used in formulation decide the pH of the final preparation. Stability of microemulsion is incredibly impacted by change in pH which might change Zeta potential of microemulsion formulation. So pH is likewise in charge of security of microemulsion. Optimized SMEDDS formulations showed similar pH values in the range of 5.5 to 6.0. So here pH is not affecting stability. So it can be anticipated that entire system showed pH of water as drug remains in oil phase only. Conductivity values of optimized SMEDDS formulation was found to be 98.34 ± 0.14 µS indicating water as continuous phase with no phase inversion. The % transmittance was also more than 99% for the optimum formulation. TEM image (Fig. 5) reveal uniform and spherical globule size for the SMEDDS indicating in line data with globule size.

In vitro dissolution

Dissolution profiles of standard Efavirenz, Marketed formulation (200mg capsules) and SMEDDS are illustrated in Fig. 6. and Fig. 7. The drug dissolution rate is markedly enhanced in the SMEDDS as 92% of the drug dissolved in 5 minute, as compared to only 4% and 45% from plain Efvirenz drug and Marketed drug product capsules respectively in 1% SLS in water dissolution medium while 86% of drug dissolved from SMEDDS in 5 minute as compared to only 2% and 32% from standard Efvirenz and Marketed drug product capsules respectively in water as a dissolution medium. Drug present in a solubilized form in this type of dosage forms and results in small droplets when comes under contact with dissolution medium which prompts the fast dissolution of drug from SMEDDS. The standard Efavirenz dissolves just 49% more than hour long time period, complete dissolution of Efavirenz not accomplished owing to large crystal size, while SMEDDS demonstrated an altogether enhanced disintegration rate with 100% of drug dissolved within 60 minute. The Mean Dissolution Time (MDT) value for SMEDDS, standard Efavirenz and marketed formulation is 3.39, 24.08 and 7.73 respectively and dissolution efficiency value is 94, 29 and 88 respectively in 1% SLS in water as a dissolution medium while in water medium MDT value for SMEDDS, standard Efavirenz and marketed formulation is 4.02, 17.47 and 12.01 respectively and dissolution efficiency value is 93, 14 and 58 respectively. Lowest MDT value and higher dissolution efficiency for the developed SMEDDS indicate faster dissolution compared to marketed preparation and plain drug.

Stability studies

The dilution may better imitate environment in the stomach following oral administration of SMEDDS pre-concentrate. To access the effect of dilution on SMEDDS pre-concentrates dilution study was executed, any precipitation or phase separation was not observed in this study after dilution of SMEDDS

in a different media. In this way, it can be inferred that dilutions in all tried media were robust. In thermodynamic stability studies, selected formulations were administered to different stress tests like centrifugation and freeze-thaw test. If the SMEDDS preparations are stable in this condition, metastable formulations can be avoided and numerous tests need not to be performed during storage. No phase separation and no change in droplet size of the optimized Efavirenz SMEDDS formulations were observed on centrifugation and freeze-thaw test. Optimized SMEDDS formulation was found to be thermodynamically stable.

Thermodynamic stability studies performed on developed optimized formulation to evaluate its stability and the integrity of the dosage form. On account of its liquid characteristics SMEDDS has an extremely confined time span of usability and there is opportunity to precipitation or phase separation of the drug from the formulation. The physical and chemical stability of SMEDDS formulation was evaluated at 2-8°C as a controlled condition, 25°C/60%RH to mimic real time condition and 40°C/75%RH to mimic accelerated condition for 6 months. It was found that there was no major change in the mean droplet size and zeta potential when SMEDDS was stored at 2-8°C, 25°C/60%RH and 40°C/75%RH over 6 months. Furthermore, the assay values were also within limits during all stability time points. No significant difference in dissolution was also observed for stability samples of SMEDDS compared to the dissolution of initial sample. These results suggest that the SMEDDS can maintain the physical as well as chemical stability of the Efavirenz during the stability period evaluated.

Risk mitigation and control strategy

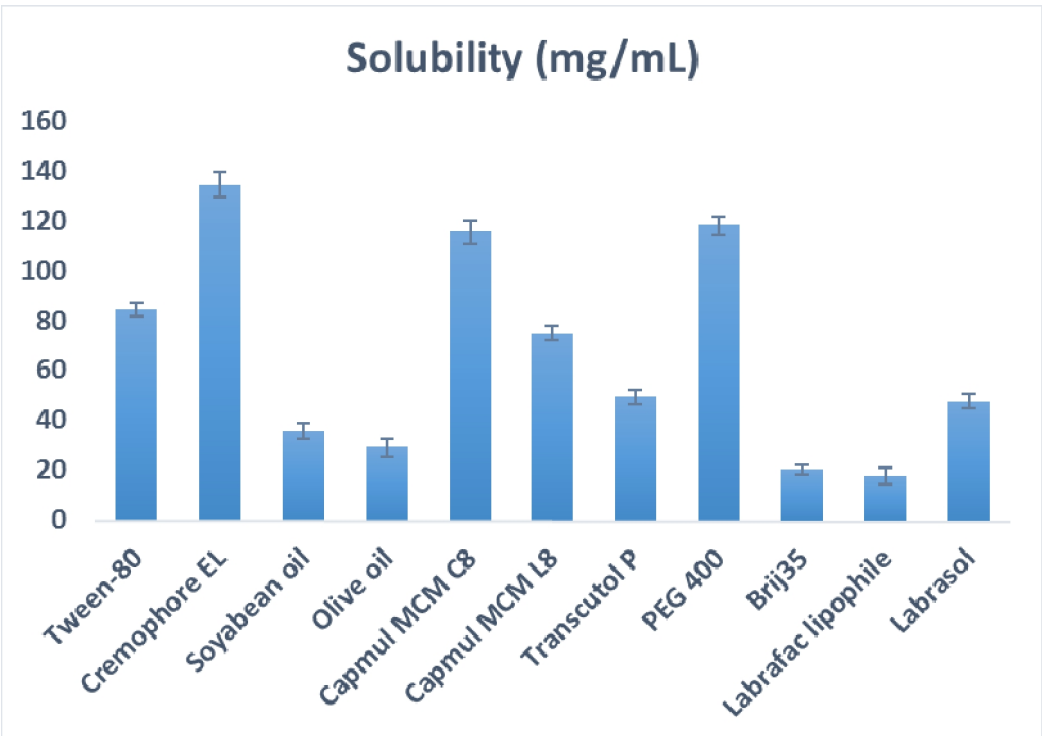
The risk mitigation and control methodology is an amalgated blueprint of in what way quality is set up grounded on existing procedure and current formulation awareness. The FMEA prior and then afterward the usage of the control technique highlighted in Fig. 8. It was examined that RPN of the imaginable failure modes were underneath 20; making them to fall under the generally low risk classification. The scalability can be further analyzed from resulting exchange from lab to pilot and after that scale up batch fabricating. Subsequently it might be additional sophisticated taking into account going with experience picked up amid the business lifecycle of the production.

CONCLUSION

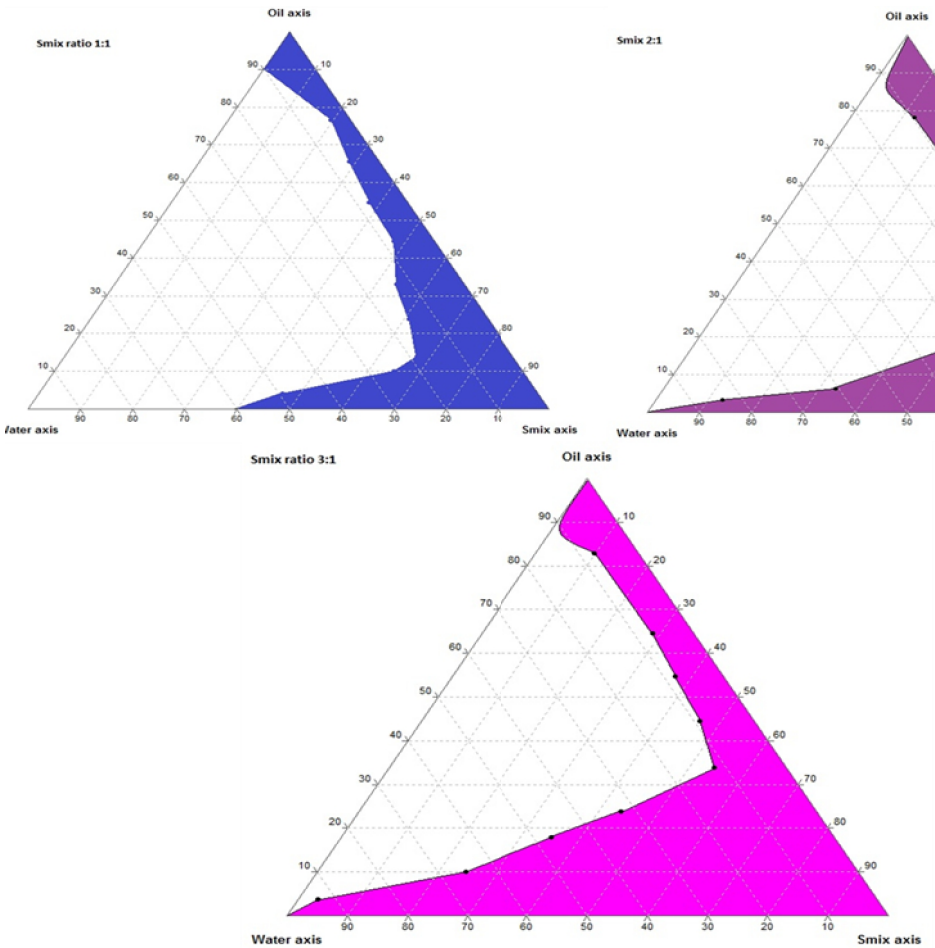
The exploration depicts the general QbD approach alongside risk assessment, risk examination and control system to alleviate the risk for fabrication of Efavirenz SMEDDS. Preliminary screening was conducted for selection of suitable oil, surfactant and co surfactant. Pseudo-ternary phase diagram and self- emulsification tests were also carried out for judicious selection of excipient ratios. The optimized formulation exhibited ratio of 7:37.5:12.5 for Capmul MCM C8 (oil): Cremophore EL (surfactant): PEG 400 as (co-surfactant) proportionately. Moreover, Efavirenz SMEDSS showed tremendous improvement in dissolution as compared to plain Efavirenz and marketed Efavirenz and also found to be stable for 6months under subjected stability conditions. The optimized formulation has shown favourable outcomes in vitro and is potential for estimating in vivo bioavailability.

FIGURES

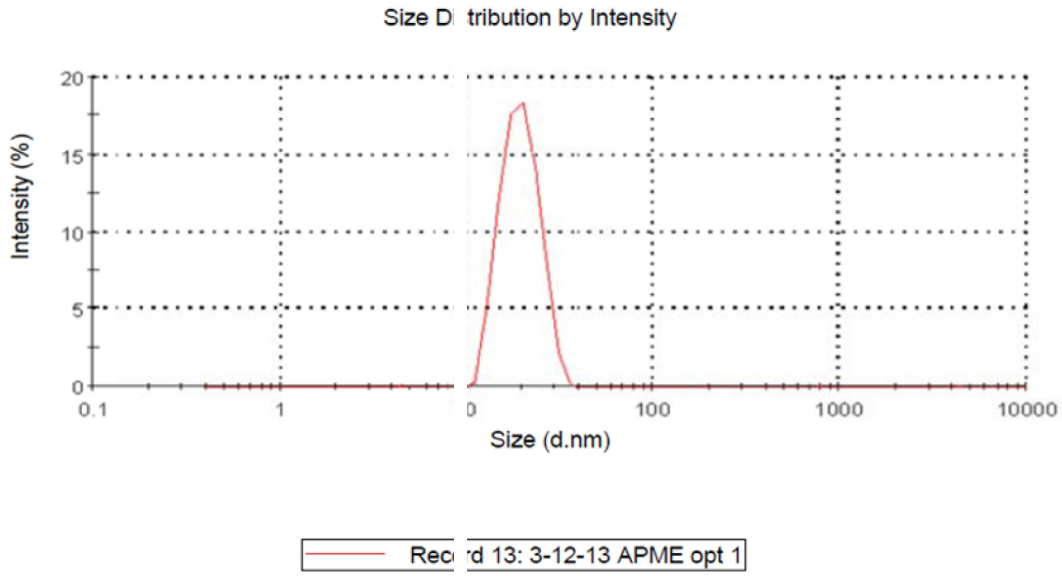
"**Fig. 1**. Results of solubility study of Efavirenz."



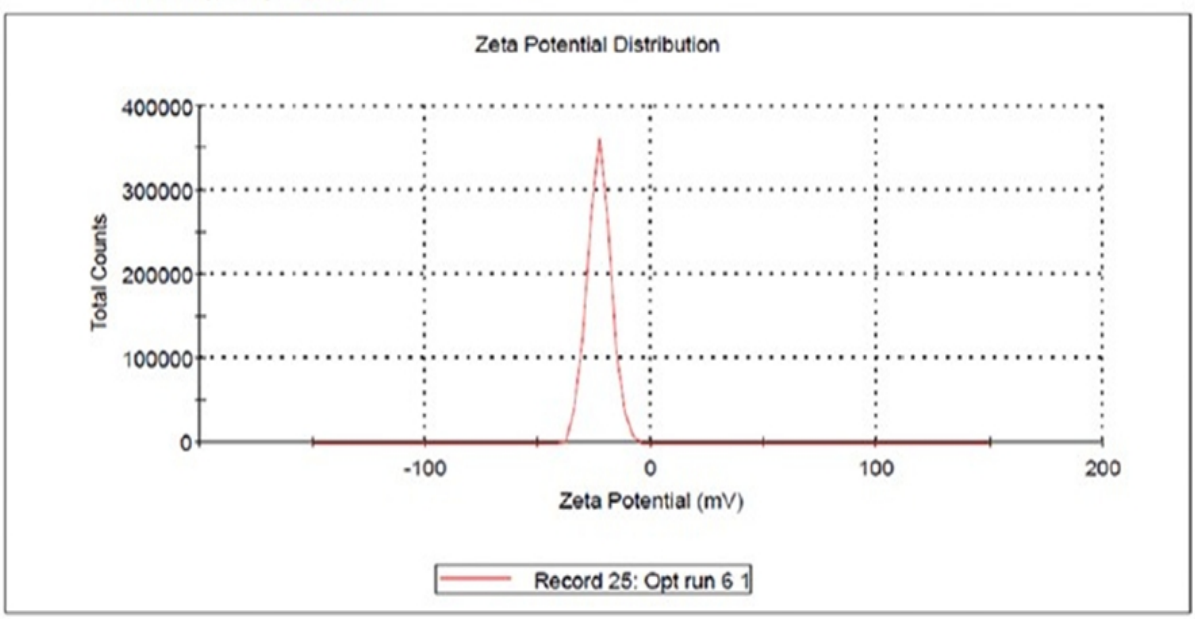
"**Fig. 2.**Pseudoternary phase diagram prepared with Oil, Surfactant and Co-surfactant. S/CoS ratio is 1:1, 2:1 and 3:1."



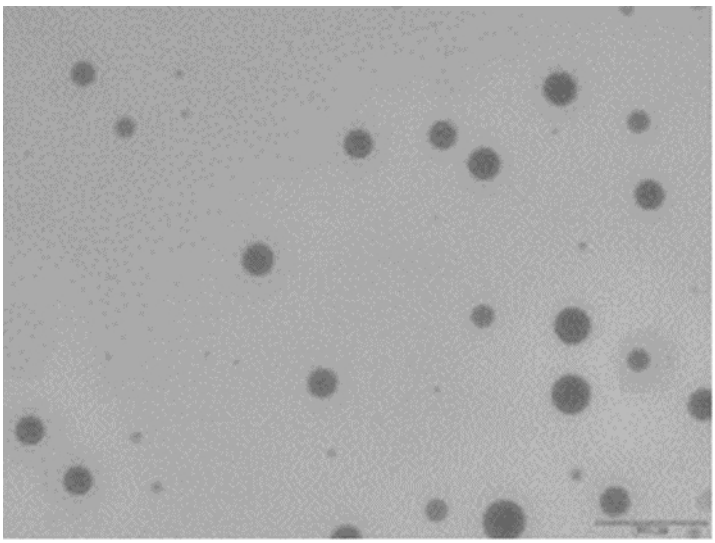
"**Fig. 3.** Droplet size distribution curve of optimised Efavirenz SMEDDS**.**"



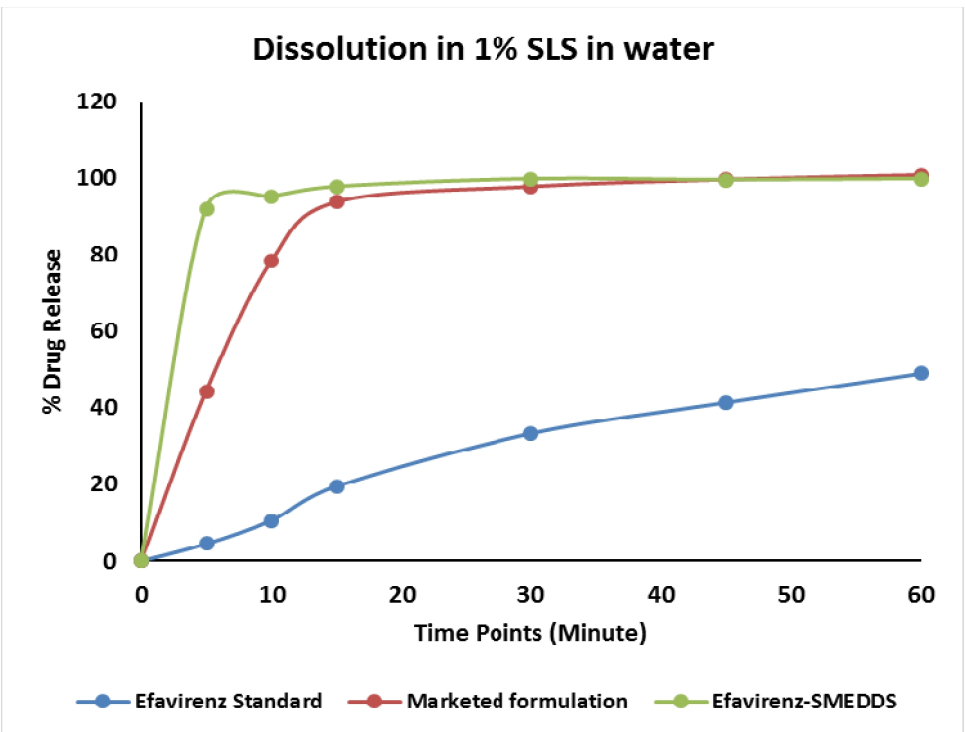
"**Fig. 4.** Zeta Potential curve of optimised Efavirenz SMEDDS**.**"



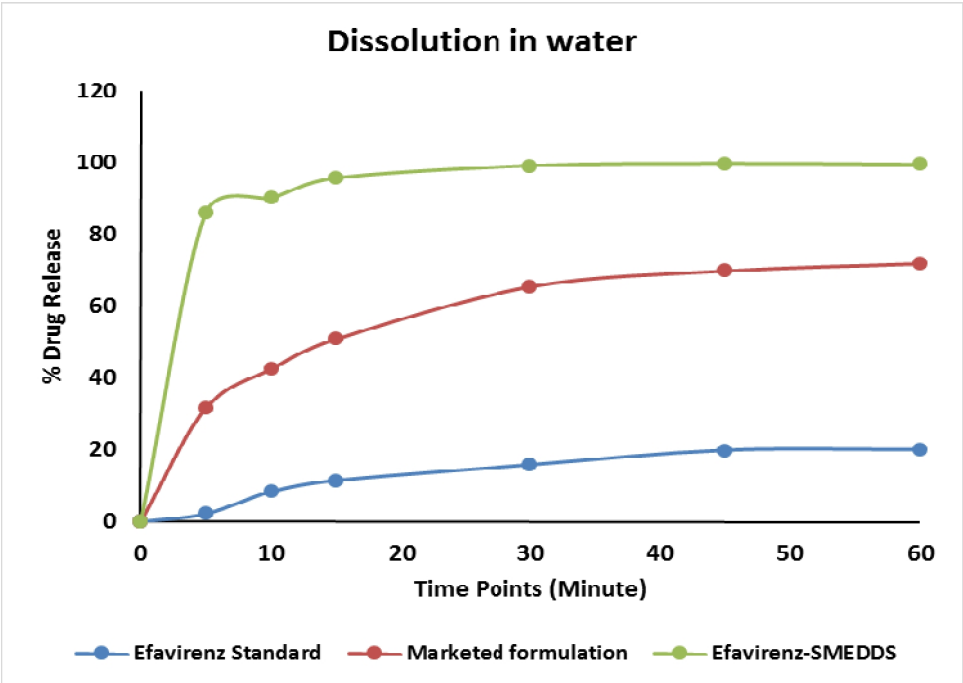
"**Fig. 5.** TramittanceElectonMicrosopyImageof diluted Efavirenz loaded SMEDDS."



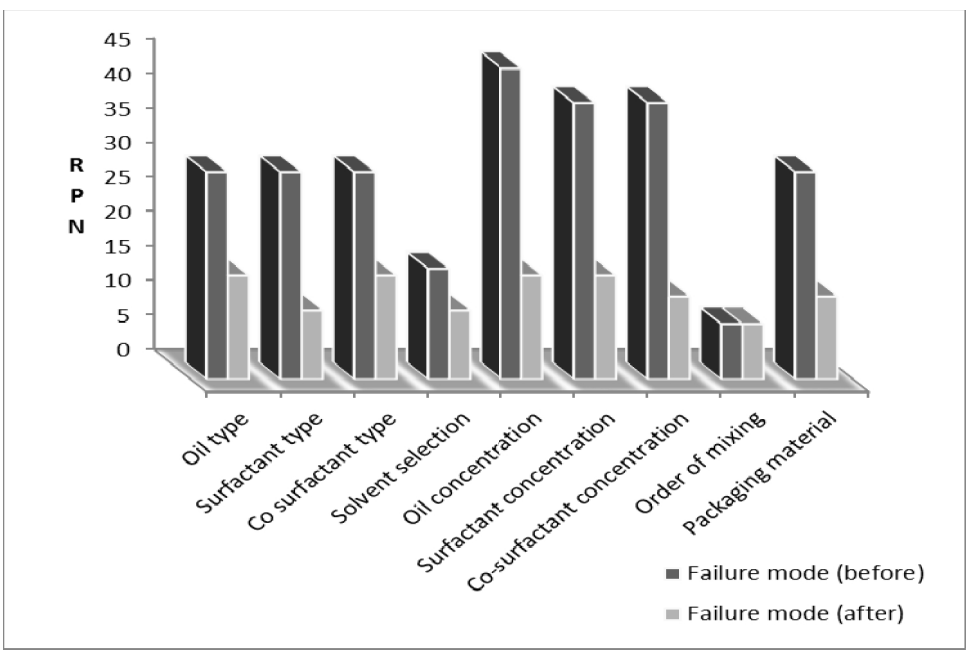
"**Fig. 6.** Dissolution profile comparison of Efavirenz SMEDDS with standard Efavirenz and Marketed formulation in 1% SLS in water."



"**Fig. 6.** Dissolution profile comparison of Efavirenz SMEDDS with standard Efavirenz and Marketed formulation in water**.**"



"**Fig. 7.** Failure mode and effects analysis of Efavirenz SMEDDS**.**"



TABLES

"**Table 1.** Quality target product profile (QTPP) hypothesized for SMEDDS of Efavirenz."

QTPP features	**Target**	**Rationale**
Formulation	SMEDDS	For solubility and bioavailability enhancement
Formulation type	Prompt release	Quicker onset of action leading to better pharmacokinetics.
Dose (Label claim)	200mg	Unit dose of Efavirenz amalgamated in a preparation of SMEDDS
Route of ingestion	Oral	Oral route is most commonly preferred route of administration.
Pharmacokinetics	Higher Maximum concentration (Cmax)and Area Under Curve	For accomplishing higher drug levels in systemic circulation.
Packaging material	Compatible during storage of formulation	To keep up product integrity and quality up to wanted timeframe of realistic usability
Stability	Accelerated and real time stability condition for period of 6 months	Minimum time frame chosen for stability investigation of final formulation.
Alternative strategies of drug delivery	Salt formation, Polymorphism, Solid dispersion, Nanocrystals, Co-crystals	Mentioned dosage forms can just upsurge the drug dissolution speed, however not the degree, which may be prompt increase drug ingestion by oral course.
Contraindications	Not any	Not any

"**Table 2.** Critical quality attributes of Efavirenz SMEDDS and their justifications."

Quality traits of drug product		**Target**	**Is it CQA?**	**Justifications**
Physical Elements	Appearance	Satisfactory to patients and no obnoxious scent	No	Color, shape and look are not specifically connected to efficacy and safety. In this manner, they are not critical. The objective is set to affirm quiet worthiness.
	Color			
	Odor			
Assay		100% w/w of strength	Yes	Variation in assay inversely affect to efficacy and safety. Thus, it will be investigated throughout the development of formulation.
Release rate of drug in 15 min:		NLT 90% in 15 min	Yes	Drug release rate from formulation is vital to accomplish drug level in systemic circulation for faster absorption. Formulation and process variable has a much influence on the drug release profile. Subsequently, these CQA will be observed all through formulation and also process advancement.
Mean size of globule		Less than 100nm	Yes	Less than 100nm globule size permits simple entrance through GI epithelial wall and paracellular pathways; subsequently was viewed as critical.
Zeta potential		-10 to -30 mV	Yes	For system to remain stable and avoid flocculation zeta potential should be between -10 to -30 mV.
% Transmittance		100%	Yes	Transmittance of SMEDDS represents system was monophasic or not on the basis of clarity of system.
Robustness to dilution		No phase separation or precipitation	Yes	Robustness to dilution reveals impact of dilution in various medias on precipitation or phase separation of microemulsion. Hence, it is critical.
Mean dissolution time (MDT)		Low	Yes	MDT is extremely critical factor indicating faster rate of release of drug in dissolution medium. Hence, it will be evaluated.
Dissolution efficiency 15 min		100%	Yes	It demonstrates essential data on in vitro dissolution and in this manner will be assessed.

"**Table 3.** Snapshot of FMEA analysis enrolling the RPN scores for the detailing variables explored."

Sr.No.	Failure modes	FMEA Rank scores			
		Severity	Occurrence	Detection	RPN
1	Oil type	5	3	2	30
2	Surfactant type	5	3	2	30
3	Co surfactant type	5	3	2	30
4	Solvent selection	4	2	2	16
5	Oil concentration	5	3	3	45
6	Surfactant concentration	5	4	2	40
7	Co-surfactant concentration	5	4	2	40
8	Order of mixing	4	2	1	8
9	Packaging material	5	3	2	30

"**Table 4.** Results of SMEDDS with varying oil concentration."

Batch No.	Oil (%)	Smix (%) (3:1)	% transmittance at 650 nm	Globule size (nm)	Zeta potential (mV)
ME1	4	40	96.70 ± 0.37	28.90 ± 1.70	-5.13
ME2	4	50	96.40 ± 1.00	24.00 ± 0.55	-5.51
ME3	4	60	98.10 ± 0.75	19.90 ± 2.75	-7.82
ME4	5	40	98.25 ± 0.60	31.22 ± 2.70	-9.96
ME5	5	50	97.44 ± 0.65	48.80 ± 0.85	-11.24
ME6	5	60	96.33 ± 1.60	51.84 ± 1.37	-12.15
ME7	6	40	98.20 ± 0.34	18.48 ± 0.47	-14.67
ME8	6	50	96.80 ± 1.40	20.92 ± 1.27	-16.28
ME9	6	60	96.30 ± 0.22	27.42 ± 1.30	-17.73
ME10	7	40	97.40 ± 0.21	20.15 ± 2.40	-20.18
ME11	7	50	99.29 ± 0.04	15.75 ± 0.55	-22.90
ME12	7	60	98.34 ± 0.55	18.51 ± 3.21	-19.47
ME13	8	40	96.35 ± 0.22	28.90 ± 1.20	-18.89
ME14	8	50	95.34 ± 0.82	26.00 ± 0.54	-16.46
ME15	8	60	96.87 ± 1.25	19.97 ± 1.80	-15.23
ME16	9	40	96.97 ± 2.10	25.00 ± 1.36	-14.67
ME17	9	50	97.78 ± 1.39	37.13 ± 1.42	-12.63
ME18	9	60	97.85 ± 1.84	41.17 ± 1.24	-10.85
ME19	10	40	96.80 ± 2.45	37.10 ± 2.43	-9.53
ME20	10	50	96.68 ± 1.71	33.00 ± 0.84	-6.83
ME21	10	60	97.54 ± 0.24	34.86 ± 0.84	-5.29

"**Table 5.** Optimised formula of SMEDDS."

Content	Quantity (% w/w)
Capmul MCM C8 (Oil)	7 %
Chremophore EL (Polyoxy-35-castor oil) (Surfactant)	37.5 %
PEG-400 (Polyethylene glycol) (Co-Surfactant)	12.5 %
Water	43 %

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

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Design & Characterization of Ritodrine hydrochloride Transdermal Delivery by Passive Diffusion & Iontophoresis

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ABSTRACT

Ritodrine hydrochloride (RTH) is a hydrophilic moiety which has been used in the treatment of premature labor. Pig ear skin was used as a model membrane for ex-vivo studies. Passive diffusion was carried out in a Franz diffusion cell. An increase of current density to the threshold value of 0.5 mA/cm^2, increased the permeation by many folds where 57% of RTH permeated at the end of 8th h in both case cathodal and anodal iontophoresis. With interrupted current (50% of electrical dose), permeation was lower but the permeation rate remained constant (cumulative permeation - 28% of RTH at the end of 8th h with current density of 0.5 mA/cm^2). RTH being a positively charged moiety gets the advantage of passive, electro repulsive as well as electro osmotic flux in anodal delivery. High cathodal flux indicated that electroporation assisted with iontophoresis could be the dominant contribution for RTH permeation.

SUMMARY

Ritodrine hydrochloride Transdermal Patch was formulated & evaluated to increase the permeation by Passive Diffusion & Iontophoresis methods.

Keywords: Transdermal, Ritrodrine hydrocloride, Pig ear skin, Franz diffusion cell, Modified Glikfield diffusion cell, Iontophoresis, Electroporation.

INTRODUCTION

In the past many years, transdermal drug delivery system (TDDS) has moved from a clinical reality to the point where it represents a viable diagnostic tool for noninvasive diagnosis and with extensive research done, it showed the promises for many more applications. However, lipophilic property of the human skin is a basic problem for limiting its widespread therapeutic use. The first challenge is to creat effectiveness of transdermal system ultimately involves adequate drug permeability through the stratum corneum (SC) (*1*).

The last many years the investigation is done and find out the specific chemical or combined chemicals that are act as penetration enhancers. According to the theory of lipid protein partitioning (LPP), the chemical penetration enhancers act by mainly three major mechanisms like Lipid matrix disruption of the SC, Interaction of intracellular protein and Improvement of the partitioning of drug or solvent into the SC. Many chemical enhancers are active for spatial disruption of normally arranged intercellular molecules (*2*).

Iontophoresis defined as the application of an electric potential that maintains constant electric current across the skin and enhances the permiation rate of ionized and unionized moieties. This technique is capable of widening range of the compounds which can be delivered transdermally (*3*).

MATERIALS AND METHODS

Chemicals:
Ritodrine hydrochloride was received as a gift sample from Neon laboratories, (Mumbai. India). Sodium chloride, Octanol given from S.D. Fine chemicals, (Mumbai. India). 1-heptane sulphonic acid sodium salt, Methanol (HPLC Grade), O-Phosphoric acid purchased from Spectrochem Pvt. Ltd, (Mumbai. India). Water (HPLC Grade) Milli Q, Millipore, AstraZeneca, (Bangalore. India). Ammonium phosphoricum™ purchased from E. Merck, (Darmstadt, Germany).

Skin Used:
Fresh Porcine skin Obtained from local slaughter's house, Gujarat.

Calibration curve of Ritodrine hydrochloride in UV:

Fig. 1. Preparation of RTH Solution for Calibration:

The absorbance was measured at 274 nm in UV spectrophotometer using saline as blank. Experiments were carried out in triplicate.

Estimation of the drug in HPLC:

System suitability test (*4*).
Fig. 2. Preparation of solution for SST

Then 50 µl of SST solution was injected into the HPLC system and the column was eluted with mobile phase as suggested in USP monograph (2009).

Calibration curve for RTH in HPLC:

Fig. 3. Preparation of RTH solution for HPLC

Then 50 µl of these standards injected into the HPLC system and the column was eluted with mobile phase as suggested in the USP monograph (2009). The standard plot was obtained by plotting the area v/s the concentration.

Mobile Phase composition (*4*):

Take 6.6 g of dibasic ammonium phosphate and 1.1 gm of 1-heptane sulphonic acid sodium salt were taken & dissolved in 700 ml of HPLC grade water; 300 ml of methanol was then added. Then pH of this solution was adjusted 3.0 using o-phosphoric acid. Then solution was mixed, filtered, sonicated and run into the column with flow rate 1.5ml/min. The drug was detected using a wavelength 214nm of UV detector. The retention time was recorded approximetly 13 min under this condition. The solvent peak eluted at the 1st minute and there was also a slight disturbance in the peak which was due to presence of skin matter. To expedite the analysis, the lipophilicity of the mobile phase was slightly increased by increasing the proportion of methanol (700:330) which resulted in a retention time of approximately 7 minutes.

Partition Coefficient (*6*):

Octanol-water partition co-efficient:

Octanol- water were mutually saturated by shaking in separating funnel, kept aside for 24 h then separated. Standard solution of drug was prepared in this presaturated water. Octanol (5 ml) was added for the equal volume of this standard drug solution in a separating funnel and solution was kept for 24 h at 37±℃ with intermediate shaking. So, the water layer was separate out, and clarified by centrifugation and assayed for drug content.

Solubility determination (*6*):

Distilled water and ethanol were taken in small glass vials & solubility was determined by preparing saturated solution of the drug. Then drug was added till solution became cloudy, which indicated the presence of undissolved RTH. These solution was put 24 h for reached the equilibrium with the undissolved drug particles. Then seprated the supernatant and filtered through Whatman filter paper. Then filtrate was proparly diluted and then absorbance was measured at 274 nm using spectrophotometer using saline as blank. Experiments were carried out in triplicate.

Ex-vivo permeation study through porcine skin:

Preparation of Skin (*5*):

The density of the hair of human skin and pig skin is similar. So, pig skin was selected for the permeation studies. Freshly excised porcine's ear skin of about 3 to 5 months old pig was obtained from a local slaughter house. Muscles, fat layer, and tissue were separated from the underlying cartilage with a scalpel. Hairs were cut short and then skin was examined for pin holes. The skin was cut into the pieces of appropriate size and was used within 2 h.

Passive permeation study (*6*):

The ex-vivo permeation experiment of the RTH was carried out using freshly excised porcine skin. Vertical type Franz diffusion cell used for the study as shown in (fig.4). The above skin was mounted on

the diffusion cell and the receiver compartment was filled with 25ml of normal saline. The ex-vivo permeation study was conducted for 8 h. The samples were assayed by using HPLC system.

Fig.4. Vertical type Franz diffusion cell

Iontophoretic permeation (*6*):

For iontophoresis, diffusion cell was modified as suggested by Glikfield et. al. The apparatus consisted of a lower (receiver) compartment and two small upper compartments connected to the receiver through skin. The receiving chamber was large (25 ml) with two parallel ports on the top and a sampling port on the side. Two upper ports were actually cylindrical extension of receiving chambers which got converted into separate compartments when skin attached to the bottom of two small cylindrical glass tubes were slipped into them. The inner and outer tubes stayed attached by glass joints forming two separate chambers with skin as the base. Both the skin touched the receptor fluid at the same depth and each chamber housed one electrode. Drug solution (5 mg/ml) was filled in one compartment and 2 ml of water in the other. In case of cathodal iontophoresis, silver/silver chloride electrode was inserted into the donor compartment containing drug whereas silver plate was inserted into anodal chamber, which served as a return electrode. In case of anodal iontophoresis, silver electrode was inserted into the donor compartment containing drug whereas silver/silver chloride plate was inserted into cathodal chamber, which served as a return electrode. For pulsed current iontophoresis softline machine was used. The C-tech DC source outlet was attached to softline timer machine to generate a modulated current with an on-off cycle of 10 sec. Other procedures were kept same as constant current iontophoresis. Modulated current was investigated only for anodal delivery. Whole course of experiment was carried out at the room temperature. The receptor fluids (5 ml) were withdrawn at hourly interval and replaced with fresh saline to maintain sink condition. The permeation study was carried out for a period of 8 h. The samples were assayed by using HPLC system.

Fig.5. Modified Glickfield Diffusion Cell for direct constant current iontophoresis.
Fig.6. Modified Glickfield Diffusion Cell for modulated current iontophoresis.

Data analysis (*6*):

Permeability coefficient:

The cumulative amount of the drug was permeated as per unit skin surface area and was plotted against time, and the slope of linear portion of the plot was estimated as the steady state flux (Jss). The permeability coefficient (Kp) was calculated as,

$$K_p = \frac{J_{ss}}{C} \ (\text{cm h}^{-1})$$

Where,
Cd = Concentration of drug in donor compartment.

Diffusion coefficient:

The diffusion coefficient of the drug was calculated using the formula,

$$D = \frac{K \cdot h}{K} \ (\text{cm}^2\,\text{s}^{-1})$$

Where, h = thickness of skin
K= partition coefficient.

RESULTS AND DISCUSSION

Standard Plot of RTH in 0.9% NaCl:

The standard calibration curve of RTH in 0.9% NaCl is depicted as Fig.7. The data of absorbance was shown in Table 1. The data had correlation coefficient of 0.999 and the equation of regressed line is Y = 0.047 X.

Table.1 Standard curve of RTH in 0.9% NaCl at 274.0 nm. (Spectrophotometric analysis)

Fig.7. Standard curve of RTH in 0.9% NaCl at 274.0 nm.

Estimation of drug in HPLC system:

System suitability test for RTH:
SST chromatogram showed distinct peaks with relative retention time of 0.3 for tyramine, 0.61 for erythro-1-(4-ketocyclohexyl)-2-{(1-hydroxyphenethyl)amino} propanol-1, 0.8 for erythro-p-hydroxyl{1-(4-ketocyclohexlethyl)amino}ethyl benzyl alcohol, 1.0 for ritodrine, 1.15 for threo diasteriomer of ritodrine. (Fig.8)

Fig.8.Chromatogram of SST for RTH.

Precision studie for RTH:

The instrument was validated for precision and it was found that the relative standard deviation (RSD) of the samples analyzed were within 3 %. The data for precision is given below in Table 2.

Table.2 Sample Area for precision studies

Fig.9.Chromatograms of RTH. (Conc. 0.05 µg/ml) n=6

Fig.10.Chromatograms of RTH. (Conc. 200 µg/ml) n=6

Fig.11.Chromatograms of RTH. (Conc. 400 µg/ml) n=6

HPLC Standard plot for RTH in 0.9 % NaCl:

The standard calibration curve of RTH in 0.9% NaCl is shown in Fig.12. The data of sample area is shown in Table.3. The data had correlation coefficient of 1.0 and the equation of the regressed line is Y = 71.08x

Table.3 Standard calibration curve of RTH.

Fig.12. Calibration curve of RTH in HPLC at 274nm

Table.4 Physicochemical parameters of RTH.

Ex-vivo permeation study through porcine skin:

The diffusate from the skin permeation studies were injected into the HPLC column in order to identify the peaks due to the molecules leached out of the skin. Fig.13. shows the chromatogram of saline and Fig.14 shows the chromatogram of blank skin diffusate obtained from blank experiments.

Fig.13. Chromatogram of saline. (Receptor fluid)

Fig.14. Chromatogram of skin diffusate. (Vehicle-Saline)

Table.5 Passive permeation data of RTH in excised pig skin.

Fig.15. Passive permeation profile of RTH in excised pig skin.

Table 6. Iontophoretic permeation (Cathodal) data of RTH under the influence of constant direct current. (Current density 0.1 mA/cm^2).

Fig. 16. Iontophoretic permeation (cathodal) profile of RTH in excised pig skin under the influence of constant direct current. (Current density 0.1 mA/cm^2)

Table 7. Iontophoretic permeation (Cathodal) data of RTH under the influence of constant direct current. (Current density 0.5 mA/cm^2).

Fig.17. Iontophoretic permeation (cathodal) profile of RTH in excised pig skin under the influence of constant direct current. (Current density 0.5 mA/cm^2).

Table.8 Iontophoretic permeation (Anodal) data of RTH under the influence of constant direct current. (Current density 0.1 mA/cm^2).

Fig.18. Iontophoretic permeation (anodal) profile of RTH in excised pig skin under the influence of constant direct current. (Current density 0.1 mA/cm^2).

Table.9 Iontophoretic permeation (Anodal) data of RTH under the influence of constant direct current. (Current density 0.5 mA/cm^2).

Fig.19. Iontophoretic permeation (anodal) profile of RTH in excised pig skin under the influence of constant direct current. (Current density 0.5 mA/cm^2).

Table 10. Iontophoretic permeation (anodal pulsed current) data of RTH under the influence of constant direct current. (Current density 0.1 mA/cm^2).

Fig.20. Iontophoretic permeation (anodal pulsed current) profile of RTH in excised pig skin under the influence of constant direct current.

Table 11. Iontophoretic permeation (anodal pulsed current) data of RTH under the influence of constant direct current. (Current density 0.5 mA/cm^2).

Fig.21. Iontophoretic permeation (anodal pulsed current) profile of RTH in excised pig skin under the influence of constant direct current.

Table. 12 Comparison of iontophoretic permeation data against passive permeation under the influence of constant current density 0.1 mA/cm^2.

Fig.22. Comparison of iontophoretic permeation profile against passive permeation under the influence of constant current density 0.1 mA/cm^2.

Table 13. Comparison of iontophoretic permeation data against passive permeation under the influence of constant current density 0.5 mA/cm^2.

Fig.23. Comparison of iontophoretic permeation profile against passive permeation under the influence of constant current density 0.5 mA/cm^2.

Table.14 Steady state flux, permeability coefficient, diffusion coefficient and enhancement ratio of ritodrine hydrochloride for passive and cathodal iontophoresis.

Table15. Steady state flux, permeability coefficient, diffusion coefficient and enhancement ratio of ritodrine hydrochloride for passive and anodal iontophoresis.

Table.16 Steady state flux, permeability coefficient, diffusion coefficient and enhancement ratio of ritodrine hydrochloride for passive and anodal pulsed current iontophoresis.

Discussion:

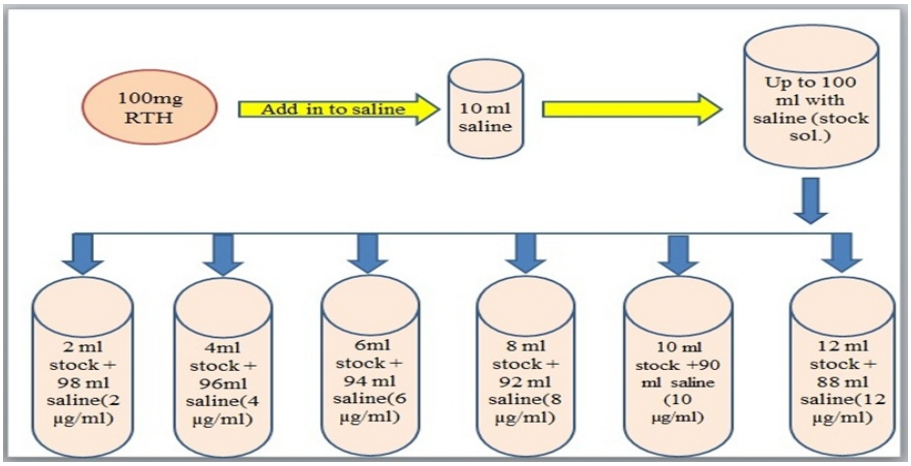
Prolonged exposure to the constant direct current is associated with the polarization of skin. Usually the resistance dips at the later period of the study and the permeation rate of drugs may become unpredictable. Hence, studies were carried out under modulated direct current too. An on-off cycle of 10 sec was employed. In constant current study, the total electrical doses for 8 hwere 48, 120 and 240 mA-min for the current densities of 0.1 and 0.5 mA/cm^2 respectively. In modulated current the doses were reduced to 24, 60 and 120 mA-min.Increase in current density to the threshold of 0.5 mA/cm^2 increased the permeation many folds where 57% of RTH permeated at the end of 8th hr.s in case of both cathodal and anodal iontophoresis study.

CONCLUSION:

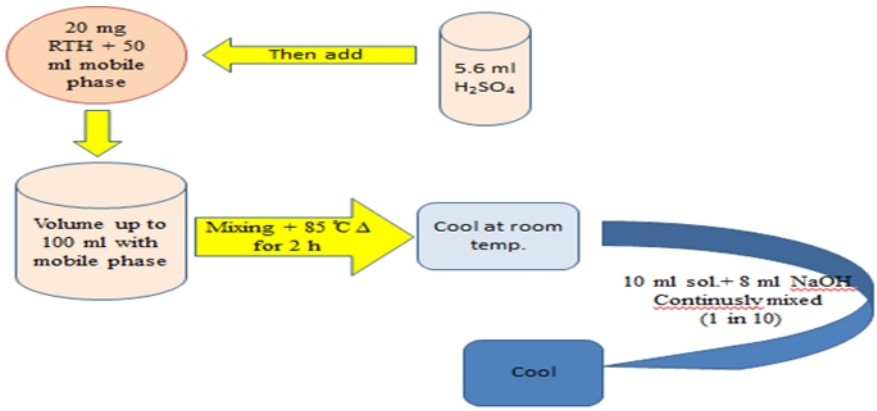
In the present study effort was put forward to assess the ex-vivo permeability of a hydrophilic drug RTH of the porcine ear skin using iontophoresis technique. The permeation profile of passive studie indicated that cumulative flux at the end of the 8th h was very low 63.809 µg/cm^2 which was very low below the target requirement. In the initial experiments both of anodal and cathodal iontophoresis were attempted with a very low current density like 0.1 mA/cm^2 and the permeation rate increased drastically and the cumulative flux at the end of 8th h was 609.598 µg/cm^2 and 713.399 µg/cm^2 respectively. Increase in current density to the threshold of 0.5 mA/cm^2 increased the permeation many folds where 57% of RTH permeated at the end of 8th hr.s in case of both cathodal and anodal iontophoresis study.

RTH on dissociation forms positively charged drug ions and its iontophoretic flux should consist of all the contribution of passive, electro repulsive flux as well as electro osmotic flux. In contrast, from the cathodal side, only passive contribution was expected. High cathodal flux indicated that electroporation assisted with iontophoresis could be the dominant contribution for RTH permeation. Since the voltage on the cathodal side was high, there was a possibility that pores had been induced on the skin surface which provided a shunt pathway of permeation and contributed towards the higher flux. So it appears that energy assisted iontophoretic drug delivery was a promising technique for transdermal delivery of hydrophilic drug RTH.

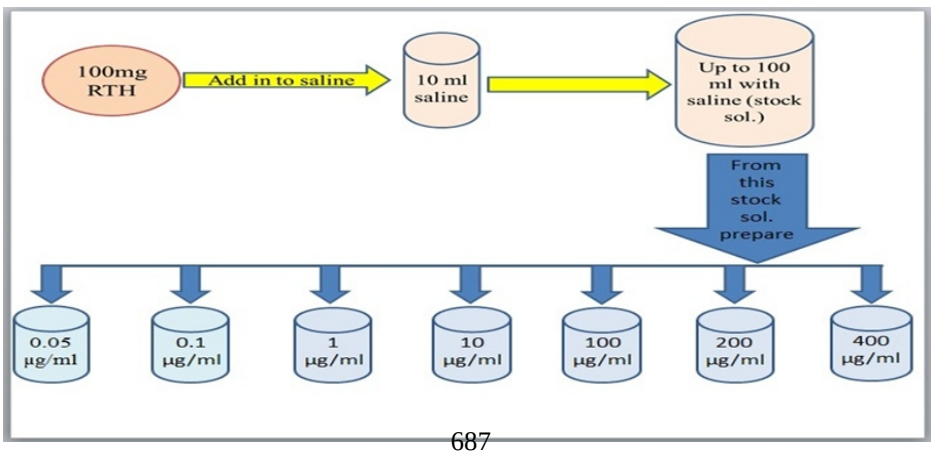
FIGURES



"**Fig. 1**. Preparation of RTH Solution for Calibration."



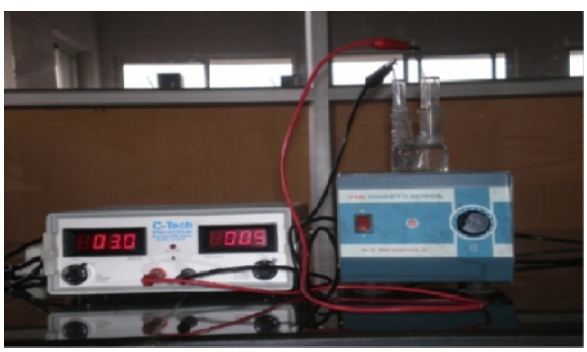
"**Fig. 2**. Preparation of solution for SST."



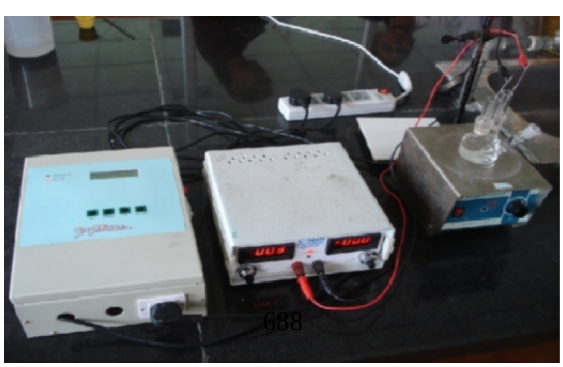
"**Fig. 3**. Preparation of RTH solution for HPLC."



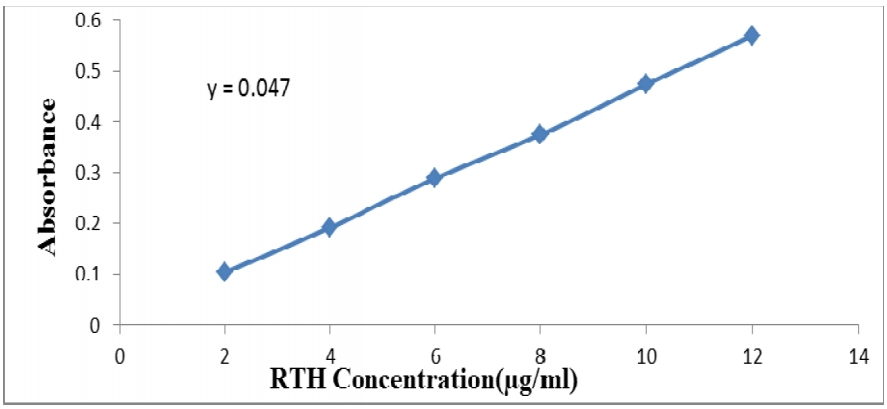
"**Fig.4**. Vertical type Franz diffusion cell."



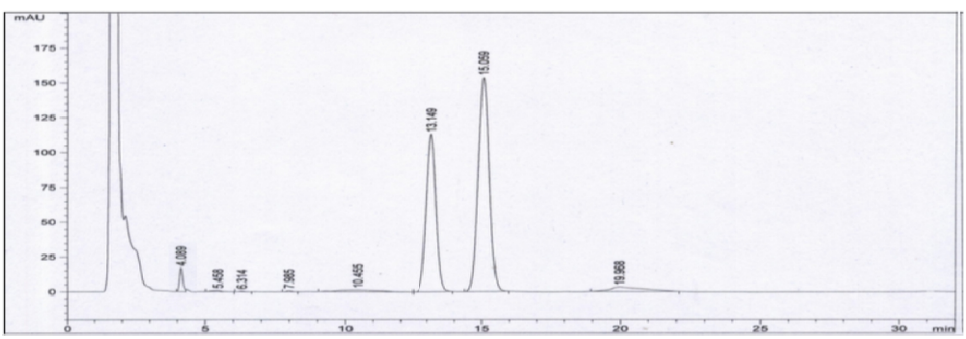
"**Fig.5**. Modified Glickfield Diffusion Cell for direct constant current iontophoresis."



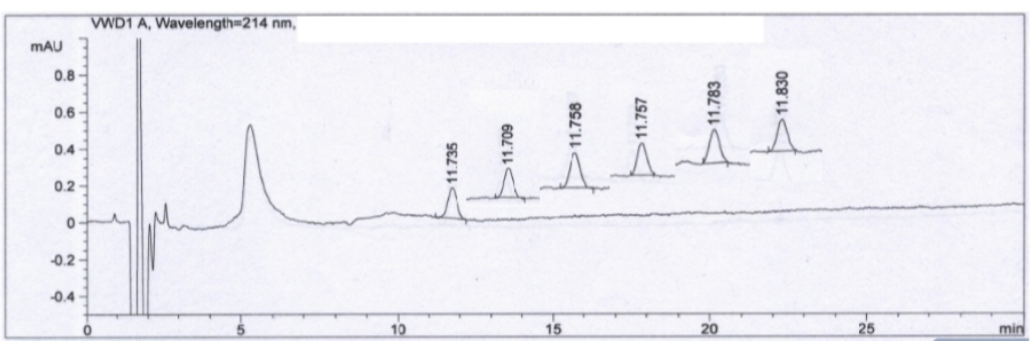
"**Fig.6**. Modified Glickfield Diffusion Cell for modulated current iontophoresis."



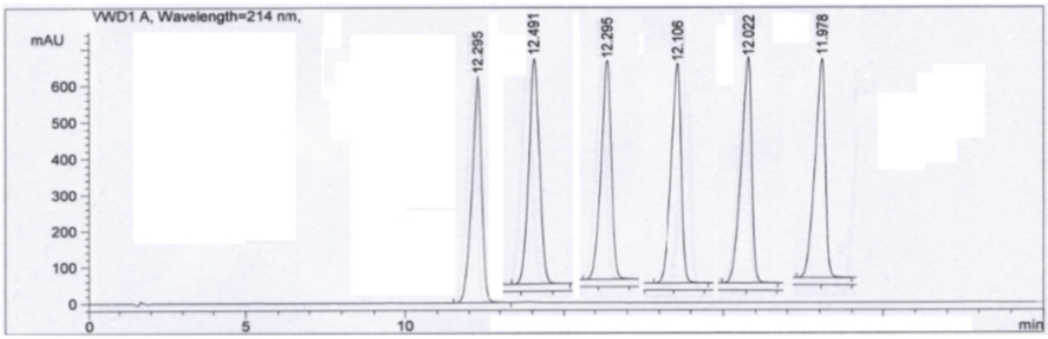
"**Fig.7**. Standard curve of RTH in 0.9% NaCl at 274.0 nm."



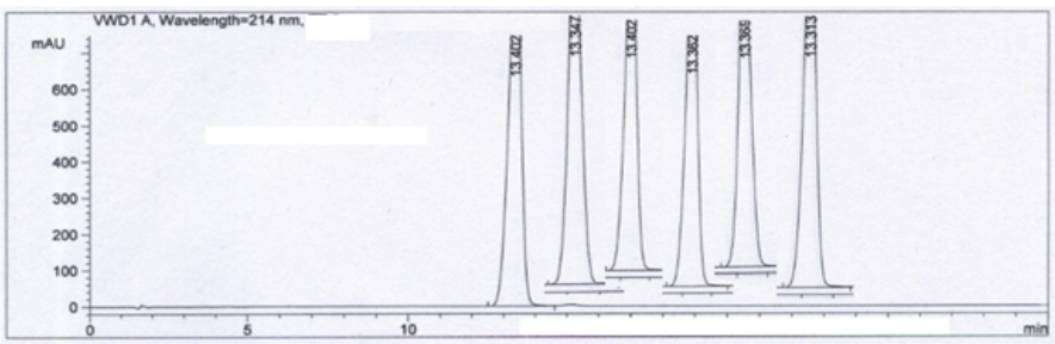
"**Fig.8**.Chromatogram of SST for RTH."



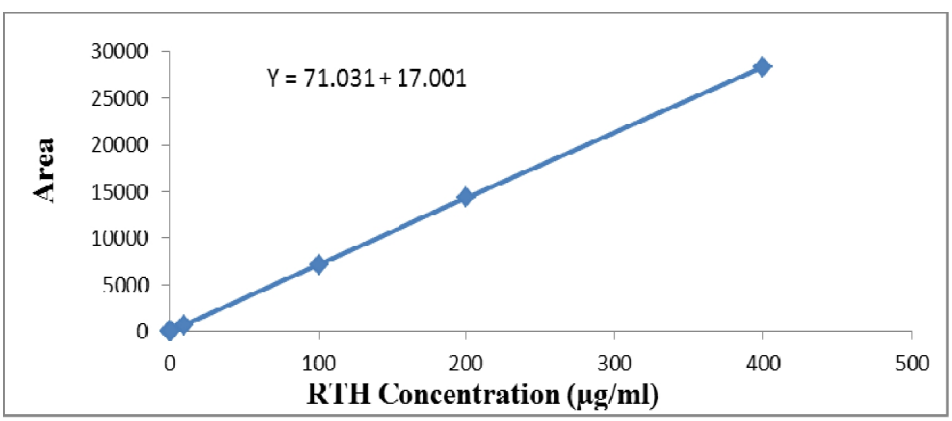
"**Fig.9**.Chromatograms of RTH. (Conc. 0.05 µg/ml) n=6."



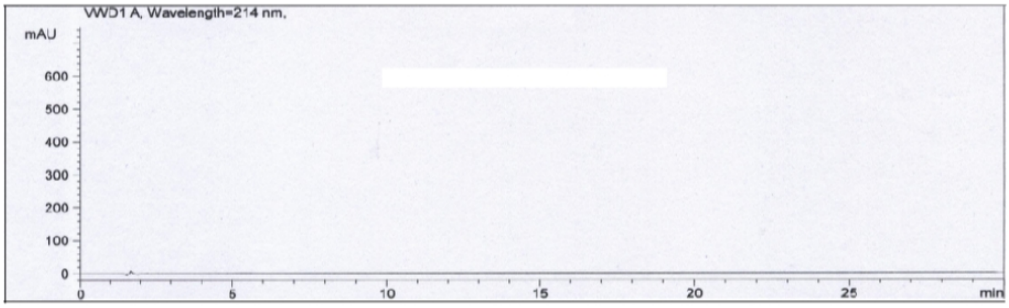
"**Fig.10**.Chromatograms of RTH. (Conc. 200 µg/ml) n=6."



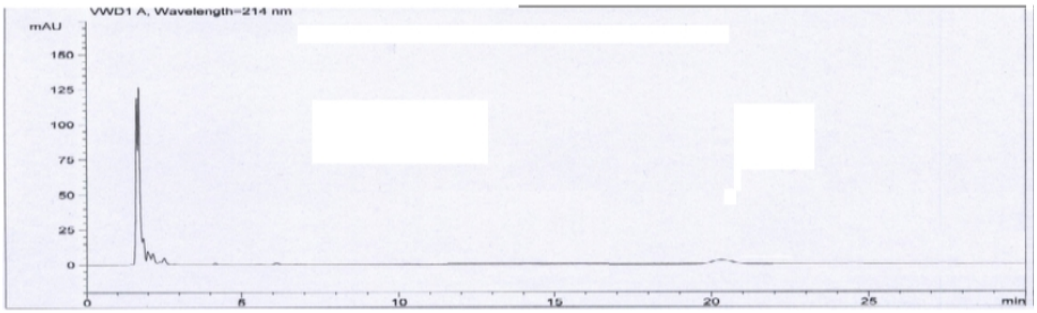
"**Fig.11**.Chromatograms of RTH. (Conc. 400 µg/ml) n=6."



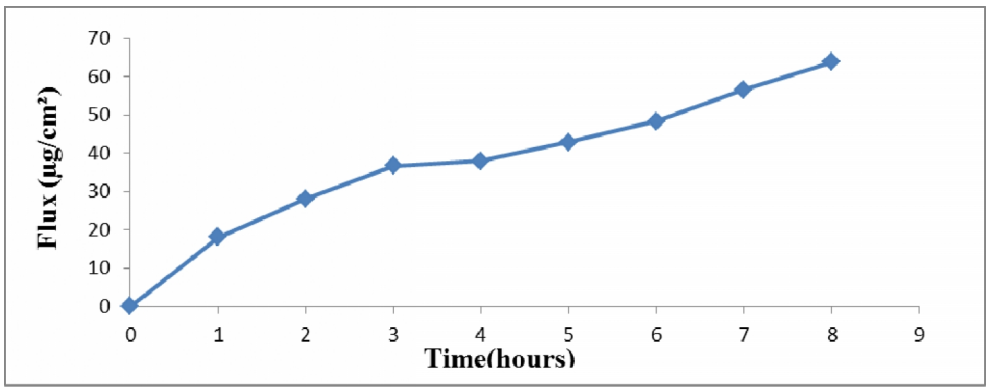
"**Fig.12**. Calibration curve of RTH in HPLC at 274nm."



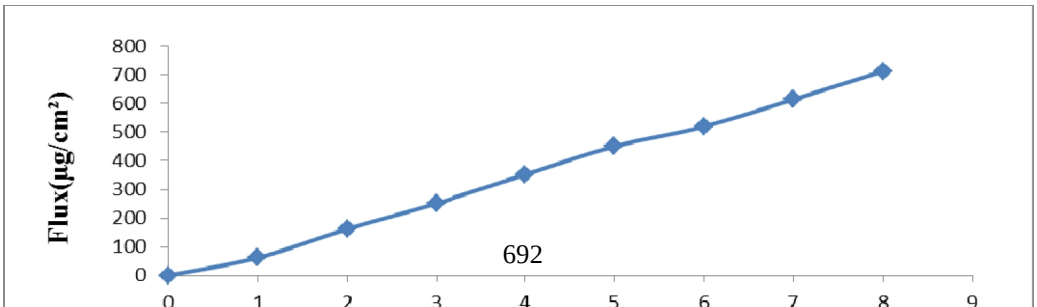
"**Fig.13**. Chromatogram of saline. (Receptor fluid)."



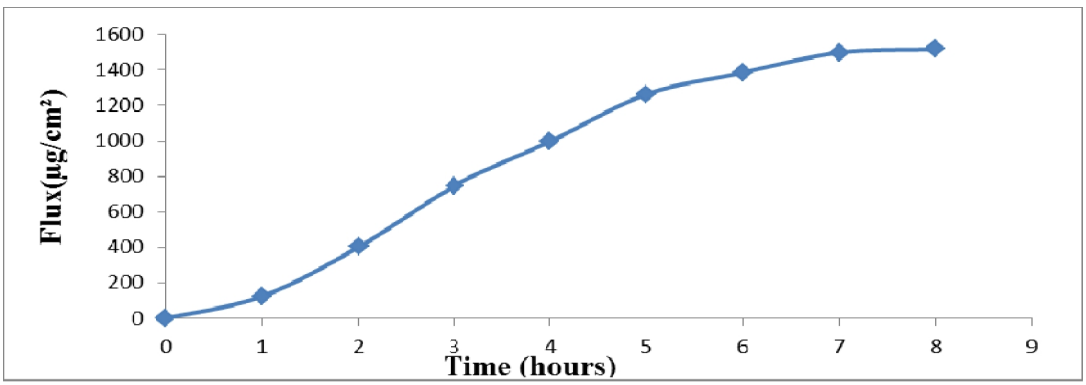
"**Fig.14**. Chromatogram of skin diffusate. (Vehicle-Saline)."



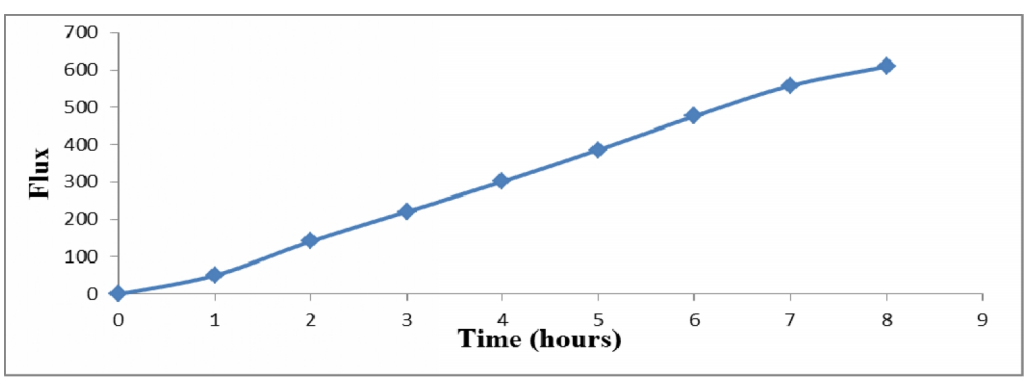
"**Fig.15**. Passive permeation profile of RTH in excised pig skin."



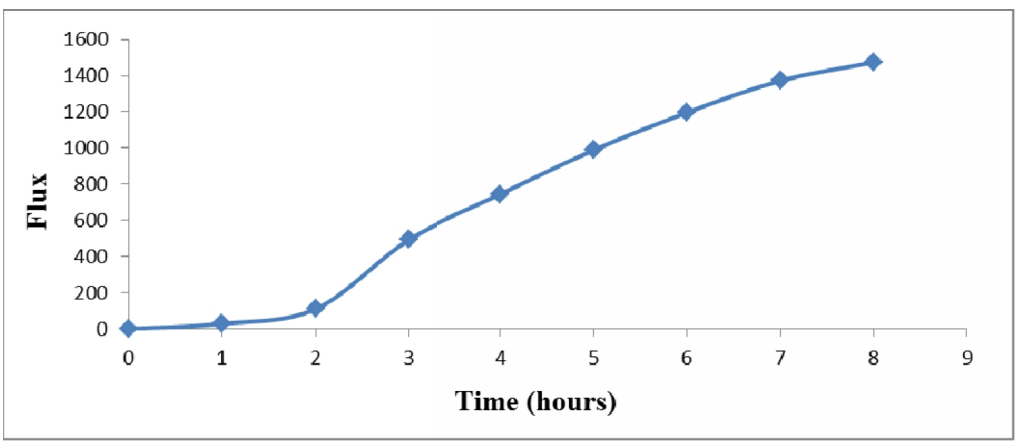
"**Fig. 16**. Iontophoretic permeation (cathodal) profile of RTH in excised pig skin under the influence of constant direct current. (Current density 0.1 mA/cm2)."



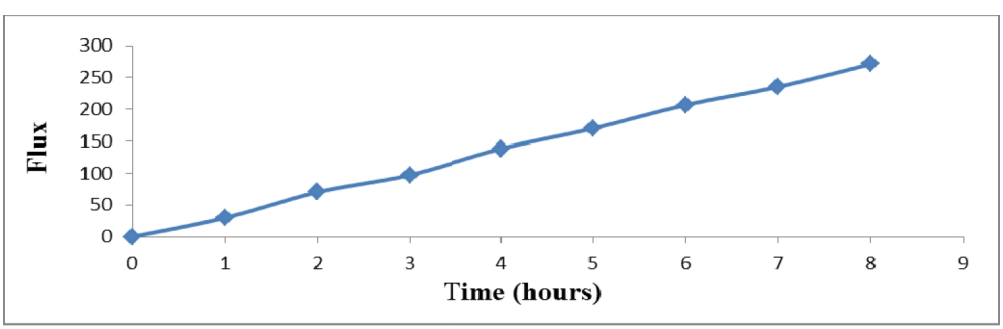
"**Fig.17**. Iontophoretic permeation (cathodal) profile of RTH in excised pig skin under the influence of constant direct current. (Current density 0.5 mA/cm2)."



"**Fig.18**. Iontophoretic permeation (anodal) profile of RTH in excised pig skin under the influence of constant direct current. (Current density 0.1 mA/cm2)."

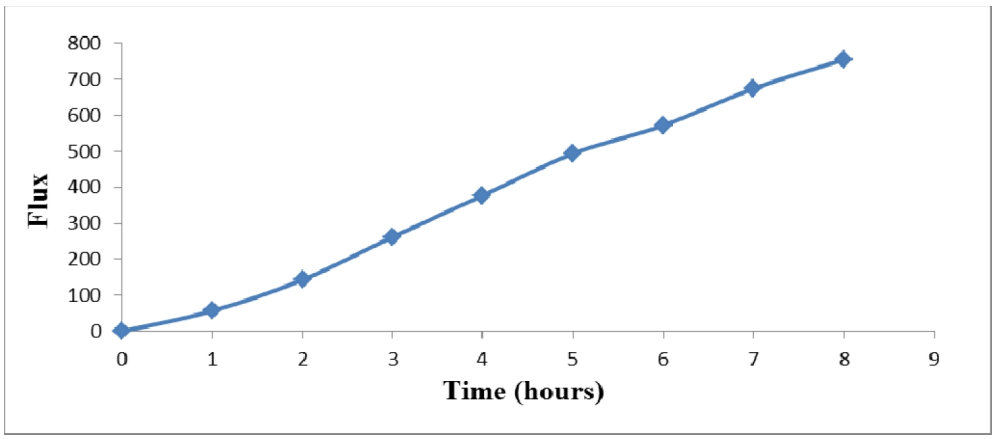


"**Fig.19**. Iontophoretic permeation (anodal) profile of RTH in excised pig skin under the influence of constant direct current. (Current density 0.5 mA/cm2)."

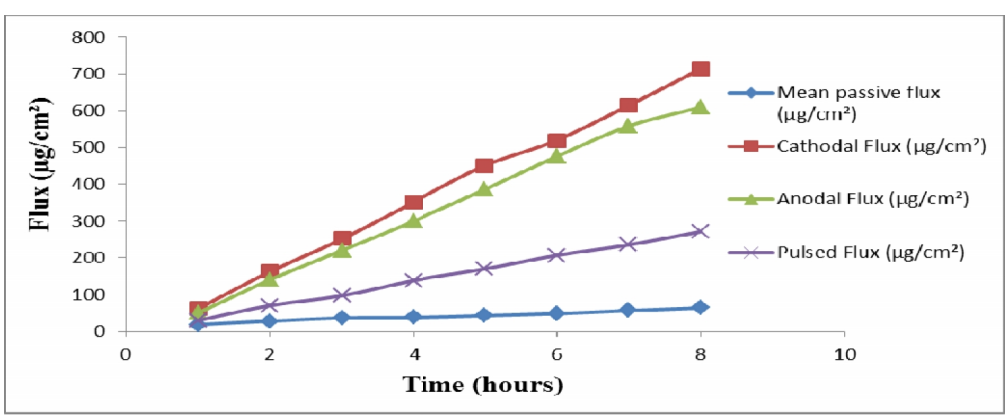


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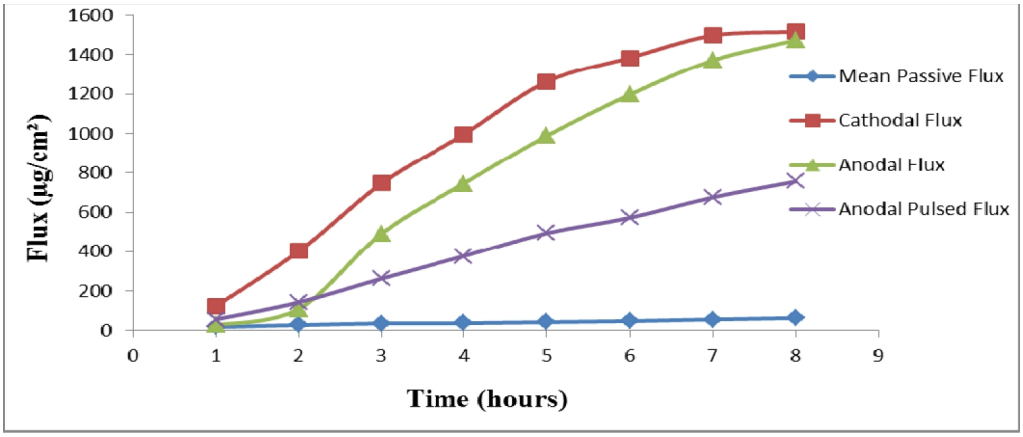
"**Fig.20**. Iontophoretic permeation (anodal pulsed current) profile of RTH in excised pig skin under the influence of constant direct current."



"**Fig.21**. Iontophoretic permeation (anodal pulsed current) profile of RTH in excised pig skin under the influence of constant direct current."



"**Fig.22**. Comparison of iontophoretic permeation profile against passive permeation under the influence of constant current density 0.1 mA/cm2."



"**Fig.23**. Comparison of iontophoretic permeation profile against passive permeation under the influence of constant current density 0.5 mA/cm2."

TABLES

"**Table.1** Standard curve of RTH in 0.9% NaCl at 274.0 nm."
(Spectrophotometric analysis)

Sr. No.	Concentration (µg/ml)	Absorbance			Average*
		Trial 1	Trial 2	Trial 3	
1	2	0.084	0.115	0.113	0.1040±0.017
2	4	0.187	0.197	0.193	0.1923±0.005
3	6	0.282	0.3	0.285	0.2890±0.010

4	8	0.376	0.378	0.37	0.3747±0.004
5	10	0.473	0.473	0.475	0.4737±0.001
6	12	0.565	0.571	0.571	0.5690±0.004

***The values represent mean ± SD**

"**Table.2** Sample Area for precision studies."

Sr.No.	**AREA OF RTH PEAK**		
	(0.05 µg/ml) mAU×S	**(0.05 µg/ml) mAU×S**	**(400 µg/ml) mAU×S**
1	3.412	14369.7	28364.0
2	3.372	14408.7	28416.8
3	3.568	14441.5	28413.1
4	3.637	14420.7	28434.9
5	3.472	14463.5	28445.3
6	3.586	14463.5	28456.4
Average	**3.5**	**14409.3**	**28421.8**
SD	0.1	42.4	32.7
RSD (%)	**3.0**	**0.29**	**0.12**

***mAU= Milli absorption unit * S = Second.**

"**Table.3** Standard calibration curve of RTH."

Sr. No.	**Concentration (µg/ml)**	**Peak area (mAU×S)***
0	0	0
1	0.05	3.57
2	0.10	6.90
3	1	64.9
4	10	718.1
5	100	7155.4
6	200	14336.6
7	400	28364.0

***mAU=Milli absorption unit * S = Seconds.**

"**Table.4** Physicochemical parameters of RTH."

Sr. No.	**Physicochemical parameters**	**Results**
1	Solubility in water	6.835 g/ml.
2	Solubility in ethanol	3.820 g/ml.
3	Partition coefficient (Octanol: Water)	0.2474 ± 0.0205*
4	pKa	8.6

***The values represent mean ± SD**

"**Table.5** Passive permeation data of RTH in excised pig skin. (Donor concentration 5 mg/ml). (Skin area= 3.8 cm^2) *n=3"

Time (hours)	Flux µg/cm^2			Mean±SD µg/cm^2
	Trial 1	Trial 2	Trial 3	
0	0	0	0	0
1	32.591	5.532	17.972	18.698 ±7.820
2	47.911	14.671	21.598	28.060 ±10.125
3	66.112	18.146	25.649	36.636 ±14.880
4	63.455	21.345	29.077	37.959 ±12.942
5	68.307	29.008	31.375	42.897 ±12.723
6	75.354	35.143	34.412	48.303 ±13.527
7	79.207	53.914	36.716	56.612 ±12.340
8	91.882	60.204	39.341	63.809 ±15.274

"**Table 6.** Iontophoretic permeation (Cathodal) data of RTH under the influence of constant direct current. (Current density 0.1 mA/cm^2)."

Time (hrs)	Sample area (mAU×S)*	Concentration jig/ml	Quantity jig/25ml	Loss jig/5ml	Cumulative permeation jigs	Total flux jig/cm^2
0	0.000	0.000	0.000	0.000	0.000	0.000
1	678.158	9.413	235.320	47.064	235.320	61.926
2	1646.016	22.847	571.164	114.233	618.228	162.692
3	2290.313	31.789	794.734	158.947	956.031	251.587
4	2931.262	40.686	1017.142	203.428	1337.386	351.944
5	3425.769	47.549	1188.735	237.747	1712.407	450.634
6	3494.678	48.506	1212.647	242.529	1974.066	519.491
7	3835.441	53.236	1330.891	266.178	2334.839	614.431
8	4152.148	57.632	1440.788	288.158	2710.914	713.399

***mAU= Milli absorption unit * S = Second.**

"**Table 7.** Iontophoretic permeation (Cathodal) data of RTH under the influence of constant direct current. (Current density 0.5 mA/cm^2)."

Time (hrs)	Sample area (mAU×S)*	Concentration jig/ml	Quantity jig/25ml	Loss jig/5ml	Cumulative permeation jigs	Total flux jig/cm^2
0	0.000	0.000	0.000	0.000	0.000	0.000
1	1356.801	18.832	470.808	94.162	470.808	123.897
2	4133.696	57.375	1434.385	286.877	1528.546	402.249
3	7078.374	98.247	2456.182	491.236	2837.221	746.637
4	8395.477	116.529	2913.215	582.643	3785.490	996.181
5	9635.386	133.738	3343.461	668.692	4798.379	1262.731

6	9045.500	125.551	3138.771	627.754	5262.382	1384.837
7	8475.967	117.646	2941.145	588.229	5692.509	1498.029
8	6991.809	97.046	2426.144	485.229	5765.738	1517.299

***mAU= Milli absorption unit * S = Second.**

"**Table.8** Iontophoretic permeation (Anodal) data of RTH under the influence of constant direct current. (Current density 0.1 mA/cm^2)."

Time (HRS)	Sample area (mAU×S)	Concentration jig/ml	Quantity jig/25ml	Loss jig/5ml	Cumulative Permeation jigs	Total flux jig/cm^2
0	0.000	0.000	0.000	0.000	0.000	0.000
1	549.067	7.621	190.525	38.105	190.525	50.138
2	1430.894	19.861	496.517	99.303	534.623	140.690
3	2023.255	28.083	702.066	140.413	839.474	220.914
4	2487.019	34.520	862.991	172.598	1140.813	300.214
5	2928.530	40.648	1016.194	203.239	1466.614	385.951
6	3324.255	46.140	1153.510	230.702	1807.169	475.571
7	3564.924	49.481	1237.022	247.404	2121.383	558.259
8	3414.162	47.388	1184.708	236.942	2316.473	609.598

***mAU= Milli absorption unit * S = Second.**

"**Table.9** Iontophoretic permeation (Anodal) data of RTH under the influence of constant direct current. (Current density 0.5 mA/cm^2)."

Time (HRS)	Sample area (mAU×S)*	Concentration jig/ml	Quantity jig/25ml	Loss jig/5ml	Cumulative permeation jigs	Total flux jig/cm^2
0	0.000	0.000	0.000	0.000	0.000	0.000
1	309.431	4.295	107.372	21.474	107.372	28.256
2	1139.603	15.818	395.440	79.088	416.915	109.714
3	5105.173	70.859	1771.485	354.297	1872.048	492.644
4	6849.153	95.066	2376.643	475.329	2831.503	745.132
5	8141.899	113.009	2825.224	565.045	3755.412	988.266
6	8794.785	122.071	3051.774	610.355	4547.007	1196.581

7	8954.708	124.291	3107.267	621.453	5212.855	1371.804
8	8289.500	115.058	2876.441	575.288	5603.482	1474.601

***mAU= Milli absorption unit * S = Second.**

"**Table 10.** Iontophoretic permeation (anodal pulsed current) data of RTH under the influence of constant direct current. (Current density 0.1 mA/cm^2)."

Time (HRS)	Sample area (mAU×S)*	Concentration jig/ml	Quantity jig/25ml	Loss jig/5ml	Cumulative Permeation jigs	Total flux jig/cm^2
0	0.000	0.000	0.000	0.000	0.000	0.000
1	324.7213	4.507	112.678	22.536	112.678	29.652
2	704.9811	9.785	244.627	48.925	267.163	70.306
3	859.2034	11.926	298.142	59.628	369.603	97.264
4	1139.1173	15.811	395.272	79.054	526.361	138.516
5	1255.521	17.427	435.663	87.133	645.807	169.949
6	1407.506	19.536	488.402	97.680	785.678	206.757
7	1439.1879	19.976	499.395	99.879	894.352	235.356
8	1544.3838	21.436	535.898	107.180	1030.734	271.246

"**Table 11.** Iontophoretic permeation (anodal pulsed current) data of RTH under the influence of constant direct current. (Current density 0.5 mA/cm^2)."
(On-off cycle= 10 sec)

Time (HRS)	Sample area (mAU×S)*	Concentration jig/ml	Quantity jig/25ml	Loss jig/5ml	Cumulative permeation jigs	Total flux jig/cm^2
0	0.000	0.000	0.000	0.000	0.000	0.000
1	622.348	8.638	215.954	43.191	215.954	56.830
2	1442.178	20.017	500.433	100.087	543.624	143.059
3	2461.819	34.170	854.247	170.849	997.524	262.506
4	3219.329	44.684	1117.101	223.420	1431.228	376.639
5	3855.147	53.509	1337.729	267.546	1875.276	493.494
6	3944.266	54.746	1368.653	273.731	2173.746	572.038
7	4282.751	59.444	1486.106	297.221	2564.930	674.981

8	4310.418	59.828	1495.707	299.141	2871.751	755.724

"**Table. 12** Comparison of iontophoretic permeation data against passive permeation under the influence of constant current density 0.1 mA/cm^2."

Time (HRS)	Mean Passive Flux (µg/cm^2)	Cathodal Flux (µg/cm^2)	Anodal Flux (µg/cm^2)	Pulsed Flux (µg/cm^2)
1	18.698	61.926	50.138	29.652
2	28.060	162.692	140.690	70.306
3	36.636	251.587	220.914	97.264
4	37.959	351.944	300.214	138.516
5	42.897	450.634	385.951	169.949
6	48.303	519.491	475.571	206.757
7	56.612	614.431	558.259	235.356
8	63.809	713.399	609.598	271.246

"**Table 13.** Comparison of iontophoretic permeation data against passive permeation under the influence of constant current density 0.5 mA/cm^2."

Time (HRS)	Mean Passive Flux (µg/cm^2)	Cathodal Flux (µg/cm^2)	Anodal Flux (µg/cm^2)	Anodal Pulsed Flux (µg/cm^2)
1	18.698	123.897	28.256	56.830
2	28.060	402.249	109.714	143.059
3	36.636	746.637	492.644	262.506
4	37.959	996.181	745.132	376.639
5	42.897	1262.731	988.266	493.494
6	48.303	1384.837	1196.581	572.038
7	56.612	1498.029	1371.804	674.981
8	63.809	1517.299	1474.601	755.724

"**Table.14** Steady state flux, permeability coefficient, diffusion coefficient and enhancement ratio of

ritodrine hydrochloride for passive and cathodal iontophoresis."

Sr.No	Parameters	Passive permeation	Cathodal Iontophoresis	
			0.1mA/cm^2	0.5 mA/cm^2
1	Steady state flux (jig cm^{-2} h^{-1})	6.173	92.360	130.280
2	Permeability coefficient (cm hr^{-1})	1.235	18.472	26.056
3	Diffusion Coefficient (cm^{2}s^{-1})× 10^{-4}	4.44	66.400	93.620
4	Enhancement ratio	1.000	14.955	21.085

"**Table15.** Steady state flux, permeability coefficient, diffusion coefficient and enhancement ratio of ritodrine hydrochloride for passive and anodal iontophoresis."

Sr. No	Parameters	Passive permeation	Anodal Iontophoresis	
			0.1mA/cm^2	0.5 mA/cm^2
1	Steady state flux (jig cm^{-2} h^{-1})	6.173	77.346	179.98
2	Permeability coefficient (cm hr^{-1})	1.235	15.470	35.996
3	Diffusion coefficient (cm^{2}s^{-1})× 10^{-4}	4.44	55.582	129.330
4	Enhancement ratio	1.000	12.520	29.130

"**Table.16** Steady state flux, permeability coefficient, diffusion coefficient and enhancement ratio of ritodrine hydrochloride for passive and anodal pulsed current iontophoresis."

Sr.No	Parameters	Passive permeation	Anodal pulsed current Iontophoresis	
			0.1mA/cm^2	0.5 mA/cm^2

1	Steady state flux (jig cm^{-2} h^{-1})	6.173	33.766	98.644
2	Permeability coefficient (cm hr^{-1})	1.235	6.753	19.730
3	fusion coefficient (cm^2s^{-1})× 10^{-4}	4.44	24.260	70.89
4	Enhancement ratio	1.000	5.464	15.966

ACKNOWLEDGEMENT

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Statistical framework to determine market potential of a pharmaceutical product

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ABSTRACT

In the era of cut throat competition, it is essential for a pharmaceutical industry to put right product at right time and in right market. In the process to determine market potential of the product, product development team considers various aspects of the product. Thus to adapt statistical method to determine market potential, a proposed statistical scale was developed comprising two main aspects, i.e. risk factors and backing factors of the pharmaceutical product. These factors were further allotted weightage according to their importance in market potential. Risk factors were given negative weightage and backing factors were given positive weightage in a way that total weightage of either risk factor or supportive factor sum up to 10. Pharmaceutical product was allotted one of the four points (0, 1, 2 or 3) for individual factors. Sum of the scores (weightage X point allotted) of all the factors gives market potential of the product.

SUMMARY

Statistical assistance to determine market potential of a pharmaceutical product.

Keywords: Market potential scale, risk factors, backing factors, point allocation

INTRODUCTION

Today, pharmaceutical industry is passing through very tight situation for balancing between the regulatory compliance and market penetration of their products to cover optimum market share and growth. Pharmaceutical product management team (PMT) utilize their prior knowledge, expertise and experience to analyse various risk factors and backing factors associated with the market potential of the product. There is a lack of process to determine market potential of a pharmaceutical product in quantitative terms. Thus it becomes tricky to the PMT to come on the firm conclusion (*1, 2*).

In present study, an arbitrary scale covering the factors affecting market potential was developed to determination the market potential of a pharmaceutical product in quantitative terms. Every factors were allotted weightage according to their importance and experience based prior knowledge. They were further divided into point allocation scale to allot specific point to the product based on the criteria of that point. Multiplication of allotted point to the allotted weightage gives the point earned by the product for particular factor. Addition of all the earned points results in the market potential of the product in numeric term. The market potential of the product is indicated by the sign and magnitude of the number.

MATERIALS AND METHODS

Materials

Microsoft Excel 2013 software utilized for mathematical treatment and related calculations.

Methods

Simple statistical method was adopted to derive market potential of a pharmaceutical product in terms of numbers. Factors influencing market potential of the product was categorized as risk factors and backing factors. **Table 1** represents the overview of weightage and point allocation criteria of risk factors. Weightage of the factor was given out of total weightage of 10 points according to their importance and prior knowledge (*3, 4*).

Similarly, **Table 2** represents the overview of weightage and point allocation criteria of backing factors. Risk factors and backing factors were allotted negative and positive sign as they impart negative and positive effect on market potential, respectively.

Flexibility of the scale is reflected in the factor selection and point allocation of scale itself. One can select any number of factors and distribute weightage according to their importance. The number of risk factors need not to be equal to the number of backing factors. Moreover, point allocation criteria can also be varied; i.e. one can construct point allocation scale according to their requirements with any number of criteria. One can remove a factor or use zero (0) point allocation criteria when the factor is not to be incorporated to the scale.

Further elaboration of the point allocation criteria for each point may be defined to get more clarity of the same (**Table 3 and Table 4**).

An example of novel modified release extended delivery system of cinnarizine was considered to determine its market potential. **Table 5** and **Table 6** represents the earned risk factor score and backing factor score of the sample product, respectively.

RESULTS AND DISCUSSION

Range of the developed scale for risk factors was ranging from 0 to -30. If none of the risk factor is present, all factors scores zero '0' and hence sum of all factors will be 0. Similarly in the case of all the factors scoring '-3', the sum of scores will be -30.

On the contrary, scale of the backing factors was ranging from 0 to 30. Thus, market potential of the product may be any number between -30 to 30. Weightage on the either side was 10. This can be a limitation of the developed scale that the total weightage on the either side as well as points of the point allocation scale must be same. In other words, the range of the scale for risk factors and backing factors must be evenly distributed on either side.

Sum of risk factor score and backing factor score gives market potential of the product. Positive market potential represents good potential of the product in the market while negative market potential represents improvement in the specific criteria scoring poor point allocation.

CONCLUSION

Risk factor score of -16 and backing factor score of 21 earned by the sample product revealed that the product has positive market potential of 5.5 (-16 + 21). But low number indicated the scope for improvement needed to increase market potential. For example, cost effective product can be produced to reduce negative impact of production cost.

FIGURES

N.A.

TABLES

Table 1. List of risk factors with weightage and point allocation criteria

Sr. no.	Risk factor	Weightage	Point allocation			
			0	-1	-2	-3
1	Competitor product availability	2.5	Factor not applicable	Not available	Available (low competition)	Available (High competition)
2	Production cost	2.5		Low	Acceptable	High
3	Stability	2.5		High	Acceptable	Low
4	Safety	1.5		High	Acceptable	Less
5	Regulatory compliance	1		High	Medium	Low
Scale range = 0 to -30						

Table 2. List of backing factors with weightage and point allocation criteria

Sr. no.	Backing factor	Weightage	Point allocation			
			0	1	2	3
1	Novelty	3.5	Factor not applicable	Not novel	Less novel	Novel
2	Market demand	2.5		Low	Average	High
3	Cost effectiveness	2.5		High cost	Moderate cost	Low cost
4	Consumer acceptability	1		Less acceptable	Either physician/ patient acceptability	Acceptable
5	Efficacy	0.5		Low	Adequate	High
Scale range = 0 to 30						

Point	Allocation criteria
1) Competitor product	
-1	No competitor product available
-2	Competitor product available but less competition due to some aspect like different dosage form or patient non-compliance etc.
-3	Similar competitor product or similar treatment available which may restrict market penetration of the product.
2) Production cost	
-1	Low production cost
-2	Acceptable/tolerable production cost
-3	High production cost
3) Stability	
-1	Highly stable product (long shelf life) under specified storage conditions
-2	Acceptable shelf life under specified storage conditions
-3	Less stability (low shelf life) under specified storage conditions
4) Safety (in terms of ADRs -Adverse Drug Reactions)	
-1	High safety (less ADRs; high benefit : risk ratio)
-2	Acceptable safety (moderate benefit : risk ratio)
-3	Less safety (more ADRs; less benefit : risk ratio)
5) Regulatory compliance	
-1	Compliant to regulatory requirements
-2	Compliant to regulatory requirements with tolerable limitations
-3	Non-compliant to regulatory requirements

Table 3. Elaborated point allocation criteria for risk factors

Table 4. Elaborated point allocation criteria for backing factors

Point	Allocation criteria
1) Novelty	
1	Product is not novel (lacks innovation)
2	Product is novel still less distinctive
3	Product is novel and unique
2) Market demand	
1	Low market demand owing to factors like limited disease spread, availability of alternative treatments etc.
2	Adequate market demand for the product
3	High market demand due to wide spread of disease, absence or inadequate of alternative treatment etc.
3) Cost effectiveness	
1	High cost treatment/therapy
2	Moderate cost treatment/therapy
3	Low cost treatment/therapy
4) Consumer acceptability	
1	Not readily acceptable by physicians and patients
2	Acceptable by only physicians or patients
3	Readily acceptable by physicians and patients
5) Efficacy	
1	Less effectiveness
2	Adequate effectiveness
3	High effectiveness

Table 5. Calculation of risk factor score for sample product.

Sr. no.	Risk factor	Weightage (W)	Point allocation (P)	Point scored (W x P)
1	Competitor product availability	2.5	-1	-2.5
2	Production cost	2.5	-3	-7.5
3	Stability	2.5	-1	-2.5
4	Safety	1.5	-1	-1.5
5	Regulatory compliance	1	-2	-2
	Risk factor score			-16

Table 6. Calculation of backing factor score for sample product.

Sr. no.	Backing factor	Weightage (W)	Point allocation (P)	Point scored (W x P)
1	Novelty	3.5	3	10.5
2	Market demand	2.5	2	5
3	Cost effectiveness	2.5	1	2.5
4	Consumer acceptability	1	2	2
5	Efficacy	0.5	3	1.5
	Risk factor score			21.5

ACKNOWLEDGEMENT

The market potential scale was developed as a part of doctoral research project in pharmaceutical sciences assisted by INSPIRE programme division of Dept. of Science and Technology, New Delhi. Authors are grateful to INSPIRE program for the same.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

(Proceedings available for download at rku.ac.in/icre)

RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

A Novel Approach-Development of a Bioanalytical Method for the Quantitation of Calcitriol in Human Plasma using various Modern Technologies

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ABSTRACT

Vitamin D helps in maintaining phosphorus and calcium level in blood which is important for bone formation. 1,25-(OH)2D3 (calcitriol) is the active form of vitamin D3. A LC–MS method was developed and validated for the quantification of Calcitriol in human plasma using combination of various modern technologies. Calcitriol was chromatographically separated by Ultra High performance liquid chromatography on analytical column having core shell technology. During sample preparation, sensitivity was enhanced with help of vitamin D slurry and Ampliflex reagent. Analyte was extracted through Solid Phase Extraction technique. 10mM Ammonium Acetate Solution and 0.1 % Formic Acid in Acetonitrile were used as mobile phase with gradient flow. A positive electrospray ionization was used on API 5500 LC-MS/MS for quantification of analyte. This method was fully validated in calibration range of 2.59 to 158.06 pg/mL and successfully applied to bioequivalence study.

SUMMARY

A Highly sensitive method for quantification of Calcitriol was developed, validated and successfully applied to bioequivalence study.

Keywords: Calcitriol, Vitamins, HPLC-MS/MS, Bioanalysis, Bioequivalence study

INTRODUCTION

Calcitriol, is 1,25-dihydroxycholecalciferol (1,25-(OH)2D3), (Figure 1). It is a secosteroid hormone that increases plasma level of calcium and phosphate. Calcium and Phosphate helps in mineralization and bone formation. Calcitriol is an active metabolite of cholecalciferol which is available in dietary foods. Calcitriol is also formed in skin after photolysis of 7-dehydrocholesterol when exposed to sunlight. Calcitriol is further hydroxylated by kidney and liver (*1, 2*).

Vitamin D deficiency is recognized as one of the most common mild chronic medical conditions in the world (*3*). Vitamin D deficiency has been linked to increased mortality in general population (*4*), many disorders of myocardial infarction (*5*), cardiac failure (*6*), stroke (*7*), infections, diabetes mellitus, auto-immune and inflammatory conditions (*8-10*).

Quantification of Calcitriol is very much clinically relevant and it has an immense market potential. A accurate, precise and sensitive method can be developed and validated to be applied on bioequivalence studies. Pharmacokinetics of Oral Calcitriol **was** studied by **Su-Eon Jin, Jeong-Sook Park and Chong-Kook Kim (2009)** in healthy human with an enzyme immunoassay in range of 2.7-63.8 pg/mL(*11*). **Andrew N. Hoofnagle, Thomas J. Laha and Thomas F. Donaldson (2010)** tried improving the throughput of extraction by using rubber transfer gasket(*12*). **Sirimas Sudsakorn, et al (2011)** determined 1,25 dihydroxy vitamin D2 in serum by using LC-MS/MS. They used Solid phase extraction method for extraction of analyte and their linearity range was 25-1000pg/mL(*13*). **Joanna Denbigh, Tony Edge, et al (2011)** did analysis of Calcitriol in biological matrix by using SLE-SPE-LC-MS/MS in range of 1-100 ng/mL. This method was having numerous steps of sample processing and sample evaporation steps(*14*). **Jan Lembcke, Axel Besa, Barbara Hoyer, et al (2011)** used immuno Tube Kit with immunoaffinity columns for sample preparation to get linearity in range of 10-1000pg/mL in serum and plasma(*15*). **Chao Yuan, Justin Kosewick, Xiang He, et al (2011)** did quantification of 1α,25-dihydroxy Vitamin D in serum by LC/MS/MS. They did a comparison of this method with RIA method on 40 subjects(*16*). **Jan H. Beumer, et al (2012)** studied local effect of Vitamin D3 catabolizing enzyme (CYP24) inhibition in lung tumor by LC-MS/MS assay, their achieved sensitivity level was 0.1 ng/mL in plasma and tumor homogenate(*17*). **Evgueni Fedorov, Hope Weiler, et al (2012)** developed a LC/MS/MS method for 25-hydroxyvitamin D3 and its C3 Epimers quantitation in infants. They used Diels-Alder derivatization to enhance the sensitivity in MS analysis(*18*). **Siddheshwar Patankar, Ashutosh Pudage, Varad Pradhan, et al, (2013)** evaluated the significance of endogenous concentration and Baseline correction in bioequivalence assessment of Calcitriol. Their achieved level of sensitivity was 5pg/mL in human Plasma(*19*).

In present study, highly sensitive HPLC–MS/MS method was developed with excellent accuracy and precision and validated for quantification of Calcitriol in human plasma and this method was used for the quantification of Calcitriol from bioequivalence study after single oral dose of Calcitriol capsules 0.5 mcg versus ROCALTROL® (calcitriol) capsules 0.5 mcg in 48 healthy subjects, under fasting conditions. In this study, we report the benefits of using an optimal solution based approach with sample preparation and LC-MS that addresses the sensitivity requirement of Calcitriol in human plasma.

MATERIALS AND METHODS

Chemicals and Reagents:

Calcitriol (mol.wt. 416.64; 99.45% w/w) was obtained from VIVAN Life Sciences Pvt.Limited, India and Calcitriol D6 (mol.wt. 422.64, 98.57 %) was procured from CLEARSYNTH LABS LIMITED, India. Human K3EDTA (Tri-Potassium ethylene di-amine tetra acetic acid) plasma was obtained from Pooja Blood Bank, Mumbai, India. Ampliflex reagent was procured from SCIEX, USA and 1, 25 dihydroxy

affinity slurry from ALPCO diagnostic. Acetonitrile (HPLC grade) was procured from Fischer Scientific, India. All other reagents/chemicals were of GR grade.

Instrumentation and Analytical Conditions:

This method was developed on API 5500 Q TRAP LC-MS/MS (MDS SCIEX, Canada) which is a triple quadrupole instrument of MS. On this instrument positive ionization was used to detect and analyse analyte. Multiple Reaction Monitoring (MRM) mode was used to analyse parent/daughter ion of analyte and internal standard. m/z was 748.50/689.40 amu and 754.70/695.50 amu for diene derivative of Calcitriol and Calcitriol D6 respectively. Kinetex C18 column (100 x 2.1 mm, 1.7µm) was used with high performance liquid chromatography instrument (Shimadzu, Japan) to chromatographically separate analyte. The autosampler temperature was maintained at 15±1ºC while the flow rate was set with gradient program from 0.35ml/min to 1.00 mL/min with 80% flow split. Acquisition and quantitation was performed with help of analyst software. The compound and source dependent parameters for Calcitriol and its internal standard are given in Table 1.

Preparation of Stock Solutions and Plasma Samples:

Calcitriol (1mg/mL) and Calcitriol D6 (1mg/mL) stock solutions were prepared in methanol. Further spiking solutions of Calcitriol were prepared in mixture of milli-Q water and acetonitrile (50:50 v/v). To prevent interference generated through endogenous Vitamin D, Plasma was stripped with help of charcoal. Spiking solutions were spiked in charcoal treated blank plasma to prepare quality control samples and calibration curve standards (Standards). Eight standards were prepared having concentration of 2.59 pg/mL, 5.14 pg/mL, 10.28 pg/mL, 17.14 pg/mL, 28.56 pg/mL, 47.60 pg/mL, 79.33 pg/mL and 158.66 pg/mL. Four level of quality control samples were prepared having concentration of 8.12 pg/mL (LQC), 33.26 pg/mL (LMQC), 83.16 pg/mL (MQC), 127.93 pg/mL (HQC). From the bulk spiked Standards and quality controls, 600 µL of samples were aliquted in pre-labeled tubes. All the samples of quality control and calibration curve standards were stored at -20±5°C in deep freezer. Method validation was performed by using these samples.

Mobile Phase Preparation:

Mixture of 10 mM Ammonium acetate for pump A and 0.1% Formic Acid in Acetonitrile in Pump B was used as mobile phase. This combination of solvents was run in a gradient program as given in Table 2.

Sample Preparation:

Eight calibration curve standards (one set) and quality control samples were retrieved from the deep freezer. Samples were thawed unassisted at room temperature. After complete thawing, 500µl of sample was aliquoted, 50µl of Calcitriol D6 (internal standard) (500.00 pg/mL) was added followed by vortexing the samples. This plasma sample was added to vials containing 170 µl of incubated slurry solution and kept on extractor for 60 minutes at 40 rpm after vortexing. Solid phase extraction was performed using Celerity Deluxe 30mg, 1 mL, DVB-LP as an extraction cartridge. Conditioning and equilibration of cartridges was done by methanol and water respectively. Sample was added to pre-conditioned cartridge. Sample washing was done with 500µl of water two times. After drying the cartridge for three minutes, sample was eluted two times with 500µl of mixture of ethanol and methanol in composition of 80:20. Eluted sample was evaporated to dryness at 40°C in nitrogen evaporator. 35µl of Ampliflex reagent was added to dried residue and kept for 30 minutes at room temperature. 90µl of water was added and mixed.

Method Validation:

USFDA guidelines were considered while validating this bioanalytical method. Parameters of sensitivity, precision, accuracy, recovery, selectivity were evaluated during course of method validation. The method was also evaluated for haemolysis effect, lipemic effect and matrix effect. Stability of analyte was evaluated for solutions of stock and working solution in refrigerated condition as well as ambient condition. For extracted samples, stability was evaluated at room temperature, in deep freezer, stability after freeze thaw cycles, dry extract stability, wet extract stability, whole blood stability, autosampler stability. Various other experiments which are done to simulate the conditions of subject sample analysis as effect of potentially interfering drugs, Lipemic Effect, Haemolysed Effect, dilution integrity, ruggedness and long batch performance were evaluated as per the USFDA guidelines (FDA, 2001) (*20*).

Selectivity and Sensitivity:

Selectivity was performed to check the endogenous interference in the assay. In this experiment, response of analyte and internal standard is compared in samples of blank and LLOQ. Samples of blank and LLOQ were also taken from Haemolyed plasma and Hyperlipidemic plasma to check the specificity of method. Sensitivity experiment is performed by spiking solution of Lower limit of quantification (LLOQ) in six different lots of plasma. Accuracy and precision of LLOQ samples is evaluated.

Matrix Factor:

Matrix effect was evaluated in terms of Matrix Factor. This experiment was performed to evaluate inherent variability of matrix during ionization in samples of analyte and internal standard. For matrix factor, 06 samples of each at concentration of LQC and HQC level were processed from 6 different plasma lots. Matrix factor is calculated as ratio of analyte or internal standard area counts with matrix ions to the analyte or internal standard area counts without matrix ions.

Accuracy and Precision:

Accuracy can be explained as closeness of calculated concentration of quality control samples to the nominal concentration of respective quality control samples (LLOQ QC, LQC, LMQC, MQC and HQC). Whereas Precision can be defined as closeness of calculated concentrations among six samples of quality control samples at each four levels (LLOQ QC, LQC, LMQC, MQC and HQC). Linearity was evaluated using regression relationship of analyte to internal standard response ratio to concentration of analyte. For the best fit of regression, $1/x^2$ weighting factor was used.

Working Solution and Stock Solution Stability:

Working solution and stock solution stability in refrigerator was evaluated for 7 days for Calcitriol and Calcitriol D6 (IS) while working solution and stock stability at ambient temperature was evaluated for 6 hours. For solution stability experiment, aqueous solutions of analyte or internal standard are prepared and kept for stability condition. After stipulated stability duration, fresh solutions are prepared and these stability and fresh samples are analysed in six replicates after appropriate dilution. The analyte or internal standard response of fresh samples was compared with stability samples.

Recovery:

To find the extraction efficiency of method, un-extracted samples at three levels of quality control (LQC, MQC and HQC) were made by reconstituting six dried blank samples with 75µL of premixed solution of recovery standard. After drying this above sample, 35µL of Ampliflex was added and kept at ambient temperature till 30 minutes. 90µL of water was added and mixed. The un-extracted samples of Calcitriol were compared with extracted samples.

Stability Experiments:

All the stability experiments were performed at levels of LQC and HQC samples. Stability of Calcitriol at ambient temperature was evaluated for 24 hours on six replicates of samples. The stability of freeze and thaw was evaluated by exposing six replicates of samples till five freeze thaw cycles. These samples were analyzed along with freshly prepared samples.
Long term stability was evaluated in plasma in Deep Freezer maintained at -20ºC±05ºC for 32 days.
Dry extract stability was evaluated by extracting samples till step of drying. Dry extract samples were stored at 2-8ºC in refrigerator for stipulated duration. After stability duration (24 hours) these samples are reconstituted and analysed along with fresh samples for stability evaluation. For wet extract stability, samples were extracted and stored at 2-8ºC. After stipulated duration (24 hours), Stability samples were processed along with fresh samples. % difference was calculated between stability samples and fresh samples. Ruggedness of method was evaluated for different analyst, different equipment and different column by analyzing one accuracy and precision batch for each different condition.

Bioequivalence Study Design:

Bioequivalence Study was performed to compare the bioavailability of test formulation of calictriol of 0.5 mcg capsule with with the reference 0.5 mcg Calcitriol formulation in healthy adult, human subjects, under fasted condition. Exclusion criteria was set to smoking, diabetes, alcoholism, smoking and allergy to Calcitriol. Other diseases related to cardiovascular, renal, gastrointestinal, respiratory, and hepatic were also considered as exclusion criteria. All the procedures were based on the USFDA guidelines. Independent Ethics Committee approved the protocol. Samples of subjects who have completed both the periods of clinical phase were analysed. All participants were informed about risks and aims of study and consent was taken in written. Inclusion criteria was set to body mass index (18.5–30.0kg/height^2), age (18–45years), meeting all laboratory test criteria, normal electrocardiogram and absence of abnormalities on physical examination. Subjects were housed from approximately 36.00 hours prior to drug administration and until 48.00 hours post dose in both the study periods. 2016 blood samples were collected in K3EDTA vacuutainers from 42 subjects in two periods. Plasma was separated after centrifugation maintained at 4ºC for 10 min at 4500 rpm. These samples were stored in deep freezer at -20ºC till analysis.

RESULTS AND DISCUSSION

Analyte and Internal Standard:

Calcitriol and its labeled internal standard Calcitriol D6 was having very close retention time as 4.51 and 4.48 respectively. Calcitriol D6 was having similar recovery as of Calcitriol. Ampliflex Diene reagent forms an adduct after reacting with Calcitriol and this adduct is used as a precursor ion. The reaction of Calcitriol with Ampliflex Diene reagent is shown in Figure 2. High sensitivity was observed with positive Electro spray ionization (ESI) mode for both analyte and IS. The precursor ion (Q1) spectra of derivatized Calcitriol is given in Figure 3.

Sample Preparation:

Sample preparation was performed by using SPE (Solid Phase Extraction) because of good extraction efficiency as well as specificity of analyte. Many extraction cartridges were tried but Celerity Deluxe DVB-LP was found to be most selective for extraction of both Calcitriol and Calcitriol D6.

Chromatographic Conditions:

Kinetex C18 column (100 x 2.1 mm, 1.7µm) with core shell technology was used during this analysis. Extracted samples were kept in autosampler at 15±1ºC while the flow rate was set with gradient program from 0.35ml/min to 1.00 mL/min with 80% flow split.

Method Validation

Selectivity and Matrix Effect:

Samples of calcitriol were found free from interference generated through endogenous substances in normal, haemolysed or lipemic plasma. Figure 4 shows representative chromatogram of a blank plasma sample, LLOQ (2.59 pg/mL) sample and a representative chromatogram of Internal Standard. The variation (%RSD) in matrix factor was 1.64% (HQC) and 1.75% (LQC) for Calcitriol. The mean matrix factor for Calcitriol samples was 1.01 at HQC and 1.00 at LQC level. The results of matrix factor were within the acceptance limit which shows that ionization and quantification of analyte is not effected by biological matrix.

Linearity and Sensitivity:

During method validation, correlation coefficients (r) were obtained more than 0.99 for linearity range of 2.59 pg/mL and 159.56 pg/mL. % RSD (Relative Standard Deviation) of LLOQ samples was found to be 3.02 in sensitivity experiment.

Accuracy and Precision:

Accuracy and precision in terms of % mean accuracy and %RSD for Calcitriol was 101.93% and 3.04% respectively at LLOQ level which depicts sensitivity of the method. Accuracy and Precision of method was calculated with back calculated concentrations of quality control samples for Calcitriol. interday and intraday accuracy and precision data is given in Table 3. The interday and intraday precision of method at levels of quality control levels was 3.88-8.91% and 2.62%-13.79%, while interday and intraday accuracy

was 98.13-103.31% and 96.54-109.34%. The data of method validation assures that method is sensitive, accurate and precise. The data of Accuracy and Precision is given in Table 3.

Recovery:

The extraction recovery of Calcitriol was 102.12 % with %RSD of 0.30 % and for Calcitriol D6, % recovery was 102.35% with a precision of 0.39%. The data of recovery indicates that Solid Phase extraction procedure recovers analyte and internal standard in an efficient manner. The data of recovery is summarized in Table 4.

Stability and other Parameters:

Stability data tabulated in Table 5 depicted that Calcitriol is stable in the expected stability environment within defined stability duration so method can be easily applied to subject sample analysis. The results of other validation parameters like effect of Ruggedness, Reinjection reproducibility, Dilution integrity and Long Batch Performance were found to be within the acceptance limit.

Study Analysis:

The method was applied on subject samples of bioequivalence study for the quantitation of Calcitriol. K3EDTA vaccutainers were used for collection of blood samples from healthy human subjects. The time points selected for study were: -18.00, -12.00, -6.00 and 0.00 hours for pre-dose and at 1.00, 1.50, 2.00, 2.50, 3.00, 3.50, 4.00, 4.50, 5.00, 5.50, 6.00, 6.50, 7.00, 8.00, 10.00, 12.00, 16.00, 24.00, 36.00, 48.00 hours post dose. Samples were analyzed and statistical evaluation was done to obtain Pharmacokinetic parameters. Table 6 shows evaluated pharmacokinetic parameters of Calcitriol capsules in human subjects. Figure 5 shows profile of mean plasma concentration vs time.

CONCLUSION

For the very first time a highly accurate, precise, sensitive method for the quantitative determination of Calcitriol in human plasma was developed using HPLC–MS/MS with positive ionization in turbo-ion spray. The method was fully validated as per USFDA guidelines and it was successfully applied to subject sample analysis for the bioequivalence study of orally administered Calcitriol capsules.

FIGURES

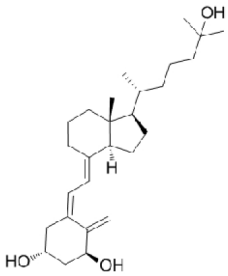


Figure 1. Structure of Calcitriol

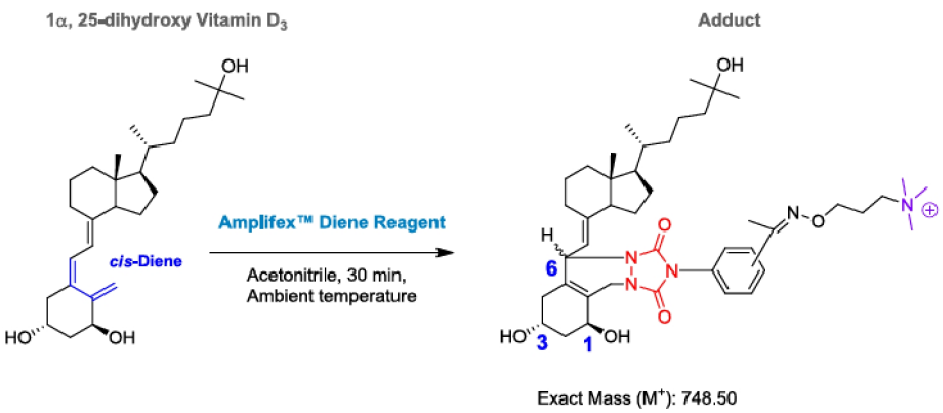


Figure 2: Chemical reaction of Calcitriol with Ampliflex Diene reagent

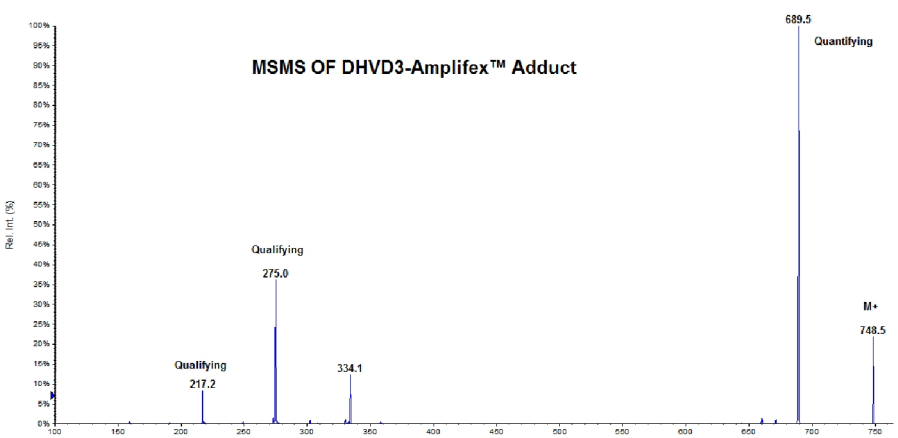


Figure 3: The precursor ion (Q1) spectra of derivatized Calcitriol

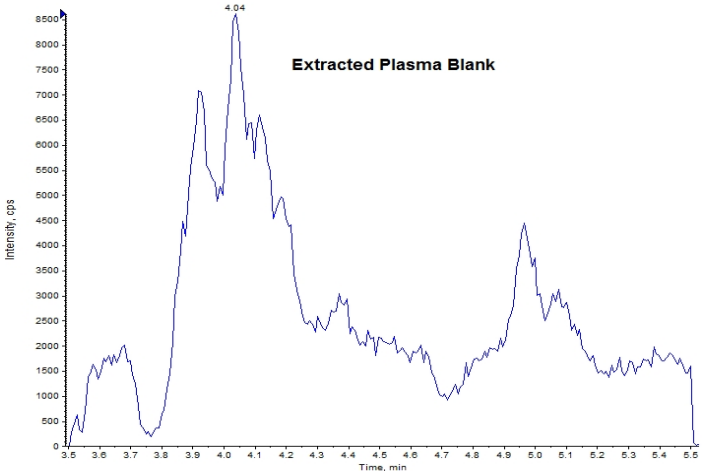


Figure 4A: Representative chromatogram of blank sample

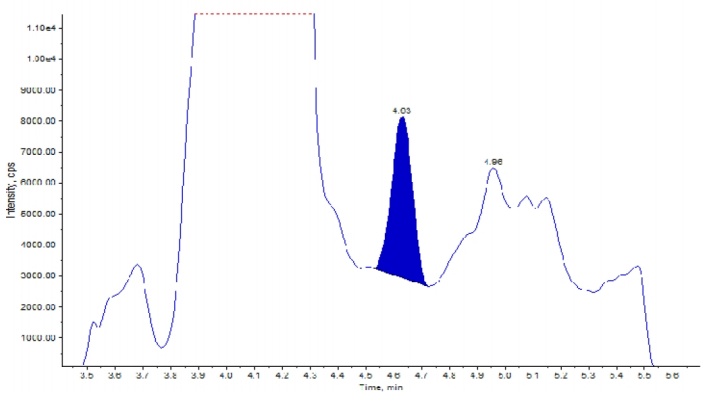


Figure 4B: Chromatogram of LLOQ (2.59 pg/mL) sample

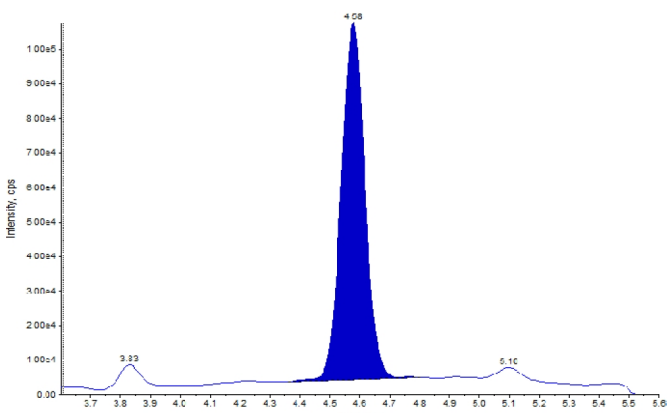


Figure 4C: Representative chromatogram of Internal Standard

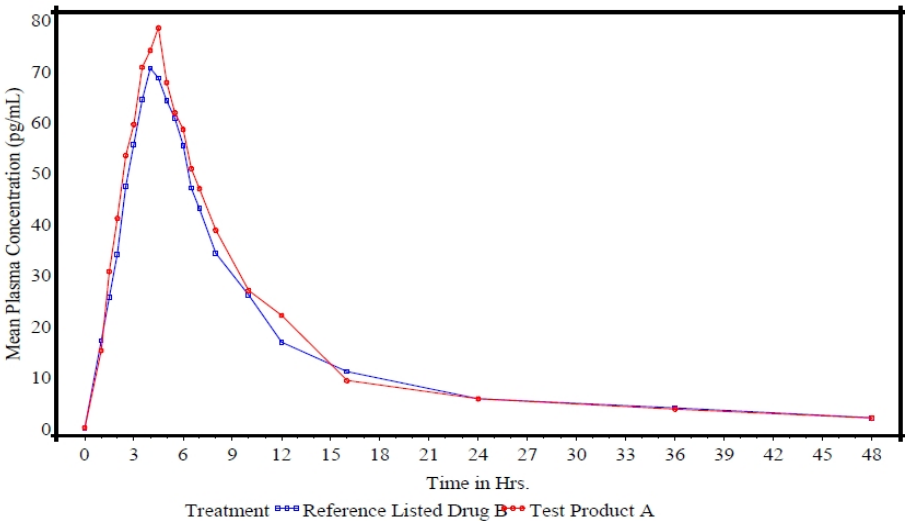


Figure 5: Profile of mean plasma concentration vs time

TABLES

Table 1: Compound and Source dependent parameters

Compound Dependent Parameters	Calcitriol	Calcitriol D6
Declustering Potential (DP)	50.00	66.00
Entrance Potential (EP)	11.00	10.00
Collision Energy (CE)	40.00	38.00
Cell Exit Potential (CXP)	20.00	31.00
Source Dependent Parameters		
Curtain gas	40.00	
IS Voltage	5500.00	
Temperature	600.00	
Collision Activation Dissociation (CAD)	Medium	

Table 2: Gradient Program for mobile Phase

Time (min)	Pump B Concentration	Flow Rate (mL/min)

	(%)	
0.01	5	0.35
0.50	5	0.35
1.00	35	0.35
5.40	54	0.35
5.50	54	0.35
5.60	98	0.35
5.65	98	1.00
6.20	98	1.00
6.25	98	1.00
6.30	98	0.35
6.40	5	0.35
8.00	Stop	0.35

Table 3: Accuracy and Precision of Calcitriol in Human Plasma (n=6)

Concentration Spiked (pg/mL)	Measured Concentration (mean±SD) (pg/mL)		Accuracy (%)		Precision (RSD %)	
	Intra-day	Inter-day	Intra-day	Inter-day	Intra-day	Inter-day
2.75	2.69±0.37	2.76±0.25	97.82	100.36	13.79	8.91
8.10	7.82±0.56	8.15±0.54	96.54	100.62	7.14	6.58
33.19	33.22±0.98	32.57±1.73	100.09	98.13	2.98	5.30
82.97	87.66±2.30	85.54±3.32	105.65	103.10	2.62	3.88
127.65	139.57±10.67	131.87±7.75	109.34	103.31	7.65	5.88

Table 4: Recovery (n=6) of assay method

Concentration (pg/mL)	Recovery	
	Mean (%)	RSD (%)
8.10	102.41	3.79
82.97	101.80	1.64
127.65	102.14	3.61

Table 5: Stability of Calcitriol in human plasma (n=6)

Storage Condition	Spiked Concentration (pg/mL)	Measured Concentration (pg/mL)	Accuracy (%)
After five	8.10	8.71	107.53

freeze/thaw cycles	127.65	124.71	97.70
Bench top stability (13 hours at ambient temperature)	8.10	8.83	109.01
	127.65	120.63	94.50
Long term stability (at -20^{o}C for 98 days)	7.71	6.91	89.62
	121.50	125.21	103.05
Dry Extract stability (at 2-8^{o}C for 27 hours)	8.10	9.07	111.95
	127.65	118.75	93.03
Wet extract Stability (at 2-8^{o}C for 25 hours)	8.10	8.85	109.26
	127.65	118.94	93.18

Table 6: Pharmacokinetic parameters

Parameter	Geometric Least-Squares means[1]		Test-to-Ref Ratio[2]	CV%[3]	90% confidence interval limits[4]		Power (%)
	Test	Reference			Lower	Upper	
C_{max} (pg/mL)	109.33	110.88	98.60	15.78	93.08	104.44	100.00
AUC_{0-t} (pg.hr/mL)	1446.39	1494.17	96.80	15.33	91.53	102.38	100.00
AUC_{0-inf} (pg.hr/mL)	2461.47	2527.24	97.40	46.80	82.70	114.70	60.83

1. For loge-transformed results (Ln), value is the least-squares geometric mean.
2. Ratio% of least-squares means or least-squares geometric means for loge-transformed results.
3. Intra-subject CV% calculated from the mean square term of the ANOVA as 100% × Sqrt (eMSE-1).
4. Confidence interval on ratio

ACKNOWLEDGEMENT

Authors wish to thank all the colleagues at Raptim research Limited and RK University for their guidance and support.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

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Antioxidant and radical scavenging activity of Mangiferin

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ABSTRACT

Mangiferin is a natural (2-C-β-D-glucopyranosyl-1,3,6,7-tetrahydroxyxanthone) abundantly present in the leaves of *Mangifera indica* L. In the present paper we evaluated the antioxidant and free radical scavenging activity of mangiferin using various *in vitro* assays such as 1,1-diphenyl-2-picryl-hydrazyl (DPPH) radical scavenging, total reducing ability determination by the Fe^{3+}–Fe^{2+} transformation method, hydrogen peroxide radical scavenging activity and anti-lipoxygenase assay. Mangiferin had effective DPPH scavenging, hydrogen peroxide radical scavenging, reducing power and lipoxygenase activity. Ascorbic acid was used as the reference compound. The present study indicated the antioxidant potential of mangiferin thereby providing evidences for its usefulness in various inflammatory conditions.

SUMMARY

Antioxidant and Radical scavenging activity of Mangiferin was evaluated using *in vitro* models.

Keywords: Antioxidant, in vitro, Mangiferin, DPPH, Radical scavenging

INTRODUCTION

In the normal physiological process reactive oxygen species (ROS) play an important element that may be produced by numerous metabolic pathways. ROS encompass various beneficial as well as harmful molecules such as hydroxyl radicals, hydrogen peroxide, superoxide radicals & singlet oxygen *(1, 2)*. Less amount of ROS are required for normal functioning such as cell growth regulation and function, signal transduction pathways, protection against invading pathogens *(3)*. Overproduction of these ROS may damage the cellular components, stimulating free radical reactions; thereby causing disease conditions such as cancer, inflammatory disorders like inflammatory bowel disease, arthritis, cardiovascular and neurodegenerative disorders. Antioxidants are compound that provide protection against these ROS; therefore for a healthy cellular state balance between ROS and antioxidant defence system is of upmost importance *(3)*. Chemically synthesized antioxidants such as butylated hydroxyl toluene (BHT), butylated hydroxyanisole (BHA) have toxic and carcinogenic effects *(4)*. Therefore safe and naturally occurring food phytochemicals are in great demand for anti-ROS activity. Antioxidant phytochemicals are defined as bioactive compounds from fruits, vegetables, grains, and other plant foods that possess impending usefulness in the management of various disease states such as diabetes, cancer, inflammatory bowel disease, arthritis, cancer *(5)*. Studies have shown various naturally occurring phytochemicals of plant origin namely, curcumin, resveratrol, quercetin, rutin with beneficial effects in oxidative stress disorders *(6, 7)*. Mangiferin (2-C-β-D-glucopyranosyl-1,3,6,7-tetrahydroxyxanthone); is abundantly present in the fruits, barks, leaves, roots of *Mangifera indica* L. (Mango) along with families including *Anacardiaceae*, *Iridaceae* and *Gentianaceae*. Mangiferin has been useful as antimicrobial, cardioprotective, gastroprotective, antidiabetic, hepatoprotective; mainly owing to its antioxidant and anti-inflammatory activity *(8)*. Thereby important objective of the experiment was to explore the antioxidant mechanism of mangiferin using various *in vitro* systems including free radical scavenging, reducing power, hydrogen peroxide & lipoxygenase assay. Also mangiferin antioxidant capacity was compared with the commercially available antioxidant ascorbic acid.

MATERIALS AND METHODS

Chemicals

Ascorbic acid, 1,1-diphenyl-2-picryl-hydrazyl (DPPH), linoleic acid, mangiferin, potassium ferricyanide [$K_3Fe(CN)_6$], trichloroacetic acid (TCA), were obtained from Sigma Chemical Company (St. Louis, MO, USA); rest all other chemicals used were of analytical grade.

Radical scavenging activity using DPPH

With slight alterations in the method described by Yang et al; radical scavenging activity of mangiferin was evaluated using DPPH as the reagent *(9)*. DPPH radical absorbs at 517 nm; but when antioxidant reacts with the radical its absorption decreases. Briefly one millilitre of the mangiferin solution in the concentration range of 10-100µg/ml was added to methanolic DPPH reagent (3ml; 0.1 mmol). The violet colour developed was proportional to the free radical generation. Using spectrophotometer the absorbance of the solution was measured at 517 nm and its percent inhibitory activity was computed using the following equation

$$\% \text{ inhibition} = \frac{A_{control} - A_{sample}}{A_{sample}} \times 100$$

Where $A_{control}$ is the absorbance without sample and A_{sample} is absorbance with sample.

Ferric cyanide (Fe^{3+}) reducing antioxidant power assay

The reducing power of mangiferin was determined using the method described by Benzie et al *(10)* with some modifications. Phosphate buffer was used for preparing solutions of mangiferin and ascorbic acid with concentrations ranging from 10-100 µg/ml. One millilitre of either mangiferin or ascorbic acid was mixed with solution containing potassium ferricyanide and phosphate buffer and then incubated at 50°C for 20 mins. Later trichloroacetic acid (2.5 ml) was added and the solution was centrifuged at 4000g for 15mins. After centrifugation distinct layers were formed. Distilled water containing ferric chloride was mixed with the upper layer of centrifuged solution and its absorbance was measured at 700 nm.

Hydrogen peroxide scavenging activity

With slight alterations in the procedure described by Gulcin et al; scavenging activity was evaluated for mangiferin using hydrogen peroxide *(11)*. The method works on the principle; as the oxidation of H2O2 proceeds there is a decrease in the absorbance of H2O2 observed. H2O2 reagent was prepared in phosphate buffer. Mangiferin and standard ascorbic acid at different concentrations ranging from 10-100 µg/mL in phosphate buffer was mixed with H2O2 and its absorbance was recorded at 230 nm. Sodium phosphate buffer without H2O2 was considered as the blank. The concentration of hydrogen peroxide in the method was calculated using a standard curve. H2O2 scavenging activity of mangiferin in terms of percentage was calculated as follows:

$$H_2O_2 \text{ scavenging effect (\%)} = \frac{A_{control} - A_{sample}}{A_{sample}} \times 100$$

Where $A_{control}$ is the absorbance without sample and A_{sample} is absorbance with sample.

Anti-Lipoxygenase Activity

Mangiferin was evaluated for the anti-inflammatory activity using enzyme lipoxidase and linoleic acid. *(12)* With slight modifications in the method of Ben-Nasr et al mangiferin at various concentration of 10, 20, 40, 60, 80, 100µg/ml and indomethacin at 60 µg/ml was dissolved in borate buffer and later lipoxidase enzyme (20,000U/ml) was added. The solution was incubated at 25°C with drop by drop addition of linoleic acid and its absorbance was measured at 234nm. The percent inhibition was calculated from the following equation:

$$\text{\% inhibition} = \frac{A_{control} - A_{test}}{A_{control}} \times 100$$

Where $A_{control}$ is the absorbance without sample and A_{test} is absorbance with sample

RESULTS AND DISCUSSION

DPPH radical scavenging activity

Free radical scavenging activity using 1,1-diphenyl-2-picryl-hydrazyl (DPPH) is most common method used for evaluating *in vitro* antioxidant activity of naturally occurring phytochemicals *(13)*. Hydrogen giving capacity of the compound contributes to the antioxidant potential of phytochemical on the DPPH radical. The current study evaluated the radical scavenging potential of mangiferin and the standard ascorbic acid. Mangiferin when added to the DPPH solution; rapidly decreased the absorbance with increase in its concentration at 517 nm. The extent of colour change from purple to yellow indicates the potential of mangiferin to scavenge the radical. Figure 1 illustrates the DPPH scavenging capacity of mangiferin. At various concentrations of 10, 20, 40, 60, 80 and 100µg/ml mangiferin scavenged the DPPH free radicals to the extent of 5.85%, 8.89%, 24.92%, 51.07%, 60.24% and 64.98% and ascorbic acid scavenged the DPPH free radicals to the extent of 3.67%, 9.01%, 15.86%, 28.66%, 41.07% and 50.86% respectively. The free radical scavenging effect of mangiferin increased with increase in its concentration. Maximum scavenging activity was observed at 100µg/ml. These results highlight mangiferin as a potential scavenger or inhibitor of free radical.

Ferric cyanide (Fe^{3+}) reducing antioxidant power assay

The reducing power assay may serve as an important indicator to evaluate the antioxidant potential of the compound. Numerous studies has revealed a straight association between the antioxidant activity of a phytochemical and its reducing power *(14)*. With slight modifications in the method of Benzie; reductive competence of mangiferin was evaluated with the conversion of Fe3+ to Fe2+. Figure 2 indicates the reductive capacity of mangiferin along with standard compound ascorbic acid at an absorbance of 700 nm. Increased absorbance indicates higher reducing power of the compound. With increase in the concentration of mangiferin; its reducing capacity also increased simultaneously. Also the reducing capacity of mangiferin was comparable to ascorbic acid.

Hydrogen peroxide scavenging activity

Excessive amounts of hydroxyl radicals can penetrate the cell membrane and cause damage to tissue and eventually cell death. Thus removing these excessive hydroxyl radicals is necessary for proper

protection of living organisms. Figure 3 illustrates the scavenging action of ascorbic acid and mangiferin at various doses of 10, 20, 40, 60, 80 and 100µg/ml. Scavenging potential of mangiferin in terms of percentage at 100µg/ml was found to be 45.82%; while at same concentration ascorbic acid showed 51.17%. Thus the scavenging activity for mangiferin was found to be in a concentration dependent manner.

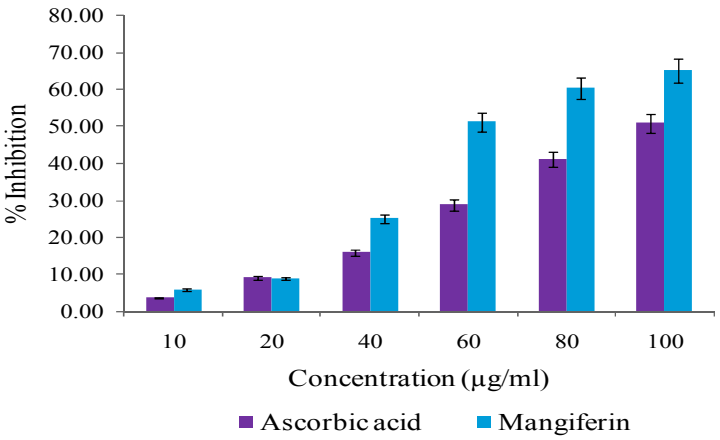
Anti-Lipoxygenase Activity

Mangiferin was evaluated for anti-inflammatory activity by *in vitro* lipoxygenase assay. Mangiferin at 10, 20, 40, 60, 80 and 100 µg showed 11.80%, 24.74%, 37%, 47.31%, 55.67% and 65.64% inhibition respectively. Reference standard indomethacin showed a 51.43% inhibition at a concentration of 60µg/ml (Figure 4).

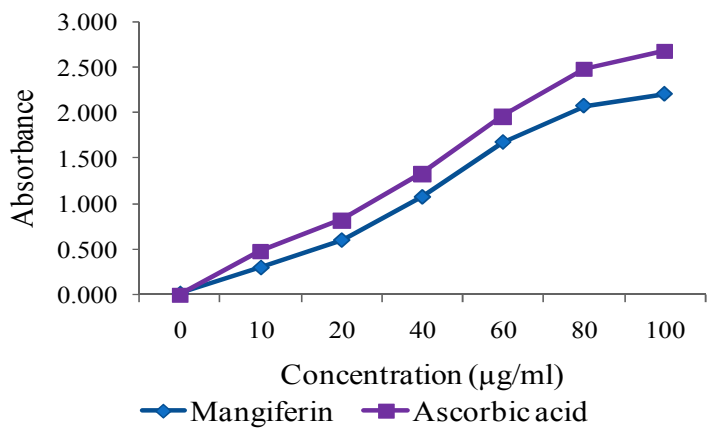
CONCLUSION

The results of the study provide evidence of mangiferin in a concentration dependent manner as a powerful antioxidant using various assays *in vitro*. More experimental evidence needs to be explored to understand the mechanism of mangiferin and its usefulness clinically as antioxidative agents in the management of numerous inflammatory conditions.

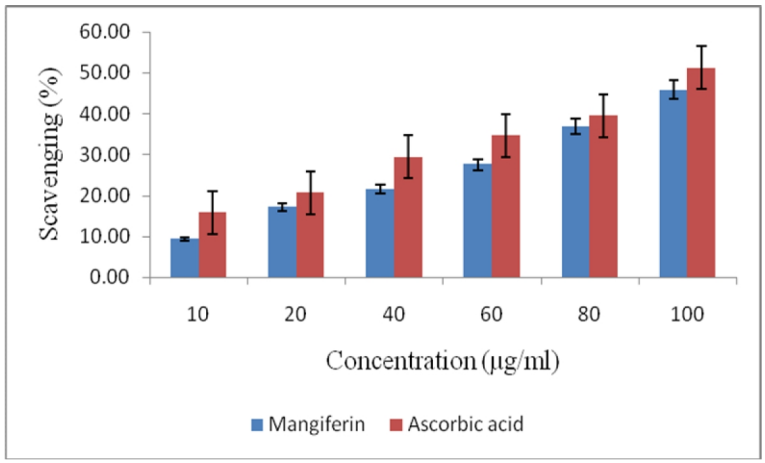
FIGURES



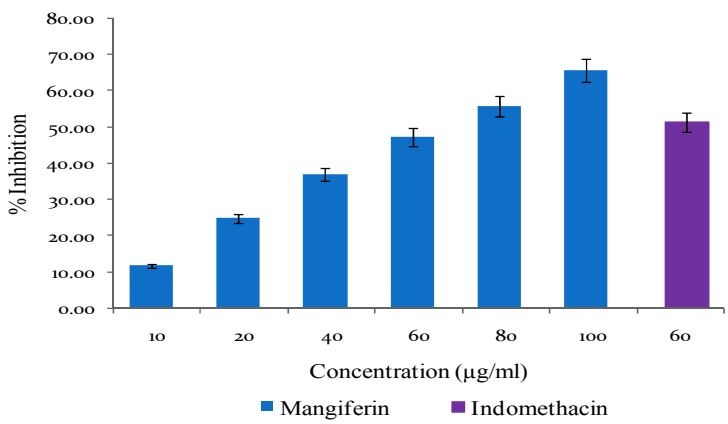
"**Fig. 1.** Radical scavenging activity (DPPH) of mangiferin."



"**Fig. 2.** Reductive capability of mangiferin."



"**Fig. 3.** Hydrogen peroxide scavenging activity of mangiferin."



"**Fig. 4.** Anti-lipoxygenase activity of mangiferin."

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

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Isolation and Identification of Polyphenolic Compound (Quercetin) from *Ipomoea aquatica* Forsk. (Convolvulaceae)

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ABSTRACT

Ipomoea aquatica is used in Indian system of medicine and many pharmacological activities reported on it. The preliminary phytochemical screening revealed presence of polyphenolic compounds in it. The hydroalcoholic extracts of leaves of *Ipomoea aquatica* was studied for isolation of flavonoid by chromatographic method. On the basis of study of physical properties, chemical tests, HPTLC and spectroscopic evaluation (UV, IR, 1HNMR and Mass Spectrometry) established the structure of the isolated compound. The isolated compound was yellow colored which was soluble in methanol, ethanol and dimethyl sulfoxide having melting point range 315-317^{o}C. Interpretations of all the spectra revealed that the isolated compound was quercetin. Thus it was concluded that many pharmacological activities reported on this plant may due to presence of quercetin.

SUMMARY

Quercetin, a flavonoid was isolated and characterized from hydroalcoholic extract of leaves of *I.aquatica.*

Keywords: Ipomoea aquatica, quercetin, convolvulaceae

INTRODUCTION

Now a day, researchers have given great attention on medicinal plants due to their safe utility. The curative properties of medicinal plants are mainly due to the presence of many complex secondary metabolites such as alkaloids, flavonoids, steroids, phenolics, terpenes, volatile oils etc. *(1)*. There are about 4000 flavonoids founds universally in plants*(2)*. Flavonoids are polyphenolic compounds and primarily recognized as the plant pigments. These flavonoids gives a remarkable biochemical and pharmacological actions *viz*., anti-inflammatory, antioxidant, anti-allergic, hepatoprotective, anti-thrombotic, antiviral and anti-carcinogenic activities *(3)*. Quercetin is also one of plant pigments having flavonoid structure. It is responsible for the colours of various parts of plants and also works as anti-inflammatory, antioxidant, anticancer agents *(4)*.

Ipomoea aquatica Forsk. (Family- *Convolvulaceae*) is green leafy perennial herb. It is commonly known as Nalanibhaji and found throughout India *(5)*. *I. aquatica* use as carminative, lessens inflammation; many other diseases in Unani system of medicine *(6)*. Many components in group of phytosterols, flavonoids, carbohydrates and proteins found to be present in preliminary phytochemical screening of *I. aquatica* leaves*(7)*. The aim of the study deals with isolation and characterization of polyphenolic compound quercetin from this plant.

MATERIALS AND METHODS

Plant material

The leaves of the plant *I. aquatica* were collected from Rajkot district of Gujarat, India. The plant material was identified by Faculty in botany, Biology Department, Gyanyagna College of Science & Management, Rajkot and voucher specimens (Voucher No. AIP/12/02) has been retained in Department of Pharmacognosy, Atmiya Institute of Pharmacy, Rajkot, Gujarat, India.

Isolation of Phytoconstituent

The dried powdered leaves of *I. aquatica* were extracted with 70% ethanol in soxhlet apparatus for 24 hrs. The extract was concentrated and fraction successively with petroleum ether (40°- 60°C), ethyl ether and ethyl acetate (each step repeated 3 times) following the method of Subramanian and Nagarajan*(8)* with some modifications. The collected fractions are considered as fraction-I, fraction-II and fraction-III respectively. Ethyl ether was used for free flavonoids while ethyl acetate for bound flavonoids.

Because of rich content of fatty materials fraction I was rejected. Fraction II was showed absence of flavonoids while fraction III showed presence of flavonoids. Fraction-III was hydrolyzed for 5 hours with 7% H2SO4. The filtrate extracted with ethyl acetate. The ethyl acetate layer was washed with distilled water till neutrality. Collected ethyl acetate fraction, dried and weighed *(*fraction IV*)*. Also collected aqueous phase with all water washes, combined them, dried and weighed *(*fraction V). Fraction V was showed presence of carbohydrate in chemical tests that also confirms the hydrolysis of glycosides to aglycon and glycone.

Fraction IV and V both subjected to chemical tests for flavonoids. Since Fraction IV showed presence of flavonoids, it was selected for Preparative Thin Layer Chromatography for further isolation of active components.

Thin layer chromatography

The activated silica gel G coated glass plates were used. Fraction IV was applied on TLC plant with standard reference compound (quercetin) and was run in solvent system of n-butanol: acetic acid: water (4: 1: 1).
Many other mobile phases such as n-butanol: acetic acid: water (4:1:5, upper layer), n-butanol: acetic acid: water (3:1:1), petroleum ether: ethyl acetate (2:1), toluene: ethyl acetate: water (5:3:2) were also tried. PTLC (Preparative Thin Layer Chromatography) was done using mobile system containing n-butanol: acetic acid : water (4: 1: 1) because it gave best results compare to other mobile systems.
Two fluorescent spots were observed in fractions IV when shown under UV light of which one spot was coinciding with those of the standard sample of quercetin (Rf: 0.87).
The developed plate was sprayed with 5% alcoholic ferric chloride solution for further confirmation. Rf value was calculated for each spot (spot IA-I have Rf: 0.87 and spot IA-II have Rf: 0.64) and compared with coinciding standard.

Preparative thin layer chromatography (PTLC)
PTLC prepared using silica gel-G coated (about 0.5 mm) glass plates (25 x 20 cm) which was activated at 100°C for 30 minutes and used after cooling.
The Fraction IV was dissolve in ethanol and applied on plate and developed dried plate was visualized under UV and fluorescent bands corresponding to standard reference quercetin was scraped. Then eluted with ethanol and it was subjected to various physical evaluation, chemical tests and spectroscopic analysis for further identification.

High Performance Thin Layer Chromatography (HPTLC) of isolated compounds
The stock solutions of standard quercetin (5, 15, 25, 35 and 45 µl) were sprayed on an HPTLC plate to obtain concentrations of 100,300, 500, 700 and 900 ng/band respectively. The stock solutions of IA-I and IA-II of 15 µl were sprayed on an HPTLC plate to obtain concentrations of each 300 ng/band. After development, the chromatograms were scanned. Densitometric detections were performed at 372 nm to confirm the purity of isolated compounds.

HPTLC-densitometry conditions and instrumentation
HPTLC was performed on 10 cm × 10 cm aluminum foil plates precoated with 0.2 mm layers of silica gel 60 F254 (E. Merck, Germany) without any pretreatment. Samples were applied as bands 6 mm wide, 10 mm apart, by use of a CAMAG (Switzerland) Linomat 5 applicator with a CAMAG microliter syringe. A constant application rate of sample 150 nl/s was employed. Plates were developed in Twin Trough Chamber 10x10cm (CAMAG, Switzerland). Plates were developed using a mobile phase: butanol: acetic acid : water (8:2:2 v/v/v) (distance of 8 cm). The optimized chamber saturation time for mobile phase was 20 min at room temperature (25 ±2 ◦C). After development, densitometric scanning was performed with a CAMAG TLC scanner 3 (CAMAG, Switzerland) in absorbance mode at 372 nm (Previously determined λ_{max} value of quercetin by UV spectroscopy). The source of radiation was deuterium lamp emitting a continuous radiation between 200 and 400 nm. The data obtained were analyzed by winCats software (CAMAG, Switzerland).

Characterization of isolated compound
Physical properties
Appearance, color, solubility and melting point of the isolated constituent was determined.

Chemical identification

The alcoholic solution of the isolated compound was subjected to shinoda test, ferric chloride test, lead acetate test and alkaline reagent test *(9,10)* .

Spectral analysis

UV spectra were determined in methanol and after using shift reagent 2M NaOH on Shimadzu UV-1700 visible spectrophotometer. FTIR spectra was measured in Bruker alpha FTIR spectrometer using KBr pellets. Also study Mass spectra. ^{1}H NMR spectra was performed on a Bruker NMR spectrometer using DMSO-*d*6 as solvent for measurement.

RESULTS AND DISCUSSION

HPTLC of isolated compounds

HPTLC chromatogram of isolated compounds IA-I and IA-II which were isolated from fraction-IV at 372 nm is given in figure 1 and figure 2 respectively. HPTLC photographs of Quercetin standard, IA-I and IA-II is given in figure 3. Isolated compound IA-I showed 1 peak at Rf value 0.87. Isolated compound IA-II showed 1 peak at Rf value 0.64. Thus the presence of one peak indicated the purity of the isolated compounds IA-I and IA-II.

Characteristics of the isolated compound

The isolated compound was yellow colour having melting point range 315-317 ^{o}C. The compound is soluble in DMSO, methanol, ethanol, aqueous alkali but practically insoluble in water.

Chemical Identification : The isolated compound gives pink colour in shinoda test**,** yellow precipitates in lead acetate test, deep blue-black color with ferric chloride test and bright yellow coloration in alkaline reagent test.

UV Spectra: The isolated compound IA-I in methanol gives 2 peaks in UV spectrum (figure 4) at λ_{max} value 259 nm and 372 nm. After addition of 2M NaOH in methanol solution λ_{max} values were shifted to 321 nm and 438 nm respectively. The UV spectra (figure 5) was shifted towards longer wavelength on addition of alkali which indicated that the compound was showed bathochromic shifts which is a characteristic of flavonoids*(9,10)*.

IR Values: FT-IR spectrum (figure 6) showed the characteristic intensities of C=O absorption band at 1653.49 cm^{-1} for the isolated compound. The OH stretch at 3508.89, 3438.67, 2929.29 and 1369.59 cm^{-1}. The C-O-C stretch at 1264.79 and 1163.81 cm^{-1}. C=C Stretch at 1515.58 and 1601.65 cm^{-1}.

Mass values: The mass spectra (figure 7) of the isolated constituent obtained by GC-MS, gave base molecular peak at 302 m/z was most abundant ions. The ^{1,3}A+ ion, which is observed for all flavonoid groups, is generally the fragment most readily formed *(11)*. The most readily formed fragment ion was ^{1,3}A+ ion which was given by all flavonoid groups at *m*/*z* 153 *(11)*. It was observed that the isolated compound also showed peak at *m*/*z* 153. Thus it was again conformed that the isolated compound was a flavonoid.

NMR values: The 1HNMR spectrum (figure 8) showed signals at δ value (400 MHz, DMSO-d_6) given in Table 1 with possible position of protons*(12-14)*. The structure of quercetin is given in figure 9. The spectrum of isolated compound IA-I showed 10 protons in their structure of which five protons on aromatic ring ranging from 6-8 ppm while remaining five protons in hydroxyl group attach to rings on

range 9-13 ppm *(15)*. These all data are similar to quercetin. Thus the isolated compound IA-I may be quercetin.

CONCLUSION

TLC and HPTLC study of isolated compound IA-I revealed that the IA-I (R_f : 0.87) was resembles standard quercetin in R_f value. The isolated compound was showed shinoda's test positive which indicates that it was flavonoid. λ_{max} from UV spectrum indicated the presence of conjugation and two chromophores which is a specific character of flavonoids. The isolated compound showed Bathocromic shift on addition of alkaline solution which is the characteristic of the flavonoid compounds that further confirmed the nature of the isolated compounds. FT-IR spectra resulted in presence of functional groups hydroxyl (-OH) stretch, C-O-C, C=O stretch for lactone and aromatic benzonoid ring. The structure of the isolated flavonoid was further elucidated by Mass spectroscopy and ^{1}HNMR spectroscopy which indicated the compound have molecular weight 302 and 1H NMR showed the aromatic proton and hydroxyl proton. These all characters are similar to quercetin. From the above study concluded that quercetin, a flavonoid was isolated and characterized from hydroalcoholic extract of leaves of *I. aquatica.* Thus it was concluded that many pharmacological activities reported on this plant may due to presence of quercetin.

FIGURES

Fig. 1. **HPTLC chromatogram of IA-I**

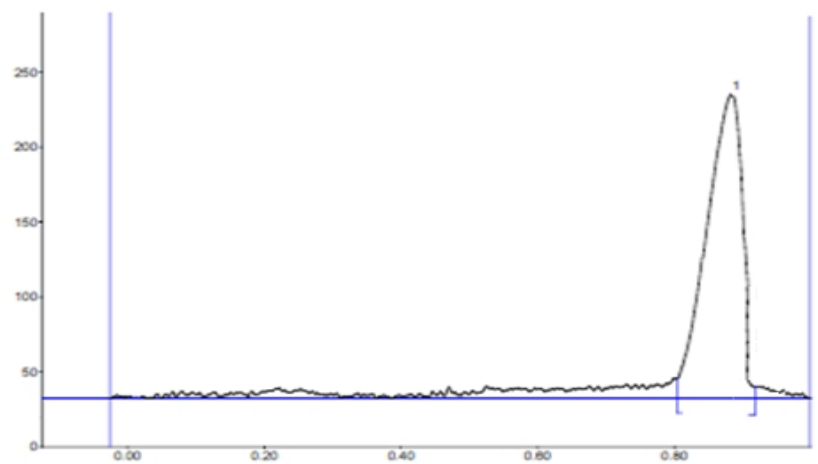


Fig. 2. HPTLC chromatogram of IA-II

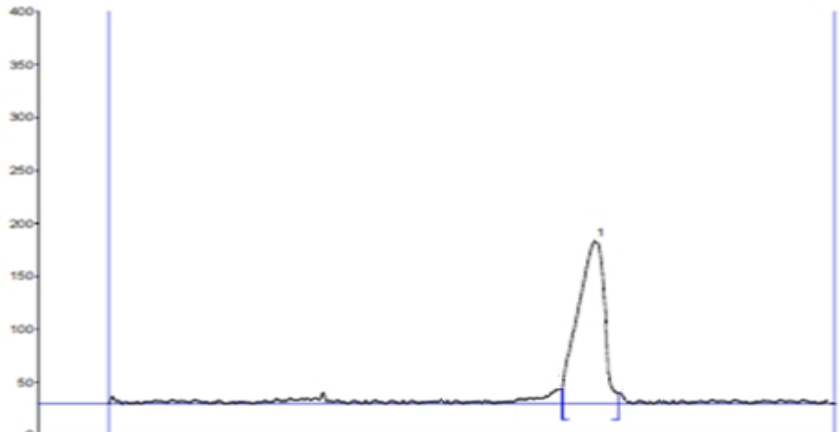
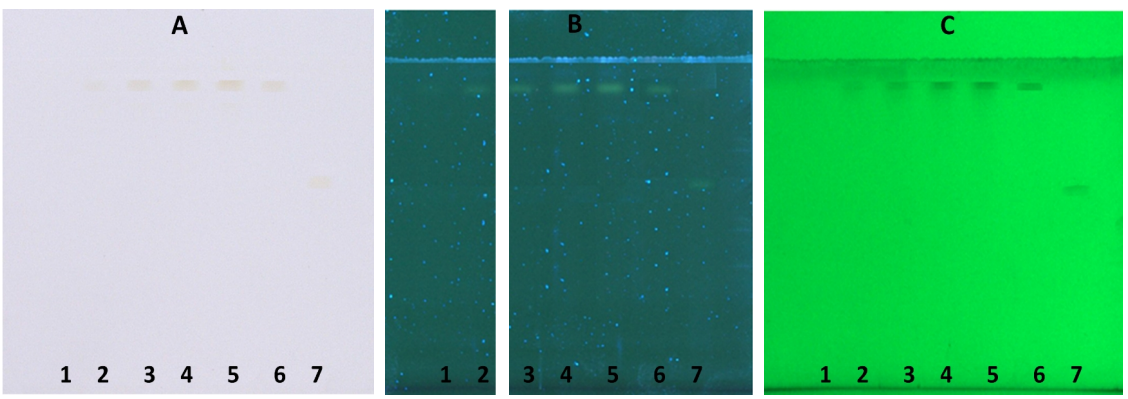


Fig. 3. HPTLC photographs of standard Quercetin, IA-I and IA-II



A: HPTLC photograph at white light, **B:** HPTLC photograph at 366 nm, **C:** HPTLC photograph at 254 nm, **Track 1:** Quercetin 100 ng/band, **Track 2:** Quercetin 300 ng/band, **Track 3:** Quercetin 500 ng/band, **Track 4:** Quercetin 700 ng/band, **Track 5:** Quercetin 900 ng/band, **Track 6:** IA-I 300 ng/band, **Track 7:** IA-II 300 ng/band.

Fig. 4. UV spectrum of isolated compound IA-1 in methanol

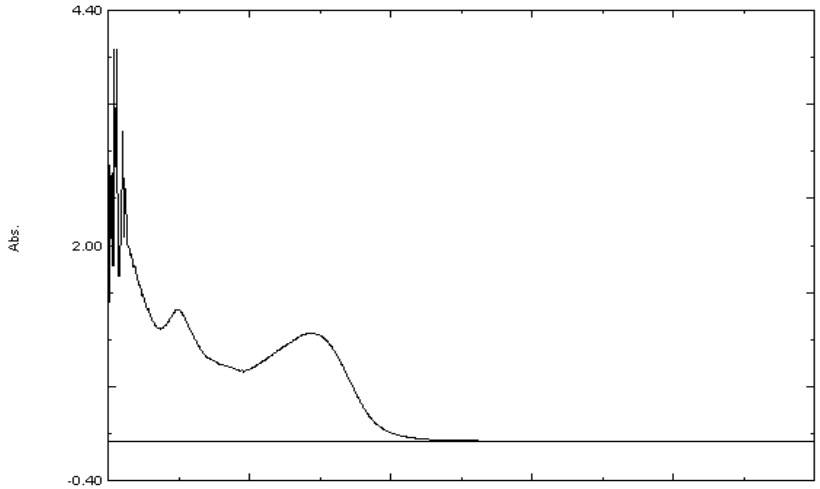


Fig. 5. UV spectrum of isolated compound IA-1 after addition of 2M NaOH in methanol solution

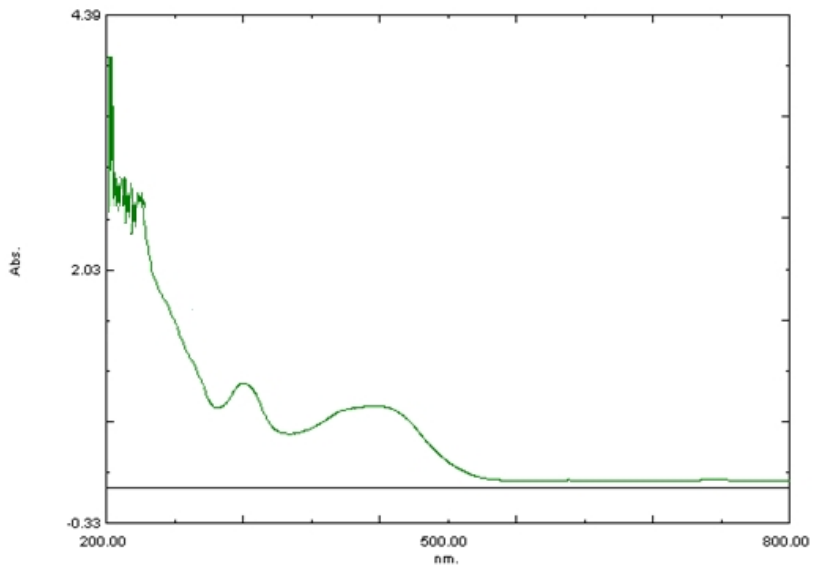


Fig. 6. FTIR spectrum of isolated compound IA-1

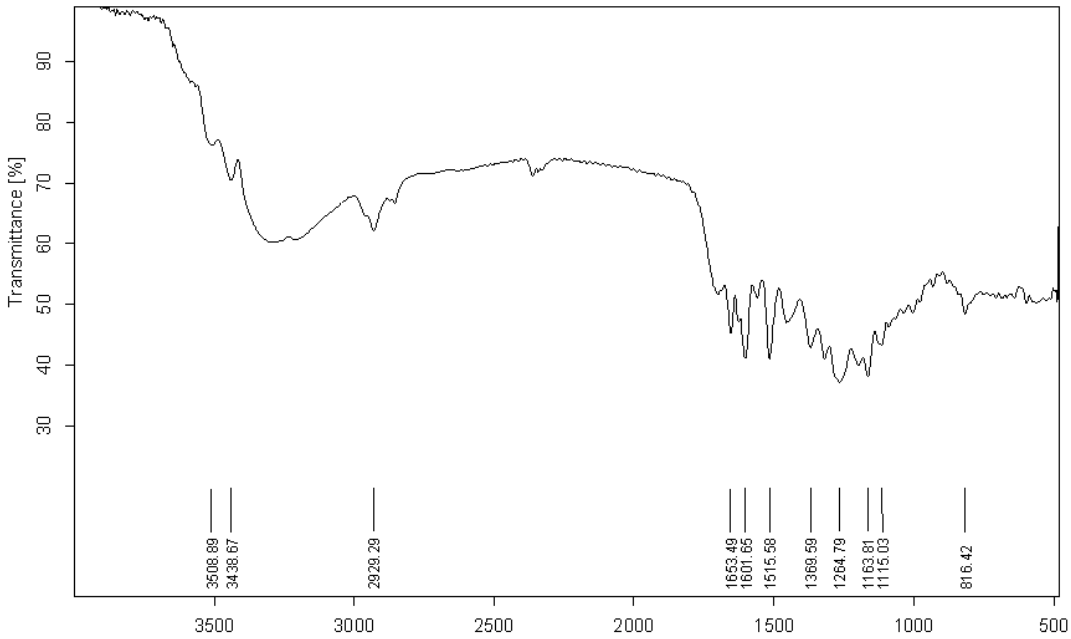


Fig. 7. Mass spectrum of isolated compound IA-1

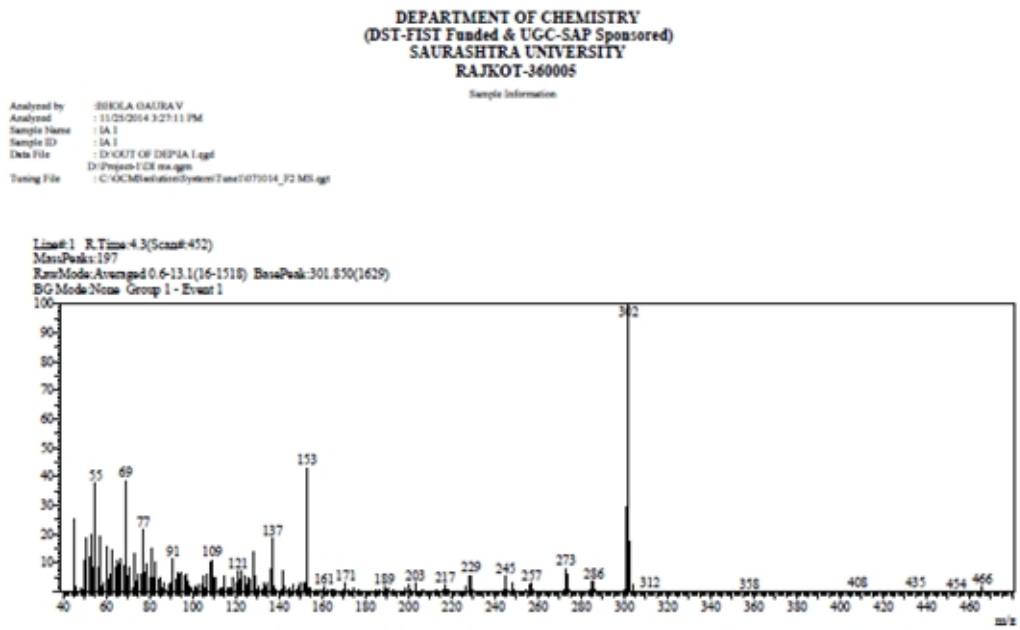


Fig. 8. ^{1}H NMR spectrum of isolated compound IA-1

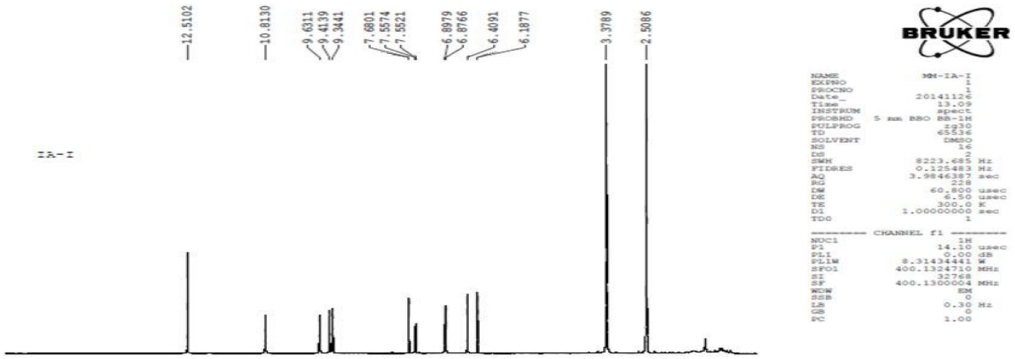
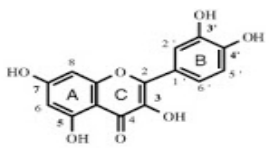


Fig. 9. Structure of quercetin



Quercetin

TABLES

Table 1. δ value in ^{1}H NMR spectrum of isolated compound and its possible position of protons

δ value (ppm)	Position of protons *(12-14)*
12.51 (1H,s)	OH-5
10.81 (1H,s)	OH-7
9.63 (1H,s)	OH-3
9.41 (1H,s)	OH-4’
9.34 (1H,s)	OH-3’
7.68 (1H,s)	H-2’
7.55 (1H,d)	H-6’
6.89, 6.87 (1H,d)	H-5’
6.40 (1H,s)	H-8
6.18 (1H,s)	H-6

ACKNOWLEDGEMENT

The authors are thankful to Department of Chemistry, M.N. Virani Science College, Rajkot, Gujarat, India, for providing the facilities for IR spectroscopy and also to Atmiya Institute of Phamacy, Rajkot, Gujarat, India for providing the facilities necessary to carry out research.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

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Stability Indicating Reverse Phase High Performance Liquid Chromatographic Method for Argatroban

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ABSTRACT

This research work is based on liquid chromatographic method development, validation and stability study for Argatroban. The method involved Acetonitrile: Water (40:60v/v) (pH 4 adjusted by o-phosphoric acid) as a mobile phase and InertSustainSwift ™C18 (250mm×4.6mm i.d, 5µm) as a column with 1 ml/min flow rate using 330 nm UV wavelength for measurement at room temperature. The retention time obtained 6.65 min. The method was linear range in concentration of 20-100µg/ml with regression coefficient (r^2) of 0.999. A method was validated as per ICH guidelines which show good recovery and reproducibility. Stability study was carried out under different degradation conditions like Acidic, Basic, Water Hydrolysis, Oxidation, Photolytic and Thermal degradation. This developed method gives the Rate of Degradation which can be useful in the routine stability study.

SUMMARY

Stability study of Argatroban by RP-HPLC

Argatroban, method validation, stability study, high performance liquid chromatography

INTRODUCTION

Argatroban is a synthetic chemical moiety act as thrombin inhibitor and used as prophylaxis action of thrombosis in patient having heparin-induced thrombocytopenia (HIT). It is new blood thinner used now days. It is synthesized from L-arginine. The main purpose of it's to control blood clotting in people who have harmful action to heparin. It is also used to prevent blood clotting during some medical treatments like coronary balloon angioplasty, percutaneous coronary intervention, stent placement etc *(1-4)*. This drug is official in Japanese Pharmacopoeia *(5)*. It is a new chemical entity so only limited analytical work has been reported for this drug *(6-9)*. No Stability method is reported for its estimation in the literature. Therefore purpose for this work was development of stability indicating chromatographic method for Argatroban.

MATERIALS AND METHODS

HPLC Instrument having UV-Visible detector SPD 10 A – LC10 AT, Shimadzu; AR grade chemicals and HPLC grade solvents were used for analysis. The separation was performed on InertSustainSwift $^{TM}C_{18}$ (250mm×4.6mm i.d.). The flow rate and volume of injection were 1 ml/min and 20µl respectively and wavelength for detection was 330 nm. The mobile phase contain Acetonitrile:Water(40:60 v/v) (pH 4 pH 4 adjusted by o-phosphoric acid) and run time was 10 min.

Preparation of standard solutions:

Preparation of standard stock solution: 1000µg/ml standard stock solution was prepared by taking 100 mg of Argatroban into 100 ml volumetric flask, first dissolved it into 25 ml of Acetonitrile and finally volume was made 100 ml with Acetonitrile.

Method Validation Parameters:

Linearity: 0.2, 0.4, 0.6, 0.8 and 1 ml of 1000µg/ml of standard solution of Argatroban were taken into different 10 ml of volumetric flasks and volume was made up to 10 ml with mobile phase to get final strength of 20, 40, 60, 80 and 100µg/ml respectively. These prepared solutions of Argatroban were injected into HPLC system under the developed chromatographic condition. Calibration curve was developed by preparing graph of peak areas versus concentrations of Argatroban.

System Suitability: 1 ml standard stock solution of Argatroban was taken into a 10 ml volumetric flask and the volume adjusted with mobile phase to get strength of 100µg/ml. This solution was injected in to HPLC system under the developed chromatographic condition and Retention time, Peak area, Tailing factor and theoretical plates in developed chromatogram was observed.

Accuracy: The %recovery was performed by 40µg/ml of Argatroban sample solution. Three samples were prepared at level of 80%, 100% and 120% and % recoveries were calculated from the calibration curve.

Precision: Intra-day and inter-day precision was calculated by three independent measurements of standard drug solution at concentrations 20, 40 and 60µg/ml of Argatroban 3 times on the same day and on three different days.

LOD & LOQ: The peak area of 10 solutions containing 40µg/ml was taken. LOD and LOQ were calculated by equation LOD = [3.3 x SD/ slope] and LOQ = [10 x SD/slope]; SD is standard deviation of response.

Robustness: It was measured by changing pH, Ratio of mobile phase and Flow rate. The pH of mobile phase was set at ±0.2, Ratio of Mobile phase was set ±5 ml and Flow rate was set at ±0.2 ml/min. Solution of both the drugs was injected three times**.**

Determination of active ingredients in synthetic mixture:

10 ml of synthetic mixture was prepared as per patent: Argatroban: 100 mg; Glacial acetic acid: 0.172 ml; Water for injection: q.s to 1 ml. Then From this synthetic mixture solution take 1 ml of solution in 100 ml volumetric flask and add 50 ml of mobile phase then sonicate it for 15 minutes and make up to 100 ml

with mobile phase to make 1000 µg/ml solution. From above solution 0.4 ml solution is taken in 10 ml volumetric flask and volume was made up to 10 ml by mobile phase to get final strength of 40µg/ml.

Degradation Study:

Acid Degradation: 10 mg of drug was taken in a volumetric flask (100 ml) and dissolved in 1 ml of methanol; volume was made by 0.01N HCl (100µg/ml). Then this solution was kept at room temperature and at definite time period pipette out 0.1 ml, neutralized by 0.1 ml 0.01N NaOH and diluted with mobile phase up to 10 ml and chromatogram was recorded. Further sampling was taken for 0, 10, 30 min, 1 Hr and 2 Hrs from above solution. Blank solution (without API) was also recorded.

Base Degradation: 10 mg of drug was taken in a volumetric flask (100 ml) and dissolved in 1 ml of methanol; volume is made by 0.01N NaOH (100µg/ml). Then this solution was kept at room temperature and at definite time period pipette out 0.1 ml, neutralized by 0.1 ml 0.01N HCl and dilute with mobile phase up to 10 ml and chromatogram was recorded. Further sampling was taken for 0, 10, 30 min, 1 hr and 2 hr from above solution. Blank solution (without API) was also recorded.

Neutral Hydrolysis: 10 mg of drug was taken in a volumetric flask (100 ml) and dissolved in 1 ml of methanol; volume is made by Distilled Water (100µg/ml). Then this solution is kept at room temperature for 2 hrs and at definite time period pipette out 0.1 ml and dilute with mobile phase up to 10 ml and chromatogram was recorded. Further sampling was taken for 0, 10, 30 min, 1 hr and 2 hr from above solution. Blank solution (without API) was also recorded.

Oxidation Degradation: 10 mg of drug was taken in a volumetric flask (100 ml) and dissolved in 1 ml of methanol and then volume was made by 3 % H_2O_2(100µg/ml). Then this solution was kept at room temperature and at definite time period pipette out 0.1 ml and dilute with mobile phase up to 10 ml and chromatogram was recorded. Further sampling was taken for 0, 10, 30, 1 hr and 2 hr from above solution. Blank solution (without API) was also recorded.

Photolytic Degradation: 100mg of standard drug was taken into the Petri dish and placed under UV Light for 2 hr. 10mg of drug was weighed and diluted with mobile phase up to 100 ml and chromatogram was recorded. Similar Procedure was followed for 2 Hr.

Thermal Degradation: 100 mg of standard drug was taken into the Petri dish and was put it into the oven at 70˚C for 2 hrs. 10mg of drug was weighed and diluted with mobile phase up to 100 ml and chromatogram was recorded. Similar Procedure was followed for 2Hr.

RESULTS AND DISCUSSION

RP-HPLC method was developed and validated as per ICH guidelines. Stability study was carried out under different condition like Acid degradation (0.01N HCl), Base degradation (0.01N NaOH), Water Hydrolysis, Oxidation degradation 3 % (H_2O_2), Photolytic degradation (UV light), Thermal degradation (70˚C). Results for developed method were represented in tables and figures.

CONCLUSION

This research work involved the development and validation of Stability indicating RP-HPLC method for Argatroban. Method was validated as per guideline of ICH. Stability study was also done under various condition like Acid degradation (0.01N HCl), Base degradation (0.01N NaOH), Water Hydrolysis, Oxidation degradation 3 % (H_2O_2), Photolytic degradation (UV light), Thermal degradation (70˚C) and from that % degradation was observed 40.10%, 44.96%, 47.98%, 53.05%, 1.14% and 1.52% Respectively. The developed method was found to be simple, robust, linear, precise and accurate for the determination of Argatroban.

FIGURES

Fig. 1. Chemical Structure of Argatroban

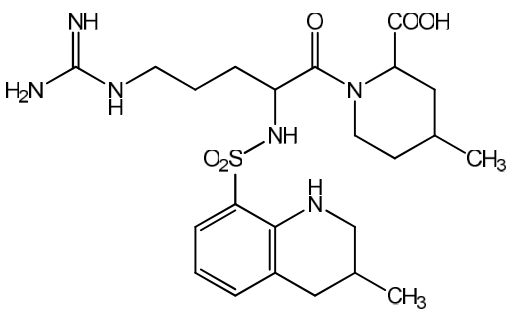


Fig. 2. Chromatogram of Linearity curve

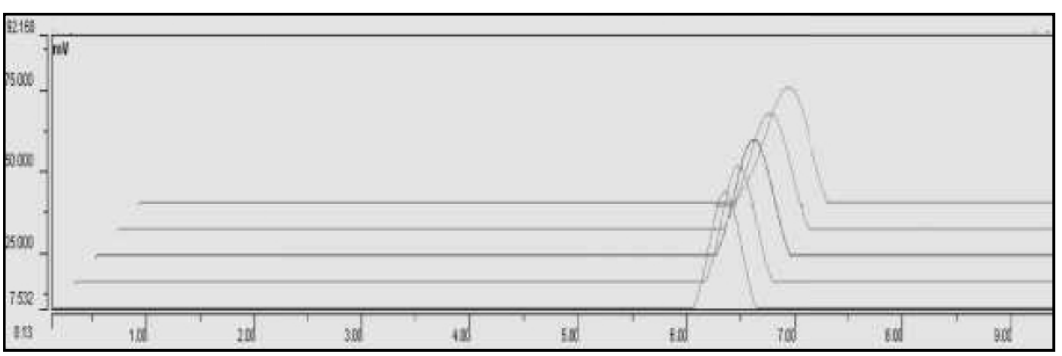


Fig. 3. Chromatogram of Argatroban Standard

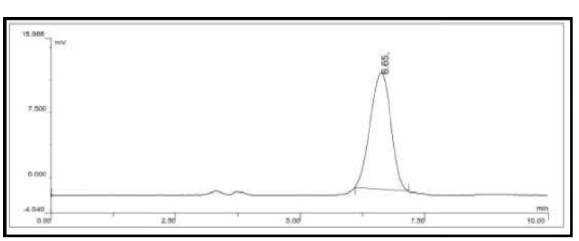


Fig. 4. Chromatogram of Argatroban Acid Degradation after 2 Hrs at Room Temperature

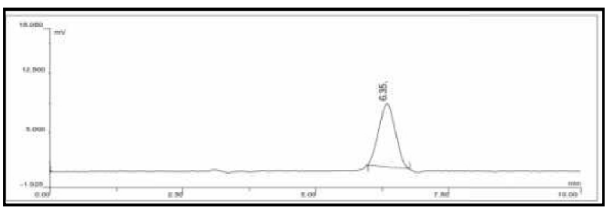


Fig. 5. Chromatogram of Argatroban Base Degradation after 2 Hrs at Room Temperature

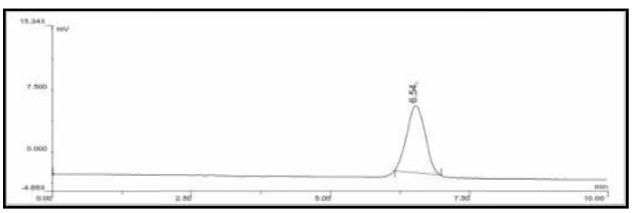


Fig. 6. Chromatogram of Argatroban Neutral Degradation after 2 Hrs at Room Temperature

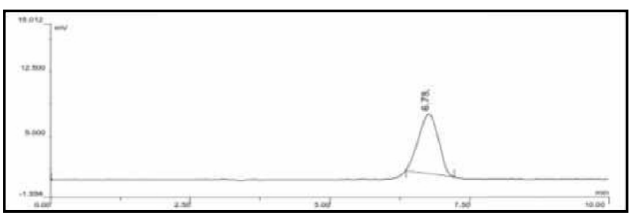


Fig. 7. Chromatogram of Argatroban Oxidation Degradation after 2 Hrs at Room Temperature

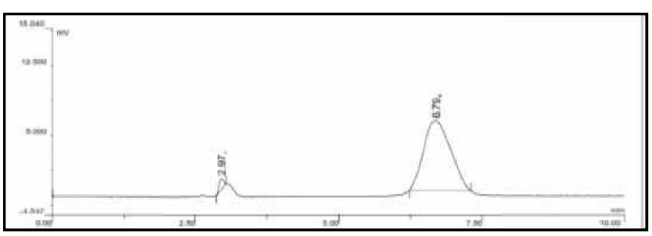


Fig. 8. Chromatogram of Argatroban Thermal Degradation after 2 Hrs at Room Temperature

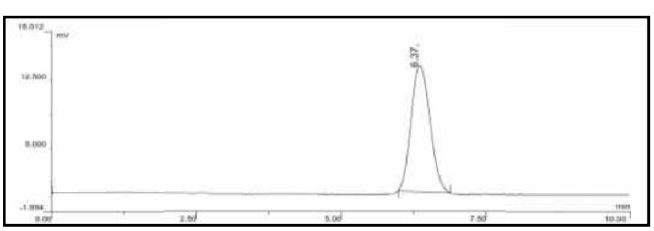
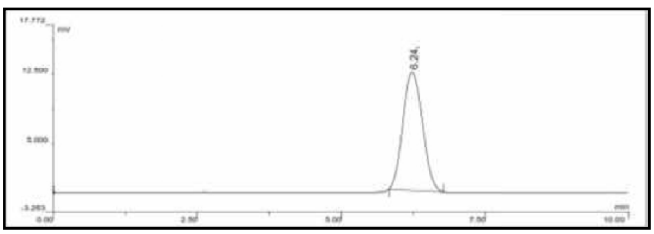


Fig. 9. Chromatogram of Argatroban Photolytic Degradation after 2 Hrs at Room Temperature



TABLES

Table 1. System suitability

Sr. No	Parameters	value obtain for Argatroban
1	No. of Theoretical Plates (N)	2103
2	Tailing Factor (Tf)	0.50

Table 2. Linearity (n=6)

Concentration (µg/ml)	Peak Area ±SD (n=6)
20	144154±839
40	324124±633
60	500200±124
80	663210±1431
100	807789±1215

Table 3. Intraday Precision

Sr. no.	**Concentration**	**Peak Area ± SD**	**% RSD**
1	20	140681±857	0.60
2	40	321428±1878	0.58
3	60	509961±2659	0.52

Table 4. Interday Precision

Sr. no.	**Concentration**	**Peak Area ± SD**	**% RSD**
1	20	141177±1070	0.71
2	40	334904±2049	0.61
3	60	513409±4046	0.78

Table 5. Accuracy

Concentration of Argatroban from formulation (µg/ml)	**Amount of Standard Argatroban added (µg/ml)**	**Total amount of Argatroban (µg/ml)**	**Argatroban obtain (µg/ml) Mean* ± SD**	**% RSD**	**% Recovery (n=3)**	**% RSD**
40	--	40	40.03 ± 0.1006	0.25	100.72±0.1100	0.10
40	32	72	71.96± 0.1401	0.19	99.94± 0.1900	0.19
40	40	80	80.58 ± 0.0900	0.11	100.08±0.2516	0.25
40	48	88	88.02 ±0.1059	0.12	100.02±0.1159	0.11

Table 6. LOD & LOQ

Standard Deviation (SD)	3461
Slope	8331
Limit of Detection (µg/ml)	0.45
Limit of Quantification (µg/ml)	1.37

Robustness Data

Table 7. Change in Flow rate

Sr. no	Flow Rate (mL/min)	Concentration (µg/ ml)	Retention Time(RT)	Mean of Peak Area ± SD (n=3)	%RSD
1	1.0	40	6.29	320656±1511	0.47
2	0.8	40	7.01	326380±1763	0.54
3	1.2	40	5.47	326374±1121	0.34

Table 8. Change in pH

Sr. no	pH change	Concentration (µg/ ml)	Mean of Peak Area ± SD (n=3)	%RSD
1	4.0	40	323160±1483	0.45
2	3.8	40	320752±1013	0.31
3	4.2	40	324044±1517	0.46

Table 9. Change in Mobile phase ratio

Sr. no	Ratio of mobile phase	Concentration (µg/ ml)	Mean of Peak Area ± SD (n=3)	%RSD
1	40:60	40	321799±1759	0.54
2	48:62	40	322184±1587	0.49
3	42:58	40	322419±2314	0.71

Table 10. Assay Data in synthetic mixture of Argatroban

Sr. no	Sample	Concentration (µg/ml)	Concentration Found (µg/ml)	% Assay
1	Argatroban	40	40.02	100.06 %
2	Argatroban Formulation	40	40.08	100.20 %

Table 11. Summary of Degradation Study

Condition	% Degradation after 2 hr at room temp.
Acid degradation	40.10%
Base degradation	44.96%
Water hydrolysis	47.98%
Oxidation	53.05%
Photolytic	1.14%
Thermal	1.52%

Table 12. Validation Parameters: Summary

Sr. no	Validation Parameter	Result
1	Linearity range (µg/ml)	20-100
2	Wavelength (nm)	330
3	Standard Regression equation	y=8331x -12011
4	Correlation coefficient	0.998
5	Precision (%RSD) Intraday Interday	 0.52-0.60 0.61-0.78
6	% Recovery (Accuracy)	100.19
7	Limit of Detection (µg/ml)	0.45
8	Limit of Quantification (µg/ml)	1.37
9	Robustness (%RSD) a. change in flow rate b. change in pH c. Ratio of mobile phase change	 0.34-0.54 0.31-0.46 0.49-0.71
10	% Assay a) API b) Formulation	 100.06% 100.20%

ACKNOWLEDGEMENT

The authors thank BDR International Pharmaceutical pvt Ltd, Baroda for kind donation of Argatroban. The authors would also like to thank Shree Dhanvantry Pharmacy College, Kim, and Gujarat for providing facilities for carrying out this research work.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

(Proceedings available for download at rku.ac.in/icre)

RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

Production purification and characterization of fibrinolytic enzyme from *streptococcus* species

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ABSTRACT

A unique thrombolytic enzyme was produced from newly isolated bacterial strain. Enzyme was purified, characterized and amino acid sequence of enzyme was resolved. Enzyme producing bacterial species was identified as streptococcus sp. was Isolated from human body swab. Fermentation technique was used for production and purified to homogeneity by three stage purification which includes salting out with ammonium sulphate, column chromatography and RP-HPLC with an 11.0 time increase in the action and 10% recovery. Molecular mass of the enzyme was found to be 49.884 kDa and *PI* of the protein found to be 6.04 by LCMS/MS QTOF. Purified enzyme was also found to exhibit fibrinolytic activity on fibrin agar plate by producing clear zones of fibrin digestion. Enzyme was found to active versus a varied range of pH from 6.5 to 8.0 with an optimum at pH 7.0 and a temperature of 37^0C to 40^0C with an optimum temperature of 37^0C

SUMMARY

Medicinally significant thrombolytic enzyme produced from bacterial species streptococcus and characterized.

Keywords: Streptokinase, molecular characterization, peptide sequence, BLAST, LCMS/MS, Q-Tof

INTRODUCTION

A thrombus is a collection of blood clot comprising fibrin, platelet and cellular rudiments trapped inside a vascular lumen(*1, 2*). The development of formation of thrombus is called thrombosis. If the thrombosis arises in arteries is named as arterial thrombus in addition if it take place in veins it is termed venous thrombosis. The vessels often involved in the arterial thrombosis are coronary, cerebral, mesenteric, renal arteries and arteries of lower extremes. Venous thrombosis can be categorized as deep vein thrombosis, portal thrombosis, renal thrombosis, jugular vein thrombosis, cerebral venous sinus thrombosis. Pathological progression of clot in either arterial or venous side requires some serious clinical attention. Various collective complaints are correlated to thrombosis, of which myocardial infraction and stroke are very usual. Thrombosis is similarly a medical problem in several cancers and postsurgery, especially after joint surgery. Blood coagulates as discussed before, are typically consist of fibrin, are produced from fibrinogen by the action of thrombin(*3*). Fibrin is hydrolyzed by plasmin which is triggered from plasminogen by plasminogen activators(*4*). Thrombolysis these times is repetitive choice not merely in the management of acute myocardial infraction but in the same way in many other indices of thromboembolic disorders (*5*). Drugs by means of thrombolytic enzymes are the finest current real method in the management of thrombosis. A series of fibrinolytic enzymes like tissue plasminogen activators (t-PA), urokinase and bacterial streptokinase have been broadly considered and used as thrombolytic agents. On the other hand these agents have several boundaries like too much cost of clinical use, adverse effects such as too much blood loss and reappearance of clot at the site of lasting thrombosis, trouble in extensive duration usage, less specificity to fibrin in the case of urokinase, streptokinase and narrow half-life of t-P, urokinase. Consequently significant studies are currently under research to boost effectiveness and specificity of fibrinolytic treatment, and bacterial fibrinolytic enzymes attracts much more medical interest during these decades(*6*). Various microorganisms are central source of thrombolytic agents. Operative thrombolytic agents today being recognized and categorized from animals, plants and microorganisms. Microorganism are chief providers of thrombolytic agents, since they have several benefits like great amount of production and oral delivery for thrombotic disorders such as acute myocardial and cerebral infractions(*7*). Our current study recognized a high titre fibrinolytic enzyme generating bacterium isolated from human body swab sample from Vadodara, Gujarat. In the current study we report production purification and characterization of plasmin like direct acting fibrinolytic enzyme(*8*).

MATERIALS AND METHODS

Chemicals and materials used in the studies were of the AR grade and fibrinogen, thrombin and agarose were bought from Hi-Media Ltd, Mumbai, India. Buffer salts like Tris, dipotassium hydrogen phosphate, ammonium sulphate, calcium chloride, magnesium sulphate, sodium chloride, HCL, bacteriological media, agar, peptone, staining dies etc. were purchased from Merck India limited, Mumbai. Tween 20, tween 80, tween 60, H2O2, triton X100 etc. were acquired from SD fine chemicals Mumbai, India.

Isolation screening and identification of bacteria for fibrinolytic activity

Around 40 diverse samples were selected from the different regions of Vadodara city, Gujarat, India. The area selected for screening were garden soil, industrial soil, mud water, body swabs and lake water. The samples were collected according to regular microbiological procedures and preserved in the freeze (4^0C) until use. Samples were initially diluted with the sterile water and screened for protease production

using skimmed milk agar plate containing (g/l): peptone 5, yeast extract 3, bacteriological agar 12.5 and skimmed milk 250ml(*9-11*). Clear zones after incubation of 72Hrs shows the protease production. Colonies generating clear regions were isolated by repetitive streaking on fresh agar plates(*12-14*). Isolated colonies were than subjected to fibrinolytic screening by fibrin plate method by means of fibrin as a substrate(*15*). The fibrin plate prepared was composed of 2.5 ml of 1.2% bovine fibrinogen in 0.1M phosphate buffer (Ph. 7.4), 7.6ml 1% agarose solution, 0.1ml of bovine thrombin (10NIH unit/ml) in to petri dish. The solution in petri dish was set aside for 1Hr(*16*) to form fibrin layer. One ml of sample was spread on the plate and as kept for incubation. Isolate producing clear zones in fibrin plate was selected and identified as Streptococcus Spp. by colony morphology, gram staining biochemical test and selective media(*6, 17*).

Enzyme production

Streptococcus Spp. was grown on the basal media containing (g/l): Glucose,20, Sucrose 30,Yeast Extract 5.0, Beef Extract 5.0, Meat Extract 5.0, Peptone 5.0, KH_2PO_4 0.5, K_2HPO_4 0.5, slight traces of salts of Mg, Cu, and Fe, $CaCl_2$ 0.5, Amphotericin B2.5mg and 1000 ml distill water. Media autoclaved for 20 minutes at 121^0C and 15Lb pressure(*18*). Media cooled to room temperature and inoculated by two ml of uniformly prepared suspension of Streptococcus Spp. Inoculated media kept on orbital shaker incubator at 37^0C, 160 RPM for 5 days(*8*).

Protein analysis

At regular interval of 24Hrs. sample was withdrawn from the flask,centrifuged at 10,000 rpm for 10 minutes at 4^0C, supernatant taken and protein concentration was estimated by biuret method utilising bovine serum albumin as a standard.

Enzyme purification

Every stages of separation were performed at low temperature except other vice stated. Centrifugation at a speed of 12,000rpm for 20 minutes at 4^0C was used to separate out the cells from the broth. Supernatant was fractionated by slow addition of ammonium sulphate at 4^0C with continuous stirring. The precipitates obtained in 0-90% saturation of ammonium sulphate were collected by centrifugation, dissolved in 20mM K-phosphate buffer and checked for the activity. Fraction showing highest activity was selected and subjected to dialysis for overnight at 4^0C using the same buffer. Sephacryl s-200 gel filtration column (1.0*64cm) previously equilibrated with K-Phosphate buffer PH 7.4 was used for the further purification of enzyme dialysate. Fractions collected were subjected to determination of activity and fraction with maximum activity was selected. Selected portion was purified by reverse phase high performance chromatography (RP-HPLC) on a water reverse phase XR ODS column (3.9*300mm). Proteins were eluted with linear gradient from 5% to 95 % (v/v) acetonitrile containing 0.1% (v/v) formic acid. Protein elution monitored at 285nm and peaks were screened for the activity(*6*).

Fibrinolytic activity assay

Fibrinolytic activity was performed corresponding to the method described formerly (*19*). To form a fibrin clot, quickly 3.0 µl of thrombin (10NIH/ml) was mixed with the 40.0 µl of 0.6% (W/V) solution of bovine fibrinogen prepared in 100mM potassium phosphate buffer PH 7.4. Mixture was kept to stand at room temperature and clot was formed. Enzyme sample added at a dose of 5.0 µg/ml and reaction mixture incubated at 37^0C for different time interval. 10.0 µl of freeze cold trichloro acetic acid (10%v/v)

was added to terminate the reaction. Mixture obtained from above step was centrifuged, supernatant was considered to determine the amount of amino acid (tyrosine at 660 nm) released using Folin- Ciocalteu's reagent spectrophotometrically. Fibrinolytic activity was calculated from the standard curve of tyrosine(*20, 21*). One unit of fibrinolytic activity is classified as 1.0 µg of tyrosine liberated per minute per ml of enzyme(*4, 22-24*)(*12*).

Invitro fibrinolytic assay

In vitro fibrinolytic activity was detected by synthetic blood clot method given by omura. Synthetic blood clot was prepared using citrated goat blood (0.3ml) and 10 µl of thrombin (10NIH unit/ml). The above mixture was kept atroom temperature to form clot and incubated with definite volume of sample for altered time intermissions at 37^0C. After a required time period the remaining clot was weighed and the activity was stated as mg of clot lysed per µg of enzyme(*6, 8, 25*).

Biochemical characterization

Activity of the enzyme was also checked for its optimal pH and temperature by inoculating enzyme at changed pH (2-11) and temperature ranges (5-70^0C)(*26*).Specificity for Substrate of enzyme was likewise checked by incubating enzyme with altered substrate including casein, bovine serum albumin, globulin, fibrin, haemoglobin in buffer at 37^0C for 30 minutes. 10µl ice cold Trichloro acetic acid (10% v/v) added to conclude the reaction and amount of free amino acid (tyrosine) released wasdetermined at 660nm using Folin- Ciocalteu's reagent. One unit of fibrinolytic activity is expressed as 1.0 µg of tyrosine liberated per minute per ml of enzyme and which was obtained from the standard curve of tyrosine.

Sodium dodecyl sulphate polyacrylamide gel electrophoresis (SDS- PAGE)

SDS PAGE was performed as defined by Lammli by means of 5 % (m/v) stacking gel and 12% (m/v) separating gel. To the purified protein identical amount of sample buffer (0.05% bromophenol blue, 5% β mercaptoethanol, 20% glycerol and 20% SDS in 0.25M Tris HCl buffer, pH6.8) was supplemented and boiled at 100^0C for 5 minutes. The resultant mixture was then applied to SDS- PAGE and electrophoresis was carried out under 100V at room temperature for 1.5Hr(*6*)(*23*).

Mass spectroscopy and protein identification

Bands of concern in SDS -PAGE gel were taken out and in processed with trypsin. The detection of protein was achieved by using liquid chromatography tandem mass spectrometry (LC-MS/MS).LC/MS study of the sample was performed using an Agilent Infinity LC system coupled with an Agilent 6530 Accurate-Mass Quadrupole Time-of-Flight (Q-TOF). LC/MS system equipped with an Agilent Jet Stream dual electrospray ionization (ESI) source. The HPLC and Q-TOF LC/MS parameters are given in **Table1& Table2.** Fragment spectra were explored and compared to NCBI non redundant protein database. Database searches were conducted using Agilent spectrum mill search engine(*22*).

RESULTS AND DISCUSSION

Isolation screening and identification of fibrinolytic enzyme producers

In the present work samples were taken from garden and industrial soil, body swabs, and water from mud and lake. Samples first plated on skimmed milk agar for protease activity and around 10 microbial strains produced clear zones around their growth. Advance isolation done to get pure colony by repetitive streaking and lastly all strains were subjected to screen for fibrinolytic action. Out of 10 strains only one strain produces a clear zone of fibrin clearance. It was finally selected and further purified by repeated streaking on agar plate. Identification of the isolated strain was done according to the Bergery's, manual of systemic bacteriology and prokaryotes. Culture gave creamy off white colonial morphology with slight promotion and uneven internal characteristic on the internal surface. Microscopically cells are gram positive and spherical shaped of 0.5 to 1.5µm in size. Growth observed in chains and moderate length chains more specifically with broth culture. After overnight growth on blood agar plate four visual colonies produce β- hemolytic (complete) pattern. Outcomes as shown in **Table No.6** of biochemical test performed typically indicates characteristic of Streptococcus Spp(*6, 27-29*).

Enzyme production and purification

The fibrinolytic enzyme from Streptococcus Spp. was purified using a combination of salting out method using ammonium sulphate precipitation and chromatographic method. Protein concentration and activity assay were kept as indicator for the purification progress.The 80% fraction showed a maximum activity, when this fraction was further purified by gel chromatography resulted in five fractions. All the fractions were checked for the activity and fraction three found to be more active. It was later separated by RP-HPLC, proteins were eluted in peaks with a retention time of 3.092, 4.188 and 6.178,minutes.The fraction with retention time 6.178 minutes shows a maximum activity. The fraction was found to homogenous, when it was re run on RP-HPLC using XR- OSD column. A summary of purification scheme is given in the **table No 3.**

Fibrinolytic activity assay

The fibrinolytic action of the Streptococcus Spp. was evaluated by incubating crude enzyme extract with artificial fibrin clot prepared by mixing thrombin and fibrinogen in test tube. Activity was calculated in terms of tyrosine amino acid released, that reacts with Folin- Ciocalteu's reagent and produce color. From the table No: 3 it clearly indicates that each purification step the activity of enzyme increase and protein concentration decreases. It is also clear from the table that purification fold is much higher with less yield at final stage of purification.

Evaluation of Invitro fibrinolytic assay

Assessment of in vitro thrombolytic activity carried out using the fermented broth of Streptococcus Spp. Comprising crude enzyme as a sample, saline solution andblood clot as a control activity evaluator respectively. Blood clot degradation observed in test tube containing sample. Clot was completely degraded after 1 hr. at 37^0C and pH 7.0.in contrast, in control solution no clot degradation was observed.

SDS-PAGE

The molecular weight of purified protein was revealed by SDS-PAGE via low molecular weight calibration kit for SDS electrophoresis (GE Health care life sciences) as indicators. The indicators used were phosphorylase b (97,000KD), bovine serum albumin (66,000KD), ovalbumin (45,000KD), carbonic anhydrase (30,000KD), trypsin inhibitor (20.100KD), and α- lacto albumin (14,400KD)(*30*). The enzyme was purified to homogeneity by three step purification and resulting in 11.0 fold purification and 10% activity recovery. As shown in the **Fig 3.** SDS-PAGE investigation gave single band of purified protein of molecular weight of around 48.5kDa.

Mass spectroscopy and protein identification

The interested protein appearing as 48.0kDa protein band on SDS-PAGE gel, the band was cut out and analyzed after tryptic digestion. The 48kDa protein was subjected to electron spray tandem mass spectroscopy analysis. The fragment spectra were exposed to the NCBI non redundant protein database search. The spectra matched four tryptic peptides that were identical to streptokinase from streptococcus pyogenes species (NCBInr.Complete Accession #: 32396322) with 24% sequence coverage. The sequence of the enzyme gives calculated mass of 49.884 and *pI*of 6.04 very related to the experimental values attained, indicates that the enzyme is monomeric protein.

Biochemical characterization

As shown in the above **Fig** no. 3, 4, 5, and 6 pH and temperature greatly influenced the action of enzyme. Enzyme was found to be very active among pH 6.5 and 8.0. There is complete loss of action also experienced in very acidic and very alkaline situations. Activity was also found to be steady when incubated in optimum pH for 3hr. at 1^0C. Similarly enzyme action was also analyzed for the effect of temperature and found that it was very active at 37^0C and activity also persisted stable between 37^0C and 43^0C. Above and below the optimum temperature, lessening in the activity was observed. From the substrate study it also shows that the enzyme is more active against fibrin matched to all other.

CONCLUSION

Above research stated the screening and isolation of bacteria which can produce fibrinolytic enzymes. The work presented above reported the production and purification of fibrinolytic enzymes using three level purification methods. Additional attempt were also made to find biochemical nature of the purified enzymes. Form the study it shows, Streptococcus Spp. Are a potential candidate for the fibrinolytic enzymes and can be isolated and screened using a simple media. Besides the enzyme produced was first checked for its proteolytic property and next to its fibrinolytic property, in both the cases the enzyme is potent proteolytic and fibrinolytic agent. Biochemical characterization similarly recommends, it can be a novel candidate for future thrombolytic disease management. Substrate specificity directs affinity of enzyme is more towards fibrin than other proteins, consequently nature of enzyme if more fibrinolytic compared to proteolytic. Invitro anti-coagulant nature of enzyme was also considered for the advancement of an anti-coagulant agent to prevent thrombosis and additional cardio vascular illnesses.

Literature examination displays that information unfolds the purification and biochemical characterization of bacterial fibrinolytic enzymes existing, very slight effort made to see the toxicity and pharmacological properties(*31-33*). Work untaken above powerfully recommend that enzyme produced from Streptococcus Spp. can be solid applicant for the safer anti thrombotic drug and it must

considered for preclinical studies by means of animal model to judge it's in vivo thrombolytic property(*5, 34-37*).

FIGURES

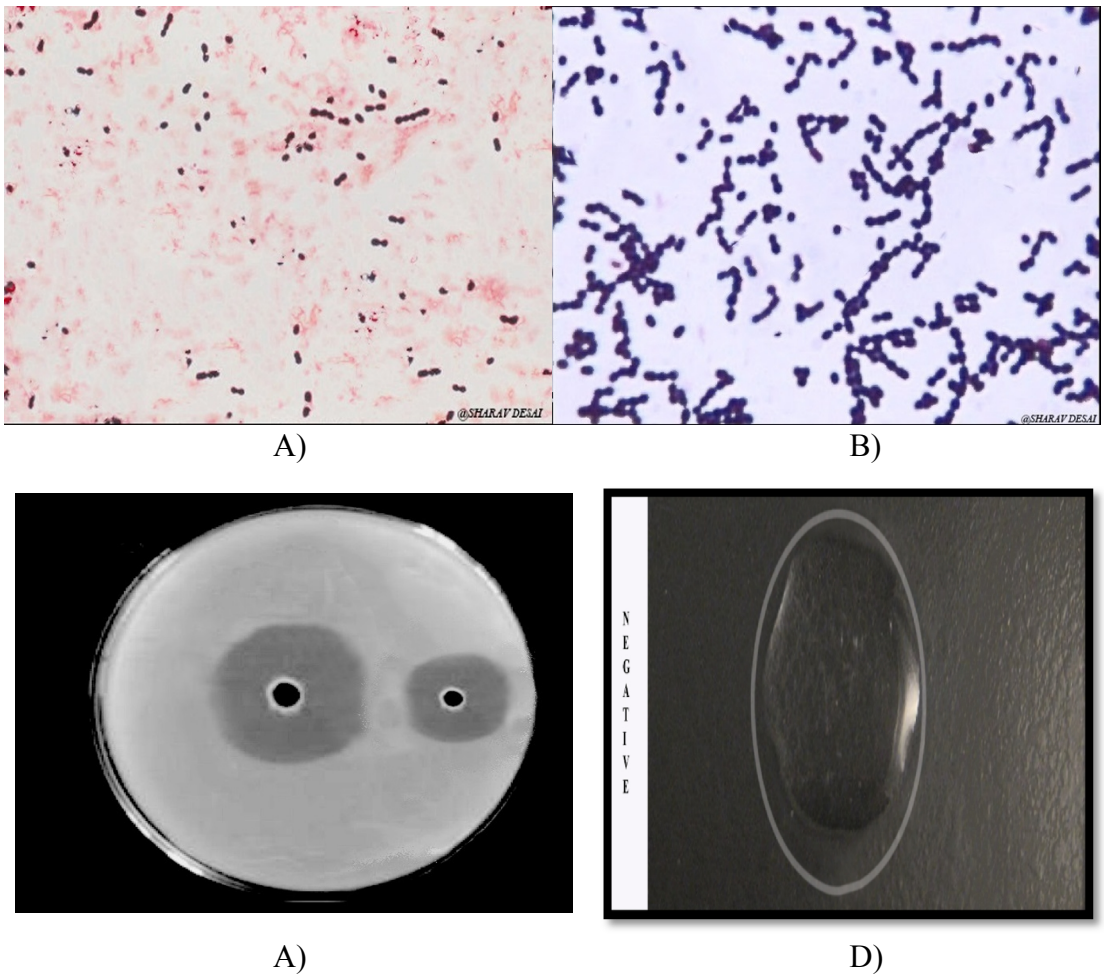


Fig 1.a) Gram Staining. B) Simple staining .C) Fibrin Plate assay D) Catalase Test

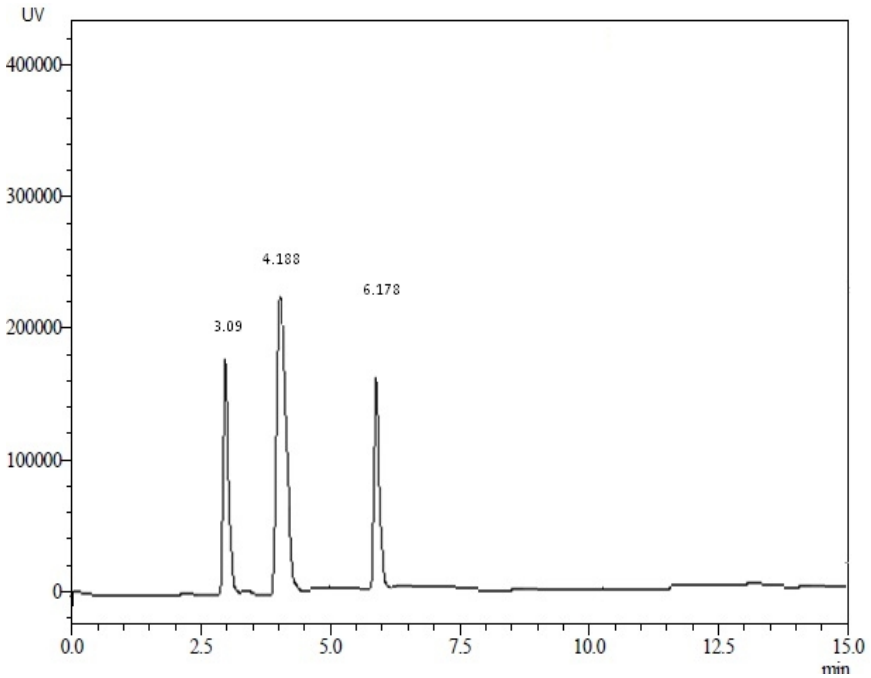


Fig 2.HPLC Chromatogram

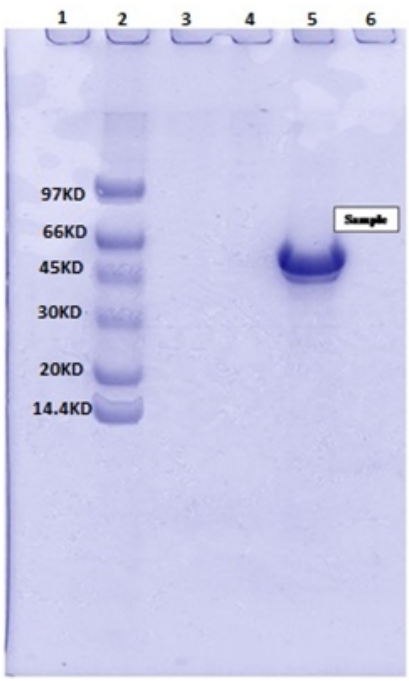


Fig 3.SDS-PAGE of purified enzyme from streptococcus spp. Lane 2: molecular mass markers; Lane 5: purified fibrinolytic enzyme.

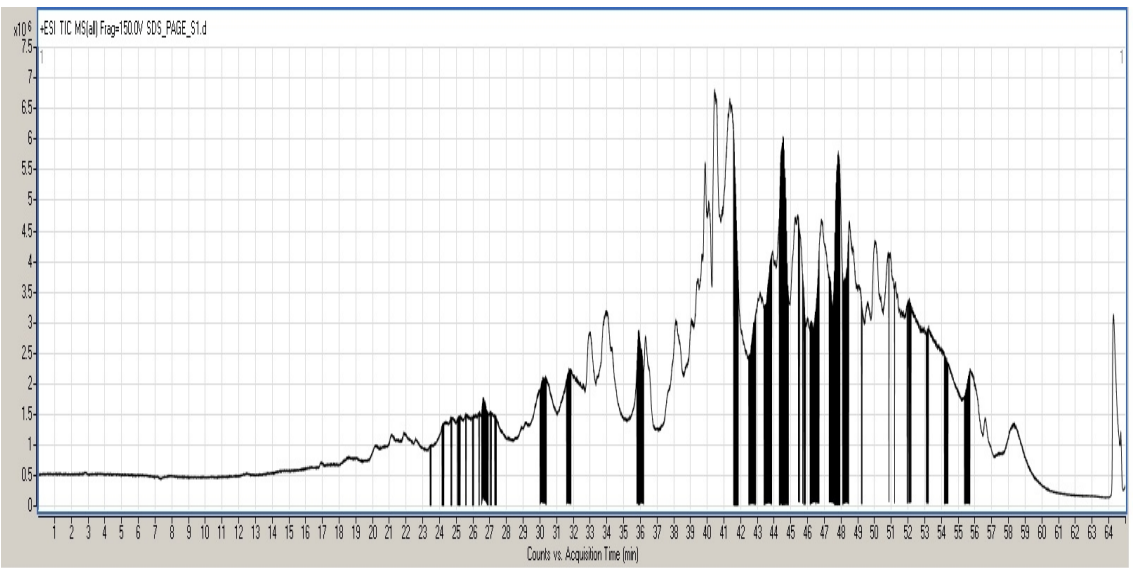


Fig 4. LCMS/MS Chromatogram

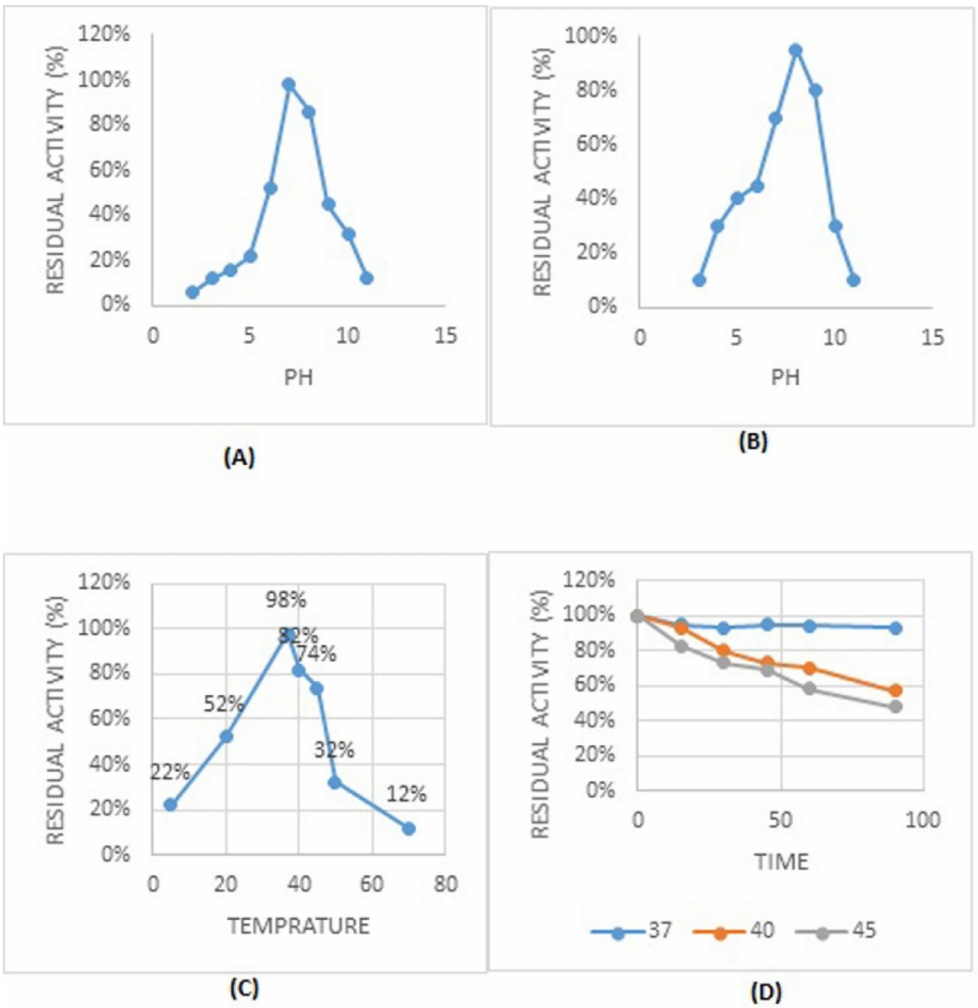


Fig 5. A) Optimum pH activity was determined by evaluating the activity at different pH

B) PH stability was determined by evaluating the residual activity with respective pH at 1^0C for 3h.

C) Optimum temperature was determined by assaying activity at different temperature for 20 minutes.

D) Thermal stability was determined by measuring the activity at 37^0C, 40^0C and 45^0C for 15 to 90 minutes.

TABLES

Table 1.HPLC parameters

Instrument	Agilent infinity LC system
Mobile phase	A) Water with 0.1% formic acid B) Acetonitrile
Gradient	0 to 50 minutes 5% solvent B 50 to 58 minutes 95% solvent B 58 to 60 minutes 5% solvent B 60 to 65 minutes 95% solvent B
Flow rate	0.4ml/ minutes
Post run column equilibration	5 minutes with 3% solvent B
Injection volume	2µl

Table 2.QTOF parameters

Instrument	Agilent 6530 Accurate-Mass Q-TOF LC/MS system
Mode	Auto
Ionization mode	Positive ion electrospray with Agilent Jet Stream technology
Mass range	100-3000m/z
Drying gas	$N_2$10 L/Min
Sheath gas	11 L/Min
Fragmentor	125V
Skimmer	65V
Gas temperature	325^0C
Ion source	Dual AJS ESI
Vcap	4000V

Table 3. Purification scheme for the fibrinolytic enzyme.

Purification step	**Total protein(mg)**	**Total fibrinolytic activity (U)**	**Specific activity (U/mg)**	**Yield (%)**	**Purification Fold**
Crude extract	610	1037	1.7	100%	1.00
NH_2SO_4	350	910	2.6	88%	1.53
Sephacryl- 200	145	696	4.8	67%	2.82
RP-HPLC	5.6	105	18.7	10%	11.00

Table 4. Amino acid sequence for the purified protein

1	MKNYL SIGVI	ALLFAL TFGT	VKPVH AIAGY	GWLPD RPPVN	NSQLV VSMAG	IVEGTD KKVF	INFFEID LTS	QHAHG GKTEQ	80
81	GLSPKS KPFA	TDNGA MPHKL	EKADL LKAIQ	KQLIAN VHSN	DGYFE VIDFA	SDATIT DRNG	KVYFA DKDGS	VTLPT QPVQE	160
161	FLLSG HVRVR	PYKEKP VQNQ	AKSVD VKYTV	QFTPLN PDDD	FRPGLK DTKL	LKTLA IGDTI	TSQEL LAQAQ	SILNKT HPGY	240
241	TIYERD SSIV	THDND IFRTI	LPMDQ EFTYR	VKDRE QAYGI	NKKSG LNEEI	NNTDL ISEKY	YILKKG ESPY	DPFDRS HLKL	320
321	FTIKY VDVNT	NELLKS EQLL	TASER NLDFR	DLYDP CDKAK	LLYNN LDAFD	IMDYT LTGKV	EDNHD KNNRI	VTVYM GKRPK	400
401	GAKGS YHLAY	DKDLY TEEER	KAYSY LRDTE	TAIPDN PKDK					440

Table 5.LCMS/MS Reference Table

Group	Subgroup	Spectra	Distinct Peptides	DS MS/MS SS	% AA coverage	Total protein spectral intensity	Protein MW (Da)	Database Accession	Protein name
1	1.1	9	8	145.34	24	6.12e+006	49941.5	32396322	Streptokinase
1	1.1	8	7	133.22	22.7	5.65e+006	49902.4	32396338	Streptokinase

Table 6. Biochemical and morphological profile for streptococcus spp.

TEST	RESULT
Gram stain	Positive
Shape	Spherical cocci
Catalase	Negative
Motility	Negative
Endospore formation	Negative
Optimum temperature	37^0C±1
Arrangement	Chain formation

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

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RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

Formulation and Evaluation of Floating *In Situ* Gel Forming System for Sustained Delivery of Tolterodine Tartrate

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ABSTRACT

This work mainly focuses on the floating in situ gel for sustained release of Tolterodine tartrate. It has short biological half-life (1.9-3.7 hrs.) and highly variable oral bioavailability (10-70 %) due to narrow absorption window. Hence, it's a proper candidate for prolonged drug delivery system. A floating drug delivery of Tolterodine tartrate was prepared to increase residence time in stomach. Different formulation was prepared by using different conc. of polymer. Box Behnken design was used to study the effect of independent variables, such as the conc. of sodium alginate (X1), conc. of gellan gum (X2) and conc. of calcium carbonate with sodium citrate (X3). The dependent variables kept are viscosity (Y1), in vitro floating lag time (Y2), drug release in 1 hrs (Y3), drug release in 6 hrs (Y4), and drug release in 12 hrs (Y5).

SUMMARY

Sustained release floating *In situ* gel of Tolterodine tartrate has been prepared successfully by using Box-Behnken designed and evaluatedto reduce problem associated with conventional dosage form.

Keywords: Box-Behnken Design, Floating in situ gel, Gellan gum, Sodium alginate, Sustained release, Tolterodine tartrate.

INTRODUCTION

Tolterodine tartrate is an anti-muscarinic drug that is used for treatment of urinary problems such as urinary incontinence or increased urinary frequency which are caused by an over-active bladder. Tolterodine tartrate has short biological half-life (1.9-3.7 hrs.) and highly variable oral bioavailability (10-70 %) due to narrow absorption window.[14,16] Hence, to overcome this limitation there is need to develop such type of doses form that specific locates in the region of stomach and deliver the drug in sustained

manner. So, floating in situ gel gives more sustained effect and retention of drug as compared to conventional dosage form.[13]

In this gelation involves formation of the double helical junction zones followed by aggregation of the double helical segments to form a three dimensional network by complexation with cations and hydrogen bonding.[7,8,11] Sodium alginate is a linear block polysaccharide copolymer made of β-D-mannuronic acid and α-L guluronic acid residues connected by 1,4 glycosidic linkages. The optimum quantities of sodium citrate that maintained the fluidity of the formulation before administration and resulted in gelation after being added to simulated gastric fluid.[5,10,12]

The objective of present research work was to formulate and evaluate floating in situ gel for providing sustained release of Tolterodine tartrate to reduce problem associated with conventional dosage form.

MATERIALS AND METHODS

Materials

Tolterodine tartrate was kindly gifted by BalPharma Ltd, Bangalore. Sodium alginate was obtained from SD fine chem. Ltd., Mumbai, Gellan gum obtained from Balaji Drugs, Baroda, Calcium carbonate and Sodium citrate was obtained from Thermo Fisher Scientific India Pvt. Ltd., Mumbai. All the other solvents and chemicals used in this study were of AR grade.

Methods

Preformulation Studies

Drug-Excipient compatibility studies

A) FT-IR Spectroscopy

FT-IR spectroscopy was employed to find out the compatibility between drug and selected polymers.

B) Differential Scanning Calorimetry study (DSC)

DSC study of drug & optimized formulation was achieved using Shimadzu DSC 60 analyzer.

Preliminary study[18]

Selection of polymer for in situ gel

Different type polymer in the same conc. were taken to select the appropriate gelling agent which showed the good characteristic. On the basis of their characteristic like gel forming capacity and viscosity they were selected.

Selection of concentration of Polymer

After selecting polymers as a gelling agent, various concentrations of polymers ranging from 0.25% to 2% w/v were taken and all the formulations containing various amounts of polymers were evaluated for viscosity and gel forming capacity, from the evaluation parameter concentration range were selected for further study.

Selection of cross linking agent

Different types of cross linking agent in the same concentration (1% w/v) were taken to select the suitable cross linking agent, which showed the good characteristic, like calcium carbonate, calcium chloride and sodium bicarbonate etc. were taken.

Selection of concentration of calcium carbonate

Concentration ranging from 0.5% to 2% w/v were taken with keeping the polymer concentration and other ingredient constant.

Selection of concentration of sodium citrate with calcium carbonate
It was selected by evaluating the formulation containing same amount of polymer and different amount of calcium carbonate. Concentration ranging from 0.5% to 2% w/v were taken with proportionally sodium citrate concentration and evaluated for gelling ability and stability for one day.

Method for preparation of floating in situ gelling solution
Polymer solutions were prepared in deionized water containing sodium citrate using stirrer. Heat the solution if needed. Different concentrations of gas forming agent & drug were added with continuous stirring. Finally both the solution were added and finally stored in amber bottles until further use.[13]

Experimental design
Box behnken design was employed. The levels and factors were calculated and experiments were performed. The dependent variables are viscosity (Y1), floating lag time (Y2), drug release in 1 hr. (Y3), drug release in 6 hrs. (Y4), and drug release in 12 hrs. (Y5) (table 1).

Evaluation parameters
pH
The pH was measured using a digital pH meter (Systronics Ltd., Ahmadabad, India).[2]

Viscosity
Viscosity was determined using a Brookfield viscometer.[4]

Determination of Drug Content
25 ml of gel equivalent to 10 mg of drug were transferred to a volumetric flask. 50 ml of 0.1 N HCl was mixed and stirred for 1 hr., sonicated for 30 min., and filter the solution. 10 ml filtrate was taken and diluted to 100 ml with 0.1 N HCl. Drug content was determined spectrophotmeterically.[4]

In vitro gelation study
The in vitro gelling capacity was graded in three categories on the basis of gelation time and time period for which the formed gel remains.[9]

(+) Gels after few minutes, dispersed rapidly
(++) Gelation immediate remains for few hours
(+++) Gelation immediate remains for an extended period.

In vitro floating study
It was determined using USP dissolution apparatus type II (paddle) having 500 ml of simulated gastric fluid (SGF, pH 1.2). Floating lag time and total duration of floating were noted.[3]

In vitro drug release
The drug release was determined using USP dissolution test apparatus with a paddle stirrer at 50 rpm by using 500 ml of 0.1 N HCl. Absorbance of samples were measured at appropriate wavelength using UV Spectrophotometer.[1]

Stability studies

Prepared formulation was stored in a high density polyethylene glass containers for one month and the stability was monitored. Periodically (initial and after one month) samples were removed and evaluated.[2]

RESULTS AND DISCUSSION

Drug excipient compatibility study

A) FT-IR Spectroscopy

FT-IR spectrum data was shown in the figure 1 to 4. The peaks were given in the table 4 can be considered as characteristic peaks of drug.Result indicates that there was no interaction between Tolterodine tartrate and polymers.

Differential Scanning Calorimetry (DSC)

DSC results is shown in Figure 5. There was no much difference in the melting point. Hence, it was concluded that, the drug was same state even in the optimized formulation without interacting with polymers.

Preliminary study

Selection of polymer for in situ gel

From the result of preliminary study (Table 5), it was decided that good gel was formed in sodium alginate and gellan gum compared to other polymers. So, from the preliminary study sodium alginate and gellan gum were selected for further work.

Optimization concentration of Polymer

From the preliminary study as shown in Table 6, different concentrations of sodium alginate and gellan gum were taken ranging from 0.25 to 2% w/v and 0.25 to 1% w/v, respectively. At very low concentration of sodium alginate 0.25 to 0.75% w/v gel was formed but they were not sufficient strength for prolonged release of drug from gel and at very high conc. of sodium alginate and gellan gum gel formed very stiff so, drug cannot diffuse from this strength of gel due to very stiff gel formation. So, concentration of sodium alginate and gellan gum were taken ranging from 1 to 1.5 % w/v and 0.25 to 0.75% w/v, respectively.

Selection of cross linking agent

Cross-linking agent also used as a gas forming agent. From the preliminary study result shown in Table 7, it was finalized that good gel was formed in calcium carbonate due to their gelling capacity, viscosity and floating lag time were in acceptable range. Finally, calcium carbonate was selected as cross linking agent for further study.

Selection of concentration of calcium carbonate

Among different concentration of calcium carbonate shown in Table 8, all the formulation having floating lag time, total floating time and viscosity were in acceptable range at high concentration. So 1 to 2% w/v concentration was selected for the further study.

Optimization of different concentration of calcium carbonate and sodium citrate

As, shown in Table 9 at very lower concentration gel was formed but during storage converted to gel and higher concentration proper gel was not formed. Hence, according to concentration of calcium carbonate, medium concentration ranging from 0.2 to 0.3% w/v was used with very good result and used for further study.

Experimental design

For all batches, all the dependent variables (viscosity, Floating lag time, Q1, Q6 and Q12) showed a variations of 315.2-922.2 cps, 49-91 sec, 39.58-55.80 %, 75.76-90.17 % and 90.99-101.74 % respectively (Table 10). The Multiple regression analysis shown in Table 11 was carried out. Values of $p<0.05$ indicate model terms are significant.

Response 1 (Y1): Viscosity

Effect of design factor on Viscosity

The experimental design results, contour plot and 3D surface plot for the viscosity of F1 to F15 formulations (Figure 7) showed strong effect of all three factors.

Polynomial equation (Table 11) of viscosity was indicated that all three factors have positive effect on the viscosity. Viscosities of the F1 to F15 formulations were found to increase with increase concentration of polymer and CaCO3. It was observed that viscosity varies from 315.22 cps to 922.2 cps for all formulations. Minimum viscosity was observed in F1 because F1 batch have minimum concentration of polymer and CaCO3. Maximum concentration of polymer in F12 formulation may be reason for maximum viscosity.

Reduced model for viscosity

For viscosity (Y1) coefficients, b23, b13, b11, b22, b33 was found to be insignificant as P values were more then 0.05 (Table 11) and hence, they were omitted from the full model to generate the reduced model. The high values of correlation coefficients for viscosity designate a good fit of design. Moreover, calculated F values (Table 12) were found to be less than the tabulated F value which suggested no significant difference among full and reduced model.

Response 2 (Y2): Floating lag time

Effect of design factor on floating lag time

The experimental design results, contour plot and 3D surface plot for the floating lag time of F1 to F15 formulations (Figure 8) showed strong effect of all three factors. Polynomial equation (Table 11) of floating lag time was indicated that two factors have positive effect and concentration of CaCO3 have negative effect on the floating lag time. Floating lag time of F1 to F15 formulations were found to increase with increase concentration of polymer and decrease with increase concentration of CaCO3. It was observed that floating lag time varies from 49 sec to 91 sec for the all formulations. It was said that floating lag time mainly depends on concentration of calcium carbonate. As the concentration increased, floating lag time decrease which is due to the formation of more gas bubbles which decreased the time to float on the surface.

Reduced model for floating lag time

For floating lag time (Y2) coefficients, b1, b2, b12, b23, b13, b11, b22, and b33 was found to be insignificant as P values were more then 0.05 (Table 11) and hence, they were omitted from the full model to generate the reduced model. The high values of correlation coefficients for viscosity designate a good fit of design. Moreover, calculated F values were found to be less than the tabulated F value (Table 12) which suggested no significant difference among full and reduced model.

Response 3 (Y3): Q1 (CPR at 1 hrs.)

Effect of design factor on Q1

The experimental design results, contour plot and 3D surface plot for the Q1 of F1 to F15 formulations (Figure 9) showed strong effect of all three factors. Polynomial equation (Table 11) of Q1 was indicated that all three factors have negative effect on the Q1. Q1 of F1 to F15 formulations were found to decrease with increase concentration of polymer. It was observed that Q1 varies from 39.58 % to 55.80 % for the all formulations. It indicates immediate drug release was due to the burst effect. Also describe that at initially, proper gelation was not occurred and drug was diffused rapidly through matrix.

Reduced model for Q1

For Q1 (Y3) coefficients, b3, b12, b23, b13, b11, b22, and b33 was found to be insignificant as P values were more then 0.05 (Table 11) and hence, they were omitted from the full model to generate the reduced model. The high values of correlation coefficients for viscosity designate a good fit of design. Moreover, calculated F values were found to be less than the tabulated F value (Table 12) which suggested no significant difference among full and reduced model.

Response 4 (Y4): Q6 (CPR at 6 hrs.)

Effect of design factor on Q6

The experimental design results, contour plot and 3D surface plot for the Q6 of F1 to F15 formulations (Figure 10) showed strong effect of all three factors. Polynomial equation (Table 11) of Q6 was indicated that all three factors have negative effect on Q6. Q6 of F1 to F15 formulations were found to decrease with increase concentration of polymer. It was observed that Q6 varies from 75.76 % to 90.17 % for the all formulations. It indicates that at Q6, gelation was occurred properly and formed matrix structure from that the drug can't diffused easily from the formulation. As concentration of sodium alginate and gellan gum and CaCO3 was increased, drug release was decreased but with only increase in the concentration of CaCO3, drug release increased due to the generation of more gas bubbles and more drug could diffuse.

Reduced model for Q6

For Q6 (Y4) coefficients, b12, b23, b13, b11, b22, and b33 was found to be insignificant as P values were more then 0.05 (Table 11) and hence, they were omitted from the full model to generate the reduced model. The high values of correlation coefficients for viscosity designate a good fit of design. Moreover, calculated F values (Table 12) were found to be less than the tabulated F value which suggested no significant difference among full and reduced model.

Response 5 (Y5): Q12 (CPR at 12 hrs.)

Effect of design factor on Q12

The experimental design results, contour plot and 3D surface plot for the Q12 of F1 to F15 formulations (Figure 11) showed strong effect of all three factors. Polynomial equation (Table 11) of Q12 was indicated that all three factors have negative effect on Q12. Q12 of F1 to F15 formulations were found to decrease with increase concentration of polymer. It was observed that Q12 varies from 90.99 %to 101.74 % for the all formulations. It indicates that at Q12, more than 95% amount of drug was dissolved and after drug release was decreased gradually due to there no more amount of drug was present in the formulations.

Reduced model for Q12

For Q12 (Y5) coefficients, b12, b23, b13, b11, b22, and b33 was found to be insignificant as P values were more then 0.05 (Table 11) and hence, they were omitted from the full model to generate the reduced model. The high values of correlation coefficients for viscosity designate a good fit of design. Moreover, calculated F values were found to be less than the tabulated F value (Table 12) which suggested no significant difference among full and reduced model.

Optimized batch analysis
Contour plots of all dependents variables were overlapped to locate the area of common interest. Optimized batch was selected on the basis of following criteria: minimum floating lag time, minimum viscosity and in range drug release. Optimized batch was selected by using DESIGN EXPERT trial version 8.0.5 (Stat-Ease. Inc. Minneapolis, USA) and overlay plot was generated (Figure 12).
To confirm the validity of design, the optimized batch was performed and % relative error was calculated which was found to be less than the 8% (Table 13) indicate goodness of fit in model.

pH
Results of pH of all the formulations were presented in Table 14.

In vitro floating study
A) In vitro floating lag time
In vitro floating study were presented in Table 14.

B) In vitro total floating time
Results of all the batches were shown in table 14.

Determination of viscosity
The viscosity of the solutions is important in view of their proposed oral administration. Results of all the formulations were shown in Table 14.

Drug content
All the formulations showed drug content in range 95.42 % to 99.58 % (Table 14). The values were acceptable as per USP standards. That indicating homogenous distribution of drug throughout gel

In vitro gelation study
Results were shown in Table 14.

In vitro drug release study
Result of in vitro drug release shown in Figure 13 of all prepared batches. A significant decrease in the rate and extent of drug release was observed with increase in gellan, sodium alginate polymer conc. and calcium carbonate. This was because of increasing the path length through which the drug molecule has to diffuse and the time needed to travel the gel matrix is increased, causing reduction in rate and extent of drug release at higher polymer conc.
The results showed that the formed gels were the capacity to retain drug for the duration of time up to 12 hrs. The initial burst effect was considerably reduced with increase in polymer conc. In all formulations, drug was gradually released up to the extent of more than 99 % in 12 hrs.

Stability study
From the overlay plot, optimized batch was found and the same was kept for stability study under controlled environment condition (40± 2 0C and 75± 5% RH). The sample were examined at interval of fifteen days and one month and analyzed.
From the Table 16, it was observed that at the end of one month viscosity of the formulation was increased from 364.7 cps to 372.6 cps which might be due to the loss of water that affect the rheological property of in situ gel. Similarly, change in pH occurred from 7.1 to 7.3, change in drug content from

99.18 % to 97.25 %, change in floating lag time from 64 sec to 66 sec. Hence, the results suggested that the formulation was not shown any significant changes in results even after one month indicate that the formulation is stable.

CONCLUSION

From all above study it can be decided that floating In situ gel for sustained release of Tolterodine tartrate having good compatibility in patient population like pediatric, geriatric and people having problem with swallowing a solid dosage form.

FIGURES

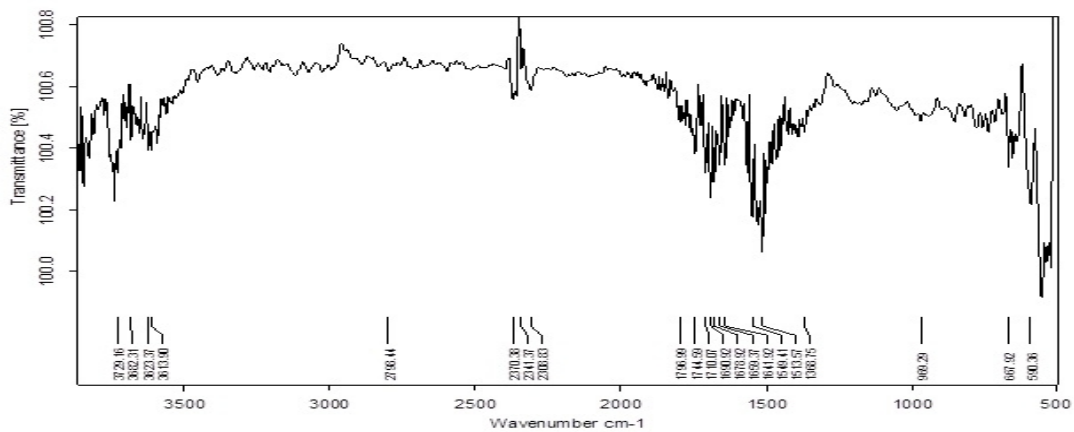


Fig. 1 FT-IR spectra of pure Tolterodine tartrate

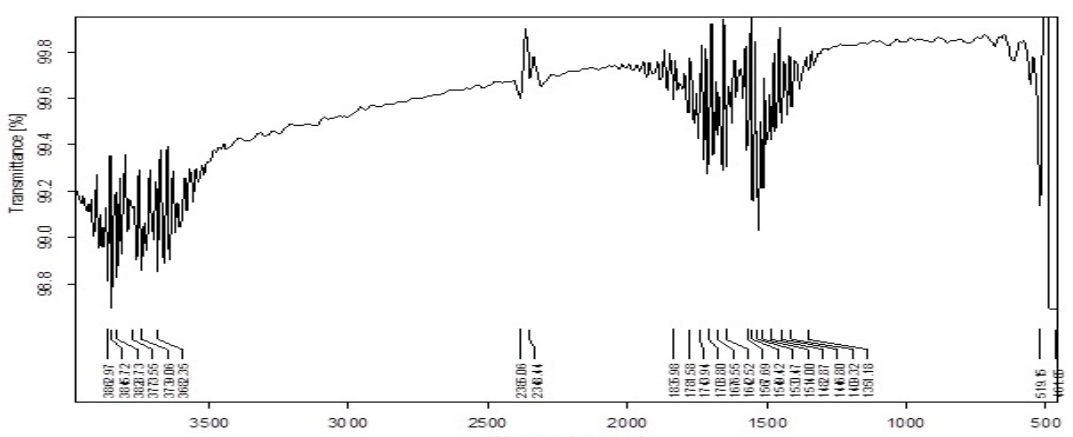


Fig. 2 FT-IR spectra of Tolterodine tartrate and Gellan gum

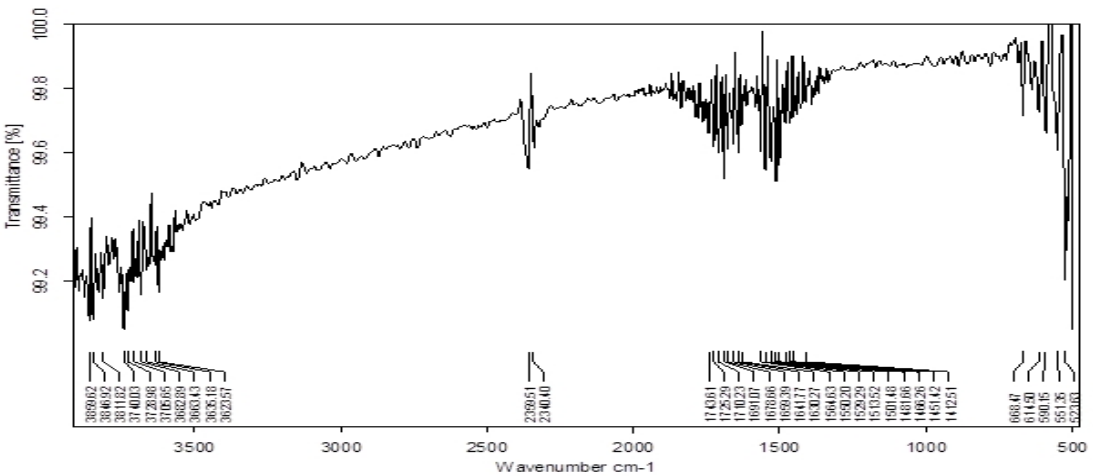


Fig. 3 FT-IR spectra of Tolterodine tartrate and sodium alginate

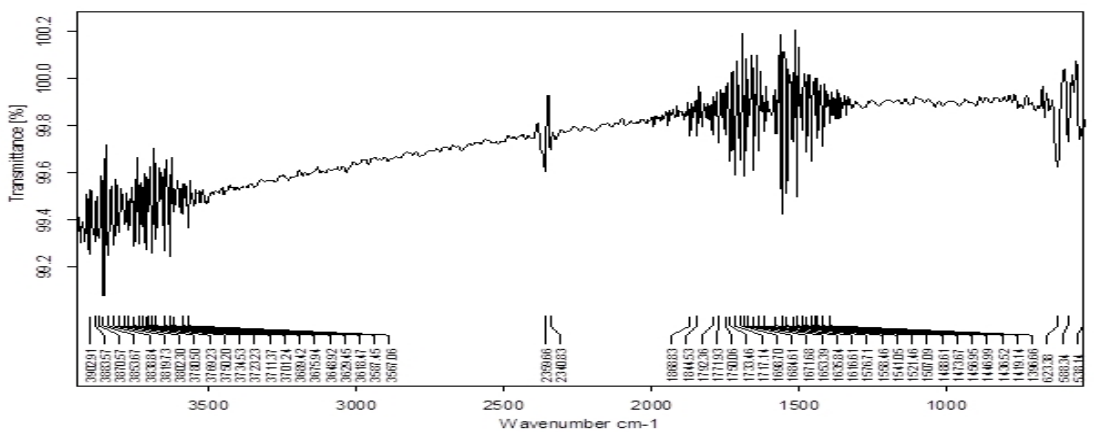


Fig. 4 FT-IR spectra of Physical mixture of pure drug, sodium alginate, $CaCO_3$ and sodium citrate

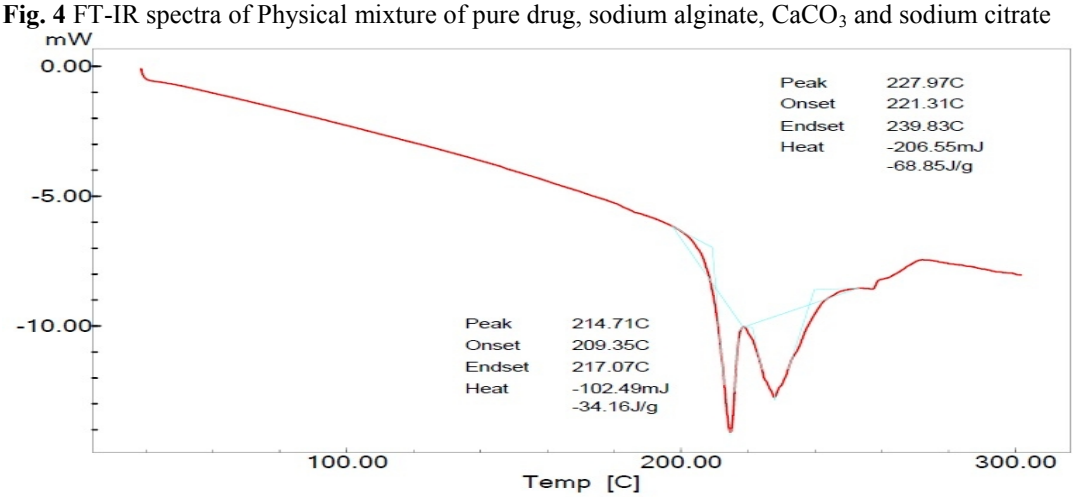


Fig. 5 DSC of pure Tolterodine tartrate

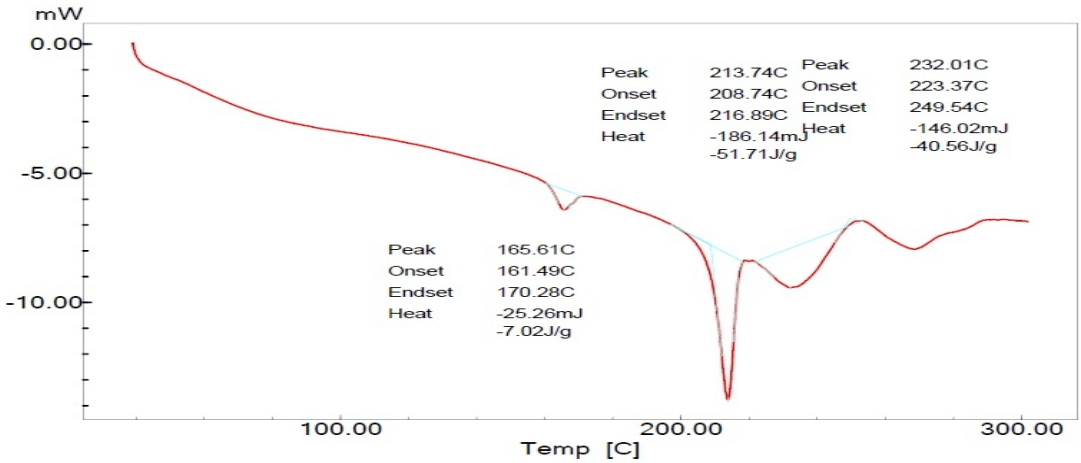
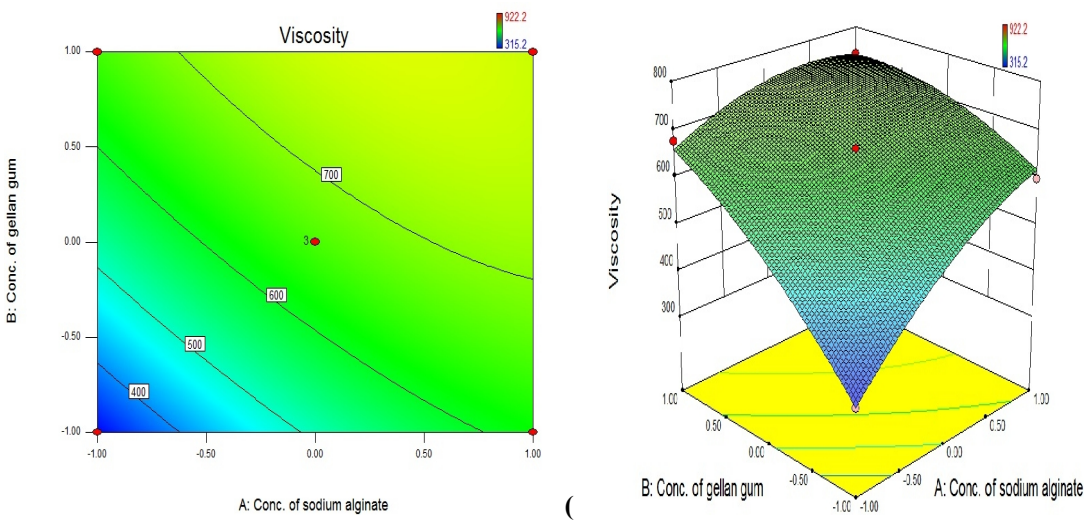
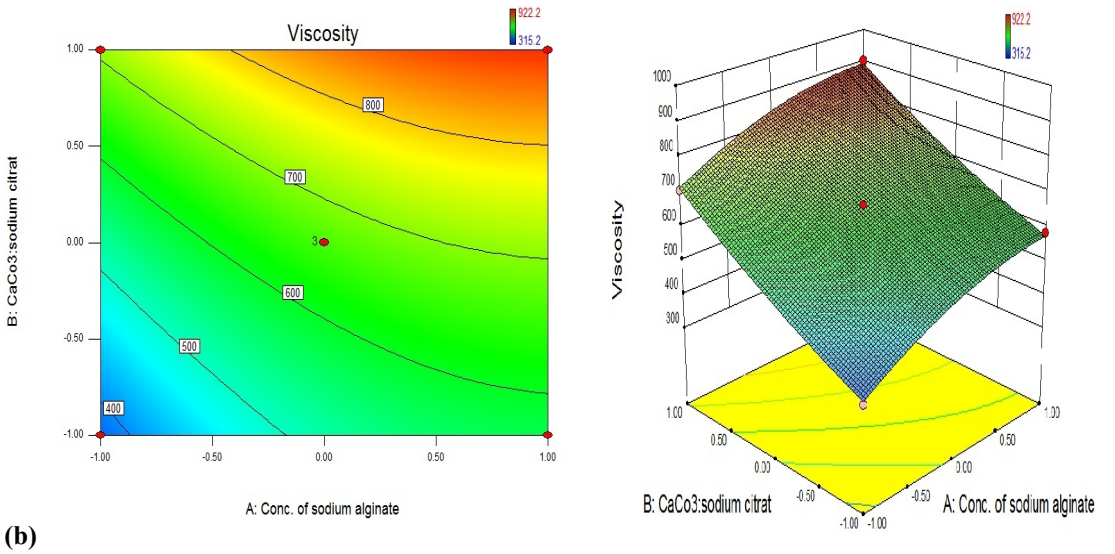


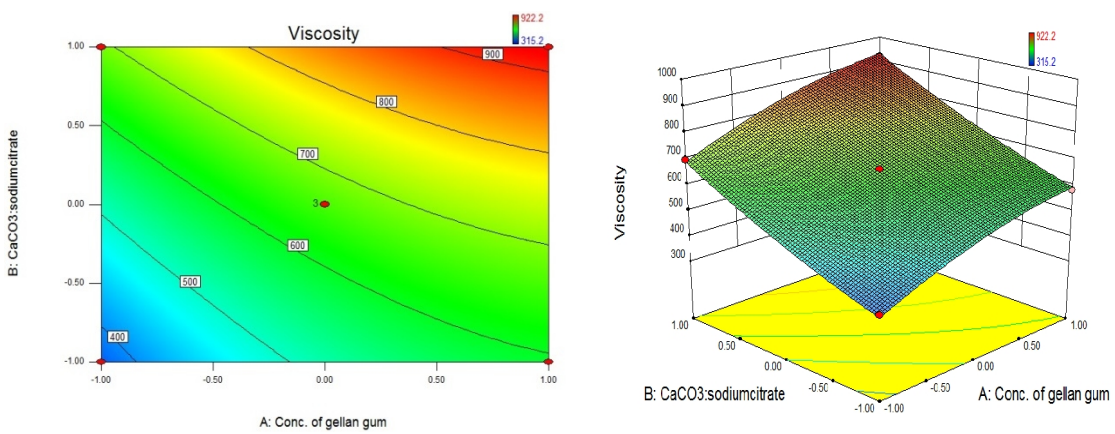
Fig. 6 DSC of optimized formulation



a) **(**

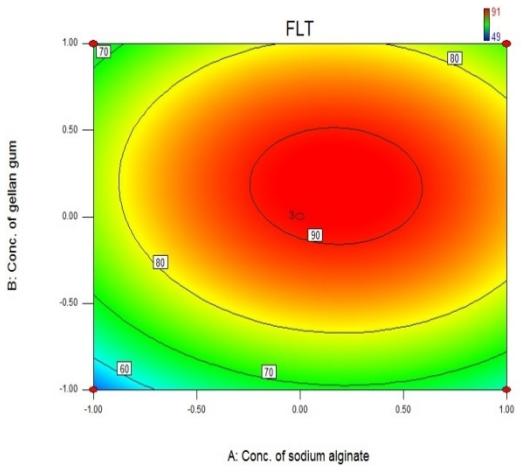


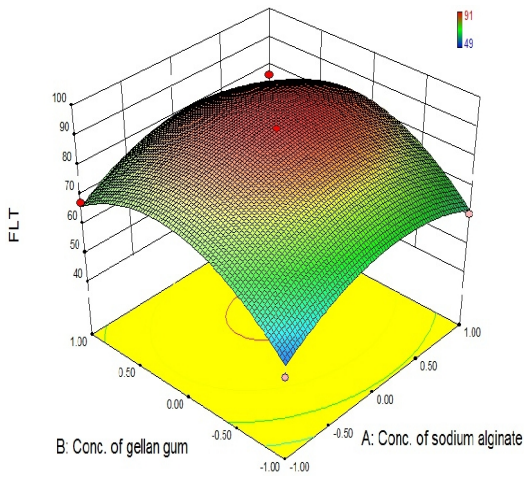
(b)



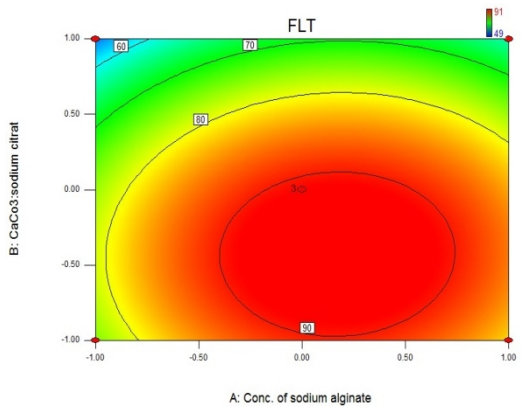
(c)

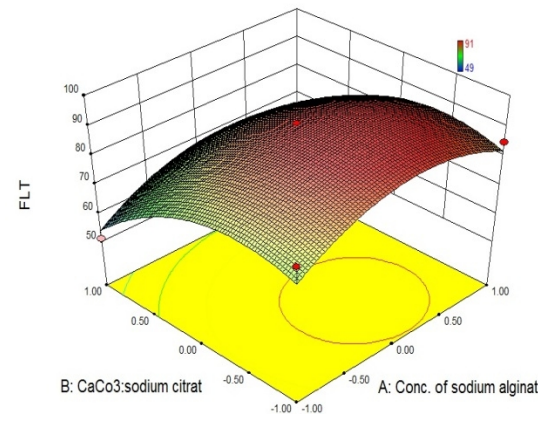
Fig. 7 Response surface and contour plots showing effect of [**(a)** Concentration of sodium alginate and gellan gum, **(b)** and Concentration of sodium alginate and $CaCO_3$ and **(c)** Concentration of gellan gum and $CaCO_3$ and sodium citrate] on viscosity.


FLT
B: Conc. of gellan gum
A: Conc. of sodium alginate
91
49
70
80
90
60
70

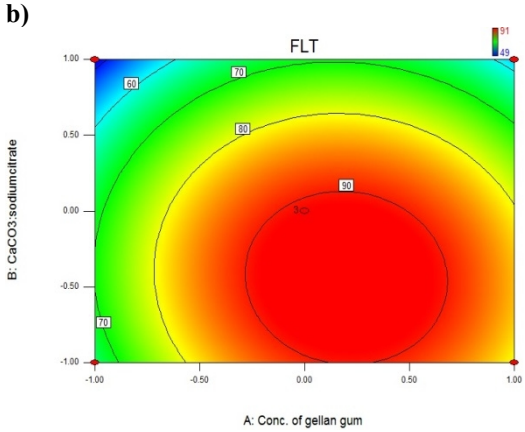

FLT
B: Conc. of gellan gum
A: Conc. of sodium alginate
91
49

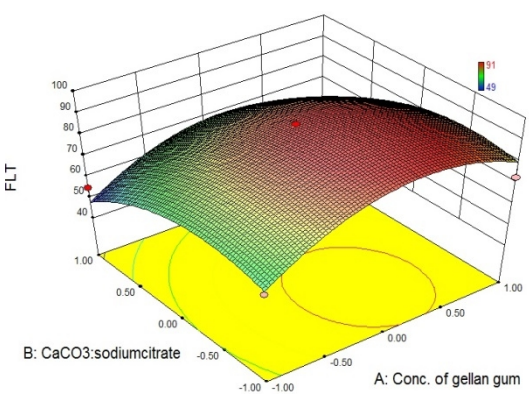
a)


FLT
B: CaCo3:sodium citrat
A: Conc. of sodium alginate
91
49
60
70
80
90


FLT
B: CaCo3:sodium citrat
A: Conc. of sodium alginat
91
49

b)


FLT
B: CaCO3:sodiumcitrate
A: Conc. of gellan gum
91
49
60
70
80
90
70


FLT
B: CaCO3:sodiumcitrate
A: Conc. of gellan gum
91
49

c)

Fig. 8 Response surface and contour plots showing effect of [**(a)** Concentration of sodium alginate and gellan gum, **(b)** and Concentration of sodium alginate and $CaCO_3$ and **(c)** Concentration of gellan gum and $CaCO_3$ and sodium citrate] on floating lag time.

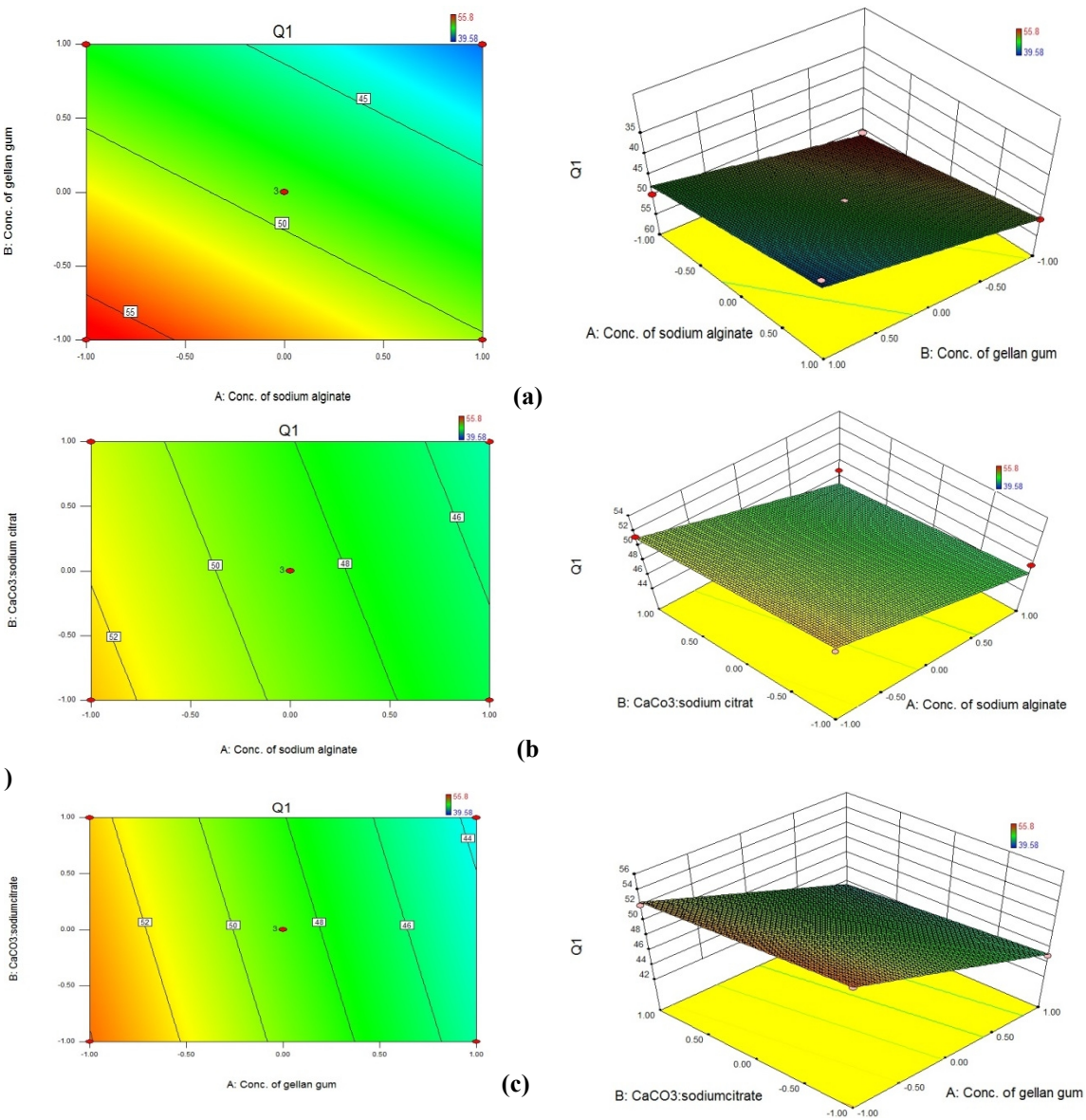


Fig. 9 Response surface and contour plots showing effect of [**(a)** Concentration of sodium alginate and gellan gum, **(b)** and Concentration of sodium alginate and $CaCO_3$ and **(c)** Concentration of gellan gum and $CaCO_3$ and sodium citrate] on Q1 (CPR at 1hr.)

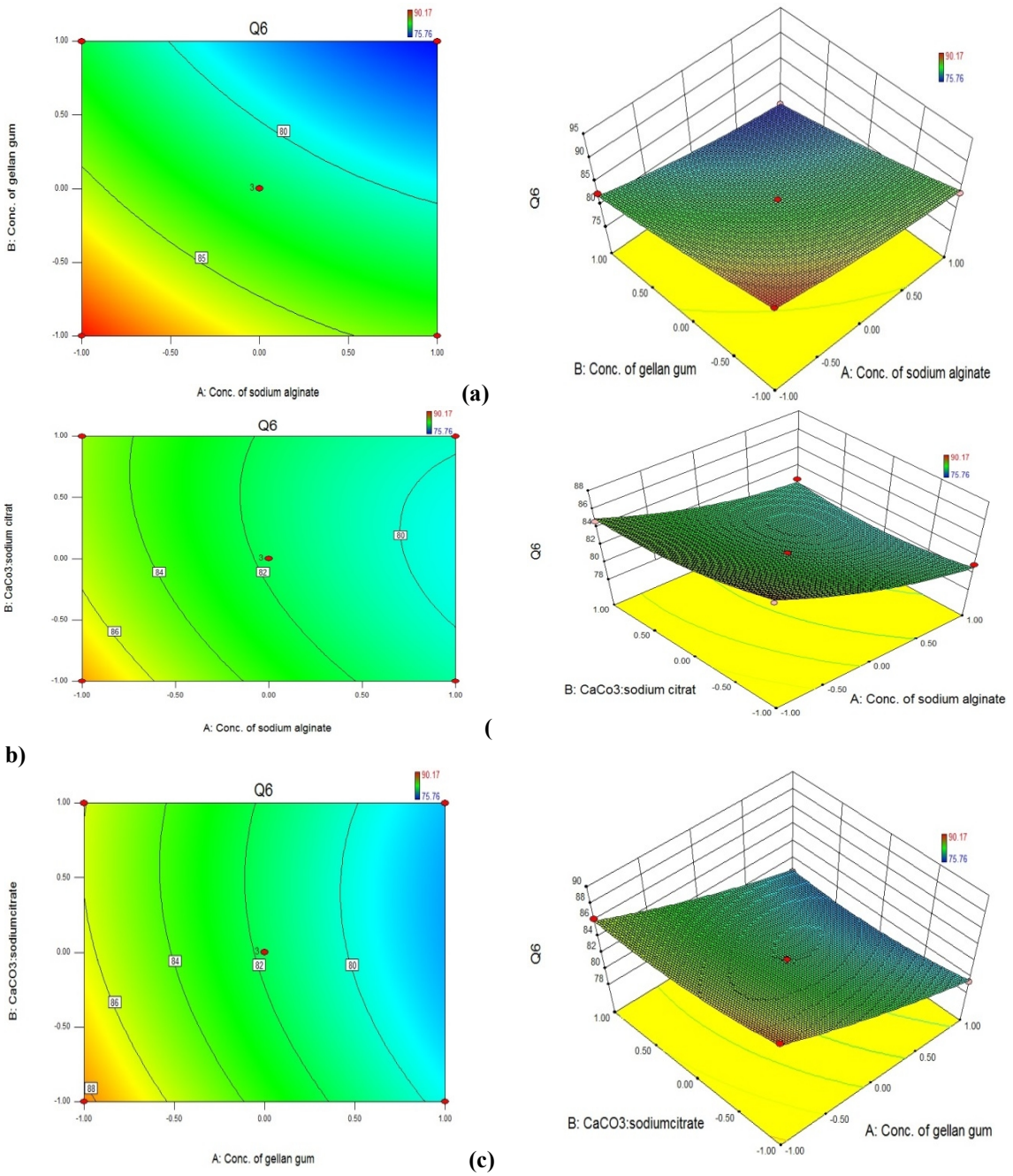


Fig. 10 Response surface and contour plots showing effect of [**(a)** Concentration of sodium alginate and gellan gum, **(b)** and Concentration of sodium alginate and $CaCO_3$ and **(c)** Concentration of gellan gum and $CaCO_3$ and sodium citrate] on Q6 (CPR at 6 hrs)

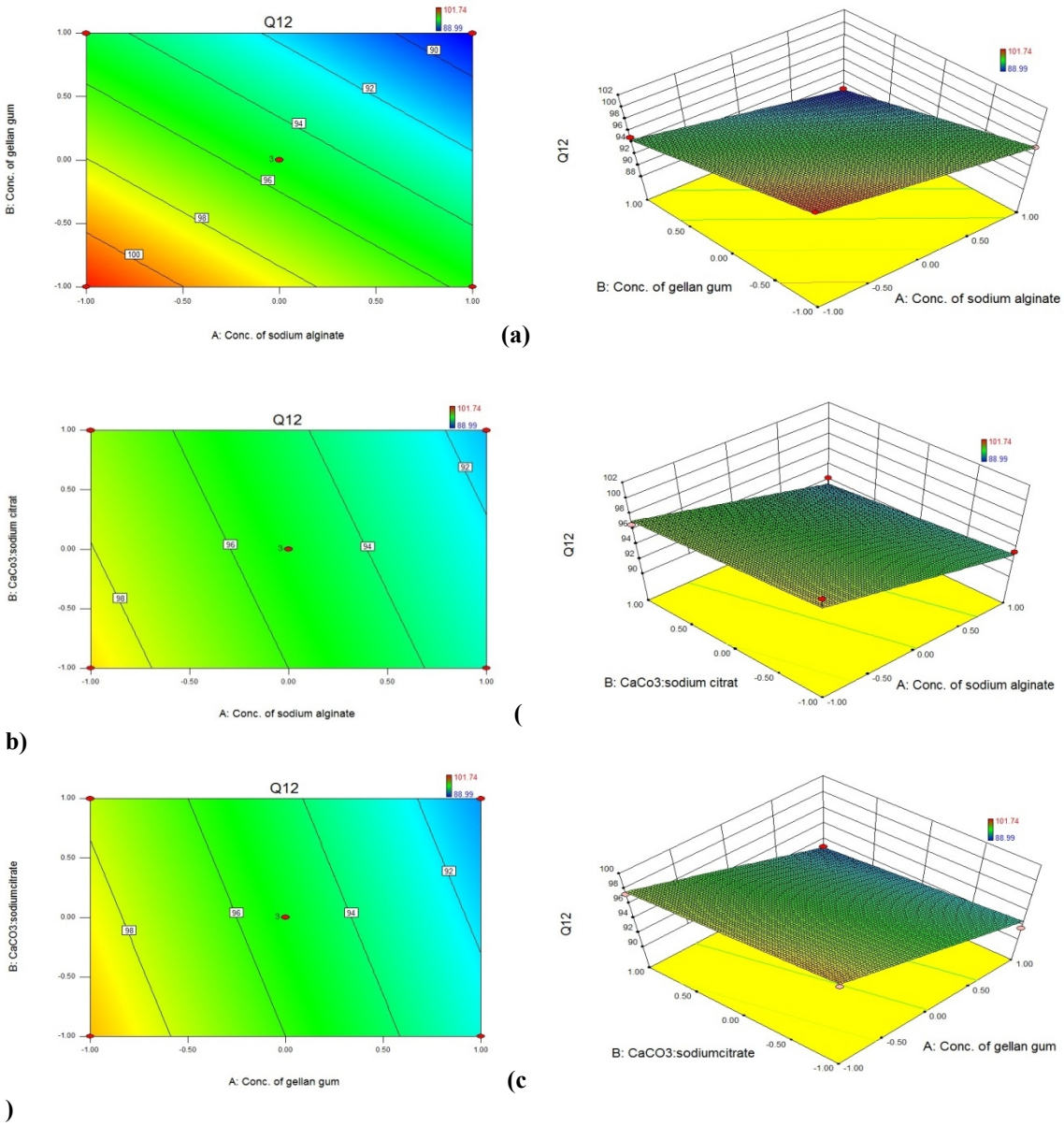


Fig.11 Response surface and contour plots showing effect of [**(a)** Concentration of sodium alginate and gellan gum, **(b)** and Concentration of sodium alginate and $CaCO_3$ and **(c)** Concentration of gellan gum and $CaCO_3$ and sodium citrate] on Q12 (CPR at 12 hrs.)

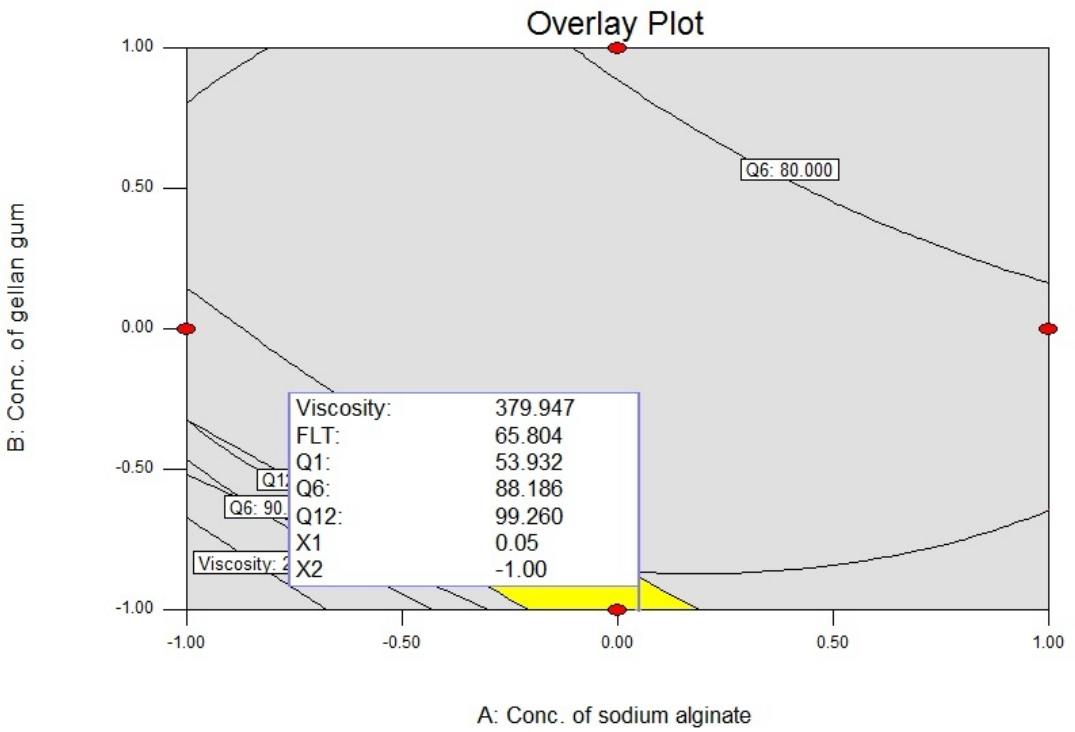


Fig. 12 Overlay plot of optimized batch

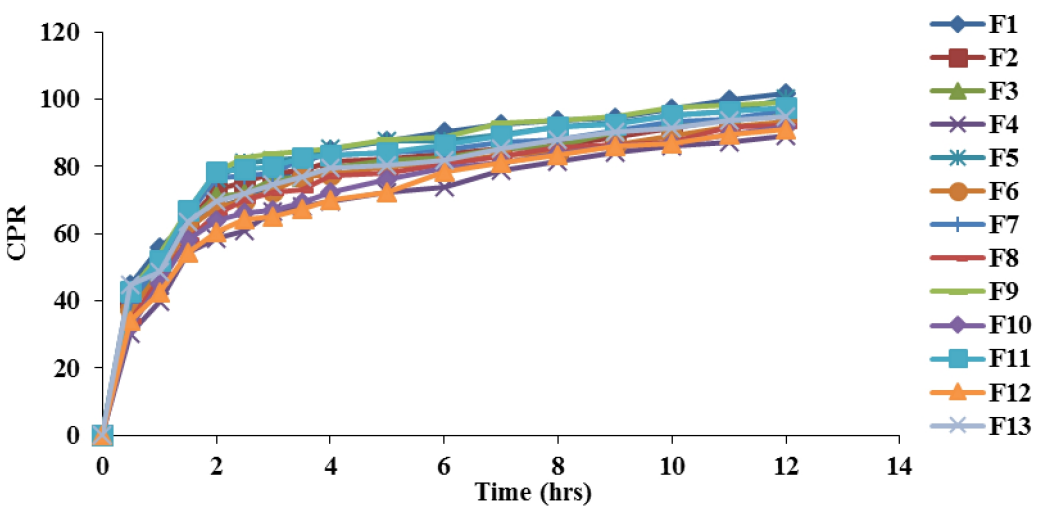


Fig. 13 *Invitro* drug release study of all experimental batches

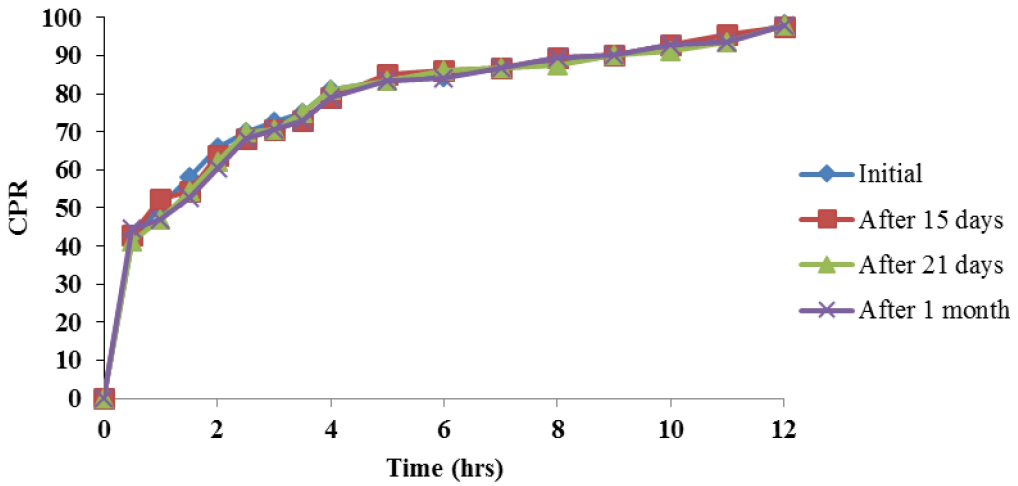


Fig 19.*In vitro* drug release study of final formulation after and before stability

TABLES

Table 1 Factor and levels for box behnken design

Independent variables	**Actual values (%)**			**Coded values**		
	Low	**Medium**	**High**	**Low**	**Medium**	**High**
Conc. of Sodium alginate (X_1)	1	1.25	1.5	-1	0	+1
Conc. of Gellan gum (X_2)	0.25	0.50	0.75	-1	0	+1
Concentration of $CaCO_3$: Sodium citrate (X_3)	1:0.2	1.5:0.25	2:0.3	-1	0	+1

Dependent variables: - Viscosity (Y_1),
- Floating lag time (Y_2),
- Drug release in 1 hr. (Y_3),
- Drug release in 6 hrs. (Y_4),
- Drug release in 12 hrs. (Y_5).

Table 2. Box-Behnken design

Batch	**Coded value**			**Actual value (%)**		
	X1	**X2**	**X3**	**X1**	**X2**	**X3**
1	-1	-1	0	1	0.25	1.5:0.25
2	+1	-1	0	1.5	0.25	1.5:0.25
3	-1	+1	0	1	0.75	1.5:0.25
4	+1	+1	0	1.5	0.75	1.5:0.25

5	-1	0	-1	1	0.50	1:0.2
6	+1	0	-1	1.5	0.50	1:0.2
7	-1	0	+1	1	0.50	2:0.3
8	+1	0	+1	1.5	0.50	2:0.3
9	0	-1	-1	1.25	0.25	1:0.2
10	0	+1	-1	1.25	0.75	1:0.2
11	0	-1	+1	1.25	0.25	2:0.3
12	0	+1	+1	1.25	0.75	2:0.3
13	0	0	0	1.25	0.50	1.5:0.25

Table 3. Formulation of experimental design batches

Ingredients	Tolterodine tartrate (mg/5ml)	Sodium alginate (%)	Gellan gum (%)	CaCO3 (%)	Sodium citrate (%)
F1	2	1	0.25	1.5	0.25
F2	2	1.5	0.25	1.5	0.25
F3	2	1	0.75	1.5	0.25
F4	2	1.5	0.75	1.5	0.25
F5	2	1	0.50	1	0.2
F6	2	1.5	0.50	1	0.2
F7	2	1	0.50	2	0.3
F8	2	1.5	0.50	2	0.3
F9	2	1.25	0.25	1	0.2
F10	2	1.25	0.75	1	0.2
F11	2	1.25	0.25	2	0.3
F12	2	1.25	0.75	2	0.3
F13	2	1.25	0.50	1.5	0.25

Table 4. Summary of FT-IR spectra of Tolterodine tartrate along with excipients

Functional group	Characteristic peaks (cm^{-1})	Observed peaks (cm^{-1})
OH	3592.70	3682
CH3	2768	2385
C-N	1368	1351
C-C	510	461
Benzene	969	519

Table 5. Result of preliminary study for selection of polymer

Polymer*	Gelling
Sodium alginate	**Gel formed**
Gellan gum	**Gel formed**
Pectin	Gel not formed
Carrageenan	Gel not formed
Xanthan gum	Gel not formed
Guar gum	Gel not formed

*Polymer concentration: 1% w/v, 1% w/v $CaCO_3$, 0.2% w/v sodium citrate

Table 6. Optimization of Polymer concentration

Name of Polymer*	Conc. of polymer (%)	Viscosity (cps)	Gelation time (sec)	Gelation Capacity#
Sodium alginate	0.25	15	29	+

Sodium alginate	0.50	26	23	++
Sodium alginate	0.75	47	13	++
Sodium alginate	**1.00**	**103**	**18**	**+++**
Sodium alginate	**1.25**	**122**	**11**	**+++**
Sodium alginate	**1.50**	**131**	**9**	**+++**
Sodium alginate	**1.75**	**195**	**8**	**+++**
Sodium alginate	2.00	289	13	+++
Gellan gum	**0.25**	**124**	**19**	**++**
Gellan gum	**0.50**	**360**	**13**	**+++**
Gellan gum	**0.75**	**538**	**6**	**+++**
Gellan gum	1.00	1138	15	+++

*All the formulations contains 0.2% w/v of sodium citrate, 1% w/v of $CaCO_3$
[#]+ = poor, ++ = good, +++ = very good.

Table 7. Selection of cross linking agent

Polymers*	Cross-linking agent (1%w/v)	Viscosity (cps)	Floating lag time (sec)	Gelation Capacity
Sodium alginate	**$CaCO_3$**	**108**	**93**	**Gel formed**
Sodium alginate	$NaHCO_3$	122	-	Gel not formed
Sodium alginate	$CaCl_2$	878	-	Gel not formed
Gellan gum	**$CaCO_3$**	**122**	**96**	**Gel formed**
Gellan gum	$NaHCO_3$	129	-	Gel not formed
Gellan gum	$CaCl_2$	472	-	Gel not formed

*All the formulations contains 1% w/v Sodium alginate and 0.25% w/v gellan gum 0.2% w/v of sodium citrate

Table 8. Selection of calcium carbonate concentration

Polymer*	$CaCO_3$ (% w/v)	Viscosity (cps)	Floating lag time (sec)	Total floating time (hrs)
Sodium Alginate	0.5	98	107	8
Sodium Alginate	**1**	**103**	**90**	**>12**
Sodium Alginate	**1.5**	**109**	**55**	**>12**
Sodium Alginate	**2**	**115**	**43**	**>12**
Gellan gum	0.5	118	115	10
Gellan gum	**1**	**121**	**105**	**>12**
Gellan gum	**1.5**	**124**	**63**	**>12**
Gellan gum	**2**	**129**	**51**	**>12**

*All the formulations contains 1% w/v Sodium alginate and 0.25% w/v gellan gum 0.2% w/v of sodium citrate

Table 9. Optimization of different concentration of calcium carbonate and sodium citrate*

Conc. of $CaCO_3$ (% w/v)	Sodium citrate (% w/v)	Gelation Capacity	Formulation after 1 day
1	0.1	+	Gel formed
1	0.15	++	Solution
1	**0.2**	**+++**	**Solution**
1	0.25	+++	Solution

1	0.3	++	Solution
1.5	0.1	+	Gel formed
1.5	0.15	++	Solution
1.5	0.2	++	Solution
1.5	**0.25**	**+++**	**Solution**
1.5	0.3	++	Solution
2	0.1	+	Gel formed
2	0.15	+	Gel formed
2	0.2	++	Solution
2	0.25	++	Solution
2	**0.3**	**+++**	**Solution**

*All the formulations contains 1% w/v Sodium alginate and 0.25% w/v gellan

Table 10. Response of experimental design formulation

Batch	Viscosity (cps)*	Floating lag time (sec)*	Q1 (%)*	Q6 (%)*	Q12 (%)*
F1	315.2±4.89	49±1.56	55.8±.2.13	90.17±1.72	101.74±2.37
F2	598.1±12.63	61±4.34	50.41±1.09	83.69±1.47	95.65±1.93
F3	677.4±856	68±4.15	49.67±1.17	82.67±3.12	95.01±1.63
F4	739.9±7.49	77±6.98	39.58±1.10	75.76±3.09	88.99±1.84
F5	367.5±4.67	81±1.45	52.19±2.40	87.57±2.64	100.03±0.70
F6	581.3±10.39	85±1.78	47.69±0.78	81.06±2.06	93.44±2.61
F7	702.8±8.65	51±2.44	51.28±3.14	84.72±1.50	96.75±2.54
F8	903.5±11.58	57±9.189	46.76±3.98	80.55±0.88	92.21±1.39
F9	382.7±10.72	64±7.43	54±1.56	88.57±1.23	99.07±2.56
F10	582.9±5.37	73±2.27	44.96±2.98	79.41±0.56	91.55±3.21
F11	698.7±6.74	553.89	52.03±3.01	86.26±1.27	97.35±2.83
F12	922.2±8.35	59±5.09	42.48±2.57	78.33±1.05	90.99±2.78
F13	661.6±9.66	91±7.57	48.66±1.83	81.76±2.38	94.81±2.39

*All results are shown in mean ± S.D. (n=3)

Table 11. Polynomial coefficient of all five responses

Coefficients	Viscosity (Y1)		Floating lag time (Y_2)		Q1 (Y_3)		Q6 (Y_4)		Q12 (Y_5)	
	FM	**RM**	**FM**	**RM**	**FM**	**RM**	**FM**	**RM**	**FM**	**RM**
b_0	97	625	91	67	48.6	48.8	81.7	83.1	94.8	95.1
b_1	18.75	94.9	3.8	-	-3.0	-3.0	-3.0	-3.0	-2.90	-2.9
b_2	14	115	6	-	-4.4	-4.4	-4.0	-4.0	-3.4	-3.4
b_3	-22.2	164	-10	-10	-0.7	-	-0.8	-0.8	-0.8	-0.8
b_{12}	-4.75	-55	-0.7	-	-1.1	-	-0.1	-	0.01	-
b_{23}	-17.7	-	0.5	-	-0.5	-	0.5	-	0.51	-
b_{13}	-8.25	-	-1.2	-	-0.1	-	0.3	-	0.29	-
b_{11}	-11.3	-	0	-	0	-	0	-	0	-
b_{22}	-17.8	-	-10.7	-	0.65	-	0.82	-	0.70	-
b_{33}	-10.8	-	-16	-	-0.45	-	0.49	-	0.16	-

Table 12. Calculation for testing the model in portions*

Viscosity(cps)					
Regression	**DF**	**SS**	**MS**	**R^2**	**DF(6,3)**
FM	10	418649.8	41864.98	0.996659	$F_{cal=2.67}$
RM	4	407333.9	101833.5	0.96972	$F_{tab=8.94}$
Error					
FM	3	1403.468	467.8225		$F_{cal}<F_{tab}$
RM	8	12719.35	1589.919		
FLT(sec)					
Regression	**DF**	**SS**	**MS**	**R^2**	**DF(9,3)**
FM	10	1937.25	193.725	0.894391	$F_{cal=2.82}$
RM	1	820.125	820.125	0.378636	$F_{tab=8.81}$
Error					
FM	3	228.75	76.25		$F_{cal}<F_{tab}$
RM	11	1345.875	122.3523		
Q1 (%)					
Regression	**DF**	**SS**	**MS**	**R^2**	**DF(8,3)**
FM	10	246.0792	24.60792	0.974651	$F_{cal=2.93}$
RM	2	233.0066	116.5033	0.974651	$F_{tab=8.85}$
Error					
FM	3	6.400075	2.133358		$F_{cal}<F_{tab}$
RM	10	19.47276	1.947276		
Q6 (%)					
Regression	**DF**	**SS**	**MS**	**R^2**	**DF(7,3)**
FM	10	214.4677	21.44677	0.994147	$F_{cal=2.78}$
RM	3	210.3097	70.10324	0.974873	$F_{tab=8.89}$
Error					
FM	3	1.262575	0.420858		$F_{cal}<F_{tab}$
RM	9	5.420552	0.602284		
Q12 (%)					
Regression	**DF**	**SS**	**MS**	**R^2**	**DF(7,3)**
FM	10	169.3698	16.93698	0.995465	$F_{cal=2.46}$ $F_{tab=8.89}$
RM	3	0.771675	0.257225	0.977021	$F_{cal}<F_{tab}$
Error					
FM	3	166.2318	55.41061		
RM	9	3.909667	0.434407		

*DF= Degree of freedom, SS= Sum of squares, MS= Mean of squares, R^2= Regression coefficient, FM= full model, RM= reduced model

Table 13. Results of check point batch

Response	Predicted value	Experimental value*	% RE
Viscosity (cps)	379.94	364.7±4.38	2.73
FLT (sec)	65.8	64±1.73	4.11
Q1 (%)	53.93	50.41±2.40	6.52
Q6 (%)	88.18	84.33±4.48	4.36
Q12 (%)	99.26	98.16±1.96	1.09

***All results were shown in mean ± S.D. (n=3)**

Table 14. Result of experimental design batches

Batch	pH*	Floating lag time (sec)*	Total floating time (hrs)	Viscosity (cps)*	Drug content (%)*	*In vitro* gelation study
F1	6.9±0.04	49±1.56	>12	315.2±4.89	97.55±1.08	++
F2	6.7±0.01	61±4.34	>12	598.1±12.63	98.75±1.76	+++
F3	7.2±0.04	68±4.15	>12	677.4±856	96.82±0.89	+++
F4	7.4±0.07	77±6.98	>12	739.9±7.49	98.29±2.67	+++
F5	7.8±0.03	81±1.45	>12	367.5±4.67	97.32±1.23	++
F6	7.1±0.05	85±1.78	>12	581.3±10.39	95.83±0.21	+++
F7	7.3±0.01	51±2.44	>12	702.8±8.65	96.04±0.57	+++
F8	7.2±0.06	57±9.189	>12	903.5±11.58	97.67±1.53	+++
F9	7.3±0.01	64±7.43	>12	382.7±10.72	99.83±1.85	+++
F10	7.4±0.03	73±2.27	>12	582.9±5.37	96.22±2.96	+++
F11	7.7±0.07	553.89	>12	698.7±6.74	99.58±1.38	+++
F12	7.5±0.05	59±5.09	>12	922.2±8.35	99.10±0.91	+++
F13	7.2±0.07	91±7.57	>12	661.6±9.66	95.42±0.67	+++

Table 16. Summary of stability studies

Evaluation parameter*	Time period for sampling			
	Initial	**15 days**	**21 days**	**30 days**
Viscosity (cps)	364.7±5.76	370.2±9.21	371.6±8.49	372.6±7.52
FLT (sec)	64±4.07	66±2.75	65±2.36	65±1.84
TFT (h)	>12	>12	>12	>12
Drug content (%)	99.18± 1.63	97.25± 0.81	97.68±0.79	97.7±1.05
pH	7.3±0.50	7.1±0.71	7.1±0.86	7.2±0.54
Gelling capacity	+++	+++	+++	+++

*All results are shown in mean ± S.D. (n=3)

ACKNOWLEDGEMENT

We would like to thank the company Bal Pharma. Ltd, Bangalore for giving us the free gift samples of Tolterodine tartrate. We would like to thanks following the institutes Shree M. N. Virani Science College, Rajkot for FTIR study and Saurashtra University Department of Pharmacy for DSC study. These authors (Dr. Biswajit Basu & Mr. Anil Kathiriya) declare that they have no conflict of interest. This article does not contain any studies with human and animal subjects performed by any of the authors.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

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RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

Preparation and Optimization of a Nanosuspension of the Poorly Soluble Drug Cilnidipine

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ABSTRACT

The present study aims at producing nanosuspension of Cilnidipine, a poorly water soluble antihypertensive drug using solvent-antisolvent method with a view to enhance its dissolution and saturated solubility. Drug solution of Cilnidipine in acetone was added to solution of stabilizer under continuous homogenization. Various process and formulation parameters were screened like homogenization speed, homogenization time, type of stabilizer, solvent to antisolvent ratio, drug concentration and stabilizer concentration. With a view to enhance physical stability of this colloidal system, nanosuspensions were freeze dried using D- mannitol. Seven different stabilizers were tried. Among them Poloxamer 407, Poloxamer 188, PVA and Tween 80 yielded nanosuspension in range of 90 to 350 nm. Freeze dried nanosuspensions were filled in capsules to make deliverable dosage form and almost 100% drug dissolved in 5 mins. The outcome of this study reveals the immense potential of nanosuspensions for delivery of Cilnidipine by improving its saturation solubility and dissolution rate.

Keywords: Nanosuspension, Cilnidipine, Dissolution

INTRODUCTION

Cilnidipine is antihypertensive drug. Cilnidipine having poor aqueous solubility. So its dissolution and solubility in aqueous media is lower. So those types of drug require enhance solubility & dissolution.
The aim of this study was, to employ the nanosuspension technique to produce cilnidipine nanoparticles for oral administration, thereby avoiding the use of harmful additives and enabling to increases the saturation solubility, dissolution and oral absorption of cilnidipine. The optimized nanosuspension formulation was evaluated for in vitro dissolution profile in comparison to the pure drug and marketed formulation (Cilacar).

MATERIALS AND METHODS

1.1. Materials

Cilnidipine was a gift from J.B. Chemicals & pharmaceutical limited (India). Poloxamer 407, 188 was purchased from BASF. PVP K29/32 was gift from signet chemicals.

1.2. Preparation of Nanosuspensions

Cilnidipine nanosuspensions were prepared by nanoprecipitation method using high Speed homogenizer. Accurately weighed Cilnidipine was dissolved in methanol (solvent) to form organic solution of drug. Specified quantities of stabilizers were dissolved in 100 ml distilled water to obtain anti-solvent system. Then, organic solution of drug was added drop by drop with the use of syringe into antisolvent system/ aqueous phase with continuous homogenization condition using high speed homogenizer (Omni PDH, USA). Various process and formulation parameters were optimized. (2,3)

2.3. Optimization of process and formulation parameters:

2.3.1 Screening of stirring speed

Accurately weighed 100 mg of drug was dissolved in 10 ml of Methanol. From the literature search, Poloxamer 188 was selected for the optimization of stirring speed. Accurately weighed 500 mg of Poloxamer 188 was dissolved in 100 ml distilled water to form antisolvent system. Then organic solution of drug was added drop by drop with the use of syringe into antisolvent system with continuous homogenization at different homogenization speeds and for different homogenization time. Different batches were prepared as shown in table 1 and screened on the basis of their appearance and stability of liquid state Nanosuspension. (4,5,6)

At 5000 rpm, nanosuspension was not formed after stirring for 10, 20 or 30 mins. Homogenization at 10,000 rpm and for 10, 20 and 30 mins produced nanosuspensions initially but they possessed very low liquid state stability i.e. 2 days only. At 15,000 rpm, after stirring for 10 mins, nanosuspension got formed with bluish tinge but it was stable for only 2 days. At 15,000 rpm after stirring for 20 mins, there was distinct bluish tinge but nanosuspension was stable for 4 days only. At 15,000 rpm after stirring for 30 mins, similar results were obtained in terms of liquid state stability. At 20,000 rpm, stirring for 10, 20 and 30 mins produced nanosuspensions with stability of more than 15 days.

The dispersion effectiveness was heavily dependent on shear applied and the time the particles spent in the shear zone. A processing time of a few minutes was sufficient to produce the desired nanosuspension. Long processing times bring only insignificant improvements; the energy expended serves merely to increase the temperature of the medium. From the above results, it was concluded that homogenization at 20,000 rpm is vital for preparation of stable nanosuspension.

2.3.2 Screening of stirring time and stabilizers

Accurately weighed 100 mg of drug was dissolved in 10 ml of Methanol to form organic solution of drug. Accurately weighed stabilizer was dissolved in 100 ml distilled water to form antisolvent system. Then solution of drug was added drop by drop with the use of syringe into antisolvent system with continuous homogenization at 20,000 rpm and for different stirring time. Batches were prepared as per protocol shown in table 2 & 3 respectively and screened on the basis of their appearance and stability of liquid state nanosuspension. (7,8)

From the above results it was concluded that homogenization at 20000 rpm and for 20 mins with stabilizer concentration (0.5% w/v) would produce nanosuspension of good quality in terms of particle size and liquid state stability. To check whether increasing the stabilizer concentration (1% w/v) would yield nanosuspensions after homogenization at 20,000 rpm and for 10 mins, further experiments were carried out. The results are shown in Table 3. (9,10)

From the above results it was concluded that, homogenization at 20,000 rpm and for 20 mins is crucial for preparation of nanosuspension. Moreover, nanosuspensions were not obtained with PVP K30, SLS and HPMC E5 while Poloxamer 407, Poloxamer 188, PVA and Tween 80 yielded nanosuspensions.

2.3.3 Selection of stabilizer

Accurately weighed 100 mg of drug was dissolved in 10 ml of acetone to form organic solution of drug. Accurately weighed 500 mg of selected stabilizers from the results of table 3 were dissolved in 100 ml distilled water to form antisolvent system. Then organic solution of drug was added drop by drop with the use of syringe into antisolvent system with continuous homogenization at 20,000 rpm for 20 mins. Batches were prepared as shown in table 4. Particle size analysis was performed using Zetasizer. Drug content was found out by suitably diluting nanosuspension (equivalent to 10 mg of drug) with methanol and measuring its absorbance at 240 nm. From the absorbance drug content was determined. Centrifugation study was performed on the prepared batches. Centrifugation was carried out at 10,000 rpm for 10 mins and observed for settling. (11,12)

Nanosuspensions of Cilnidipine were obtained successfully with Poloxamer 188 & 407, PVA and Tween 80 while PVP K30, SLS and HPMC E5 were unable to produce Nanosuspensions

Both the Poloxamers; **Poloxamer 188 and Poloxamer 407** have the same overall molecular structure.

On the other hand, Cilnidipine and that of **Tween 80** were substantial for formation of smaller particles and stabilization of nanosuspension. As reported by Sepassi et a1111 Tween 80 is a molecule of small size (Molecular weight 1310 g/mol) so it forms a thin adsorption layer on the drug nanoparticles and offers a less effective steric stabilization than higher molecular weight polymers. This may be the reason for relatively larger particles of Cilnidipine with Tween 80 (i.e. Mean particle diameter 317.5 nm) than that of with Poloxamers. Vinyl groups of **PVA** (Polyvinyl alcohol), due to their hydrophobic nature tend to adsorb onto the hydrophobic part of Cilnidipine nanoparticles while –OH extend themselves outside into the aqueous environment and thus providing stabilization to the nanoparticles and preventing agglomeration. –OH bonds of PVA makes hydrogen bonding with water molecules (antisolvent system) and thus viscosity of it increases. This may be due to presence of sufficient extending polymer chains that provide comparatively better coverage and therefore better coverage to the nanoparticles allowing homogeneous particle distribution. (13)

SLS is an anionic surfactant which provides electrostatic stabilization. Cilnidipine is a very non-polar molecule (log P 8.6). So ionic stabilizer such as SLS could not provide sufficient affinity with particle surface and thus it was unable to produce nanosuspension.

Among all the stabilizers tried, **PVP K30** possesses the highest molecular weight (50,000 g/mol). So due to its higher molecular weight, PVP K30 may exert more kinetic restriction in process of adsorption on the surface of drug nanoparticles and slower diffusion resulting in their inability to produce nanosuspension.

The reason for inability of HPMC E5 to produce Cilnidipine nanosuspension remained unknown. As Tween 80 interferes with the drug in assay, it was dropped and not considered for further studies. Centrifugation studies at 10,000 rpm for 10 mins did not show any settling of particles. This might be due to Brownian motion exhibited by colloidal systems like nanosuspensions which opposes the settling force.(14,15)

2.4. Experimental Design

In full factorial design, only a limited number of factors can be investigated because an increase in the number of experiments to be carried out. The central composite design allowed us to evaluate two factors at five levels by preparing only thirteen batches.

The Poloxamer 407 concentration and Stirring time play a crucial role in the preparation of Cilnidipine nanosuspension. Center composite design (CCD) was employed for systemic study of Independent variables Poloxamer 407 concentration (X_1) and Stirring time (X_2)] on responses such as particle size, saturation solubility study and cumulative percentage release at 5 min ($CPR_{5\ min}$). The selections of dependent variables were done on the basis of the aim of the present investigation (enhanced solubility

and dissolution rate of cilnidipine). preliminary trials, two factors were determined as follows. A design consists of thirteen runs. A second- order quadratic model incorporating interactive and polynomial terms was used to evaluate the responses.

$Y_1 = Bo + b_1X_1 + b_2X_2 + b_3 X_1X_2 + b_4X_1^2 + b_5 X_2^2 + b_6X_1^3 + b_7 X_2^3$

Where, Y_1 was the dependent variables, b_0 was arithmetic mean response of the 13 runs and b_1 was the estimated coefficient for factor X_1. The main effects (X_1 and X_2) represent the average result of changing one factor at a time from its low to high value. The interaction terms (X_1X_2) show how the responses changes when two factors simultaneously changed.

Data were further analyzed by Microsoft Excel® 2010 version (Microsoft Corp. USA) for regression analysis. Analysis of variance (ANOVA). Response surface and contour plots were plotted to study responses variations against two independent variables using Design Expert® 7.1.5 (Stat- Ease, Inc. Minneapolis,USA). (16)

2.4.1 Criteria for optimized formula

The criterion for selection of optimum formula was primarily based on the desired values of the response parameters, i.e. Y_1 particle size (nm), Y_2 saturation solubility study (mg/ml), Y_3 cumulative percentage release at 5 min (CPR_{5min}). (%). (see Table 8). The formulation corresponding to optimum responses were prepared and evaluated for % drug release.

As shown in Table 9 the approximations of response values Y_1 particle size (nm), Y_2 saturation solubility study (mg/ml), Y_3 cumulative percentage release at 5 min (CPR_{5min}).

Contour plots and response surface analysis

Two-dimensional contour plots and three-dimensional response surface plots are presented in Figure 5, 6, 7 , which is useful to study the interaction effects of the factors on the responses. The relationship between the dependent and independent variables was elucidated by constructing response surface plots.

- **Influence of formulation composition factor on particle size**

- For dependent variable Y_1 if, X_1 from −α to +α level increased and keeping X_2 at lower level particle size decreases from 195 to 114 nm. If keeping X_1 constant and X_2 level increased from -α to +α angle of repose will decreases up to 160 to 99 nm. A lowest particle size of 91.45 was observed with Poloxamer concentration 0.50 and stirring time-20 min (Batch 5) which suggests good particle size. (17,18)
- **Influence of formulation composition factor on saturation solubility study**

- For dependent variable Y_2 if, X_1 from −α to +α level increased and keeping X_2 at lower level particle size increases from 30 to 41 mg/ml. If keeping X_1 constant and X_2 level increased from -α to +α angle of repose will increases up to 32 to 48 mg/ml. A highest saturation solubility of 51 mg/ml was observed with Poloxamer concentration 0.50 and stirring time-20 min (Batch 5) which suggests good saturation solubility.
- **Influence of formulation composition factor on cumulative percentage release at 5 min (CPR_{5min}). (%)**
- For dependent variable Y_3 if, X_1 from −α to +α level increased and keeping X_2 at lower level particle size increases from 70 to 84 %. If keeping X_1 constant and X_2 level increased from -α to +α angle of repose will increases up to 86 to 95% A highest cumulative percentage release at 5 min of 100% was observed with Poloxamer concentration 0.50 and stirring time0 min (Batch 5) which suggests cumulative percentage release at 5 min.

2.4.1. Optimization of formula and Validation of CCD

After generating the polynomial equations relating the dependent and independent variables, the in situ formulations were optimized for the responses Y_1, Y_2 and Y_3. The desirable ranges of these responses were described in Table 10 and Figure 8. Therefore, to verify the evolved models, the optimum formulation was prepared according the above values of the factors and subjected to the analysis of

responses. As shown in Table 10 and Figure 8**.** It was demonstrating that the observed value of a new batch was quite closer to predicted value.
The overlay plot of optimized batch is given in Table 10 Figure 8**.** The predicted batch shows significant reproducibility within the percentage deviation. From the result shows that the predictive value close to the experimental value so design is significant.

2.5. Lyophilization of optimized nanosuspensions:

Further transformation into solid products is often required for physical stability and/or patient convenience reasons. Generally, there are two methodologies to convert aqueous dispersions to dry powders, i.e. lyophilization and spray drying.
Nanosuspensions contained in a petridish with the addition of cryoprotectant (100 %w/w of drug) were frozen in deep freezer at -40°C for 2 hr for primary freezing. The petridish were then transferred to freeze dryer and the lyophilization was carried out under a vacuum at 15 mTorr -54 0 C for 24 hr.

2.5.1 Selection of cryoprotectant

Furthermore, the crystallization of ice may exercise a mechanical stress on nanoparticles leading to their destabilization. For these reasons, special excipients must be added to the suspension of nanoparticles before freezing to protect these fragile systems. These excipients are usually added in order to protect the product from freezing stress or drying stress and also to increase its stability upon storage. The immobilization of nanoparticles within a glassy matrix of cryoprotectant can prevent their aggregation and protect them against the mechanical stress of ice crystals. (19,20)
lyophilization, three different cryoprotectants D-mannitol, Sucrosse, MCC were tried. Cryoprotectant selection was carried out using OP 1. Lyophillization was carried out according to procedure described in . Protocol of experiments for selection of cryoprotectant is shown in table. Based on visual inspection of quality and quantity of the lyophilized product, cryoprotectant was selected.
As shown in table 11, D-Mannitol served the purpose of cryoprotection very well while sucrose gave waxy film and microcrystalline cellulose gave brittle film. This may be due to recrystallization of mannitol around drug nanoparticles during water removal process and thus preventing particle agglomeration. The immobilization of nanoparticles within a glassy matrix of cryoprotectant can prevent their aggregation and protect them against the mechanical stress of ice crystals. So from the above results, it was concluded that for the freeze drying of cilnidipine nanosuspensions, D-Mannitol would be the most suitable cryoprotectant.(21)

2.5.2 Optimization of cryoprotectant concentration

On the basis of results of batches L1, L2 and L3, cryoprotectant was selected. For optimization of the cryoprotectant concentration, three different concentrations of cryoprotectant (50 %, 100 % and 250% w/w of drug) were taken. Batches were taken as shown in table 12
D-Mannitol 50 % w/w of drug could not provide sufficient cryoprotection while fluffy powder with good yield was obtained with 100 and 250 % D- Mannitol. At low concentration D- Mannitol could not provide adequate cryoprotection. This may be due to formation of eutectic with ice which can cause phase separation in the cryoconcentrated portion of the frozen nanoparticles suspension with no opportunity for a stabilization interaction. Moreover, the growing crystals of water and mannitol may exert mechanical forces on the nanoparticles leading to their fusion. So, it is required that at least some of the mannitol remains molecularly dispersed. Unavailability of molecularly dispersed D-Mannitol, in case of 50%w/w of drug can be the reason for film formation. From the above results, it was concluded that d- Mannitol 100% w/w of drug would be sufficient for cryoprotectant effect.(22,23)

2.5.2. Characterization of optimized lyophilized nanosuspension:

1. Average particle size

It is based on the measurement of the Brownian motion of particles. The Brownian motion is the random movement of particles in suspension. The smaller the particle, the faster the Brownian motion. The computer can provide the mean size and the distribution width of the nanoparticles in the batch.

Nanosuspension was added to the sample cell (quartz cuvette) and put into sample holder unit and measurement was carried out with the help of software.

2. XRD analysis

XRD study was performed to determine the change in crystalline nature of the pure drug after formulation of lyophilized nanosuspension. The samples were irradiated with the monochromatized CuKα radiation (1.5406 A0) and analyzed between 2 to 50 0 θ. The voltage and the current used were 30 kV and 30 mA respectively. Moreover, XRD study of lyophilized blank batches (without drug, only with mannitol and Poloxamer 407) was also performed to ascertain the change in crystalline nature of drug.

From the data obtained from XRD analysis, decrease in peak height was observed.

3. DSC studies

DSC studies were performed to determine the change in crystallinity of the pure drug after formulation of lyophilized nanosuspension. The DSC thermograms of samples were recorded by weighing nearly 2 mg of sample and hermatically sealing it in an aluminium pan. The pan was pin holed to facilitate the escape of vapours. The sample was heated at a constant rate 5 0 C/min over a temperature range of 30 0 to 300 0 C. Inert atmosphere was maintained by purging nitrogen gas at a flow rate of 200 ml/min.

To confirm the results of XRD studies, DSC studies were carried out. Thermograms of pure drug, optimized formula and lyophilized formula are shown in figure 26.

The pure drug shows sharp peak at 48.30 C (with an enthalpy of 89.78 J/g) corresponding to its melting point. On the other hand, F1 showed peak at 40.8260 C with enthalpy of 23.204 J/g and F2 showed peak at 41.737 0 C with enthalpy 23.957 J/g. This reduction in enthalpy and melting point can be due to decreased crystallinity of pure drug which is supported by results of XRD analysis. (24,25)

4. SEM

Lyophilized nanosuspensions were dispersed in water and then poured on carbon tape. Water was allowed to evaporate. This sample was placed on an aluminum stub. Then Platinum/ Palladium coating was applied for 2 cycles of 30 secs. This stub was placed in the vacuum chamber of the instrument. The sample was observed for morphological characterization using a gaseous secondary electron detector working at acceleration voltage of 25.00 kV. Raw drug was also observed for morphological characterization by Scanning Electron Microscope.

5. Drug content

Lyophilized nanosuspensions equivalent to 10 mg of drug was taken in 100 ml volumetric flask and diluted upto 100 ml with methanol. The absorbance of resulting solution was measured at 240.0 nm and drug content was calculated.(26)

6. Saturation solubility determination

Saturation solubility of lyophilized nanosuspensions were carried out in phosph. ate buffers 6.8 known excess of lyophilized nanosuspension (200 mg) were added to 10 ml distilled water. The sample was rotated at 20 rpm in an orbital shaker at 25± 0.50 C for 24 hr. The stirred samples were further taken in test tubes and centrifuged at 10,000 rpm for 15 minutes. Supernants was then filtered (0.45 μm, Gelman, Mumbai) suitably diluted and analyzed spectrophotometrically at 243 nm. Triplicate determination was perform.

7. In vitro Dissolution

Dissolution studies of nanosuspensions were performed in triplicate using USP Type II dissolution apparatus. nanosuspensions equivalent to 10 mg of cilnidipine were taken and placed in dissolution vessels containing 900 ml of 1% SLS in maintained at 37 ± 0.50 C and stirred at 75 rpm. Samples were withdrawn using 0.22μ nylon merck filter at 1 to 10 mins and replaced with fresh dissolution medium. Samples were suitably diluted and concentration of cilnidipine was determined spectrophotometrically at 243 nm.

For lyophilized nanosuspension equivalent to 10 mg of cilnidipine were taken and placed in dissolution vessel.(27,28)

2.6. Development of dosage form

Lyophilized nanosuspension equivalent to 10 mg of drug was filled in hard gelatin capsule shells and they were evaluated for parameters.

Disintegration time

Place 1 capsule in each of the six tubes of the basket and, place a disc. Operate the apparatus, using purified water as media maintained at 37 ± 2 °C. The disintegration time was noted when there was no residue on the screen of apparatus.(29,30)

Dissolution studies

Dissolution studies were performed as describe.

FIGURES

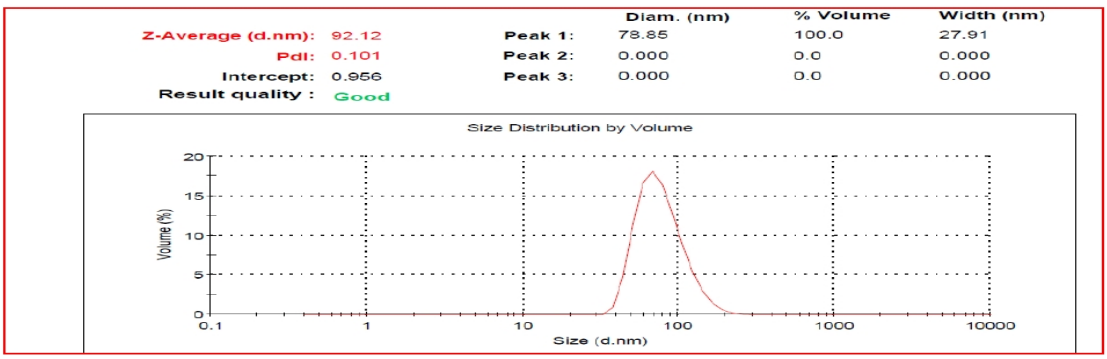


Fig 1: Particle size analysis of B1 (stabilizer Poloxamer407)

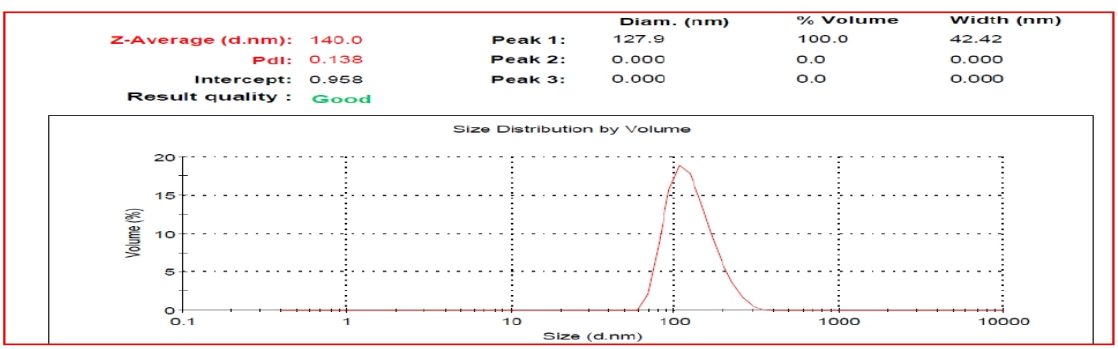


Fig 2: Particle size analysis of B2 (stabilizer Poloxamer 188)

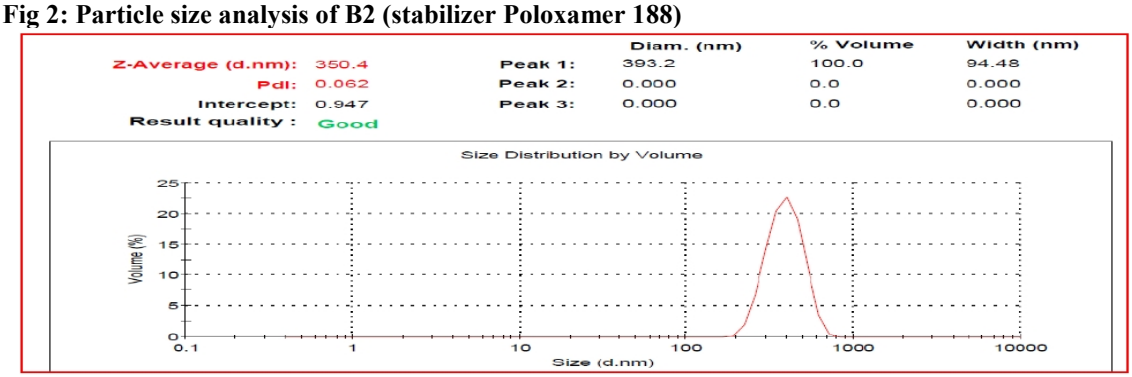


Fig 3: Particle size analysis of B3 (stabilizer PVA)

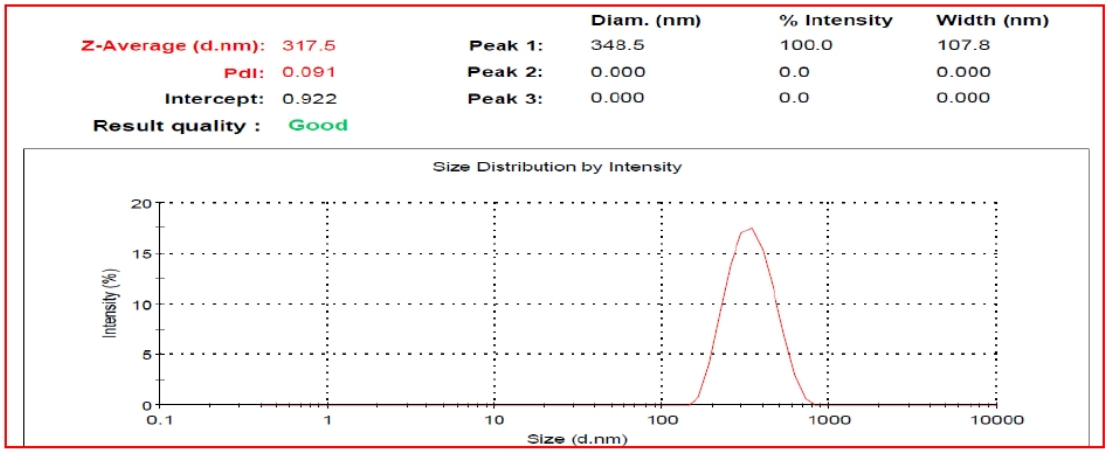


Fig 4: Particle size analysis of B4 (stabilizer Tween 80)

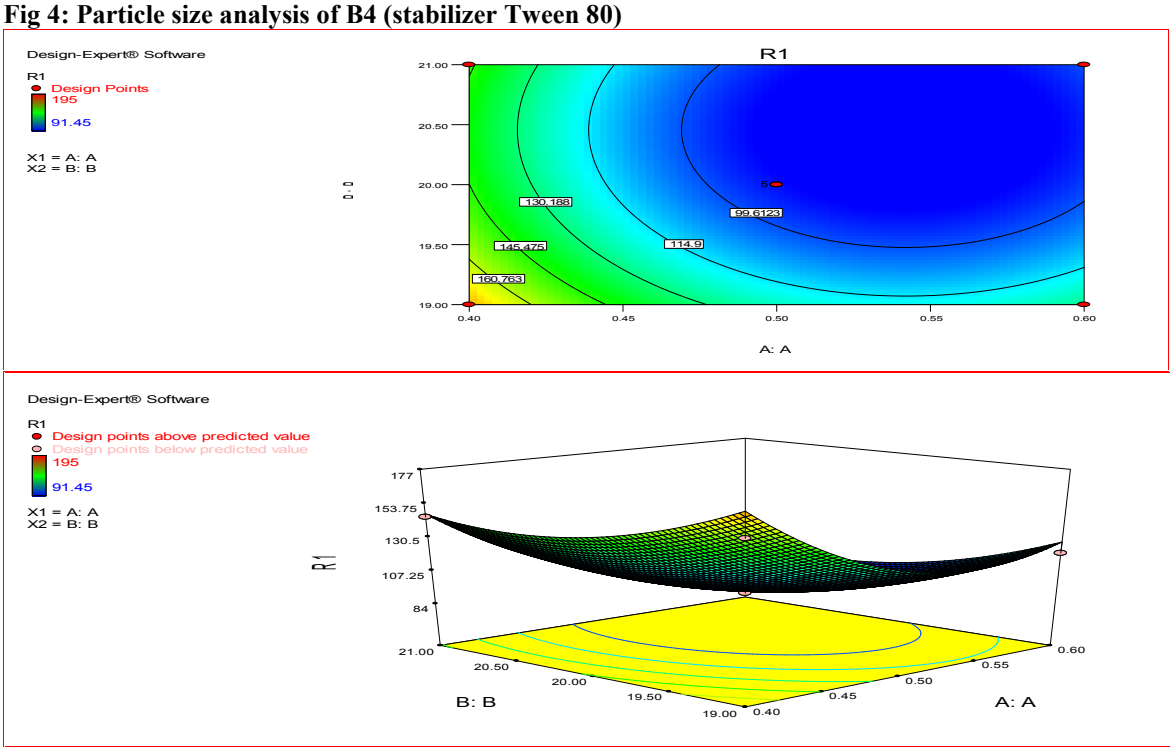
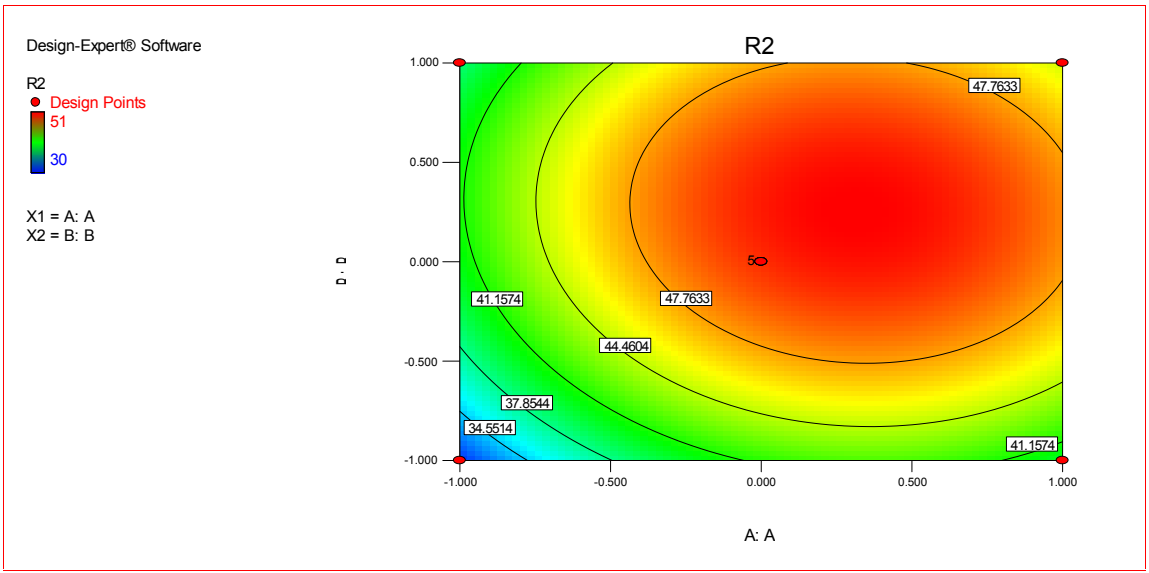


Fig 5: Response surface plot and contour plot showing the effect of X_1 and X_2 on particle size



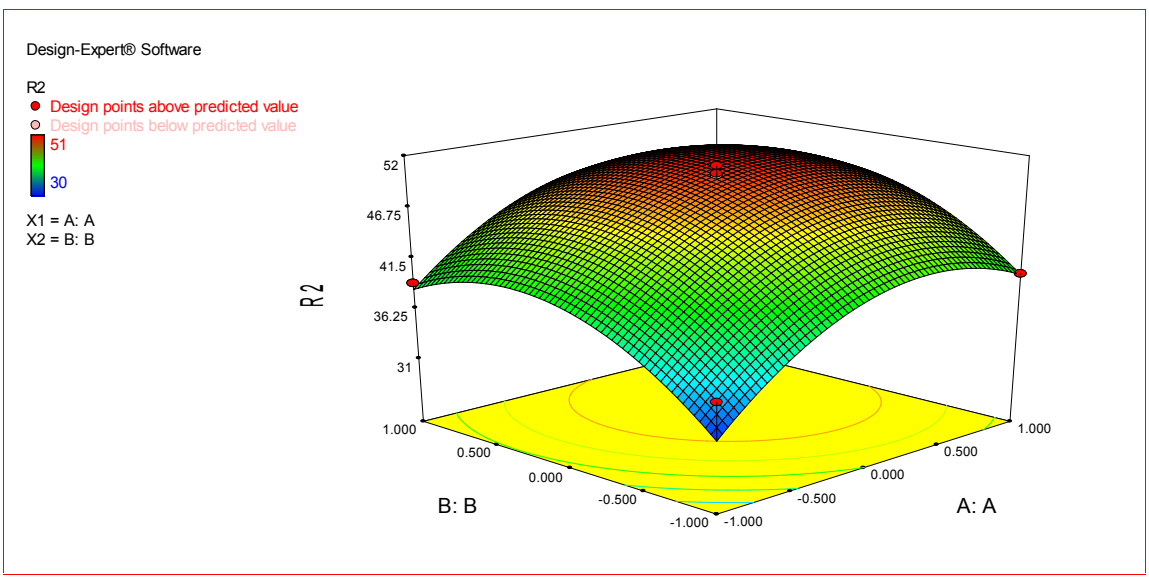
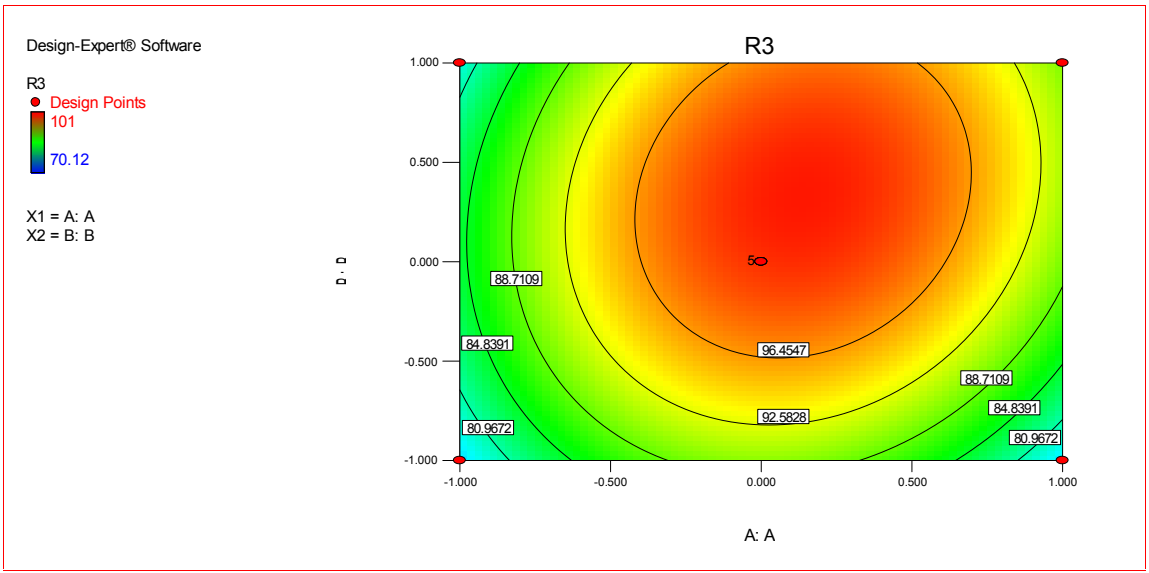


Fig 6: Response surface plot and contour plot showing the effect of X_1 and X_2 on saturation solubility



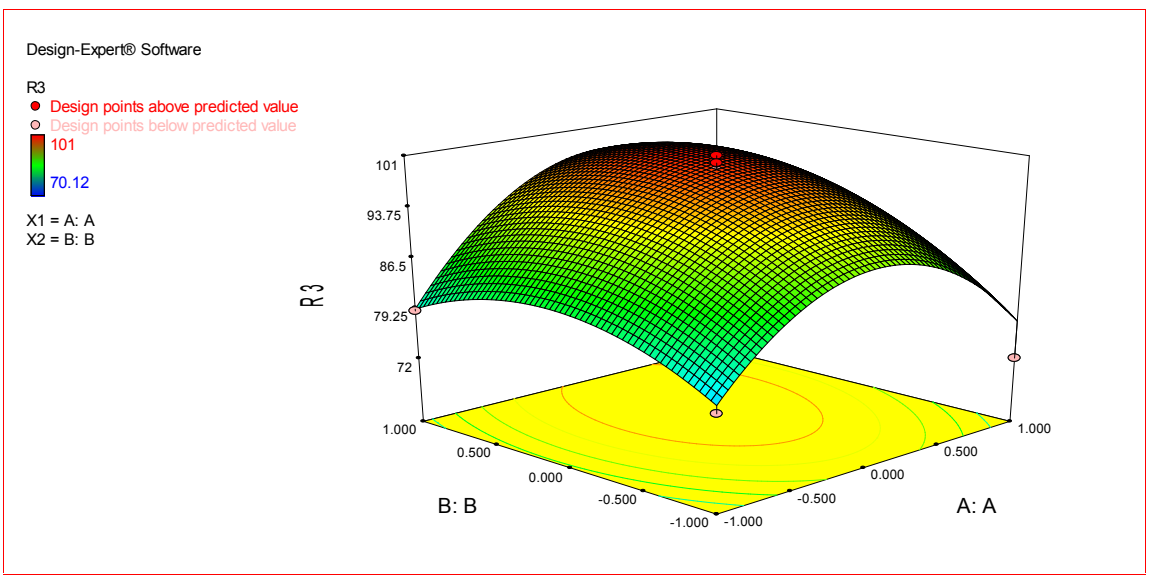
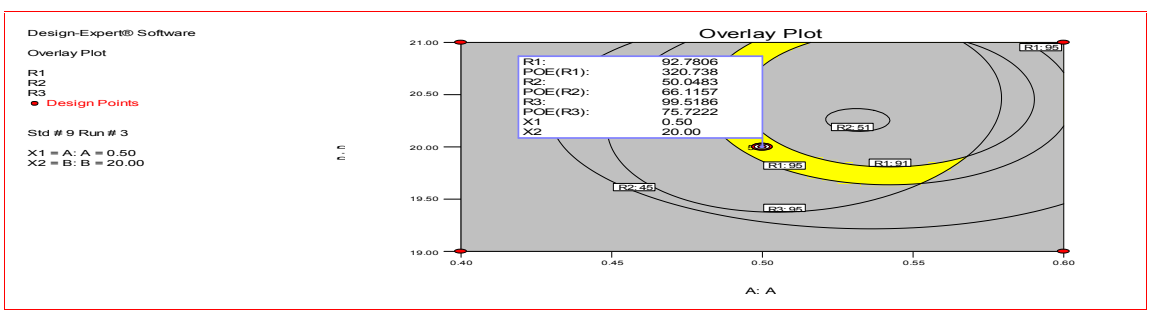
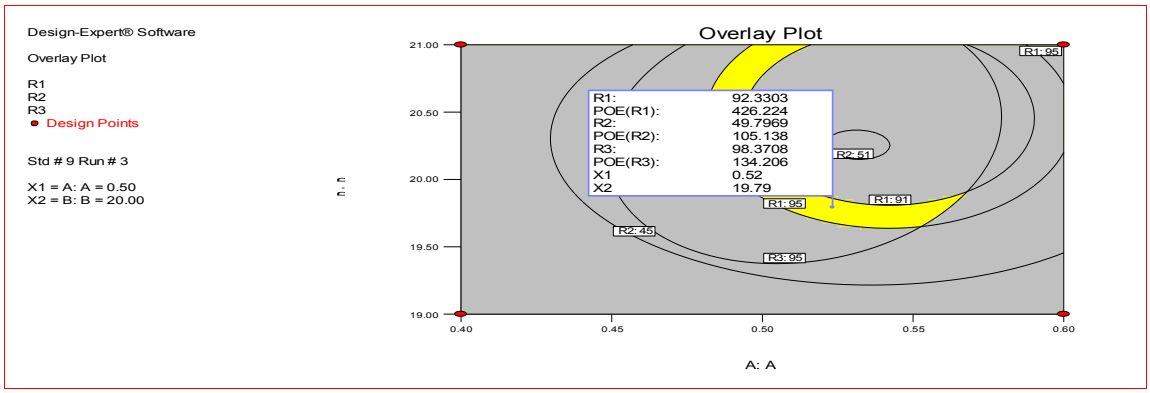


Fig 7: Response surface plot and contour plot showing the effect of X_1 and X_2 on cumulative percentage release at 5 min (CPR_{5min}). (%)





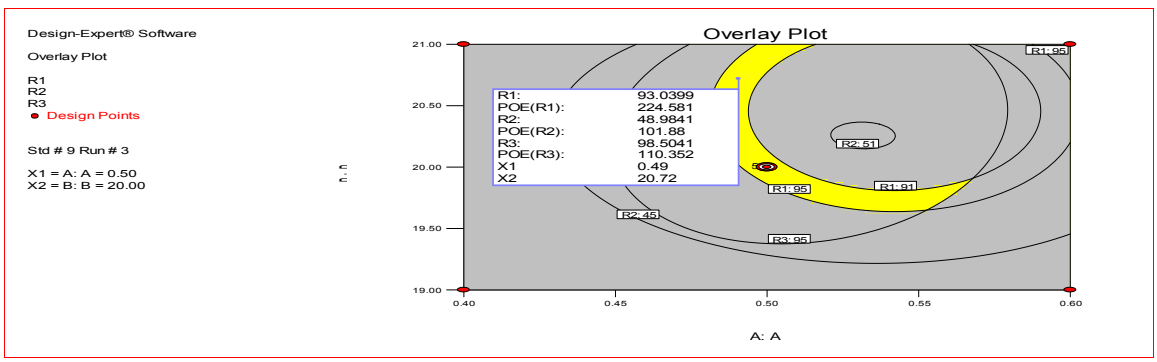
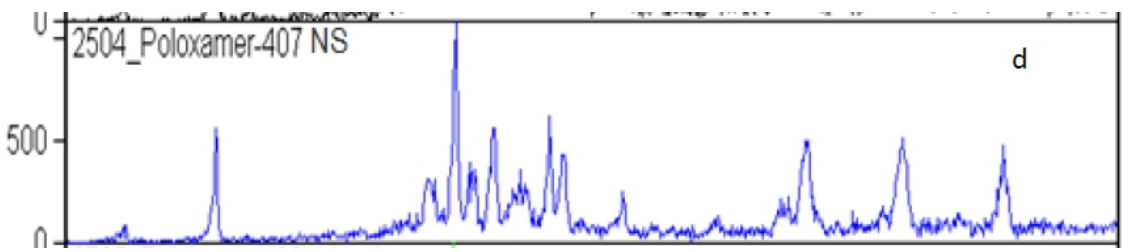


Fig 8: Overplay plot showing combined effects of factors X_1 and X_2 on Y_1, Y_2, Y_3



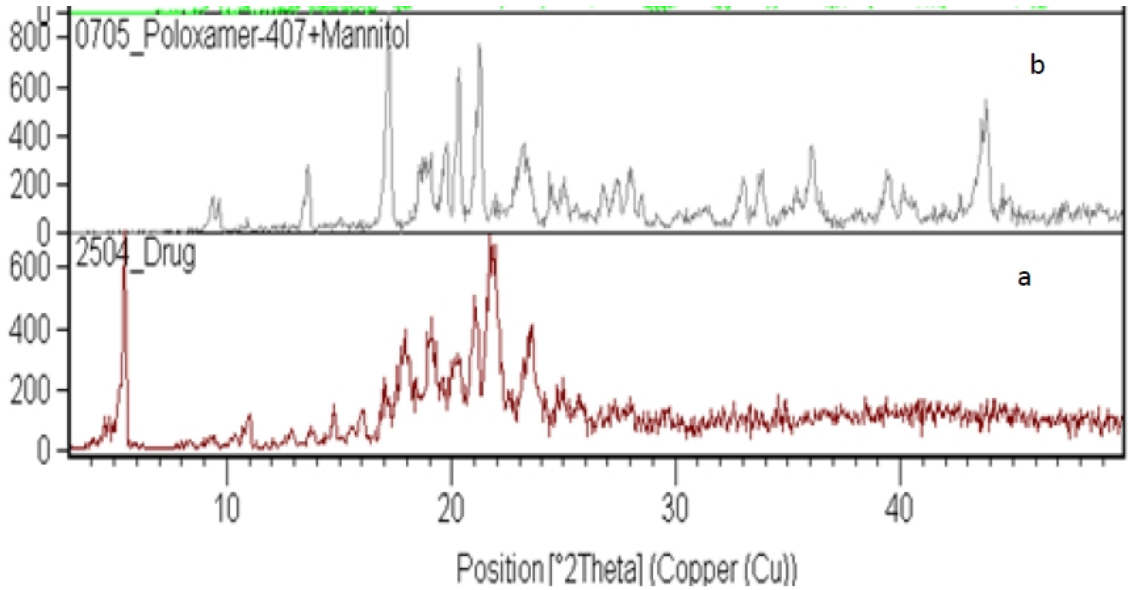


Fig 9: XRD analysis of pure drug, optimized nanosuspension, optimized lyophilized suspension

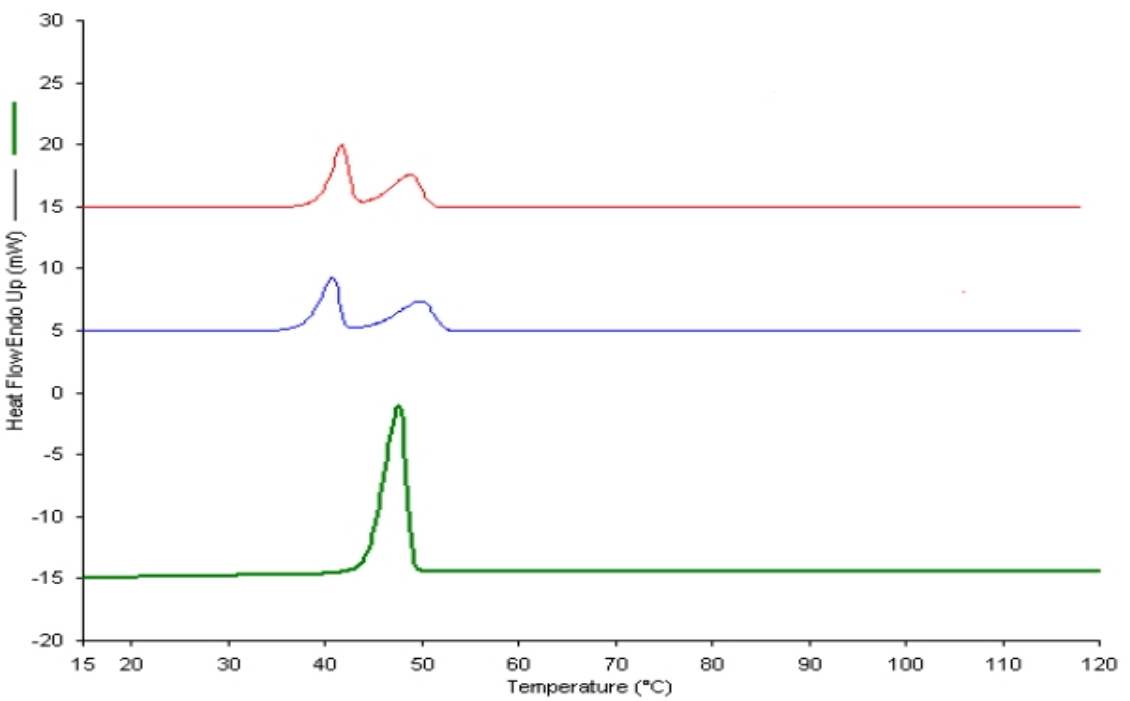


Fig 10: DSC thermogram of Pure drug optimized nanosuspension, optimized lyophilized suspension

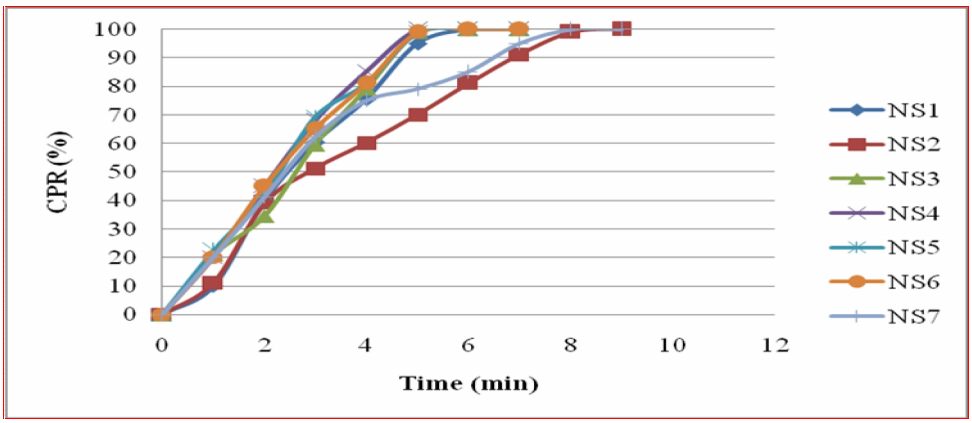


Fig 11: Dissolution profile of nanosuspension NS_1 to NS_8

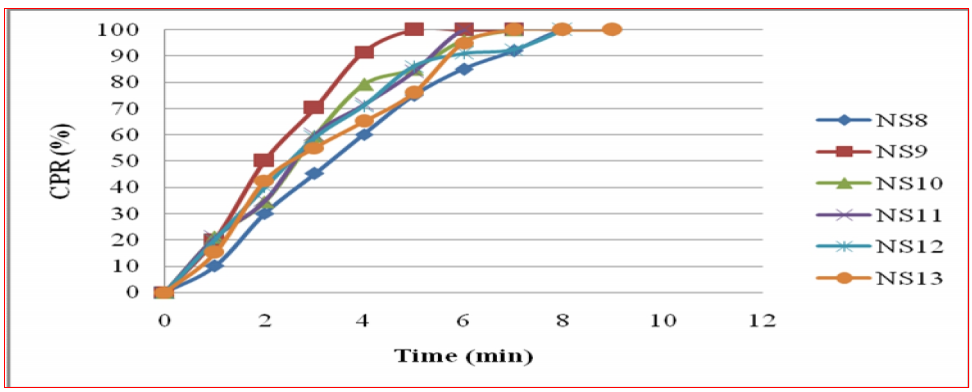


Fig 12: Dissolution profile of nanosuspension NS_8 to NS_{13}

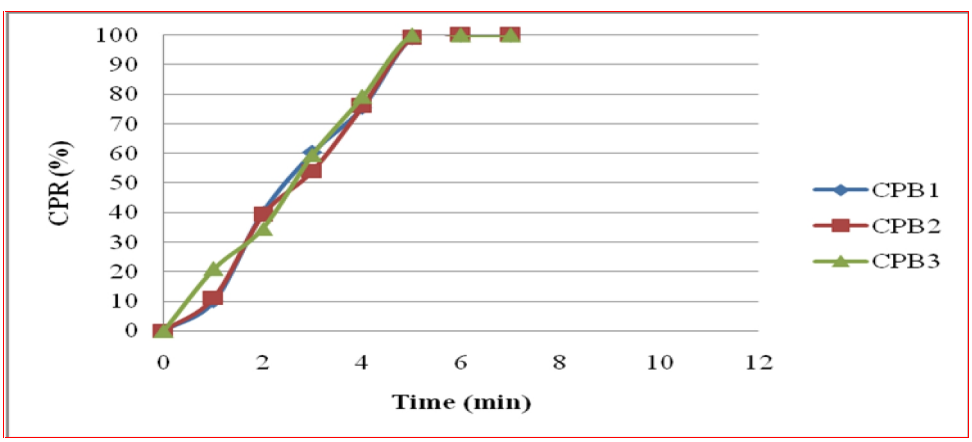


Fig 13: Dissolution profile of nanosuspension CPB_1 to CPB_3

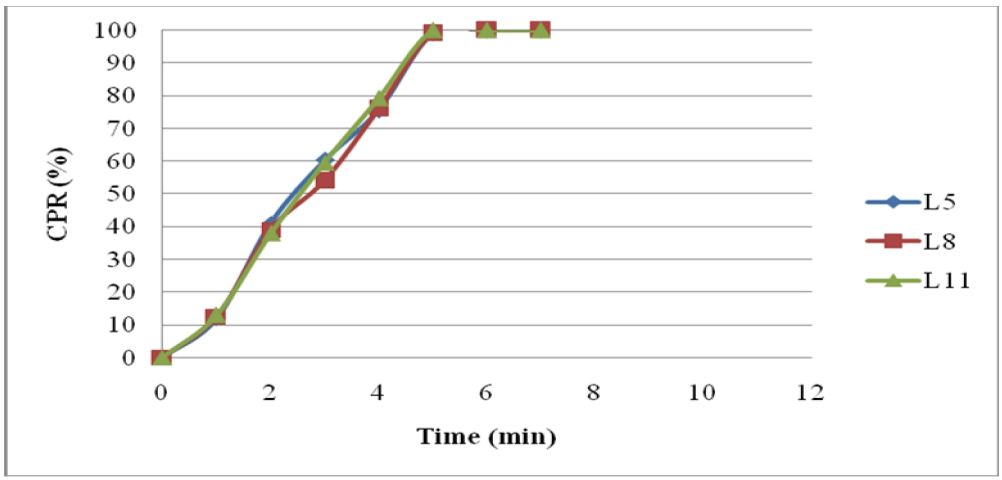


Fig 14: Dissolution profile of nanosuspension L_5, L8 & L_{11}

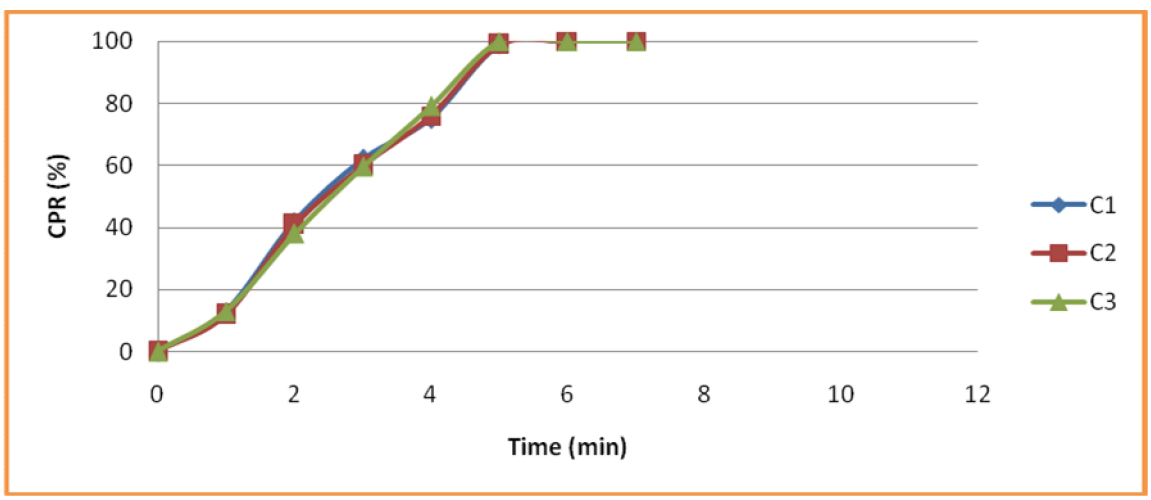


Fig 15: Dissolution profile of capsule

TABLES

Table 1: Protocol of experiments for optimization stirring speed

Preliminary trial batch	Stirring speed (rpm)	Stirring Time (min)	Initial observation	Liquid state Stability
P1	5000	10	Aggregates	--
P2	5000	20	Aggregates	--
P3	5000	30	Aggregates	--

P4	10000	10	Bluish tinge	1 Day
P5	10000	20	Bluish tinge	2 Days
P6	10000	30	Watery	2 Days
P7	15000	10	Bluish tinge	2 Days
P8	15000	20	Bluish tinge	4 Days
P9	15000	30	Watery	2 Days
P10	20000	10	Bluish tinge	>15 days
P11	20000	20	Bluish tinge	>15 days
P12	20000	30	Orange yellow	>15 days

Table 2: Protocol of experiments for optimization of stirring time and selection of stabilizers

Batch	Stabilizer concentration (0.5% w/v)	Stirring Time (min)	Initial observation	Liquid state stability(days)
T1	PVP K30	10	Watery	1
T2	PVP K30	20	Watery	2
T3	Poloxamer 407	10	Bluish tinge	5
T4	Poloxamer 407	20	Bluish tinge	>15
T5	Poloxamer 188	10	Bluish tinge	>15 (no bluish tinge)
T6	Poloxamer 188	20	Bluish tinge (distinct)	>15
T7	HPMC E5	10	aggregates	-
T8	HPMC E5	20	Bluish tinge	2
T9	PVA	10	Bluish tinge	5
T10	PVA	20	Bluish tinge	>15
T11	Tween 80	10	Bluish tinge	4
T12	Tween 80	20	Bluish tinge	>15
T13	SLS	10	aggregates	--
T14	SLS	20	Watery	1 hour only

Table 3: Protocol of experiments for optimization of stirring time and selection of stabilizers

Batch	Stabilizer concentration (1% w/v)	Stirring Time (min)	Initial observation	Liquid state stability(days)
T15	PVP K30	10	Watery	1
T16	PVP K30	20	Watery	2
T17	Poloxamer 407	10	Bluish tinge	6
T18	Poloxamer 407	20	Bluish tinge	>15
T19	Poloxamer 188	10	Bluish tinge	>15 (no bluish tinge)
T20	Poloxamer 188	20	Bluish tinge (Distinct)	>15
T21	HPMC E5	10	Aggregates	--
T22	HPMC E5	20	Bluish tinge	3
T23	PVA	10	Bluish tinge	5
T24	PVA	20	Bluish tinge	>15
T25	Tween 80	10	Bluish tinge	5
T26	Tween 80	20	Bluish tinge	>15
T27	SLS	10	Aggregates	--
T28	SLS	20	Watery	4 hours

Table 4: Protocol of experiments for selection of stabilizers

Batch	Stabilizer concentration (0.5% w/v)
B1	Poloxamer 407
B2	Poloxamer 188
B3	PVA
B4	Tween 80

Table 5: Results of stirring time and stabilizers

Batch	Stabilizer concentration (0.5% w/v)	Particle size nm	PDI	Centrifugation Study	Drug Content %
B1	Poloxamer 407	92.12	0.101	No settling	98.98 %
B2	Poloxamer 188	140.0	0.138	No settling	99.12 %
B3	PVA	350.4	0.062	No settling	99.45%
B4	Tween 80	317.5	0.091	No settling	99.78%

Table 6: Factors and their different levels for Central composite design for preparation liquisolid tablets

Independent Variables	Levels				
	Lowest (-α)	Low (-1)	Medium (0)	High (+1)	Highest (+α)
Poloxamer 407 concentration (%) (X_1)	0.36	0.4	0.5	0.6	0.64
Stirring time (X_2) Min	18.59	19	20	21	21.41
Transformed values	-1.414	-1	0	+1	+1.414
Dependent variables	Y_1 particle size (nm)				
	Y_2 saturation solubility study (mg/ml)				
	Y_3 cumulative percentage release at 5 min (CPR_{5min}). (%)				

Table 7: Experimental matrix and results

RUN	Independent Variables		Responses		
	X_1 (Poloxamer 407 concentration %)	**X_2 (Stirring time)**	**Y_1 particle size (nm)**	**Y_2 saturation solubility study (mg/ml)**	**Y_3 cumulative percentage release at 5 min (CPR_{5min}). (%)**
NS_1	0.00	1.414	99.00	45.00	95.00
NS_2	-1.414	0.00	195.00	30.00	70.12
NS_3	0.00	0.00	92.12	50.00	99.00
NS_4	0.00	0.00	94.52	49.12	100.00
NS_5	0.00	0.00	91.45	51.00	98.74
NS_6	0.00	0.00	93.00	50.00	99.00
NS_7	-1.00	1.00	145.00	39.00	79.00

NS_8	1.00	-1.00	120.00	40.00	72.00
NS_9	0.00	0.00	92.45	50.22	101.00
NS_{10}	1.00	1.00	105	42.00	85.00
NS_{11}	1.414	0.00	115	46.00	84.00
NS_{12}	0.00	-1.414	160	32.00	86.00
NS_{13}	-1.00	-1.00	160	45.00	76.00

Tablet 8: Dependent variables with constraints in Central Composite Design

Response variables	Constraints
Y_1 particle size (nm)	$91 \leq Y_1 \geq 95$
Y_2 saturation solubility study (mg/ml)	$45 \leq Y_2 \geq 51$
Y_3 cumulative percentage release at 5 min (CPR_{5min}). (%)	$95 \leq Y_2 \geq 101$

Table 9: Regression analysis of central composite design batches

Model	Coefficient	Y_1 particle size (nm)	Y_2 saturation solubility study (mg/ml)	Y_3 cumulative percentage release at 5 min (CPR_{5min}). (%)
	β0	+92.71	+50.07	+99.55
	β1 (X_1)	-24.14	+3.83	+2.70
	β2 (X_2)	-14.53	+3.05	+3.59
	β12 (X_1X_2)	+0.00	-0.50	+2.50
	β3 (X_1^2)	+28.71	-5.85	-12.69
	β4 (X_2^2)	+15.86	-5.60	-5.97
Cubic	r^2	0.9475	0.9242	0.9286
	Adjusted r^2	0.9100	0.8701	0.8776
	PRESS	5150.64	337.34	766.49

Source	D.F.	Sum Square	Mean Square	F Value	p value
Particle size(Y1) (nm)					
Model	7	13674	1953.52	49.97	**0.0002**
X_1	1	3200	3200.00	81.85	**0.0003**
X_2	1	1860.50	1860.00	47.59	**0.0010**
X_1X_2	1	0.000	0.000	0.000	1.0000
X_1^2	1	5733.41	5733.41	146.64	**<0.0001**
X_2^2	1	1771.64	1771.64	45.31	0.0011
$X_1^2 X_2$	1	395.75	395.75	10.12	0.0245
$X_1 X_2^2$	1	137.26	137.26	3.51	0.1198
Y_2 saturation solubility study (mg/ml)					
Model	7	641.65	91.66	156.71	**<0.0001**
X_1	1	128.00	128.00	218.83	**<0.0001**

X_2	1	84.50	84.50	177.46	**<0.0001**
X_1X_2	1	1.00	1.00	1.71	**<0.0001**
X_1^2	1	237.78	237.78	406.51	**<0.0001**
X_2^2	1	217.88	217.88	372.49	**<0.0001**
$X_1^2 X_2$	1	19.17	19.17	32.78	0.0023
$X_1 X_2^2$	1	26.75	26.75	45.72	0.0011
Cumulative percentage release at 10 min ($CPR_{10\ min}$) (Y_3) (%)					
Model	7	1478.60	211.23	15.01	**0.0044**
X_1	1	96.33	96.33	6.84	0.0473
X_2	1	40.50	40.50	2.88	0.1506
X_1X_2	1	25.00	25.00	1.78	0.2401
X_1^2	1	1120.07	1120.07	79.57	**0.0003**
X_2^2	1	247.85	247.85	17.61	0.0085
$X_1^2 X_2$	1	1.34	1.34	0.095	0.7703
$X_1 X_2^2$	1	38.85	38.85	2.76	0.1575

Table 10: Results of optimized batches

Sr. No.	Responses	Experimental Values	Predicted Values	%Relative Error*
CPB_1	Y_1 particle size (nm)	92	92.33	0.35
	Y_2 saturation solubility study (mg/ml)	49.50	49.79	0.58
	Y_3 cumulative percentage release at 5 min (CPR_{5min}).(%)	99	98.37	0.63
CPB_2	Y_1 particle size (nm)	92.00	92.0399	0.04
	Y_2 saturation solubility study (mg/ml)	48.50	48.98	0.97
	Y_3 cumulative percentage release at 5 min (CPR_{5min}).(%)	99	98.50	0.50
CPB_3	Y_1 particle size (nm)	92.00	92.70	0.76
	Y_2 saturation solubility study (mg/ml)	50.00	50.08	0.15
	Y_3 cumulative percentage release at 5 min (CPR_{5min}).(%)	100	99.55	0.45

***** Relative Error (%) = (predicted value - Experimental value)/predicted value×100 %.

Table 11: Selection of cryoprotectant

Batch	Cryoprotectant	Appearance of freeze dried product
L1	D-mannitol	Fluffy powder
L2	Sucrose	Waxy film
L3	MCC	Brittle film

Table 12: Selection of cryoprotectant

Batch Code	Optimize batch	Cryoprotectant Conc (%)	Appearance
L4	OP1	50	Film
L5	OP1	100	Fluffy powder
L6	OP1	250	Fluffy powder
L7	OP2	50	Film
L8	OP2	100	Fluffy powder
L9	OP2	250	Fluffy powder
L10	OP3	50	Film
L11	OP3	100	Film
L12	OP3	250	Film

Table 13: Particle size of nanosuspension

Batch	Z. Avg (d.nm)	Dia. (nm)	PDI
NS_1	99.00	100.12	0.120
NS_2	195.00	200.15	0.138
NS_3	92.12	90.12	0.101
NS_4	94.52	95.45	0.105
NS_5	91.45	92.00	0.109
NS_6	93.00	95.00	0.108
NS_7	145.00	160.12	0.138
NS_8	120.00	125.45	0.128
NS_9	92.45	93.52	0.101
NS_{10}	105.00	110.00	0.108
NS_{11}	115.00	116.12	0.184
NS_{12}	160.00	165.45	0.085
NS_{13}	160.00	168.23	0.086
CPB_1	92.33	93.23	0.102
CPB_2	92.03	92.12	0.108
CPB_3	92.70	92.15	0.102

Table 14: Drug content of nanosuspension

Batch	Drug content (%)
NS_1	98.98±0.12
NS_2	99.16±0.13
NS_3	99.45±0.56
NS_4	99.12±0.01
NS_5	102.01±0.04
NS_6	101.01±0.1
NS_7	100.02±0.4
NS_8	99.12±0.09
NS_9	100.14±0.2
NS_{10}	98.23±0.5
NS_{11}	100.24±0.2
NS_{12}	98.56±0.1
NS_{13}	99.67±0.13
CPB_1	101.01±0.1
CPB_2	100.02±0.4
CPB_3	99.12±0.09
L_5	101.01±0.1
L_8	100.02±0.4
L_{11}	101.01±0.1

Table 15: Saturation solubility of nanosuspension

BATCH	Saturation solubility determination (mg/ml)
L_5	10.00
L_8	9.52
L_{11}	9.81
Pure drug	2.0

Table 16: Disintegration time of lyophilized batches

Batch	Disintegration time (min)

C_1	2 min 30 sec
C_2	1 min 59 sec
C_3	2 min 10 sec

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

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Evaluation of hepatoprotective and antioxidant potential of *Melilotus officinalis* on iron dextran induced hepatotoxicity in Sprague Dawley rat model

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ABSTRACT

Melilotus officinalis (Fabaceae) reported to have various pharmacological activities. It contains flavonoids and phenolic compounds which have hepatoprotective, antioxidant and iron chelation properties. Present study deals with evaluation of hepatoprotective and antioxidant potentials of *M. officinalis*. Hepatotoxicity was induced by iron dextran (12.5 mg/100g). Different fractions of *M. officinalis* were given orally and Deferoxamine (DFO) subcutaneously for 30 days. Biochemical parameters were estimated on 15th and 30th day whereas antioxidant parameters on 30th day of treatment. Methanolic fraction of methanolic extract (MFME) and methanolic fraction of aqueous extract (MFAE) of *M. officinalis* significantly ($P<0.01$) decreases Superoxide Dismutase (SOD), Catalase (CAT) and Glutathione (GSH) whereas increases Malondialdehyde (MDA) as compared to disease control rats. There were significant ($P<0.01$) hepatoprotective effect shown by MFME and MFAE. It was concluded that MFME and MFAE of *M. officinalis* have hepatoprotective effect due to antioxidant potential by scavenging free radicals through chelating excess iron.

SUMMARY

The MFME and MFAE of *M. officinalis* have hepatoprotective and antioxidant potential against iron dextran induced hepatotoxicity in Sprague Dawley rats.

Keywords: Melilotus officinalis, Fabaceae, Hepatoprotective effect, Antioxidant potential and Iron overload

INTRODUCTION

Iron is essential for many metabolic processes, but it become toxic when accumulated. It encourages oxidative stress in the liver of overloaded conditions based on Fenton and Haber–Weiss reactions (*1*). Oxidative stress leads to tissue damage and contribute to development of numerous diseases and disorders (*2*). Iron intoxication in animals is an experimental model that mimics oxidative stress in many pathophysiological conditions (*3*). Studies showed that various herbs protect organs against iron dextran induced oxidative stress (*4, 5*).

Melilotus officinalis (Fabaceae) commonly known as yellow sweet clover reported to have antioxidants (*6*), anticoagulant (*7*), antibacterial, antitumor (*8*), antiinflammatory (*9*), antihypertensive (*10*) and astringent activity (*11*). *M. officinalis* is aromatic, emollient, styptic and carminative. The small fruits of plant are used as demulcent, maturant, tonic and aphrodisiac, there for useful in leucoderma (*12*). The seeds were reported to be poisonous (*13*). *M. officinalis* contains flavonoids and various phenolic compounds, coumarines, melilotin, triterpenes, tannin, sterols, carotenoids, mucilage (*14*). It also contains fatty acid, essential oil and glycosides (*15, 16*). Flavonoids are found in the various plants and have antioxidant, hepatoprotective and cytoprotective effect (*17)*. Hence the present study deals with the evaluation of hepatoprotective and antioxidant potential of different fractions of *M. officinalis* against iron dextran induced hepatic oxidative stress in Sprague Dawley rats.

MATERIALS AND METHODS

Plant collection and authentication

The fresh arial part of *M. officinalis* was collected in the flowering stage from the fields of Choaglamsar, Leh, Jammu and Kashmir, India during august 2013. It was authenticated by Mr. Akhtar H. Malik, Curator, Centre for Biodiversity and Taxonomy, Department of Botany, University of Kashmir, Jammu and Kashmir, India (Specimen No. 1915-KASH).

Preparation of extracts and fractions

The fresh aerial part of *M. officinalis* was shade dried and powered coarsely. The powdered material was extracted with methanol and water by soxhlet extraction method. The extracts were concentrated and dried to obtain residue. Both the dried extracts were dissolve in water and subjected for the fractionation

with following solvents in increasing polarity viz petroleum ether, benzene, chloroform, acetone, methanol and water. The fractions were concentrated and dried to obtain residue. The dried methanolic and aqueous fractions were weighed and required quantities of the same were dissolved in appropriate solvents for further investigations (*18*).

Animals

Twelve weeks old male Sprague-Dawley rats weighing about 200 to 250 g, procured from Zydus research centre, Ahmedabad, India were used in this study. The animals were housed at ambient temperature (23±2^{o}C), relative humidity (55±5%) and 12h/12h light dark cycle at animal house, Department of Pharmacology, School of Pharmacy, RK University, Rajkot, India. All animals were feed standard pellets diet and water *ad labium*. The research protocol was approved by IAEC as per the guidelines of CPCSEA (protocol no. RKCP/COL/RP/15/63). The animals were observed and the body weight was recorded at regular interval.

Instruments

A UV spectrophotometer, model UV-1800 (Shimadzu, Japan) and fully automated clinical chemistry analyzer, model C71 (BeneSphera diagnostic solutions USA) were used for the study.

Drug and chemicals

Desferal® (Deferoxamine mesylate, Novartis Pharmaceuticals Corporation, USA) and Imferon® (Iron dextran, Shreya life sciences Pvt. Ltd. India) were purchased. Various standard analyzing kits were commercially obtained from ERBA diagnostic mannheim, Germany. All other reagents and chemicals were commercially available and of analytical grade.

Induction of hepatotoxicity and treatment of animals with different fractions of *M. officinalis*

The rats were divided in to eleven groups each of six rats. All the groups except normal control (NC) were given six intraperitoneal injections of iron dextran (12.5 mg/100g) uniformly distributed over a period of 30 days (*19*). Group I, NC rats were given only dextran intraperitoneally at the same time. Group II, disease control (DC) rats were subjected only to iron dextran; Group III received subcutaneously standard iron chelator Deferoxamine (DFO) (40 mg/kg/day) (*20*); Group IV received methanolic fraction of methanolic extract of *M. officinalis* (150 mg/kg/day, p.o.) (MFME 150 mg/kg); Group V received methanolic fraction of methanolic extract of *M. officinalis* (300 mg/kg/day, p.o.) (MFME 300 mg/kg); Group VI received aqueous fraction of methanolic extract of *M. officinalis* (150 mg/kg/day, p.o.) (AFME 150 mg/kg); Group VII received aqueous fraction of methanolic extract of *M. officinalis* (300 mg/kg/day, p.o.) (AFME 300 mg/kg); Group VIII received methanolic fraction of aqueous extract of *M. officinalis* (150 mg/kg/day, p.o.) (MFAE 150 mg/kg); Group IX received methanolic fraction of aqueous extract of *M. officinalis* (300 mg/kg/day, p.o.) (MFAE 300 mg/kg); Group

X received aqueous fraction of aqueous extract of *M. officinalis* (150 mg/kg/day, p.o.) (AFAE 150 mg/kg); Group XI received aqueous fraction of aqueous extract of *M. officinalis* (300 mg/kg/day, p.o.) (AFAE 300 mg/kg).

Collection of samples

The study was carried out for the period of 30 days. The blood samples were collected on 15th and 30th day of treatment under the fasting conditions. The blood samples were collected by retro orbital plexuses in clean dry centrifuge tube, under light chloroform anaesthesia.

Estimation of liver biochemical parameters

The various serum biochemical markers for liver function like AST, ALT, Total Protein and Albumin were determined on 15th and 30th day of treatment.

Estimation of antioxidant parameters

At the end of the study, the overnight fasted rats were scarified. The liver was collected and weighted. The liver was preserved in 5% formalin solution for further investigation. The liver homogenates were prepared and were used to estimate antioxidant parameters like SOD (*21*), CAT (*22*), GSH (*23*) and MDA (*24*).

Histopathological examination of liver

The histopathological slides of liver were prepared by H & E staining method (*25*). The histopathological examination was performed to study the hepatoprotective potential of different fractions of *M. officinalis* on iron dextran induced hepatotoxicity. The slides were assessed by light microscopy under 40 X magnifications.

Statistical analysis

The results were expressed as mean ± standard deviation. The data were analyzed using one-way analysis of variance (ANOVA) with Dunnett's post test to determine statistically significant differences.

RESULTS

Effect of different fractions of *M. officinalis* on iron dextran induced hepatotoxicity

The beneficial effect of different factions of *M. officinalis* on liver had been determined on 15th and 30th day of treatment. The DC rats shows significant ($p<0.01$) increase in serum levels of AST and ALT whereas decline in serum levels of Total Protein and Albumin as compared to NC rats, which indicates that iron dextran induced hepatotoxicity had been developed. Rats treated with DFO and different fractions of *M. officinalis* shows significant ($p<0.01$) decline in serum levels of AST and ALT whereas increase in serum levels of Total Protein and Albumin as shown in Table 1. The MFME 150 mg/kg, MFME 300 mg/kg, MFAE 150 mg/kg and MFAE 300 mg/kg of *M. officinalis* shows more

significant hepatoprotective potential in iron dextran induced hepatotoxicity on 30th day of treatment as compared to other treatment groups.

Effect of different fractions of *M. officinalis* on iron dextran induced oxidative stress

The liver homogenates of DC rats demonstrate significant ($p<0.01$) decline in levels of SOD, CAT and GSH levels whereas increase level of MDA as compared to NC rats. The liver homogenates of rats treated with different fractions of *M. officinalis* shows significant ($p<0.01$) improvement in SOD, CAT and GSH levels whereas decline level of MDA. The results suggest that the different fractions of *M. officinalis* have antioxidant potential in iron dextran induced oxidative stress (Figure 1 to 4). There were more significant ($p<0.01$) antioxidant potential observed in MFME 150 mg/kg, MFME 300 mg/kg, MFAE 150 mg/kg and MFAE 300 mg/kg of *M. officinalis* treated rats over other fractions of *M. officinalis*.

Effect of different fractions of *M. officinalis* on body weight and liver weight

The effect of iron overload on body weight of rats was shown in Table 2. The body weight of DC rats significantly ($p<0.01$) decreases as compared to NC rats on 30th day of treatment which indicate that the iron dextran induced toxicity had been developed. The rats treated with DFO and different fractions of *M. officinalis* like MFME 150 mg/kg, MFME 300 mg/kg, MFAE 150 mg/kg and MFAE 300 mg/kg of *M. officinalis* shows more significant ($p<0.01$) improvement in body weight as compared to other treatment groups on 30th day of treatment.

The DC rats reveals the significant ($p<0.01$) increase in liver weight and liver weight to body weight ratio as compared to NC rats as shown in Table 2, which specify that iron dextran induced hepatotoxicity had been developed. Animals treated with DFO and different fractions of *M. officinalis* shows significant ($p<0.01$) decreases in liver weight and liver weight to body weight ratio.

Effect of different fractions of *M. officinalis* on histopathology of liver

The transverse section of liver reveals the normal hepatocytes (NH) and central vein (CV) in NC rats (Fig. 5-A) as compared to DC (Fig. 5-B) which shows the disarrangement and degenerations of hepatocytes near the central vein with formations of vacuoles (V), fibrosis (Fi), accumulation of iron in kupffer cells (KC), infiltration of inflammatory cells and inflammation of hepatocytes (IH), which reveals that the excess iron deposition take place in liver which leads to liver damage. The rats treated with DFO (Fig. 5-C) and different fractions of *M. officinalis* (Fig. 5-D to K) revealed the significant improvement in hepatotoxicity due to iron dextran. Our results suggest that the both the dose of MFME and MFAE shows more significant hepatoprotective potential as compared to other fractions of *M. officinalis*.

DISCUSSION

Flavonoids are found in the various plants and have antioxidant, hepatoprotective and cytoprotective effect (*17*). *M. officinalis* contains flavonoids and various phenolic compounds (*26*). Hence the present study was design to evaluate of hepatoprotective and antioxidant potential of different fractions of *M. officinalis* against iron dextran induced hepatic oxidative stress in Sprague Dawley rats.

Iron is an important element of the body which play vital role in various biochemical mechanisms. The normal level of iron is regulated by feedback mechanism between its requirement and absorption from intestine. Iron overload is unavoidable because there is no physiological mechanism for excretion of excess iron from human body (*27*). Excess of iron accumulation causes organ dysfunction through the production of reactive oxygen species (*28*). Liver is extremely sensitive to toxic agents. The study of enzyme activities such as AST and ALT where as liver protein such as Total protein and Albumin have been found to be of great value in the evaluation of clinical and experimental liver damage (*29*).In the present study it was observed that the rats treated with iron dextran resulted in significant hepatic damage as revealed by the elevated levels of serum AST and ALT where as decline levels of Total protein and Albumin. Various studies show that herbs with hepatoprotective potential reduce the levels of AST and ALT where as increases Total protein and Albumin levels (*30, 31*). Rats treated with the different fractions of *M. officinalis* significantly ($p<0.01$) attenuated the elevated levels of the serum AST and ALT where as the levels of Total protein and Albumin are increases. These changes can be considered as an appearance of improvement in hepatocytes which were damaged due to iron dextran.

The increased level of end product of lipid peroxide (MDA) leads to generation of ROS in iron overload condition. This leads to tissue damage and failure of natural antioxidant defence mechanisms to avoid formation of excessive free radicals. To neutralize oxidative stress, liver produces various enzymatic and non-enzymatic antioxidants such as SOD, CAT and GSH. These prevent the generation of free radical and protect the cells from oxidative damage (*32*). Antioxidant therapy reduces the level of MDA and increases SOD, CAT and GSH levels (*33*). The present study reveals that MFME and MFAE of *M. officinalis* significantly ($p<0.01$) decreases the level of MDA and increases the levels of SOD, CAT and GSH in iron dextran induced liver damage.

Excess supplement of iron reduces body weight, increases not only the liver weight but also liver weight to body weight ratio. These effects are due to its accumulation in hepatocytes that result in to liver damage (*34*). Treatment of hepatotoxicity with polyherbal formulation help in gaining body weight and decline the liver weight as well as liver weight to body weight ratio (*35*). Our results indicate that *M. officinalis* prevents the excess loss of body weight and reduces the liver weight as well as liver weight to body weight ratio which proves hepatoprotective effect of *M. officinalis*.

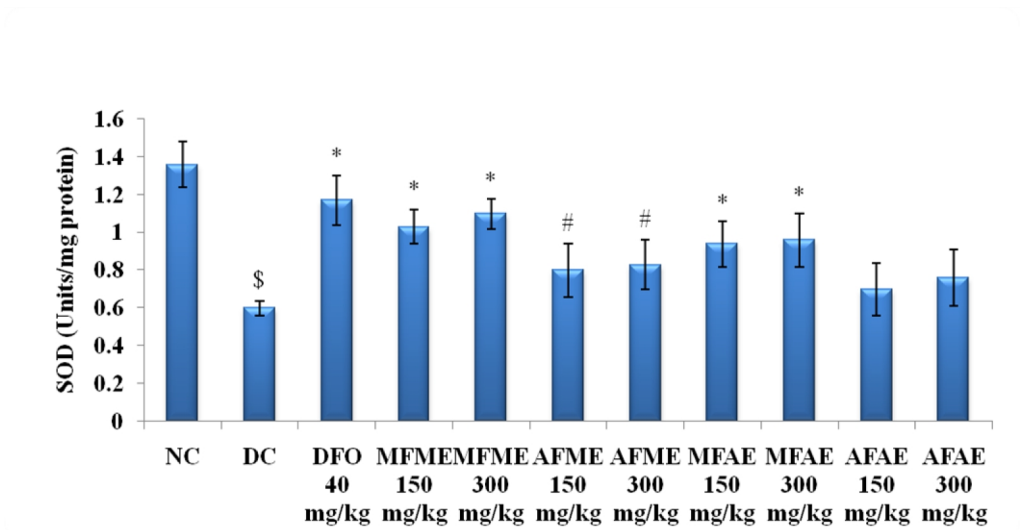
Iron overload leads to degenerations of hepatocytes, formations of vacuoles, fibrosis, accumulation of iron in kupffer cells and infiltration of inflammatory cells that causes inflammation of hepatocytes (*36*). Flavonoids restore the liver damage induced by iron overload (*37*). The rats treated with MFME and MFAE of M. officinalis shows reduced histological alteration.

The results suggest that higher dose of *M. officinalis* (300 mg/kg) has more significant hepatoprotective and antioxidant potential as compared to lower dose (150 mg/kg). *M. officinalis* shows better results on 30th day of treatment as compare to 15th day.

CONCLUSION

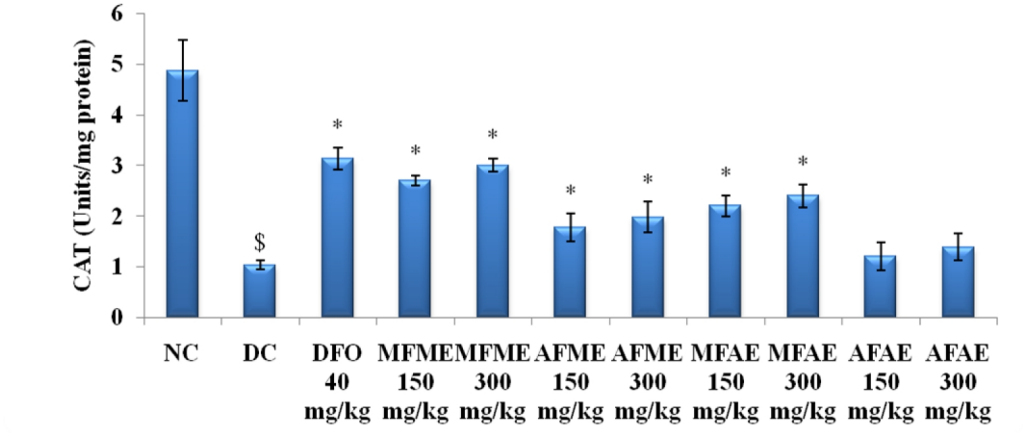
The present investigation indicates that hepatoprotective effect of *M. officinalis* could be due to antioxidant potential by scavenging the free radicals through chelating excess of iron. The antioxidant potential of *M. officinalis* could be extended for further isolation, characterization and biological evaluation of active constituents for the development of new herbal antioxidant.

FIGURES

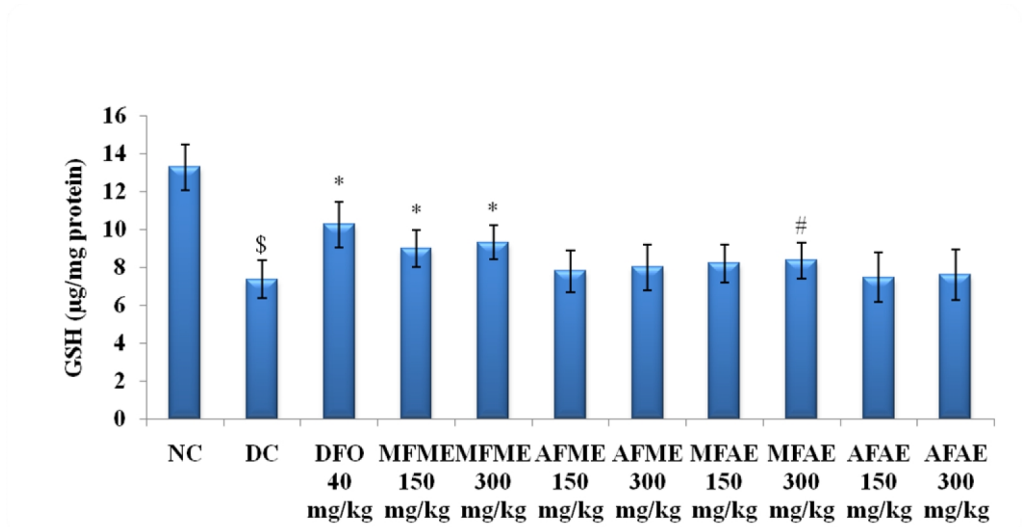


"Fig. 1. Effect of different fractions of *M. officinalis* on SOD"

The results were expressed as Mean ± S.D (n = 6), * $p < 0.01$ when compared to diseases control rats, $^{\#}$ $p < 0.05$ when compared to diseases control rats and $^{\$}$ $p < 0.01$ when compared to normal control rats.

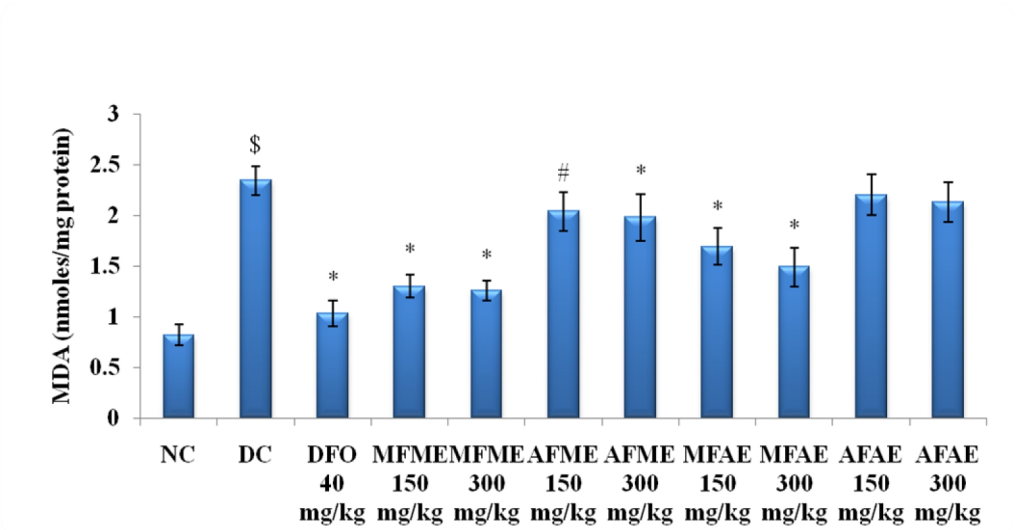


"Fig. 2 Effect of different fractions of *M. officinalis* on CAT"
The results were expressed as Mean ± S.D (n = 6), * $p < 0.01$ when compared to diseases control rats and $^{\$}$ $p < 0.01$ when compared to normal control rats.



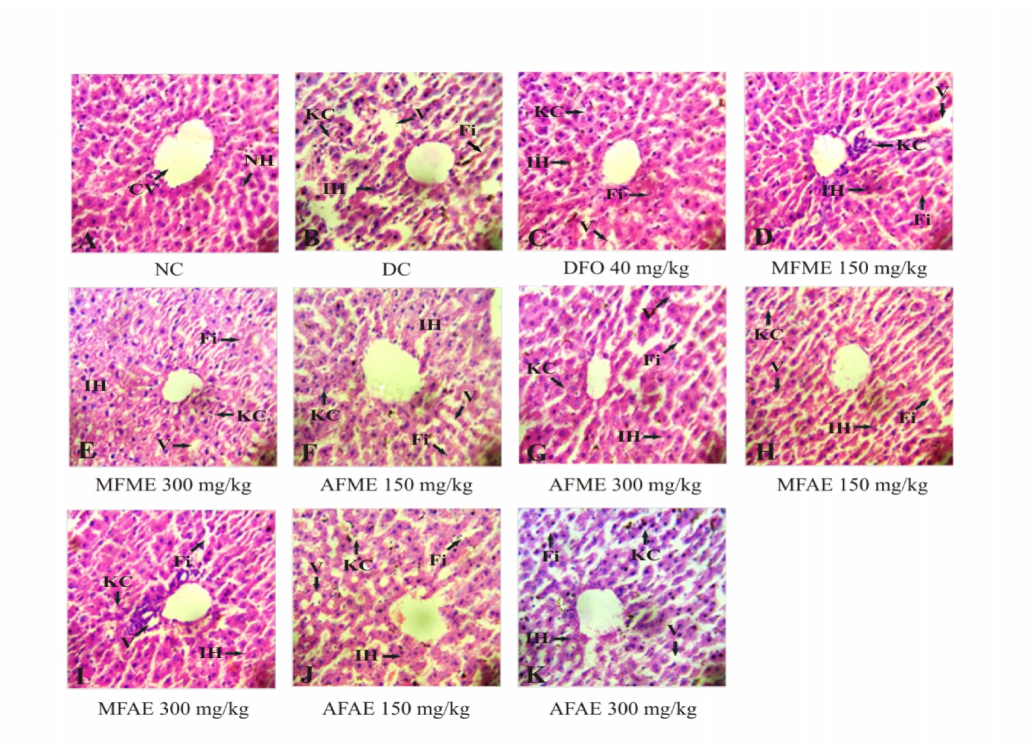
"Fig. 3 Effect of different fractions of *M. officinalis* on GSH**"**

The results were expressed as Mean ± S.D (n = 6), * $p < 0.01$ when compared to diseases control rats, $^{\#}$ $p < 0.05$ when compared to diseases control rats and $^{\$}$ $p < 0.01$ when compared to normal control rats.



"Fig. 4 Effect of different fractions of *M. officinalis* on MDA**"**

The results were expressed as Mean ± S.D (n = 6), * $p < 0.01$ when compared to diseases control rats, $^{\#}$ $p < 0.05$ when compared to diseases control rats and $^{\$}$ $p < 0.01$ when compared to normal control rats.



"**Fig. 5** Effect of different fractions of *M. officinalis* on histopathology of liver"

TABLES

"Table 1. Effect of different fractions of *M. officinalis* on liver biochemical parameter in iron dextran induced hepatotoxicity in rats"

Groups	AST (IU/L)		ALT (IU/L)		Total Protein (g/dL)		Albumin (g/dL)	
	15th day	30th day	15th day	30th day	15th day	30th day	15th day	30th day
NC	52.00 ± 3.10	54.67 ± 2.88	19.00 ± 1.41	22.05 ± 1.46	6.22 ± 0.74	6.50 ± 0.57	4.07 ± 0.36	4.42 ± 0.37
DC	238.50 ± 17.83$	255.67 ± 16.57$	102.50 ± 4.37$	131.83 ± 4.83$	1.87 ± 0.12$	1.68 ± 0.10$	1.25 ± 0.14$	1.13 ± 0.08$
DFO 40 mg/kg	103.50 ± 2.59*	90.00 ± 5.90*	61.17 ± 2.93*	56.00 ± 2.90*	5.05 ± 0.14*	5.43 ± 0.21*	3.70 ± 0.22*	3.95 ± 0.21*
MFME 150 mg/kg	148.00 ± 9.27*	137.83 ± 7.17*	79.67 ± 4.41*	74.00 ± 4.15*	4.00 ± 0.34*	4.20 ± 0.38*	2.90 ± 0.26*	3.18 ± 0.25*
MFME 300 mg/kg	123.12 ± 7.62*	110.83 ± 7.70*	73.33 ± 2.58*	69.83 ± 1.94*	4.40 ± 0.24*	4.62 ± 0.25*	3.30 ± 0.33*	3.52 ± 0.31*
AFME 150 mg/kg	200.50 ± 11.10*	197.17 ± 13.18*	94.17 ± 4.07*	90.83 ± 4.31*	2.47 ± 0.27#	2.65 ± 0.23*	1.70 ± 0.22#	1.88 ± 0.19*
AFME 300 mg/kg	194.33 ± 10.63*	190.00 ± 10.14*	92.50 ± 3.62*	89.67 ± 3.61*	2.83 ± 0.24*	2.77 ± 0.22*	2.17 ± 0.23*	2.38 ± 0.21*
MFAE 150 mg/kg	175.50 ± 7.79*	163.33 ± 5.20*	87.50 ± 2.95*	83.33 ± 2.42*	3.30 ± 0.38*	3.07 ± 0.40*	2.50 ± 0.24*	2.73 ± 0.21*
MFAE 300 mg/kg	161.33 ± 7.09*	149.67 ± 10.89*	84.33 ± 2.34*	79.83 ± 2.48*	3.60 ± 0.28*	3.23 ± 0.26*	2.70 ± 0.20*	3.08 ± 0.20*
AFAE 150 mg/kg	224.67 ± 14.47	213.17 ± 12.12*	98.17 ± 1.94*	97.00 ± 2.28*	2.00 ± 0.17	1.92 ± 0.23	1.33 ± 0.18	1.43 ± 0.23
AFAE 300 mg/kg	221.50 ± 14.54	209.83 ± 17.50*	97.50 ± 1.05*	96.17 ± 0.98*	2.40 ± 0.09#	2.13 ± 0.12	1.48 ± 0.21	1.48 ± 0.21

The results were expressed as Mean ± S.D (n = 6), * $p < 0.01$ when compared to diseases control rats, # $p < 0.05$ when compared to diseases control rats and $ $p < 0.01$ when compared to normal control rats.

"Table 2. Effect of different fractions of *M. officinalis* on body weight and liver weight"

Groups	Body weight (g)		Liver weight (g)	Liver weight to body weight ratio (%)
	Initial body weight	Final body weight		
NC	220.83 ± 7.55	234.33 ± 3.39	14.10 ± 0.98	6.02 ± 0.38
DC	231.17 ± 4.88	201.83 ± 3.76$	17.78 ± 0.64$	8.82 ± 0.45$
DFO 40 mg/kg	243.67 ± 2.16	222.83 ± 3.43*	15.05 ± 0.68*	6.75 ± 0.28*
MFME 150 mg/kg	244.17 ± 3.06	232.33 ± 7.66*	15.97 ± 0.79*	6.87 ± 0.29*

MFME 300 mg/kg	240.00 ± 5.87	233.83 ± 3.06*	15.73 ± 0.82*	6.73 ± 0.42*
AFME 150 mg/kg	239.00 ± 3.29	206.67 ± 6.15	16.83 ± 0.29	8.15 ± 0.30
AFME 300 mg/kg	236.33 ± 4.48	208.67 ± 2.58	16.68 ± 0.95	8.00 ± 0.48*
MFAE 150 mg/kg	242.50 ± 3.02	229.50 ± 3.62*	16.08 ± 1.39#	7.01 ± 0.59*
MFAE 300 mg/kg	239.33 ± 4.08	234.50 ± 5.28*	15.88 ± 1.20*	6.77 ± 0.38*
AFAE 150 mg/kg	240.17 ± 3.19	208.17 ± 2.64	16.63 ± 0.91	7.99 ± 0.46*
AFAE 300 mg/kg	239.33 ± 4.76	209.00 ± 2.53#	16.97 ± 0.80	8.12 ± 0.43#

The results were expressed as Mean ± S.D (n = 6), * $p < 0.01$ when compared to diseases control rats, # $p < 0.05$ when compared to diseases control rats and $ $p < 0.01$ when compared to normal control rats.

ACKNOWLEDGEMENT

The authors are grateful to School of Pharmacy, RK University, Kasturbadham, Rajkot (Gujarat), India for providing the facility to conduct the experimental work.

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A NEW GENERATION OF PAEDIATRIC PATIENT CENTRIC SPRINKLE CAPSULES DESIGNED FOR THE TREATMENT OF MALARIA

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ABSTRACT

The research work was intended to develop stable formulation of Artemether and Lumefantrine with improved solubility and ease of acceptability to paediatric patient. Formulation of sprinkle pellets was done using ratio of MCC PH 101 and HPMC E5 as binder, MCC PH 101 as spheronozing agent, CCS as disintegrat and Stevia as sweetening agent. Sprinkle formulation were developed by extrusion and spheronization technique. Phase solubility study confirmed 1:1 and 1:2 ratios for the complexation of drug and β-CD. From the 2^3 factorial design 4% concentration of HPMC E5 was optimized and for RPM and spheronization time, the values were 1200 and 5 min respectively. The preformulation parameters of the powder mixture were found to be within range. The dissolution study showed that both the drugs were released in about 90 min. Comparison with marketed tablet showed f_1=3.42 value and f_2=80.94 value.

SUMMARY

The current research work was intended to develop pellets having improved stability as well as solubility by carrying out β-CD complexation and optimization of formulation parameter done by 2^3 factorial design.

Keywords: Artemether, Lumefantrine, Pellets, β-CD, 2^3 full factorial design

INTRODUCTION

Malaria is parasitic disease transmit to people due to bites of infected *Plasmodium* parasites mosquitoes(*1*). Artemether is the drug of choice to treat malarial condition (*2, 3*). Generally combination therapy is recommended by the physician.

Cyclodextrin is unique compound with lipophilic inner cavity and hydrophilic outer surface that resembles a molecular container which holds non polar, non-ionic guest molecules in its inner cavity. This results the formation of inclusion complex that confers unique property (enhanced solubilization capacity) on guest molecules due to hydrophilic outer surface of molecular container.

MATERIALS AND METHODS

Materials:

Artemether (Baroque Pharmaceuticals, Khambhat), Lumefantrine (Baroque Pharmaceuticals, Khambhat), Avicel PH101 (Yarrow chem Pvt Ltd., Mumbai), β-Cyclodextrine (Astron chemicals, Ahmedabad), Stevia (Bharat parenteral, Vadodara), CCS (Astron chemicals, Goa), HPMC E5 (ColorconAsiaPvt. Ltd., Goa).

Methods:

Preparation of sprinkle dosage form:

Accurately weighed amount of Artemether and β-CD were mixed in mortar and were uniformly mixed with the help of pestle. Same procedure was also followed in the case of Lumefantrine. Accurately weighed MCC PH 101, CCS and Stevia were added to the physical mixture and mixed uniformly. HPMC E5 was dissolved in water and the binder solution was prepared. Binder solution was added drop by drop to the powder mixture and the mass of desired consistency was obtained. Prepared mass was extruded through the house hold extruder. Then extrudes were air dried.

After the desired drying is achieved. Extrudes are spheronised in the lab scale Spheronised at different RPM for different time period. The prepared pellets were dried in oven for 6-8 hr. and were sieved from different sieve. A prepared dried pellets can be filled in transparent as wellas colouredHPMC capsules with different sizes of 00, 0, and 1.House hold extruder and Lab scale Spheronizer are shown in figure 1. Extrudes and pellets are shown in figure 2.

Optimization of formulation:

The optimization of formulation was done by 2^3 statistical factorial design. The percentage concentration of binder (X1), the spheronization speed (X2) and time of spheronization (X3) were studied to achieve desired product profile. The percentage yield (Y1), and pellet size (Y2) were chosen as dependent variables. The summary of independent and dependent factors were depicted in Table 1. The QTPP parameters for the pellet preparation are shown in below table 1.

Evaluation of micromeritic properties (4-6):

The drug powder and drug-excipients blends were further studied for various micromeritic parameters.

Compatibility study:

The compatibility between actives and excipients was checked by thermal analysis using DSC and infra-red spectroscopy.

Evaluation of pellets:

Solubility determination of artemetherand lumefantrine:

(a) Phase solubility:

An excess amount of drug was placed into 25-mL separate glass flasks containing different concentrations of β-cyclodextrine in 5 ml distilled water. All flasks were stoppered to avoid solvent

loss. The content of the suspension was kept for shaking for 48hrs in Orbital Shaking Incubator at 37°C and 100rpm. After attainment of equilibrium of 48 hrs. 1ml of supernant was withdrawn and filtered through polypropylene membrane filter (0.45µm) and analysed using a UV–visible spectrophotometer (Shimadzu UV-1650PC (E) 230V, Tokyo, Japan) at 254 and 234 nm respectively. All solubility measurements were performed in triplicate. The phase solubility determination of a molar ratio with inclusion complex of drug and β-cyclodextrine1:1, 1:2, 1:3, 1:4. The apparent stability constant for artemether and Lumefantrine with the β-Cyclodextrine complex.

(b) Physical molecular complex:
Accurately weighed quantity of polymer was mixed with sufficient quantity drug to obtain a smooth and homogeneous mixture. The inclusion complex was prepared in the following ratio:artemether: β-CD in the ratios of 1:1 to 1:4; lumefantrine: β-CD in the ratios of 1:1 to 1:4.

(c) Saturation solubility study:
An excess amount of drug was placed into 25-mL glass flasks containing different concentrations of β-Cyclodextrine in 5 ml distilled water. All flasks were stoppered to avoid solvent loss. The content of the suspension was kept for shaking for 48 hr in Orbital Shaking Incubator at 37°C and 100 rpm. After attainment of equilibrium of 48 hrs. 1ml of supernant was withdrawn and filtered throughpolypropylene membrane filter (0.45 µm) and analysed using a UV–visible spectrophotometer (Shimadzu UV-1650PC (E) 230V, Tokyo, Japan) at 254 and 234nm. All solubility measurements were performed in triplicate.

Particle size analysis:
The pellet size analysis was carried out by standard sieving method. Aperture size was chosen in between0.5 and 4 nm.

Surface characterization:
The morphological characterization of pellets was performed by using a scanning electron microscopy technique.

Flowability and friability of pellets:
The freely flowing attribute of the pellets was checked by assessing micromeritic property.Roche friabilator was used to check friability of optimized pellets formulation as per pharmacopoeial specification.

Drug assay:
Accurately weighed pellets containing capsule equivalent to 20 mg Artemether and 120 mg Lumefantrine were powdered. Powder of the pellets were dissolved in appropriate volume of methanol. The 15 min stirring was done and then filtered. After suitable dilutions, resulted solution was analysedspectrophotometricallyat 254 and 338 nm.

In-vitro dissolution (7, 8):
The in-vitro*drug* release of the sprinkle pellets was determined using dissolution type-I apparatus (Electro lab TDT 08L, Mumbai, India). The volume of dissolution medium was 900 ml, 0.1 N HCl solution containing 1% SLS kept at 37 ± 0.5ºC.The basket rotation speed was 100 rpm. 5 ml of dissolution sample was withdrawn at an interval of 10,20,30,and 40, up to 90 min. Equivalent volume of sampled media was replenished with fresh medium. After suitable dilutions, resulted solution was analysed spectrophotometricallyat 254 and 338 nmfor Artemether and Lumefantrine respectively.

Stability testing:
Short term stability testing of prepared pellets was performed at extreme temperature and relative humidity condition as per guidelines. The pellets were wrapped in aluminium foil then kept in humidity chamber with well controlled conditions of temperature and humidity and then the samples were issued

after a period of 10, 20 and 30 days. Withdrawn samples were tested for drug content and release behaviour.

Simulated oral cavity model to assess extent of taste-masking (9-11):

Design of simulated oral cavity model apparatus:

The modified apparatus (figure 3) consist mainly 3 parts;

1. The artificial salivary fluid reservoir.
2. The simulated oral cavity.
3. Digital monitoring system.

The reservoir contains artificial salivary juice; the liquid was transferred through a tube which control flow rate by regulator. Fluid enters into the oral cavity surrounding the tongue. The simulated oral cavity, which is an adult dental set of lower and upper jaw. It was assembled on the tray which is connected with sampling tube. The artificial spongy tongue was placed at the lower jaw. A tube connected with reservoir was supplying fluid to the tongue at controlled rate, which mimicked the secretion rate of saliva. Previously wetted whatmann filter paper was kept on spongy tongue to facilitate base for formulation retention. The digital monitoring system consists of webcam was used to capture flow of salivary fluid as well as internal view of oral cavity.

Webcam connected to a computer for recording the process as video images. In this modified apparatus the testing can be initiated by putting the three different formulations i.e. pure drug powder, Marketed formulation (LUMETHER®) and Pellet formulation over the artificial tongue.

Assessment of taste masking:

Evaluation of extent of Taste masking study was performed using simulated oral cavity model. This simulated oral cavity model mimic the oral cavity environment. Simulated oral cavity model equipped with reservoir system containing artificial salivary fluid secretion, regulated with 1 ml/min using flow regulator. Pure drug, Marketed formulation and prepared Pellet formulation were placed on pre-wetted Whatmann filter paper one after another. After predetermined time (0 to 10 sec.) formulation was scrapped and removed from tongue. Dissolved drug in the ASF and retained on tongue was collected. Drug content was estimated using UV Spectrophotometer.

RESULTS AND DISCUSSION

Evaluation of pellets:

All the powder mixtures exhibited excellent flowability as well as other micromeritic properties.

Compatibility study:

FTIRstudy of mixture:

The infra-red spectra of all the tested samples showed the prominent characterizing peaks of both the drugs, which indicated the absence of any significant interaction in the physical mixture of drugs and excipients.

DSC study of mixture:

The thermogram of the investigated physical mixture exhibited the characteristic exothermic peaks, indicating the absence of interaction between the two components present in the physical mixture.

Solubility determination of artemetherand lumefantrine:

Phase solubility:

As concentration of β-Cyclodextrine increased from1:1 to 1:4 the solubility of both drug increased from 0.0583 mg/ml to 0.0747 mg/ml at 1:2 β-CD 1.28 folds enhancement of solubility of Lumefantrine and

was found as that of in distilled water. Table no 2 shows the Solubility and ΔG° value of artemether and Lumefantrine using different molar ratio of β-CD.

Particle size analysis:
Formulated pellets were subjected to size distribution study and are represented in figure 4.

Surface characterization:
The characterization of the pellets using SEM as shown in figure 5.

Drug assay:
From the preliminary batch F_{17} gives the highest drug content up to 99.89 ± 0.02 %.

In-vitro dissolution:
The percentage drug release studied from optimized formulation, as depicted in figure 6. From the dissolution profile, batch A6 showed comparatively higher release than other batches. Batch A6 was prepared with 4% HPMC E5 concentration, 1200 RPM and 5 min of spheronization time. Batch A6 also produced highest % yield as well as maximum amount of pellets in 1.2 mm size. So batch A6 was considered as best amongst all the batches in the design.

Optimization of the formulation:
The independent and dependent variables were correlated using mathematical statistical model evolved through Design-Expert® software 8.0.7.1. Y_1 (% Yield)& Y_2 (size) showed excellent fit with R-square value of greater than 0.99. The influence of binder concentration and RPM is shown in above Figure, which shows that pellet size was influenced by increasing binder concentration and RPM linearly, but in decreasing order. Desired range of pellet size was 1.2-1.4 mm.

From the dissolution profile, batch A6 showed comparatively higher release than other batches. Batch A6 was prepared with 4% HPMC E5 concentration, 1200 RPM and 5 min of spheronization time. Batch A6 also produced highest % yield as well as maximum amount of pellets in 1.2 mm size. So batch A6 was considered as best amongst all the batches in the design. Polynomial equation were obtain for Y1 and Y2 respectively $Y_1 = +50.50 + 4.75\ X_1 + 3.25\ X_2 + 6.75\ X_3 + 1.50\ X_1X_2 + 1.00\ X_1X_3 + 2.0\ X_1X_3$ and $Y_2 = +1.40 + 0.060\ X_1 + 0.045\ X_2 + 0.080$ X3.Counter plot of size and % yield shown in figure 7 and 8 respectively.

Drug release kinetic study:
It showed drug release kinetic of higuchi model. Linearity was observed with the plots, because the correlation coefficient found to be 0.9557, compared to other release kinetic models.

Stability testing:
The optimized batch of the prepared pellets A6 was exposed to the temperature and humidity condition as per stipulated guidelines by regulatory body. The samples were taken at the interval of 10, 20 and 30 days and the drug release was measured. The release is shown in table 3.

Assessment of taste masking:
The results clearly reveal that pellet formulation has ability to mask effectively the bitter taste of Artemether and Lumefantrine. Hence taste masking could be achieved to a large extent by the use of stevia and β-CD. The formulated product has shown much superiority to the pure drug from the perspective of bitterness. Figure 9 Percentage Drug released from pure drug, Marketed formulation and Pellet formulation in the simulated oral cavity.

Comparison with market formulation:

Marketed tablet showed similar dissolution profile as prepared pellet formulation. The values of f_1 and f_2 are 3.42 and 80.94.

CONCLUSION

Phase solubility studies confirmed ratio of 1:1 and 1:2 for Artemether and Lumefantrine respectively for drug: β-CD. Factorial design confirmed 4% concentration of HPMC E5, 1200 RPM and 5min Spheronization time as optimum parameters for the preparation of the pellets in desired size and %yield. Flow property studies showed good floe property of the powder blend. Dissolution of both the drug showed release in 90 min and zero order kinetic was followed for artemether and higuchi model was best fitted for Lumefantrine. Comparison with marketed formulation was carried out by model dependent approach and f_1 and f_2 values were 3.77 and 77.92 respectively which confirmed similarity between both the formulation and it was also stable. Hence we can conclude that prepared formulation was stable and the solubility was increased with good release profile. Simulated oral cavity method confirmed less amount of drug exposure to oral cavity in the prepared pellets as compared to pure drug and marketed formulation. Relative % bitterness value of prepared pellets was less as compared to marketed formulation and pure drug.

FIGURES



Fig. 1. (a) House hold extruder (b) Lab scale Spheronizer



Fig. 2. (a) Extrudes (b) pellets filled in HPMC “00” size capsule

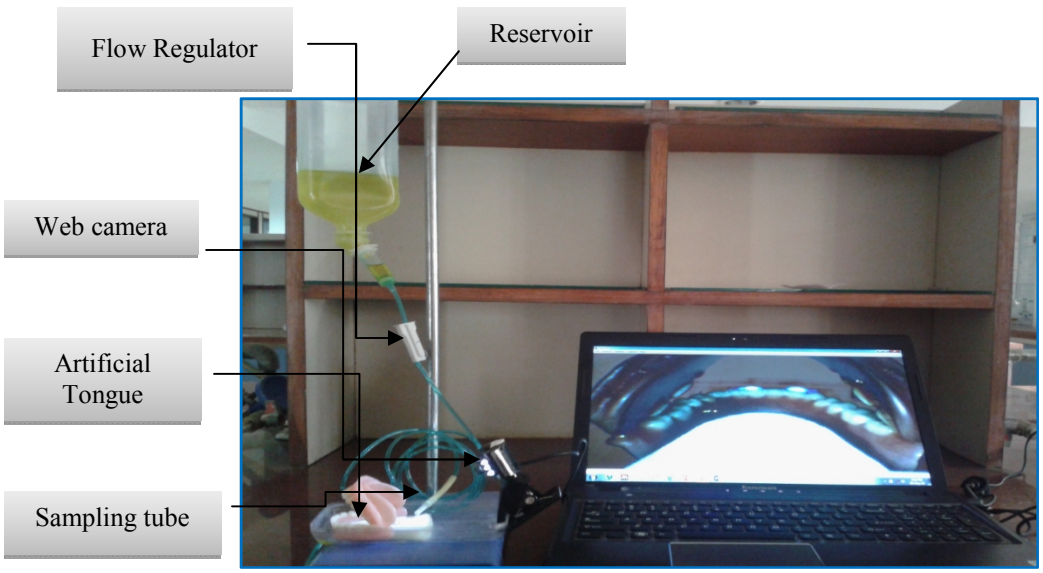


Fig. 3. Simulated oral cavity model

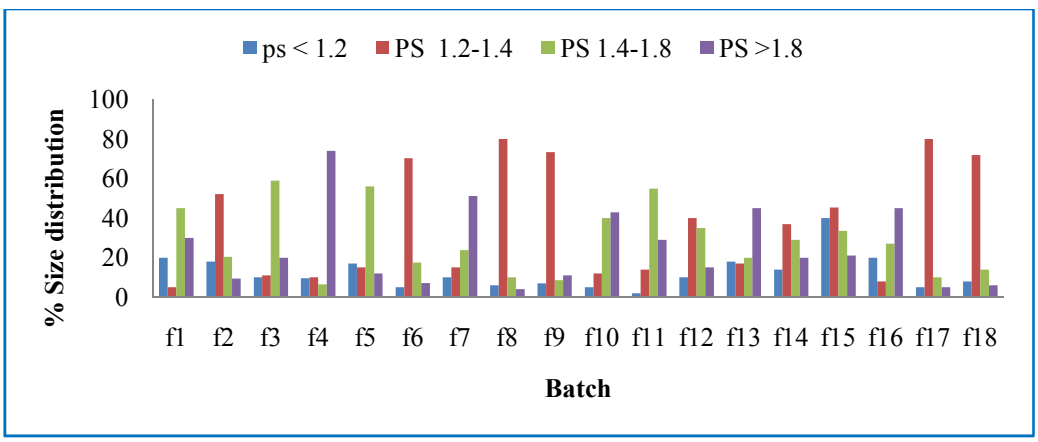


Fig. 4.Size distribution chart of formulated pellets

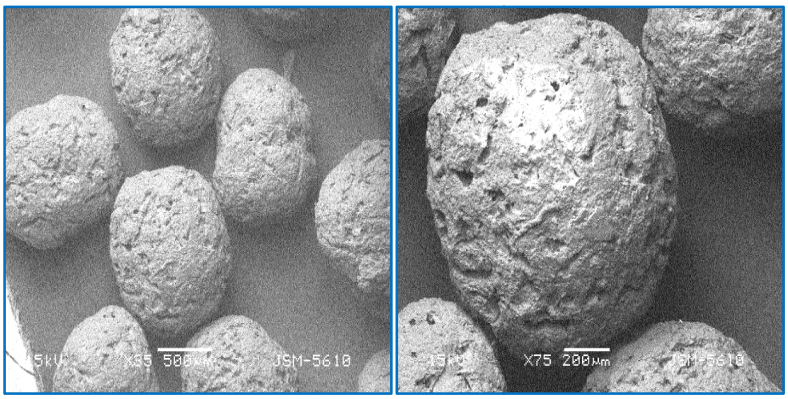


Fig. 5. SEM analysis of prepared pellets in 35X and 75X

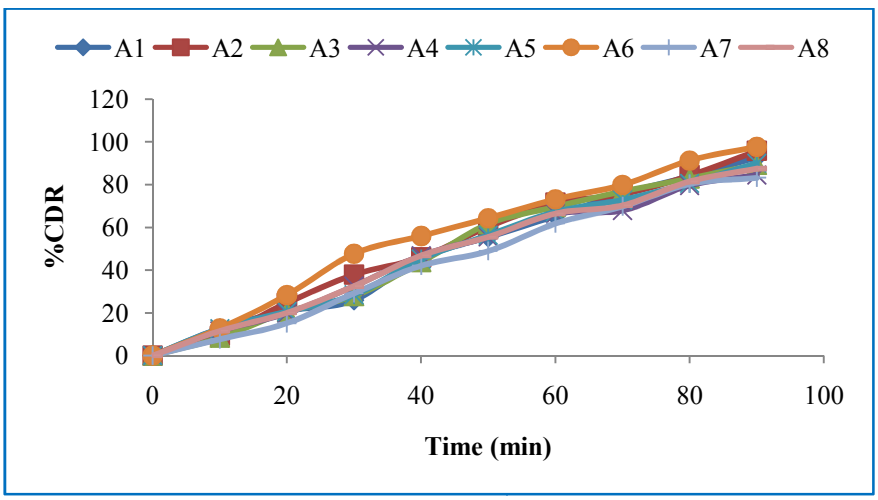


Fig. 6. Dissolution profile of factorial batches

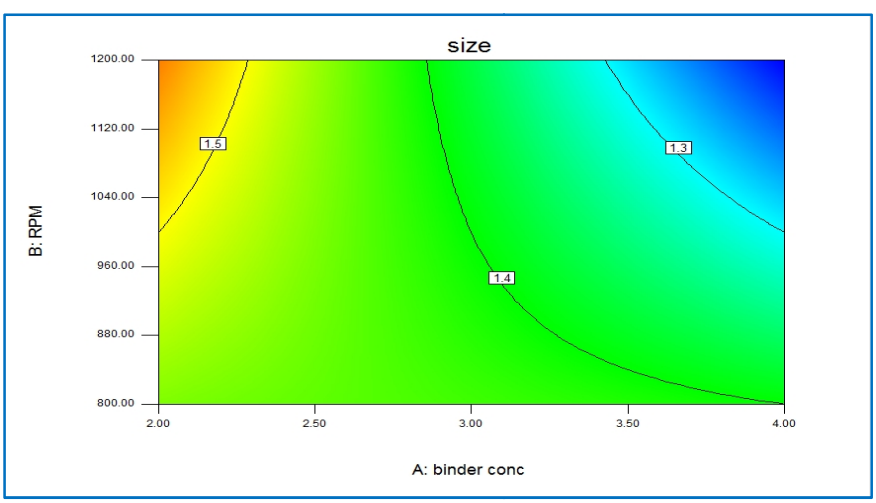


Fig. 7.2D contour plot of size

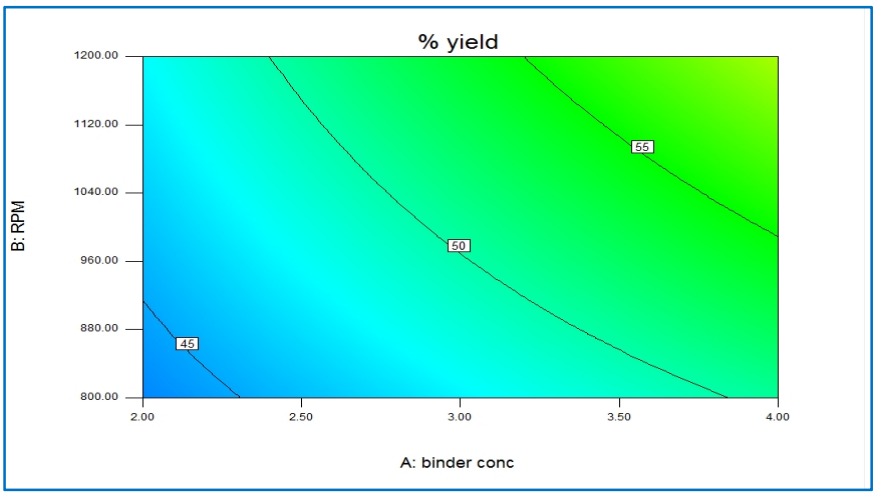


Fig. 8.2D contour plot of % yield

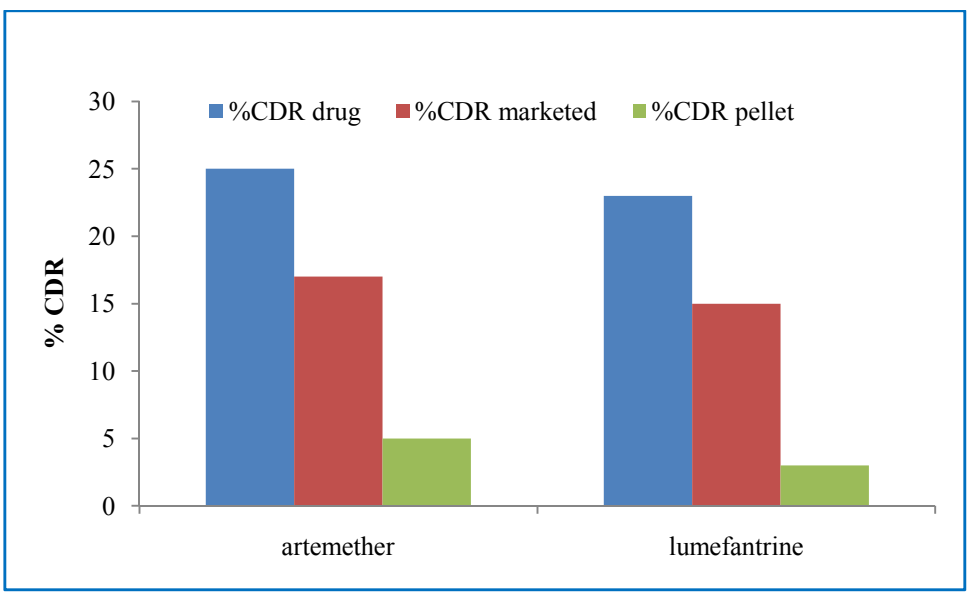


Fig. 9. Percentage Drug released from pure drug, marketed formulation and pellet formulation in the simulated oral cavity

TABLES

Table 1. Factors and levels with coded value, values of dependent variables and QTPP parameters of response variables

Run	X1	X2	X3	Y_1 (% Yield)	Y_2 size (mm)
A1	+1	-1	+1	56	1.4
A2	+1	+1	-1	50	1.2
A3	-1	+1	+1	55	1.5
A4	-1	+1	-1	40	1.6
A5	+1	-1	-1	45	1.4
A6	+1	+1	+1	70	1.2
A7	-1	-1	-1	40	1.5
A8	-1	-1	+1	48	1.4

Factor	**low (-1)**	**high (+1)**
X1 (Conc. Of binder) (%)	2	4
X2 (RPM)	800	1200
X3 (Time for spheronization) (min)	2	5

Parameter	**QTPP**
% yield Y1	>60%
pellet size Y2 (mm)	1.2-1.4

Table 2. Solubility of Artemether and Lumefantrine using different molar ratio of β-CD

Molar ratio of β-CD	Solubility of Artemether (mg/ml)	ΔG° of Artemether	Solubility of Lumefantrine (mg/ml)	ΔG° of Lumefantrine
1:0	0.0961	-	0.0583	-
1:1	0.154	-1134.03	0.0691	-0.389
1:2	0.145	-989.4	0.0747	-0.594
1:3	0.143	-956.1	0.0745	-0.561
1:4	0.142	-939.46	0.0737	-0.420

Table 3. Stability study of pellets

Time (days)	Drug content of artemether	Drug content of Lumefantrine	% CDR of Artemether	% CDR of Lumefantrine
7	96.16 ± 0.002	96.78 ± 0.001	96.06± 0.002	96.75±0.003
14	96.14 ± 0.002	96.78±0.001	96.00±0.002	96.75±0.001
21	96.14± 0.002	96.74± 0.003	95.98±0.001	96.70±0.001
30	96.13± 0.001	96.74±0.002	95.98±0.001	96.68±0.002

ACKNOWLEDGEMENT

Authors would like to thank Anand Pharmacy College for furnishing instrumentals facility and resource materials for smooth execution of present study. Also, thank Baroque Pharmaceuticals, Khambhat, for providing Artemether and Lumefantrine as a gift sample.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

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RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

MEDICATED CHOCOLATE CONTAINING CEFPODOXIME PROXETIL: A NOVEL SOLID DOSAGE FORM FOR PAEDIATRIC PATIENT

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ABSTRACT

Medicated chocolate are more preferable for paediatric and diphasic patient. The objective of present study was to develop chocolate formulation of Cefpodoxime proxetil for paediatric dosage form. Solubility and taste of bitter drug was enhance by β-CD complexation method using kneading technique. Prepared medicated chocolate was optimized by 2^3 full factorial design on the basis of drug content (mg), in-vitro drug release (%), hardness (kg/cm^2). Multiple regression analysis was carried out and response surfaces were obtained. Dissolution of the optimized batch was compared with intact and crushed formulation. The quantitative effect of independent variables on dependent variables at different levels was predicted by polynomial equation. Optimized batch F7 showed 95% CDR in 30 min (crushed) and 38% in 80min (intact). Stability study of optimized batch was revealed that no change in chemical and physical characteristics.

SUMMARY

In the present research work, attempt was made to develop medicated chocolate having improved solubility and taste masking by carrying out β-CD complexation. 2^3 factorial design was used to optimize the formulation parameters.

Key words: Cefpodoxime proxetil, medicated chocolate, modified taste masking apparatus.

INTRODUCTION

Pediatric formulations can be quite scientifically challenging to develop due to unique requirements and limitations (*1*). A Different challenges for developing pediatric formulation includes Diversity of children, Taste masking, Stability – physical, chemical, microbial, Achieving global regulatory acceptability, Providing rapid patient access and Accelerated development timelines (*2*).

One of the greatest challenges in pediatric pharmacology has been optimization of oral drug delivery. Crushing tablets may change the rate or extent of drug absorption and hence bioavailability. Cutting tablets, another common practice, may be acceptable for some drugs, however this practice can introduce considerable variability between doses. Extemporaneous oral suspension or solution can also having issue with stability, handling, addition of flavoring agent or use of a different brand, may alter the stability of the final product or the absorption characteristics of the drug. Commercially available oral liquid medications provide a more reliable, ready-to-use preparation for infants and children, but bioequivalence with solid oral dosage forms is still not assured. The problems encountered with currently available formulations highlight the need for the development of new products that are both easy to administer and capable of providing reliable serum drug concentrations and better taste (*3*).

Chocolate is highly sophisticated a versatile food that is combined to create completely different taste and texture sensations. Chocolate is also an anhydrous medium and is therefore resistant to microbial growth and to hydrolysis of water-sensitive active agents. Chocolate is well-suited as a vehicle for delivering active agents in many aspects. For example, the organoleptic characteristics of chocolate are excellent for masking unpleasant flavours associated with some active agents and giving a smooth and creamy texture to compositions of active agents that are otherwise undesirably gritty. Chocolate abundantly contains compounds such as saturated fat, polyphenols, sterols, di and tri terpenes, aliphatic alcohols, and methyl xanthines. Cocoa is the principle ingredient of chocolate and it is rich in polyphenols, particularly in flavan-3-ols such as epicatechins, catechins, and procyanidins. Research studies suggest that a high intake of dietary flavonoids, a subgroup of polyphenols, Chocolate may reduce the risk of coronary heart disease. The antioxidant properties of flavonoids may partially account for the protective effect.

In continuous of above aspect, Cefpodoximeproxetil is a most commonly prescribed medicine in various diseases condition in paediatrics population. Cefpodoximeproxetil having issue of poor solubility, bitter in taste. Bioavailability can be enhancing in present of food also.

MATERIALS AND METHODS

Materials and methods:

Cefdoximeproxetil (Bharat parenteral Pvt. Ltd, Vadodara), Cocca powder (Pruthvi foods Pvt. Ltd, Ahmedabad), Cocca butter (Pruthvi foods Pvt. Ltd, Ahmedabad), Skim Milk Powder (Pruthvi foods Pvt. Ltd, Ahmedabad), Sorbitan Tri Stearate (Savannah Sufactants Limited, Goa), Perlitol 25C (Signet Chemical Corporation Pvt. Ltd, Mumbai), β-Cyclodextrin (Mapro Pharmaceuticals Pvt. Ltd, Mumbai).

Development and Preparation of Medicated chocolate (4)

Preparation of chocolate base (5, 6)

Double boiler was taken for the preparation of chocolate base. Ingredient for chocolate base was weighed and sieved (sieve no: 30). In the mixture of ingredients melted cocca butter was added. With the help of glass rod the mixture was stirred for about 30 min for getting pourable consistency. Careful attention was paid to the chocolate manufacturing process to ensure that the temperature of the mixture was not too high. The pourable chocolate mass was then transferred in to the mould. Then above mixture of chocolate base was cooled up to semisolid consistency.

Preparation of medicated chocolate (4)

Oven was preheated at 50°C. Then chocolate base was melted to obtained free pourable liquid. Cefpodoxime proxetil + β-CD were in chocolate base for uniform mixing. Above mixture was filled

into pre calibrated polycarbonate set mould (Internal cavity=3.5gm) and refrigerated for 15 min till it become solid. Figure 1 and Table no. 1 showing the preparation of medicated chocolate.

Evaluation parameters:

Evaluation of inclusion complex:

Phase solubility (7, 8):

An excess amount of Cefpodoximeproxetil was placed into 25-mL glass flasks containing different concentrations of beta-Cyclodextrin in 5ml distilled water. All flasks were stopper to avoid solvent loss. The content of the suspension was kept for shaking for 24hrs in Orbital Shaking Incubator at 37°C and 100rpm. After attainment of equilibrium of 72 hrs. 1ml of supernatant was withdrawn and filtered through Whatmann filter paper (No.1) and analysed using a UV–visible spectrophotometer (SHIMADZUUV-1650PC (E) 230V, Tokyo, Japan) at 224nm. All solubility measurements were performed in triplicate. The phase solubility study was done to determine the Cefpodoxime with complexing agent β-CD. The phase solubility of a molar inclusion complex of the drug with β-cyclodextrin 1:1, 1:2, 1:3, 1:4. The apparent stability constant for Cefpodoximeproxetil with the β-cyclodextrin complex.

Inclusion complex prepared by kneading method (9):

Solid dispersion was prepared with Cefpodoximeproxetil (CFP) in β-CDin1:1, 1:2, 1:3, and 1:4 molar ratio by kneading method. Accurately weighed quantity of β-CD was mixed with sufficient quantity of water to obtain a smooth and homogenous paste. The drug was slowly added to the paste and the mixture triturated for 1hr. During the process the water content of the paste was empirically adjusted to maintain the consistency of the paste. The paste was dried at 45°C for 48hrs. Pulverized and passed through sieveNo.100. Collected samples were stored in a screw-capped glass vial until use. The inclusion complex was prepared in the following ratios.

Saturation solubility study:

An excess amount of Cefpodoximeproxetil was placed into 25-mL glass flasks containing different concentrations of β-Cyclodextrin in 5ml distilled water. All flasks were stopper to avoid solvent loss. The content of the suspension was kept for shaking for 24hrs in Orbital Shaking Incubator at 37°C and 100rpm. After attainment of equilibrium of 72hrs. 1ml of supernatant was withdrawn and filtered through Whatmann filter paper (No.1) and analysed using a UV–visible spectrophotometer (ShimadzuUV-1650PC (E) 230V, Tokyo, Japan) at 224nm. All solubility measurements were performed in triplicate.

Evaluation of medicated Chocolate

Texture and consistency evaluation:

Texture of the medicated chocolate in terms of stickiness and grittiness was evaluated by visual inspection of the product after mildly rubbing the chocolate sample between two fingers.

Hardness:

Chocolate crushing strength, which was the force required to break the tablet, was measured with a Pfizer tablet hardness tester. The hardness (crushing strength) of three medicated chocolate per batch was determined and mean were taken.

Determination of drug content in the medicated Chocolate:

Drug content of a medicated chocolate was determined by using UV Spectrometer. For Cefpodoximeproxetil cube Medicated chocolate was taken in 25ml beaker and dissolved in 10ml of methanol & sonicated. Then this sonicated mixture was poured in a centrifuge tubes. It was then centrifuged for 15min at 2500rpm. Upper layer having clear liquid containing dissolved drug and solid part of chocolate base was settle down on bottom. This supernatant was then filtered to remove any traces of chocolate remaining in it. Then this liquid sample was analysed using by UV

spectrophotometer against methanol as a blank.

Blooming test:
Fat bloom: Fat bloom was caused by the recrystallization of the fats and/or a migration of a filling fat to the chocolate layer. Storage at a constant temperature was delay the appearance of fat bloom. *Sugar bloom:* Each sample was subjected to treatment cycles compressed (1) 30°Cfor 11hours, (2) temperature shifting for one hour, (3) 18°C for 11hours, and (4) temperature shifting for 1hour. A test chocolate formulation observed, after the step at18°C for 11hours, whether or not blooming has taken place.

Estimation of free and complex drug for the assessment of taste perception:
The inclusion of complex was estimated by dissolving complex in salivary fluid (10ml) for the estimation of free drug and complexed drug by UV spectroscopic method. The release study was conducted by stimulated oral cavity model.

Simulated oral cavity model for Evaluation of extent of Taste-masking study (11):
Taste masking is generally evaluated by testing the product on human volunteers. Bitterness is judged on the basis of the amount of drug released in the salivary juice. If more amount of drug is released in a stipulated time, more bitterness is felt by the volunteer. However, this method (testing on men) suffers two major limitations:

- Approval from ethics committee is to be taken, which is a time consuming & costly approach, and
- The method is subjective (the same results cannot be expected in different population of volunteers).

Another method is use of electronic tongue. This method demands the availability of expensive instrument. Hence, in the present study a simple taste masking test was developed using a set of denture, computer and a web camera. The test was conducted in artificial salivary juice and the basic device was procured from dentist to mimic the area of the oral cavity.

- Design of Simulated oral cavity model apparatus:

The modified apparatus consistmainly3 parts;

1. The Artificial salivary fluid reservoir.
2. The simulated oral cavity.
3. Digital monitoring system.

In Figure no. 2 showing the house modified stimulated oral cavity model. The reservoir contains artificial salivary juice; the liquid was transferred through a tube which control flow rate by regulator. Fluid enters into the oral cavity surrounding the tongue. The simulated oral cavity, which is an adult dental set of lower and upper jaw. It was assembled on the tray which is connected with sampling tube. The artificial spongy tongue was placed at the lower jaw. A tube connected with reservoir was supplying fluid to the tongue at controlled rate, which mimicked the secretion rate of saliva. Previously wetted Whatmann filter paper was kept on spongy tongue to facilitate base for formulation retention. The digital monitoring system consists of web cam was used to capture flow of salivary fluid as well as internal view of oral cavity. Show in Figure no. 3different parts of stimulated oral cavity model. Web cam connected to a computer for recording the process as video images. In this modified apparatus the testing can be initiated by putting the two different formulations i.e. Pure Cefpodoximeproxetil drug powder and Medicated chocolate formulation of Cefpodoximeproxetil over the artificial tongue.

In-vitro Dissolution Study:
The in-vitro dissolution studies was performed using USP Method II (rotating paddle apparatus) in 900ml of distilled water thermostatically maintained at 37±0.5°C at a rotation rate of 50 rpm by USP Dissolution Tester(Electrolab,TDT-08L, Mumbai,India.). Cefpodoximeproxetil and β-cyclodextrin

complex was subjected to dissolution. At predetermined time interval, 5ml of dissolution medium was withdrawn, filtered through Whatmann filter paper (No.1). At each time of withdrawal, 5ml of fresh corresponding medium was replaced into the dissolution flask. Samples were spectrophotometrically assayed for drug content at λ-max (232 nm). Each test was performed in triplicate. From results average Cumulative Percentage Release (%w/v) vs Time (min) graphs was plotted. Optimum ratios of SDs were selected on the bases of dissolution parameters. The in-vitro dissolution of optimum ratios of SDs prepared by various methods was done in three dissolution.

Stability Study:
Stability studies were done according to short term stability study. Optimized formulation was packed in aluminium foil and kept in wide mouth air tight container, kept in a stability chamber at specified temperature (25 ±5°C) and refrigerated condition (0-8°C) for one month. The chemical stability of the formulation was assessed by the estimation of %CDR in the formulation and physical stability was evaluated by monitoring any change in hardness, melting point and organoleptic property.

RESULTS AND DISCUSSION

Drug Identification and Drug-Polymer Compatibility Study

Fourier Transform Infrared Spectroscopy (FTIR):
FTIR studies were carried out for pure Cefpodoximeproxetil and along with inclusion carriers. There was no significant shifting in the physical mixture of all excipients in the final formulation. So, it confirmed that chemical modification of the drug had been taken place due to complexation with β-CD. This indicated that there was significant difference between the internal structures and conformation of these samples at the molecular level due to formation of H-bond during complexation. Thus it can be concluded that there is no any chemical interaction between drug and excipients.

DSC study:
The DSC patterns of pure Cefpodoximeproxetil, kneading mixtures of Cefpodoximeproxetil & β-CD and final formulation were shown below. Pure Cefpodoximeproxetil showed a sharp endotherm at 125.38˚C. There was a change in the melting endotherm of Cefpodoximeproxetil due to complexation with β-CD and it was observed at 221.11°C. Final formulation showed sharp endotherm at 213.81˚C of Cefpodoximeproxetil, and 169.23˚C of mannitol (Pearlitol 25C). There was a slight change in the melting endotherm of final formulation compared to drug + β-CD. This result showed minor of interaction in this formulation so final formulation of Cefpodoximeproxetil medicated chocolate was thermo-dynamically stable.

Solubility Determination of Cefpodoximeproxetil:
Solubility studies were carried out according to Higuchi and Connors (1965). The water solubility of Cefpodoximeproxetil is assessed by preparation of its saturated solution. An excess amount of Cefpodoximeproxetil was placed in 10ml of water and the mixture was stirred for 24hrs. At 37°C. After removing the insoluble substance by filtration, the absorbance of the filtrate was recorded at 224nm in UV at 37°C and the concentration was found by the standard curve of Cefpodoximeproxetil. The results show that the water solubility of the Cefpodoximeproxetil is 0.0014±0.0022 mg/ml.

Phase Solubility study:
The influence of β-CD on solubility of Cefpodoximeproxetil in distilled water at 37°C is prepared in Figure No.4. The solubility of Cefpodoximeproxetil was increased 5.42 fold during complexation with β-CD. The enhancement insolubility might be due to the formation of H-Bond during the complexation and drug molecules are entrapped into the polymer void space.

Optimization:

The 2^3 full factorial design was used for the optimization of Medicated chocolate formulation. A total of 8 experiments was performed for three factors at two levels each. The experimental runs with independent variables and the observed responses for the 8 formulations are shown in Table no.2. Suitable polynomial equation involving the individual main effects and interaction terms was selected based on the estimation of several statistical parameters, such as the multiple correlation coefficient (R^2), adjusted multiple correlation coefficient (adjustedR^2) and the predicted residual sum of squares (PRESS), provided by the Design-Expert software®(8.0.7.1) Trial Version. Suitable model was selected according to the data which is mentioned in the table and validated.

Characterization of medicated Chocolate:

Physicochemical properties of Cefpodoximeproxetil medicated chocolate:

The physical properties of the different medicated chocolate formulations are summarized in Table no. 3 . The drug content was found to be in acceptable range (96.30-99.72%) for all the formulation, indicated uniform drug distribution of drug. In determination of medicated chocolate weight, all the formulations were found to be within the Pharmacopeia limits (i.e.5%). Thickness value of the medicated chocolate vary depending upon flow characteristics (i.e.viscosity of the formulations).The thickness of medicated chocolate were slightly in varied in all prepared formulations because moderately deviation in die cavity. Friability values of all formulation batches from F1 to F8 were less than 1% small values in friability imply much less friability during transportation.

In-vitro drug release study:

The drug release profile of the Cefpodoximeproxetil medicated chocolate from the Intact and crushed formulations comparison was shown in Figure no.5 and 6 and the data shown in Table no.4. The drug release study from the medicated chocolate containing Cocoa powder, milk solids, Pearlitol 25C, Stevia and Cocoa butter clearly indicated that the release of the drug was influenced by the concentration of cocoa butter upto 30min. Table no.4 shows %CDR in 30min of all the Batches which had %CDR was found to be up to 95%. To study of the drug release mechanism of Cefpodoximeproxetil from medicated Chocolate, the released were compared with intact and partially crushed formulation. Comparison of release profile between two different forms shown in Figure No. 7**.** On the basis of drug release comparison from the below graph. It was concluded that partially crushed formulation shows better drug release within 30min then the intact formulation.

Influence of critical process parameters on quality of product:

Hardness:

A) Contour Plot

Two-dimensional contour plot is presented in Figure no. 8 . To show the relationship between the dependent and independent variables for Y1 response i.e. Hardness if, X_2 increased from−1to+1level Hardness was increased and similar if X_3 was decreased, the %yield value was found to be increased.

Melting Point:

A) Contour Plot

For the response Y_2 i.e. Melting point, the interaction between factors X_1 and X2 can be elucidated by using contour plot as illustrated in Figure no.9 and respectively. When the factor X1 was changed from −1 to +1 level increased, Melting point was increased. Similarly if X2 was increased, Melting point value was increased.

Percentage CDR:

A) Contour Plot

For the response Y3 i.e. %CDR, the interaction between factors X_2 and X_3 can be elucidated by using contour and response surface plot as illustrated in Figure no. 10 . As the factor X2 increased from −1 to +1 level, %CDR was found to decrease. Similarly when the factor X3 was increased, %CDR was

found to increase.

Optimization of parameters and Validation of 2^3 factorial design

After generating the polynomial equations relating the dependent and independent variables were optimized for the responses. The optimum values for the variables were obtained by graphical and numerical analysis using the Design-Expert software which is based on criterion of desirability. Percentage error was measured so as to find out the procedure. As shown in Table no.5, the observed value was found quite closer to the predicted value. Linearity observed between actual and predicted values of response variable shows excepted ability of RSM as the percentage error was obtained less than 2% so, formulation was validated by experimental design. Check point batches obtained from extensive grid search. All check point batches having predicted %CDR 91.43, 91.26, 91.45 and actual %CDR 90.71, 90.21, 90.33 respectively, for batch 1, 2, 3. Also that shows less error. The check point 1 was formulated using a cocoa butter 25gm, concentration of Pearlitol 25C 20gm and blending time was 40min getting predicted %CDR 91.26%. For the check point 2 has concentration of Cocca butter 21.02gm, concentration of Pearlitol 25C, 19.89gm and blending time was 40.17 min getting predicted %CDR 91.43%. For the check point 3 has concentration of coca butter 24.52gm, concentration of Pearlitol 25C 19.59gm and blending time was 40.98min getting predicted %CDR 91.45%. All check points having %CDR in an appropriate range and %Error was found to be less than 2%.

Evaluation of an extent of taste masking:

The complexation efficacy was determined on the basis of different solubility of complex drug. The amount of complex drug was found 0.0076mg/ml solubility by kneading method and pure drug having 0.0014mg/ml solubility during phase solubility**.** Principle ingredient like cocca butter, Pearlitol 25C and stevia has also ability to mask the taste perception of prepared medicated chocolate. The dosage form was kept on the surface of the wet artificial tongue for a period of 10Sec. This time was similar to the time used in studies on human volunteer. Finally the amount of dissolved drug was determined after care ful removal of the test sample (pure CP and CP medicated Chocolate formulation). The amount of drug dissolved in the artificial salivary juice (in the wet tongue) was determined by spectroscopic method at 232nm. During the taste assessment study the free drug concentration was found to be a very less amount. So, on the basis of free drug concentration. It was predicted that the taste of optimized batch was masked with the help of β-CD and sweetening agent.

Evaluation of bloom test:

Optimized batch passed the bloom test after evaluated at different condition. Concentration of Emulsifier played important role to retarded sugar blooming and fat blooming in the optimized batch. Sorbitan tristearate (STS) used as emulsifier helped to retard sugar and fat bloom in the formulation at different temperature. Table no. 6 showing the data of bloom test of optimize batch.

Short-term Stability study of optimized batch:

Short term stability study was done on the optimized for 1 month at room temperature and refrigerating temperature. The sample was evaluated for hardness, in vitro drug release, and melting point. Values obtained for parameters were found to be within ± 5% of initial values. Stability study did not reveal any degradation of the Cefpodoximeproxetil and also no changes in release profile of the optimized formulation. Table no. 5 showing the data of short-term stability study of optimize batch.

CONCLUSION

Systemic formulation enabled us to develop a formulation as per the current requirements of paediatric dosage form. As per 2^3Factorial Design it is concluded that Batch containing 0.2gm cocoa powder, 0.4gm milk solid, 1.8gm cocoa butter, 2.5gm Pearlitol, 0.2% stevia and 0.3% STS was optimized. On the basis of Solubility study with β-CD was ensured, the basic objective of taste masking the bitter taste of Cefpodoximeproxetil was observed. Since it is cost effective, has better business potential in the formulation.

FIGURES



Fig.1. Preparation of medicated chocolate

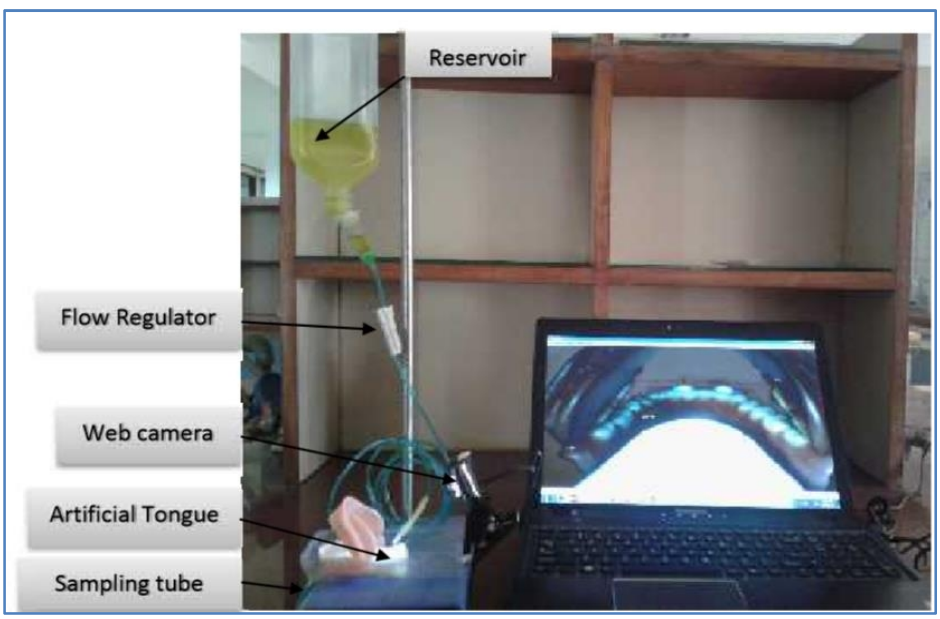


Fig.2. In house modified stimulated oral cavity model



Fig.3. Different parts of simulated oral cavity model (A)Front view of model (B) Artificial salivary media reservoir (C) regulator

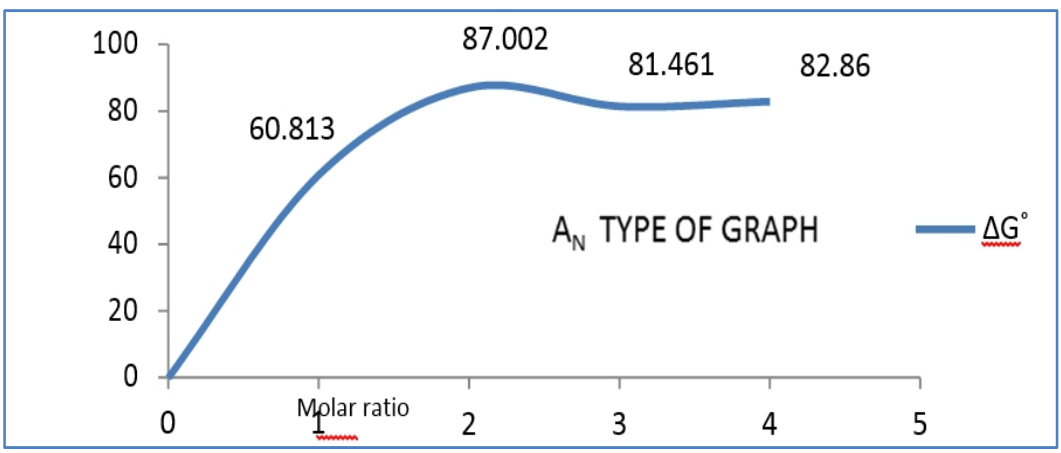


Fig.4. Phase solubility diagram for Cefpodoximeproxetil in the presence of β-CD

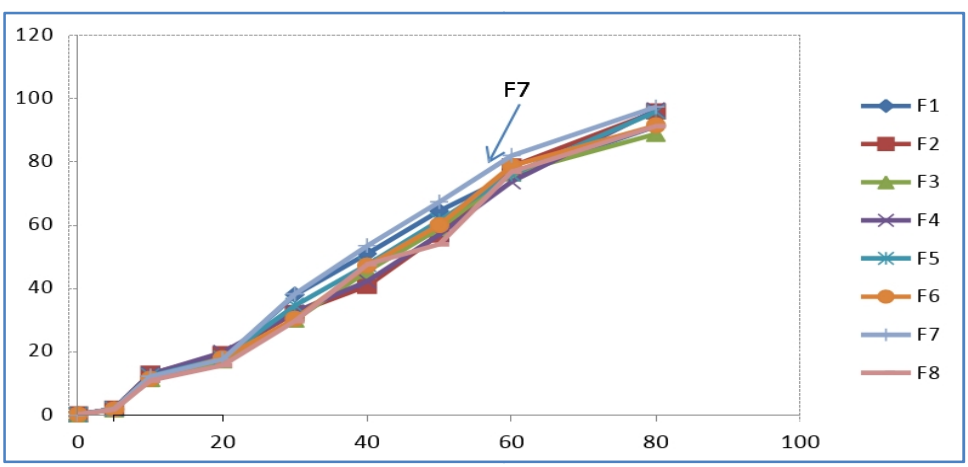


Fig.5. In-vitro Drug Release of intact Formulations

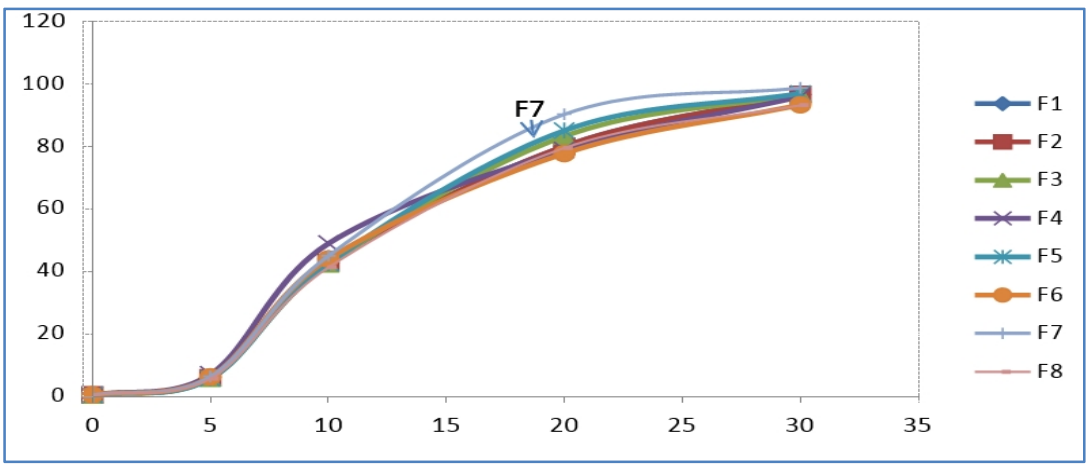


Fig.6. In-vitro Drug Release of partially crushed formulation

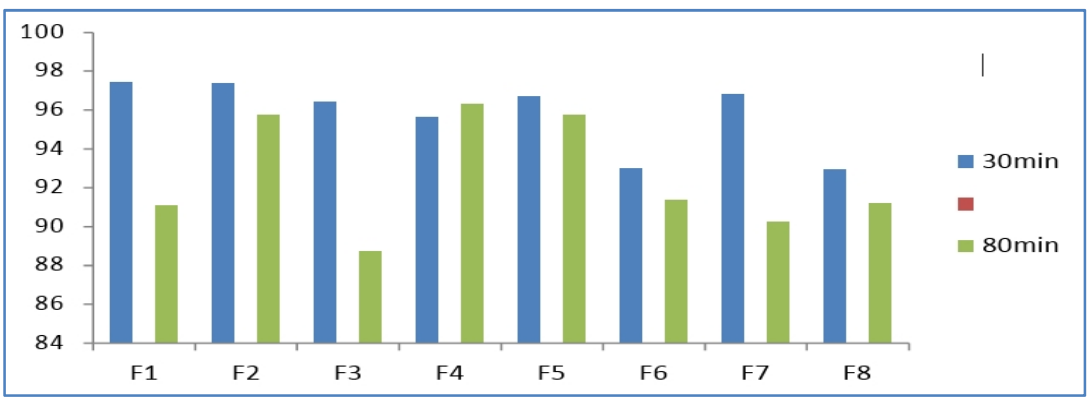


Fig.7. Comparative In-vitro release study of intact and partially crushed formulation

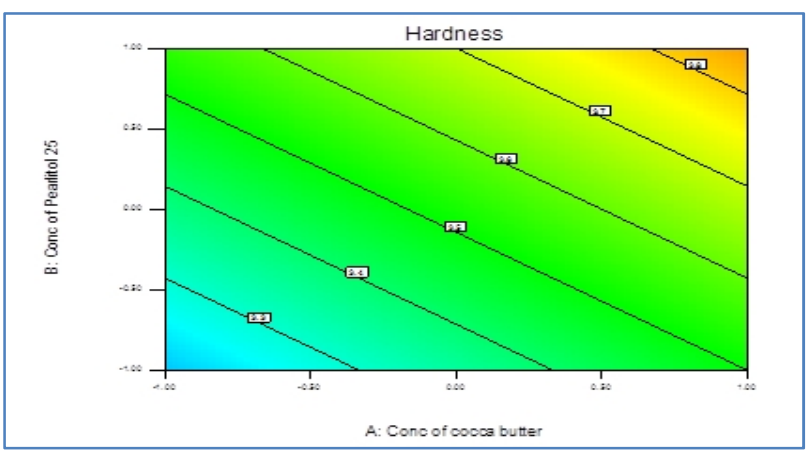


Fig.8. Contour plot for Hardness

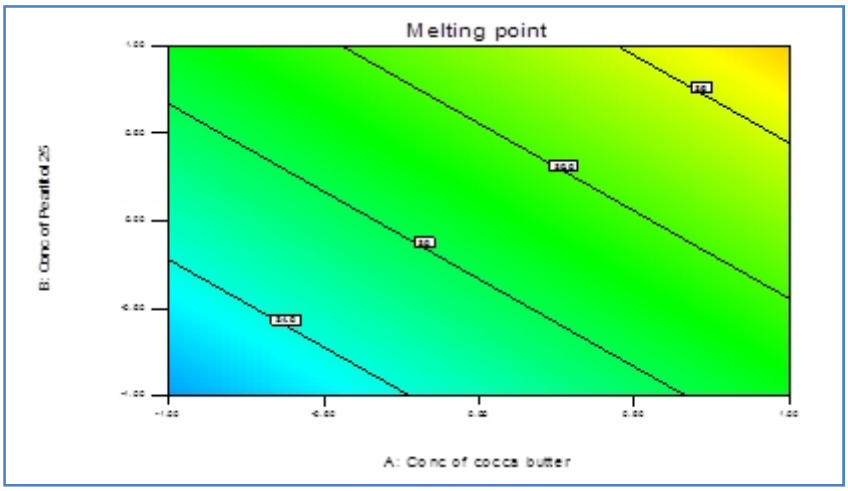


Fig.9. Contour plot for Melting point

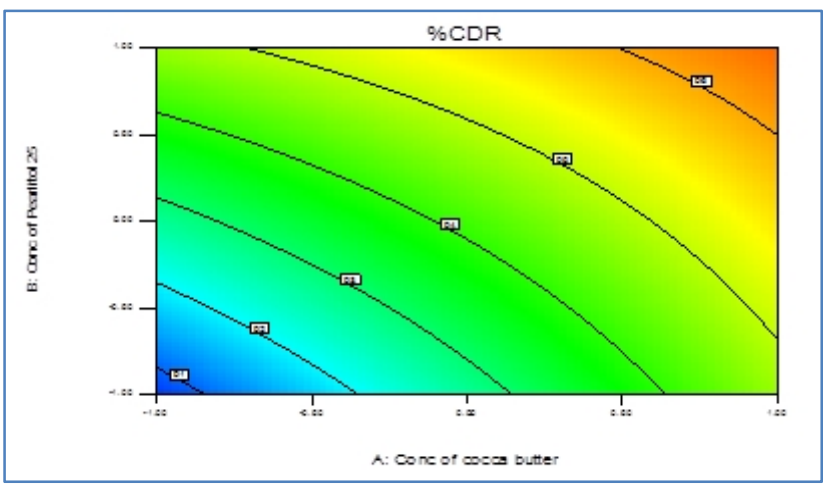


Fig.10. Contour plot for %CDR

TABLES

Table 1. Composition of Medicated chocolate Of Cefpodoximeproxetil

Sr no.	Ingredients	Amount
1	Cefpodoximeproxetil	100 mg
2	β-Cyclodextrin	200 mg
3	Cocca powder	0.2 gm
4	Milk solid	0.4 gm
5	Cocca butter	1.8 gm
6	Perlitol 25C	2.5 gm
7	Stevia sugar	0.011 gm
8	Sorbitan Tri Stearate (STS)	0.016 gm
9	Methyl Paraben	0.027 ml

Table 2. Process variables and observed responses from the randomized run in 2^3fullfactorial design

Batch	X1	X2	X3	Hardness (Kg/cm2)	Melting point (°C)	%CDR (%) (t3)
F1	-1	-1	-1	3	33.5	91.097
F2	-1	1	-1	3.5	35	95.787
F3	1	1	-1	3.6	35	95.758
F4	-1	-1	1	3.3	34	90.265
F5	1	-1	1	3.6	36	96.339
F6	1	-1	-1	3.5	35	93.123
F7	1	1	1	4	37	96.507
F8	-1	1	1	3.7	36	93.731

X_1=Conc of Cocca Butter, X_2=Conc of Pearlitol 25C, X_3=Blending Time

Table 3. Physicochemical properties of Cefpodoximeproxetil medicated chocolate

Batch No.	Drug Content	Weight Variation(%)	Friability (%)	Thickness (mm)
F1	98.52±0.02	3.37±0.06	0.55±0.011	1.1±0.02
F2	98.62±0.01	3.37±0.07	0.52±0.012	1.0±0.04
F3	99.72±0.01	3.32±0.05	0.50±0.01	1.1±0.04
F4	97.70±0.01	3.39±0.06	0.40±0.04	1.0±0.03
F5	98.23±0.08	3.33±0.06	0.41±0.012	1.0±0.04
F6	98.63±0.01	3.37±0.02	0.30±0.019	1.2±0.05
F7	98.65±0.01	3.4±0.016	0.27±0.01	1.1±0.03
F8	96.30±0.01	3.39±0.02	0.29±0.011	1.1±0.04

Table 4. In-vitro Drug release of Cefpodoximeproxetil medicated chocolate from both intact and crushed formulation

%CDR								
Time	F1		F2		F3		F4	
	Intact	Crushed	Intact	Crushed	Intact	Crushed	Intact	Crushed
0	0	0	0	0	0	0	0	0
5	1.778±0.08	5.522±0.06	1.858±0.05	5.522±0.03	1.592±0.04	5.336±0.11	1.805±0.082	6.663±0.04
10	12.032±0.04	42.386±0.17	12.899±0.01	42.386±0.04	11.159±0.02	41.991±0.03	12.710±0.11	48.569±0.015
20	17.415±0.0	79.725±0.05	19.032±0.11	79.725±0.12	17.026±0.011	82.973±0.01	19.622±0.01	78.548±0.0

	1							4
30	37.817±0.07	96.351±0.05	32.094±0.04	96.351±0.03	29.941±0.04	96.401±0.04	32.359±0.13	95.657±0.03
40	50.855±0.04	-	40.678±0.05	-	45.368±0.01	-	42.153±0.05	-
50	64.274±0.09	-	56.899±0.03	-	58.799±0.01	-	56.899±0.04	-
60	75.639±0.05	-	76.43±0.04	-	77.224±0.011	-	76.563±0.03	-
80	91.097±0.03	-	90.787±0.01	-	88.731±0.05	-	91.332±0.02	-

Time	F5		F6		F7		F8	
	Intact	Crushed	Intact	Crushed	Intact	Crushed	Intact	Crushed
0	0	0	0	0	0	0	0	0
5	1.778±0.05	5.336±0.04	1.592±0.011	5.787±0.012	1.778±0.011	5.920±0.01	1.566±0.03	5.256±0.01
10	12.191±0.14	42.256±0.011	11.292±0.023	43.595±0.011	12.032±0.12	44.598±0.11	10.77±0.05	40.938±0.04
20	17.796±0.12	84.793±0.012	17.764±0.03	77.454±0.03	17.469±0.01	79.991±0.12	15.53±0.11	79.235±0.11
30	34.483±0.04	96.725±0.011	30.206±0.06	93.014±0.06	38.112±0.06	98.489±0.03	29.58±0.14	92.917±0.05
40	47.374±0.03	-	47.056±0.09	-	53.359±0.06	-	47.52±0.08	-
50	61.244±0.11	-	59.899±0.14	-	67.236±0.016	-	53.79±0.014	-
60	75.935±0.011	-	77.604±0.012	-	75.882±0.01	-	74.72±0.014	-
80	90.758±0.012	-	91.392±0.016	-	91.507±0.05	-	91.18±0.03	-

Table 5. Stability study data of optimized batch

Sr. no.	Stability Interval of Factorial Responses					
	Condition	Testing (Day)	Hardness	Melting point	%CDR	Appearance
1	Room temperature	Initial	3.5	34	95.00	Acceptable
		15 days	3.4	35	95.30	Acceptable
		30 days	3.4	36	94.90	Acceptable
2	Refrigerated condition (2°C-8°C)	Initial	3.4	32	95.40	Acceptable
		15 days	3.5	33	95.29	Acceptable
		30 days	3.6	34	94.50	Acceptable

Table 6. Blooming test data of optimized batch

Sr.	Storage Condition	Evaluated Condition	Fat Bloom	Sugar Bloom
1	Refrigerated Condition (2-8°C) for 24 hr.	At Room Temperature	No Blooming	No Blooming
2	Room Temperature (25°C) for 24 hr.	At Refrigerated Condition	No Blooming	No Blooming

ACKNOWLEDGEMENT
Authors would like to thank Anand Pharmacy College for providing instrumentals facility and resource materials for smooth execution of present study. Also, thank Bharat parenteral Pvt. Ltd, Vadodara, for providing Cefpodoxime proxetil as a gift sample.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

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Development and validation of Spectrophotmetric methods for determination of Diclofenac sodium, Paracetamol and Chlorzoxazone in tablet dosage form

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ABSTRACT

The spectrophotometric methods namely successive ratio-derivative and Double divisor methods were proposed for simultaneous determination of ternary mixtures of Diclofenac sodium, Paracetamol and Chlorzoxazone, without preceding separation steps. Method I is based on successive derivative of ratio spectra in two steps whereas method II is based on convolution of double divisor ratio spectra, obtained by dividing the absorption spectrum of the ternary mixture by a standard spectrum of two of the three compounds in the mixture. All the drugs exhibited good linearity over the reported concentration range with acceptable correlation coefficient. The methods were validated according to ICH guidelines for evaluation of accuracy, repeatability, reproducibility, sensitivity showing acceptable % RSD of less than 2. The proposed methods demonstrated that these are simple, rapid, accurate and precise methods and can be applied for routine analysis in quality control laboratories eliminating the need of prior separation of the pharmaceutical mixtures.

SUMMARY

Newly developed and validated Spectrophotometric methods were applied for simultaneous estimation of Diclofenac sodium, Paracetamol and Chlorzoxazone in marketed formulation.

Keywords: Successive ratio-derivative method, Double divisor method, UV Spectrophotometry, Diclofenac sodium, Paracetamol, Chlorzoxazone, validation

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INTRODUCTION

Diclofenac sodium (DICLO, 2-(2,6-dichloroanilino)phenyl]acetate)), non-steroidal anti-inflammatory drug having antipyretic and analgesic actions, is used to treat pain, dysmenorrhea, ocular inflammation, osteoarthritis, rheumatoid arthritis and ankylosing spondylitis. Paracetamol (PCM, N-acetyl-p-aminophenol), an analgesic and antipyretic drug, is used to treat fever, headache, and other minor ache and pain. Chlorzoxazone (CLR, 5-chloro-2-(3H)-benzoxazolone), centrally acting muscle relaxant, is used for relieving painful muscle spasms occurring in musculoskeletal and neuromuscular disorders, acts on the spinal cord by depressing reflexes. (*1,2*)

Official methods were reported individually for DICLO, PCM and CLR. Literature review reports several non-official methods including HPLC*(3-6*), HPTLC(*7-10*) and UV spectrophotometry(*11-14*) to determine DICLO, PCM and CLR in individually or in combination with other drugs. In addition, HPLC and HPTLC methods were reported for simultaneous estimation of DICLO, PCM and CLR (*2,15,16*). However, the literature shows that development of successive ratio derivative and double divisor UV spectrophotometric method is lacking for the combination of said drugs. In context to this, research work focusses on the development of above methods to determine the ternary mixture of DICLO, PCM and CLR in bulk and tablet formulation.

The use of spectrophotometric techniques using wavelength transformation and mathematical algorithms has brought an easy, quick and novel way of methodology to determine the analytes in complex samples without prior separation. Additionally, several sophisticated instrumental techniques like HPLC, capillary electrophoresis etc. can be used to determine or separate the ternary mixtures but its expensive and time consuming (*17*). Hence, application of mathematical algorithm has proved to be an official way for determination of drugs in ternary or quaternary mixture. Hence, this research work aims to develop and

validate successive ratio derivative and double divisor UV spectrophotometric method for simultaneous estimation of DICLO, PCM and CLR ternary mixture in bulk and tablet dosage form.

MATERIALS AND METHODS

Instruments

A double beam UV-Visible Spectrophotometer (Shimadzu, Japan) model UV-1800 connected with computer system with a quartz cell of 1 cm path length and fixed slit width (2nm), UV probe software, Shimadzu version 2.34; Electronic analytical balance (AUX-220), Shimadzu were used in the study.

Chemicals and materials

Paracetamol (PCM), Diclofenac sodium (DICLO) and Chlorzoxazone (CLR) were supplied as a gratis sample from Jenburkt Pharmaceuticals Limited, Bhavanagar, India. All solvents and chemicals, of analytical grade, were purchased from Merck Specialities Pvt. Ltd., India. Marketed formulation, Powergesic MR tablets (Jenburkt Pharmaceuticals Ltd., Bhavanagar) was procured from local market.

Procedure

Preparation of standard solutions

Accurately weighed 10 mg of DICLO, PCM, and CLR were transferred, dissolved and diluted up to the mark into three separate 10 ml volumetric flask with 0.1 N NaOH to prepare a 1000 µg/ml stock solution. Working standard solution of 100 µg/ml of each drugs were prepared with appropriate dilution with 0.1 N NaOH. From this, appropriate aliquots were taken to prepare final concentration of each drugs DICLO (1-20 µg/ml), PCM (4-22 µg/ml) and CLR (4-22 µg/ml). Additionally, mixed standard stock solution was prepared using 10 mg of each drugs into 10 ml volumetric flask and diluted with 0.1 N NaOH. From this solution, appropriate dilutions were made to prepare mixed working standard solutions of drugs of 100 µg/ml.

Successive ratio derivative method (method I)

The Zero order (D_0) UV spectra of different concentrations for mixture of DICLO, PCM, and CLR were recorded in the range of 200-400 nm. For calibration curve of DICLO, the zero order UV absorption spectra of the solutions of different concentration of mixture recorded in the calibration range were divided by standard spectrum of CLR (10 µg/ml) for obtaining first ratio spectra, then converted to first order derivative. Further, the stored spectra of standard solution of PCM was divided by standard solution of CLR to record ratio spectra that was further converted mathematically to first order

derivative. Finally, the outputs of first order derivative were further divided to obtain second ratio spectra and the output was further transformed to first order derivative. The minimum or maximum of the first derivative second ratio spectra with respect to wavelength, the calibration graph of DICLO was constructed. Similar procedure was used for the other drugs; PCM and CLR by using divisor concentration of 8 µg/ml DICLO, 4 µg/ml PCM, and 10 µg/ml CLR. For the determination of the concentration of drugs, various concentrations as divisor were also tested like 4, 16, 22 µg/ml for DICLO, 8, 20, 24 µg/ml for PCM as well as 10, 20, 24 µg/ml CLR, but only selected concentrations shown results in range and maintain the linearity. (*18*)

Double divisor ratio spectra method (Method II)

For DICLO, the stored spectra of ternary mixture were divided by sum of standard spectrum of PCM (4 µg/ml) and CLR (10 µg/ml) to record ratio spectra, followed by first derivative transformation of these vectors with respect to wavelength. For constructing calibration curve, the maximum or minimum of the first derivative was recorded. Similar procedure was followed for the other two components; PCM and CLR by using divisor concentration of 8 µg/ml DICLO, 4 µg/ml PCM, and 10 µg/ml CLR. (*19*)

Validation of method

Various analytical method parameters like linearity, limit of detection (LOD), limit of quantification (LOQ), precision and accuracy according to ICH guidelines Q2(R1) were performed to validate both the methods.(*20*) In method I, linear relationship between absorbance and concentration were evaluated over the concentration range of DICLO (2 – 20 µg/ml), PCM (8-22 µg/ml) and CLR (4-22 µg/ml) by making five replicate measurements. Similarly, calibration curve were constructed by plotting the absorbance versus the concentration range of 1-10 µg/ml for DICLO as well as 4-22 µg/ml for PCM and CLR, in five replicates. From the standard deviation of the response (σ) and slope of calibration curve (S) of each drug, LOD and LOQ of the developed method were calculated using formula,

$$LOD = 3.3 \times \sigma/s,$$

$$LOQ = 10 \times \sigma/s,$$

Intraday and interday precision were performed in three replicates using 10, 14 and 18 µg/ml concentration for each drugs in Method I. Whereas in method II, Intraday and Interday Precision was evaluated in three replicates in the concentration range for PCM and CLR (10, 14 and 18 µg/ml) and for DICLO (4, 6 and 8 µg/ml). Moreover, accuracy was ascertained by performing standard addition method for recovery study at three concentration levels (80%, 100% and 120%) in triplicate. For PCM and CLR,

three different concentrations of standards i.e. 8, 10 and 12 µg/ml were spiked to the formulation (10 µg/ml). Similarly, three different concentrations of DICLO standards i.e. 0.8, 1 and 1.2 µg/ml were spiked to the formulation (1µg/ml).

Analysis of marketed formulation

Twenty tablets (Powergesic MR tablets) were ground in fine powder form and powder equivalent to PCM, CLR (10 mg each) and DICLO (1 mg) accurately weighed and 15 ml of 0.1 N NaOH was added in 100 ml volumetric flask and sonicated for 15 minutes. Then, to obtain sample stock solution, the mixture was diluted up to the mark with 0.1 N NaOH, mixed and filtered. For the determination, stock solution was diluted to volume methanol to obtain a final concentration 10 µg/ml of PCM, 10 µg/ml of CLR and 1 µg/ml of DICLO. Procedure similar to standard mixture was followed in UV probe software for recording and scanning of the spectra of DICLO, PCM, and CLR and repeating analysis in triplicate.

RESULTS AND DISCUSSION

Development and Optimization of methods

Zero-order absorption (D_0) overlay spectra of DICLO, PCM, and CLR (Fig. 2) shows considerable overlapping of bands and therefore, zero order absorption spectra was converted to first order and second order derivative spectra. Both overlay spectra of DICLO, PCM, and CLR shows interference of other drug at zero-cross over point (ZCPs) of each selected drug, revealing that their simultaneous determination is difficult in their combined dosage form by first order and second order derivative spectrophotometric method.

In context to said problems, as described above, two methods successive ratio derivative (method I) and double divisor method (method II) are proposed. The linear regression parameters like intercept, corelation coefficient and slope were tested to chek the effect of divisor concentration. The selected divisor concentrations shows good results for these parametrs. The wavelength selected for estimation of DICLO (239.21 nm), PCM (312.60 nm), and CLR (222.41 nm) gave good correlation coefficient for method I (Fig. 3a, 3b and 3c). Similarly, acceptable correlation coefficient was obtained for the wavelength selected for estimation of DICLO (315.2 nm), PCM (314.2 nm) and CLR (320.4 nm) using method II respectively (Fig. 4a, 4b and 4c).

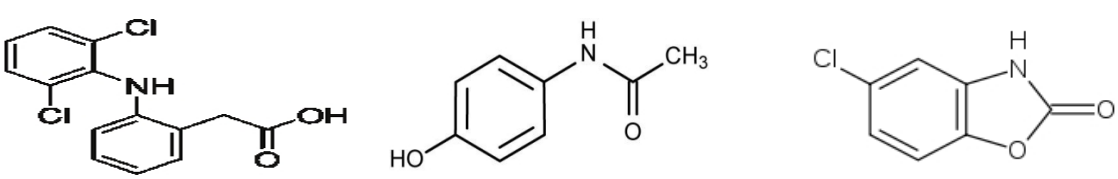
Validation of methods

For proposed methods, method validation parameters were performed according to the ICH guidelines. Good linearity is evident from the high value of the correlation coefficient (Table 1). The LOD and LOQ of developed method shows high sensitivity of both the methods. From the results of precision study of both methods, %RSD was found to be less than 2 indicating reproducibility of both the developed methods as evident from Table 1. Accuracy was assessed by the standard addition method at three concentration levels in tablet formulation, showed mean percentage recovery at all three levels in the range of 98.26% to 102.99%, suggesting suitability of method to perform routine drug analysis (Table 2). Percentage amount found for all the three drugs from marketed formulation were within the range of 99.94-100.85% and 99.66-101.49% for method I and II respectively, shows zero interference by excipients and good accuracy of the proposed methods (Table 3).

CONCLUSION

Two novel and simple spectrophotometric methods namely successive ratio derivative and double divisor ratio derivative were developed for the determination of PCM, DICLO and CLR in ternary mixture and tablet formulation using 0.1 N NaOH as a solvent without prior separation. From the results of validation parameters, it is proved that the method is simple, precise, reliable, sensitive and accurate. These methods showed good recovery for all the three drugs and hence can be used in the routine quality control for simultaneous determination of ternary mixture and pharmaceutical formulation.

FIGURES



A B C

Fig. 1: Chemical structures of **A**: Diclofenac sodium **B**: Paracetamol and **C**: Chlorzoxazone

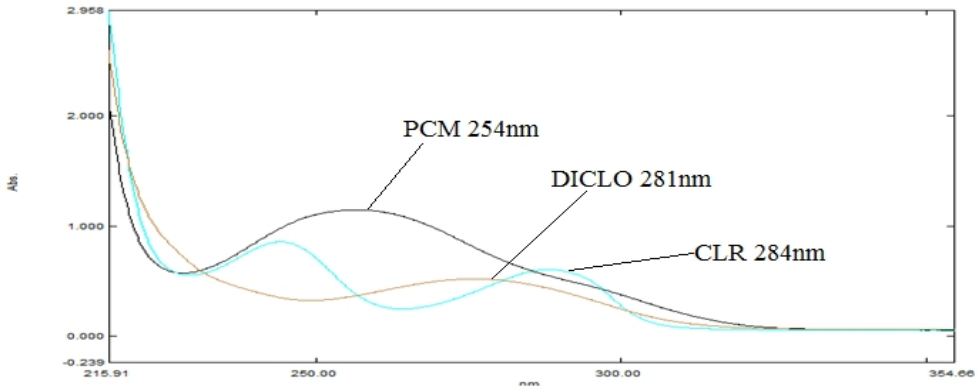
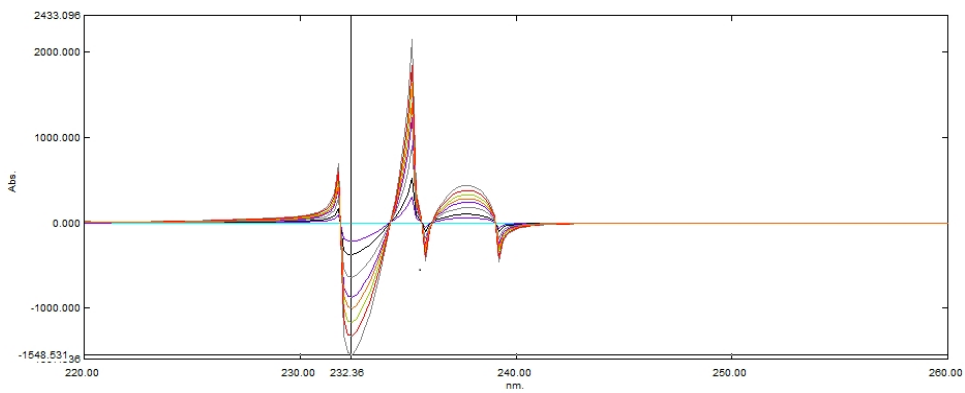
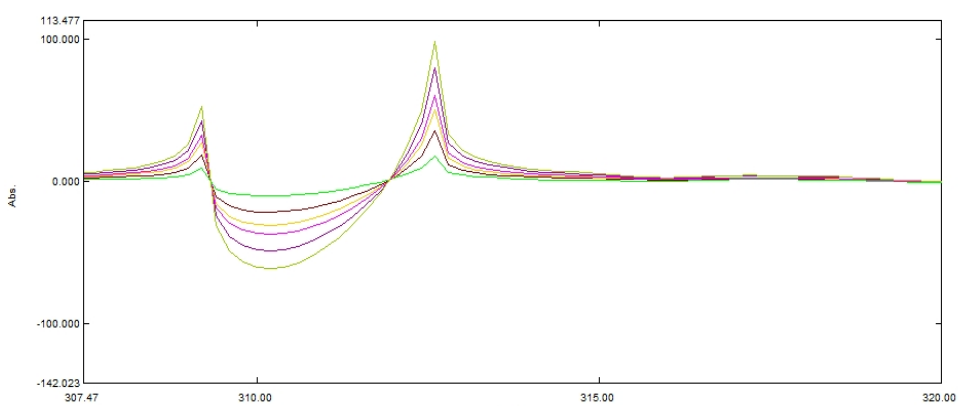


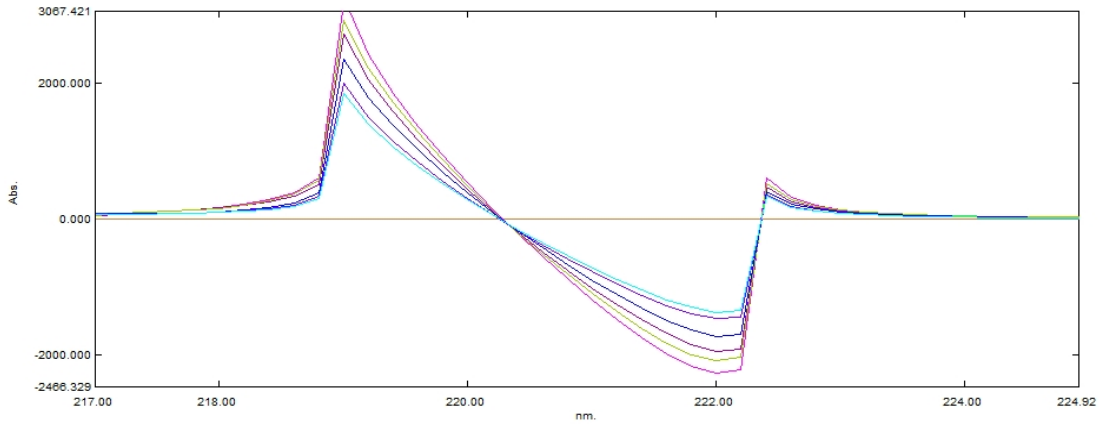
Fig. 2: Zero order Overlay spectra of DICLO (10 µg/ml), PCM (10 µg/ml) and CLR (10 µg/ml)



(a)

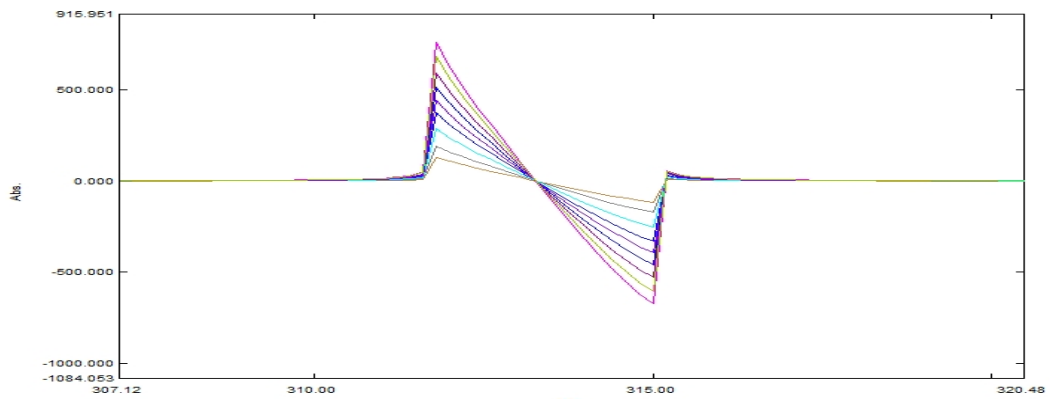


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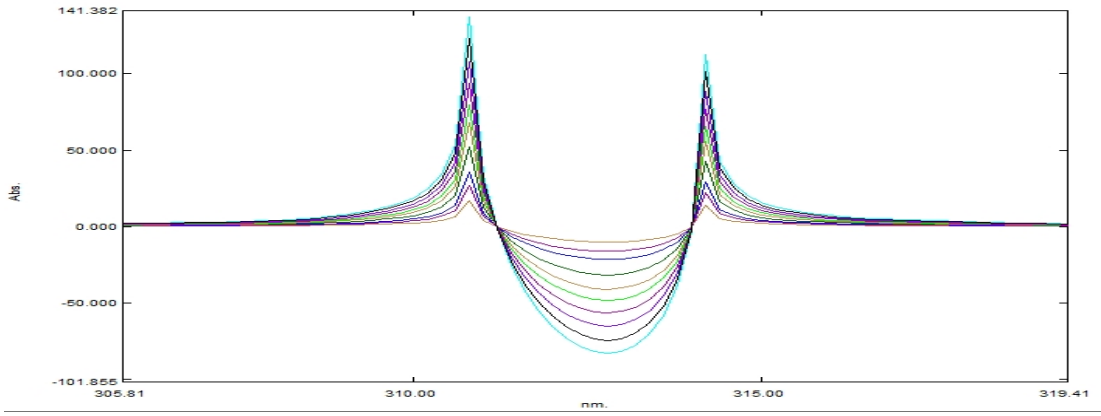


(c)

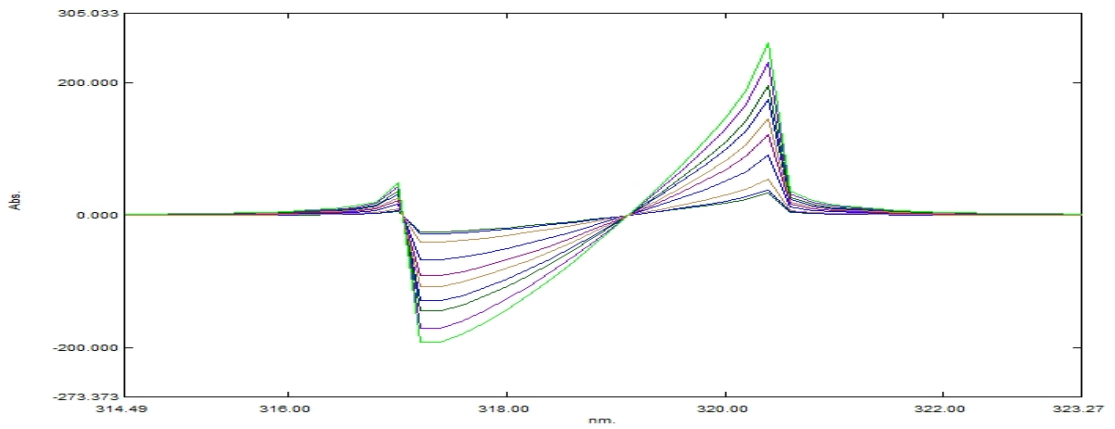
Fig. 3: **(A)** First order derivative (D_1) of second ratio spectrum of DICLO (2-20 µg/ml); **(B)** First order derivative (D_1) of second ratio spectrum of PCM (8-22 µg/ml); **(C)** First order derivative (D_1) of second ratio spectrum of CLR (4-22 µg/ml)



(a)



(b)



(c)

Fig. 4: **(A)** First-order derivative (D_1) Ratio spectra of DICLO (1-10 µg/ml); **(B)** First order derivative (D_1) ratio spectra of PCM (4-22 µg/ml); **(C)** First order derivative (D_1) ratio spectra of CLR (4-22 µg/ml)

TABLES

Table 1: Validation parameters for DICLO, PCM and CLR by proposed methods

Parameters	**Method I**			**Method II**		
	DICLO	**PCM**	**CLR**	**DICLO**	**PCM**	**CLR**
Linearity[a]						
Wavelength (nm)	239.21	312.60	222.41	315.20	314.20	320.40
Calibration range(µg/ml)	2 – 20	8 - 22	4 – 22	1 – 10	4 – 22	4 – 22

Correlation coefficient (r^2)	0.9914	0.9961	0.9937	0.9982	0.9974	0.9989
Regration equation	Y = 108.4012x – 259.7802	Y = 7.4776x – 58.322	Y = 22.2471x + 62.0407	Y= 2.9991x + 2.6328	Y = 5.238x – 8.9416	Y = 13.013x – 36. 884
SD of slope	0.0901	0.5830	0.3014	0.0595	0.1004	0.2271
SD of intercept	1.5562	0.0441	0.0368	0.0097	0.0192	0.0093
Sensitivity						
LOD (µg/ml)	0.0473	0.0025	0.0011	0.0122	0.04	0.0028
LOQ (µg/ml)	0.1435	0.0078	0.0034	0.0371	0.1212	0.0086
Precision[b] (%RSD)						
Intraday Precision	0.0041-0.0905	0.3261-0.6994	0.0187-0.4796	0.048-0.07	0.012-0.206	0.026-0.108
Interday precision	0.0324-0.1586	0.7575-1.083	0.0763-0.2791	0.2870-0.6460	0.2050-0.7750	0.1030-0.2810

a = Average of five determinations, b = Three concentrations/three replicates, % RSD = Relative Standard Deviation.

Table 2 : Recovery study at three concentration levels of DICLO, PCM and CLR.

Concentration of Standard added	Mean % Recovery[a] ± SD			%RSD		
	DICLO	PCM	CLR	DICLO	PCM	CLR
Method I						
80	100.20 ± 0.0061	99.04 ± 0.0368	100.04 ± 0.0305	0.7622	0.4602	0.3819
100	100.5 ± 0.2980	100.40 ± 0.0395	100.26 ± 0.0057	0.2980	0.3907	0.0571
120	99.78 ± 0.0021	100.09 ± 0.0254	100.05 ± 0.251	0.1781	0.2121	0.2095
Method II						
80	100.75 ± 0.017	100.37 ± 0.039	100.7 ± 0.005	0.94	0.22	0.547
100	100.18 ± 0.018	100.22 ± 0.107	100.17 ± 0.005	0.94	0.53	0.528
120	100.41 ± 0.0091	100.20 ± 0.060	100.10 ± 0.003	0.44	0.27	0.364

a = Mean of three determination at three concentration level of standard, % RSD = Relative Standard Deviation

Table 3 : Analysis of DICLO, PCM and CLR in marketed formulation

	Method I			Method II		
	DICLO	PCM	CLR	DICLO	PCM	CLR
Label claim (mg)	50	500	500	50	500	500
Mean %Assay[a]	100.310	100.140	100.410	100.120	100.430	100.420
%RSD[a]	0.501	0.389	0.595	0.539	0.402	0.402

a = average of three determination, % RSD = Relative Standard Deviation

ACKNOWLEDGEMENT

The authors are grateful to Jenburkt Pharmaceuticals Limited, Bhavanagar, India for providing gratis sample of Paracetamol, Diclofenac sodium and Chlorzoxazone.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

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Development and Validation of RP-HPLC Method for Analysis of Olmesartan, Amlodipine and Hydrochlorothiazide in Biological Fluid

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ABSTRACT

Sensitive and selective high performance liquid chromatography method was developed and validated from olmesartan, amlodipine and hydrochlorothiazide in biological fluid after eliminating protein by protein precipitation method. The samples were analysed using Phenomenex C18 column and mobile phase consists of 10Mm potassium dihydrogen phosphate Buffer: methanol (30:70 v/v) pH 3. Concentration range of 25–4000 ng/mL for olmesartan, 10 - 4000 ng/ml for Amlodipine and hydrochlorothiazide. Thus the present research gives an accurate method for the determination of all the three drugs in biological fluid.

SUMMARY

Proposed study describes a sensitive and selective RP-HPLC method for determination of OLM, AMLO and HTZ in human plasma was developed and validated. Developed method was optimized for extraction procedure, mobile phase composition, flow rate, etc. Chromatographic run time was 9.0 min and the average retention times of OLM, AMLO and HTZ were 5.01, 6.427 and 3.5 min, respectively.The developed method was validated in human plasma and linearity range of 25 to 4000 ng/ml For OLM, 10 to 400 ng/ml for AMLO and HTZ which is at very sensitive level. The method was validated as per ICH guideline.

Keywords: Amlodipine besylate, hydrochlorothiazide, Olmesartan, RP-HPLC method, Bioanalytical Method

INTRODUCTION

Cardiovascular diseases are the disorders of heart and blood vessels.[1]. The major risk factors underlying cardiovascular diseases are low density lipoprotein (LDL) cholesterol, hypertension, platelet aggregation, diabetes, smoking and obesity, which are primarily caused by unhealthy diet and physical inactivity [2]. Angiotensin receptor blockers, angiotensin converting enzyme inhibitors, Calcium channel blockers, and diuretics are commonly used for management of hypertension [3,4] . The effective therapy for moderate to severe hypertension requires multiple antihypertensive agents from different classes of drugs. Many dual and triple combinations are available for management of hypertension and cardiovascular diseases. One of the triple combinations amongst various available and widely used is olmesartan medoxomil, amlodipine besylate and hydrochlorthiazide.

Olmesartan (OLM) chemically (5-methyl-2-oxo-2H-1,3-dioxol-4-yl)methyl-4-(2-hydroxypropan-2-yl)-2-propyl-1-({4-[2-(2H-1, 2, 3, 4-tetrazol-5-yl)phenyl)phenyl}methyl-1H-imidazole-5-carboxylate as shown in [Figure 1]a. OLM is an angiotensin II receptor (type AT1) antagonist [3,5] . OLM has not yet been officially described in any pharmacopoeia. Amlodipine besylate (AML), a beta-blocker, chemically 4-[2-hydroxy-3-[(1-methylethyl)amino]propoxy] benzeneacetamide as shown in [Figure 2][3,4,6] . Hydrochlorothiazide (HCTZ), a thiazide diuretic, is chemically known as 6-chloro-3,4-dihydro-2H-1, 2, 4-benzothiadiazine-7-sulfonamide-1,1-dioxide [Figure 3]. [3,4,6] .

Literature reviewed reports several analytical methods including HPLC [7-,9] , a stability- indicating HPLC [10-14] , HPTLC [15,16] and UV-spectrophotometry [17-19] for the determination of OLM, AML and HCTZ either single or in combination with other drugs. Many analytical methods have been reported individually for AML [20,21] and HCTZ [20-22] . However, development of a high performance liquid chromatographic (HPLC) method for simultaneous estimation of OLM, AML and HCTZ in biological fluid has not been reported that why this combination was selected for research.

Recently, HPLC is widely employed for the quantification of drugs because of low maintenance cost, lower analysis time. It facilitates automated application of sample. Moreover, it is flexible enough to analyze different kinds of samples [23]. Hence, this research paper describes the development of HPLC method for estimation of OLM, AML and HCTZ in biological fluid

MATERIALS AND METHODS

RP-HPLC instrument (Shimadzu, SPD 20AV, Japan,) equipped with a UV-Visible detector, Phenomenex (Torrance, CA) C_{18} column (250 mm × 4.6 mm id, 5 µm particle size) and LC-solution software. analytical balance (shimadzu, zapan), Ultra sonic cleaner (Life care eq. PVT. LTD, India)
Corning volumetric flasks, pipettes of borosilicate glass. Digital pH meter (systonic).Pharmaceutical grade of Amlodipine besylate (AML), olmesartan (OLM) and Hydrochlorothiazide (HTZ) were supplied as a gift samples from Torrent Pharmaceutical Ltd, Gujarat (India) with 99.98 % purity. The pharmaceutical formulations containing 20 mg OLM, 5 mg AML and 12.5 mgHTZ of brand OLMAT – AMH tablet was procured from the local pharmacy(india), HPLC grade acetonitrile (Merck Chemicals Ltd., India). HPLC grade Water (Merck Chemicals Ltd., India). Human Plasma (Nathani Blood Bank, Rajkot),Trichloroacetic acid (Rankem Ltd, India), Potassium dihydrogen phosphate (Rankem Ltd, new Delhi, India), Nylon 47 mm - 0.45 µm membrane filter (Gelman Laboratory, India),Whatman filter paper no. 41. (Whatman International Ltd., England).

Preparation of Mobile Phase

Preparation of 10mM potassium dihydrogen phosphate Buffer in 250 ml volumetric flask, take 0.34 gm of potassium dihydrogen phosphate and dissolve it in 50 ml HPLC grade water, and make up to volume with HPLC grade water. Mobile Phase is prepared by taking 30 ml of 10Mm potassium dihydrogen phosphate Buffer and 70 ml of methanol. Adjust pH 3 of final mixture by glacial acetic acid as required.

Preparation of OLM, AML and HTZ Standard Stock Solutions

Accurately weighed OLM (10 mg), Amlodipine besylate (13.86 mg, which is equivalent to AML 10 mg) and HTZ (10 mg) were transferred to a 100 mL separate volumetric flask and dissolved. Diluted upto the mark with methanol, this solution are further diluted 10 time so that standard solution having concentrations of OLM (10 µg/mL), AML (10 µg/mL) and HTZ (10 µg/mL).

Sample preparation

In Spike Plasma protein was precipitate with Trichloroacetic acid (sample: TCA, 9:1, v/v) and centrifuged at 10000 rpm for 10 min. The upper layer was filtered through a 0.45-µm Millipore syringe filter. 20 µL of the supernatant was injected for analysis.

Determination of analytical wavelength

The standard solution of OLM, AMLO and HTZ were injected above mention chromatographic condition. Detection was carried out at different wavelength and best response was achieved at 230 nm with PDA detector. So three drugs were detected at this analytical wavelength.

Method validation [23]

Calibration curves were constructed by plotting concentrations Vs peak areas of OLM, AMLO and HTZ). 5 ml of human plasma was transferred into a series of centrifuge tube and spike with different concentration of OLM, AMLO and HTZ) to give final concentration of 25–4000 ng/mL for OLM, 10 - 4000 ng/ml for AMLO and HTZ. TCA (5%) was added to the spike Plasma (sample: TCA, 9:1, v/v) for protein precipitation, vortexed and centrifuged at 10000 rpm for 10 min. The upper layer was filtered through a 0.45-µm Millipore syringe filter. 20 µL of the supernatant were injected.

For specificity and selectivity, Three different lots of plasma were taken, depreoteinized it with 5 % TCA solution (plasma: 5 % TCA solution in proportion of 9:1 %v/v) and being analyzed in HPLC for any interfering peak at retention time of OLM, AMLO and HTZ were compared separately with sample plasma (at LOQ level) at retention time of individual drug separately and % of peak area response are calculated.

Sensitivity study was carried out by using Five different plasma sample were taken, deproteinized it with 5 % TCA solution (plasma: 5 % TCA solution in proportion of 9:1 %v/v) and spiked with LLOQ level of OLM (25 ng/ml), OF AMLO and HTZ (10 ng/ml) concentration subsequently extracted in solvent and than analyzed in HPLC and % CV was calculated for both drug individually which described sensitivity of method to analyte.

For determination of accuracy, plasma sample was spiked with 3 different concentrations of OLM, AMLO and HTZ (100 ng/ml, 1500ng/ml and 3000 ng/ml) with 5 times determination OLM, AMLO and HTZ from plasma and subsequent analysis in HPLC. Peak area appropriate to that extracted sample was recorded and concentration was calculated by placing peak area value in calibration curve equation $Y = mx + c$, which gives concentration to that extracted OLM, AMLO and HTZ and % nominal concentration was calculated by comparing peak area of sample to that standard calibration curve peak area for each drug.

Precision was studied by within run precision involves single analytical run. The three different concentration (100 ng/ml, 1500 ng/ml and 3000 ng/ml) of OLM, AMLO and HTZ were spiked with plasma sample and being subjected to protein precipitation and were being analyzed by HPLC and % CV was calculated.Between run precision involves analytical run by time, i.e., samples are analyzed in different days with freshly preparation of all solution on each day and procedure was carried out for

different 5 days and samples are analyzed in each day. The three different concentration (100 ng/ml, 1500 ng/ml and 3000 ng/ml) of OLM, AMLO and HTZ were spiked with plasma sample

For Recovery mean peak area of 3 different spiked samples with freshly prepared un-extracted samples with same concentration. It involves spiking of 3 different concentrations of OLM, AMLO and HTZ (100 ng/ml, 1500 ng/ml and 3000 ng/ml) than being subjected to protein precipitation. Analyzed in HPLC and peak area obtained for each solution for each drug were compared with freshly prepared un-extracted samples with same concentration for each drug and for each solution.

Matrix Effect was performed to ensure the effect of matrix throughout the application of the method; three different lots of Blanks plasma were spiked with OLM, AMLO and HTZ at two different concentration levels (100 ng/ml, 3000 ng/ml for OLM, AMLO and HTZ). And calculate % Nominal concentration of the sample

Stability of the developed method was checked by Stock Solution Stability, Process Stability and Bench Top Stability**.** Stock solution stability was determined by comparing the peak areas of freshly prepared solutions with stability samples. Main stock of OLM, AMLO and HTZ stock solution (4 µg/ml) was freshly prepared and aliquots of OLM, AMLO and HTZ were kept at 2-8°C for 5 days (stability samples). Area of stability samples and freshly prepared samples were compared to determine mean % change during stability period.Process stability of OLM, AMLO and HTZ after 6 hours at room temperature OLM, AMLO and HTZ (100 ng/ml and 3000 ng/ml) sample was prepared and kept for 6 hours at room temperature that sample was analyzed after 6 hours along with freshly prepared sample. Concentrations were calculated to determine mean % change during stability period. For Bench Top Stability**,** Two different samples of OLM, AMLO and HTZ (100 ng/ml and 3000 ng/ml) were spiked in human plasma and kept at room temperature for 13 hours, extracted in methanol separately and were analyzed along with freshly prepared 100 ng/ml and 3000 ng/ml samples. Concentration was calculated to determine % change during stability period.

RESULTS AND DISCUSSION

HPLC method is considering the efficient; an attempt has been made to develop a precise, simple, rapid, accurate, and economic method for the estimation of OLM, AMLO and HTZ in the biological method. Thus, the method enables to the quantification of OLM, AMLO and HTZ. Simplicity of sample preparation and the low costs of reagents is advantage of HPLC method. For the HPLC method development mobile phase consists of methanol: Buffer (70: 30, v/v) at pH 3 with flow rate of 1.0 mL/min. The selected optimum wavelength was 230 nm. Under these chromatographic conditions described, OLM, AMLO and HTZ peaks were well resolved and plasma endogenous components did not give any interfering peaks. The average retention times of OLM, AMLO and HTZ were 5.02, 6.43 and 3.5 min, respectively. Chromatogram obtained after drug spick in plasma is given fig. 4.

In plasma was linear over the range 25-4000 for OLM and 10-4,000 ng/mL AML and HTZ. The relative standard deviation (RSD) values of the slope were equal to or better than 5%.This procedure was repeated 6 times and 6 calibration curves for each drug are taken and mean R2 value was calculated and than those concentration range for each drug was selected for this bioanalytical method

For specificity and selectivity**,** Three different lots of plasma were taken, depreoteinized it with 5 % TCA solution (plasma:5 % TCA solution in proportion of 9:1 %v/v) and blank plasma were analyzed in HPLC for any interfering peak at retention time of OLM, AMLO and HTZ were compared with sample plasma (at LLOQ level) Absence of interfering components is accepted because the response is ≤ 20% of the lower limit of quantification (LLOQ) for OLM, AMLO and HTZ (Table- 2) respectively at their retention time individually.

Sensitivity of this method was determine by Five different plasma sample those spiked with LLOQ level of OLM (25 ng/ml), AML and, HTZ (10 ng/ml) after protein precipitation and than analyzed in HPLC and % CV was found to be 4.74 for OLM, 3.21 for AMLO and 3.27 for HTZ. The % nominal concentration for OLM is ranged from 88.22 to 93.84 (Table No. 3), the % nominal concentration for LLOQ samples of AMLO is ranged from 85.5 to 92.2 and the % nominal concentration for LLOQ samples of HTZ is ranged from 85.3 to 93.2 (Table No. 3) which describes good sensitivity of method to analytes.

For determination of accuracy, plasma sample was spiked with 3 different concentrations of OLM, AMLO and HTZ (100 ng/ml, 1500ng/ml and 3000 ng/ml) with 5 times determination OLM, AMLO and HTZ from plasma and subsequent analysis in HPLC. Mean concentration was calculated and % nominal concentration of 3 different concentration OLM (100 ng/ml, 1500ng/ml and 3000 ng/ml) were found to be 90.3%, 90.32% and 89.72% respectively (Table No. 4), for AMLO (100 ng/ml, 1500ng/ml and 3000 ng/ml)were found to be 90.69%, 88.93%, 92.4% respectively (Table No. 4) and for HTZ (100 ng/ml, 1500ng/ml and 3000 ng/ml) 88.51% ,92.11%, 90.11% respectively (Table No. 4) and finally % CV was determined for each drug. Hence, the mean value should be within 15% of the actual value.

Within run precision involves single analytical run. Three different concentration (100 ng/ml, 1500 ng/ml and 3000 ng/ml) of OLM, AMLO and HTZ were spiked with plasma sample and were being subjected to protein precipitation and were being analyzed by HPLC and % CV was calculated and % CV were found to be 1.51, 1.88 and 1.81 for three different concentration of OLM respectively, 1.41, 1.18 and 1.38 for three different concentration of AML respectively AND 1.04, 1.75, 169 for three different concentration of HTZ respectively. Within rug precision study which was carried out in single day analytical run.

Between run precision was carried out for 5 different days and samples are analyzed in each day. The three different concentration (100 ng/ml, 1500 ng/ml and 3000 ng/ml) of OLM, AMLO and HTZ were spiked with plasma sample and % CV were found to be 2.56, 1.70 and 1.99 for three different concentration of OLM respectively, 2.02, 2.07 and 2.66 for three different concentration of AMLO respectively and 2.45,2.86, 2.5 for three different concentration of HTZ respectively which described that this method is precise for between-run precision study which was carried out for 5 different days with same procedure in each day with freshly preparation of all solutions.

The mean % recovery for OLM (100 ng/ml, 1500 ng/ml and 3000 ng/ml) are found to be 89.51%, 88.63% and 89.08% respectively.(Table No. 5). The % CV of Extracted samples of for OLM (100 ng/ml, 1500 ng/ml and 3000 ng/ml) are 1.09, 2.43 and 2.09 respectively. The % CV of recovery across 3 different levels for OLM (100 ng/ml, 1500 ng/ml and 3000 ng/ml) is 0.49 which is presented in Table No. 5. The mean % recovery for AML (100 ng/ml, 1500 ng/ml and 3000 ng/ml) are found to be 89.56%, 88.96% and 92.23% respectively.(Table No. 5). The % CV of Extracted samples of for AMLO (100 ng/ml, 1500 ng/ml and 3000 ng/ml) are 1.49, 1.73 and 1.99 respectively. The % CV of recovery across 3 different levels for AMLO (100 ng/ml, 1500 ng/ml and 3000 ng/ml) is 1.92 which is presented in Table No. 5. The mean % recovery for HTZ (100 ng/ml, 1500 ng/ml and 3000 ng/ml) are found to be 90.14%, 89.91% and 88.91% respectively.(Table No. 5). The % CV of Extracted samples of for HTZ (100 ng/ml, 1500 ng/ml and 3000 ng/ml) are 1.89, 1.43 and 1.05 respectively. The % CV of recovery across 3 different levels for HTZ (100 ng/ml, 1500 ng/ml and 3000 ng/ml) is 0.65 which is presented in Table No. 5.

Matrix Effect study by blanks Plasma from three lots was spiked with OLM, AMLO and HTZ at two different concentration levels. The % Nominal concentration of the OLM for 100 ng/ml and 3000 ng/ml was 90.24%, 90.06 % respectively. For AMLO, % nominal concentration found to be 92.08 % and 90.69% for 100 mg/ml and 3000 ng/ml concentration respectively. For HTZ, % nominal concentration found to be 87.09% and 89.06% for 100 mg/ml and 3000 ng/ml concentration respectively.

Stock solution stability was determined by comparing the peak areas of freshly Prepared solutions with stability samples.OLM, AMLO and HTZ stock solution was stable at 2-8°C for 5 days with mean % change of 2.73, 2.98, 3.01 which did not showed any degradation peak of OLM, AMLO and HTZ.

For process stability OLM, AMLO and HTZ (100 ng/ml and 3000 ng/ml) sample was prepared and kept for 6 hours at room temperature that sample was analyzed after 6 hours along with freshly prepared sample. OLM is found to be stable at 100 ng/ml and 3000 ng/ml samples for 6 hours at room temperature with mean % change of 1.46 and 2.14. AMLO is found to be stable at 100 ng/ml and 3000 ng/ml samples for 6 hours at room temperature with mean % change of 1.83 and 2.44. HTZ is found to be stable at 100 ng/ml and 3000 ng/ml samples for 6 hours at room temperature with mean % change of 1.99 and 2.81

For bench top stability, Two different samples of OLM, AMLO and HTZ (100 ng/ml and 3000 ng/ml) were spiked in human plasma and kept at room temperature for 13 hours and analyzed with freshly prepared 100 ng/ml and 3000 ng/ml samples. OLM, AMLO and HTZ were found to be stable at 100 ng/ml and 3000 ng/ml samples for 13 hours at room temperature with mean % change of 1.93 and 2.04, 1.81 and 2.42, 1.29 and 2.58 respectively

CONCLUSION

Proposed study describes a sensitive and selective RP-HPLC method for determination of OLM, AMLO and HTZ in human plasma was developed and validated. Developed method was optimized for extraction procedure, mobile phase composition, flow rate, etc. Chromatographic run time was 9.0 min and the average retention times of OLM, AMLO and HTZ were 5.01, 6.427 and 3.5 min, respectively.The developed method was validated in human plasma and linearity range of 25 to 4000 ng/ml For OLM, 10 to 400 ng/ml for AMLO and HTZ which is at very sensitive level. The method was validated as per USFDA guideline.

FIGURES

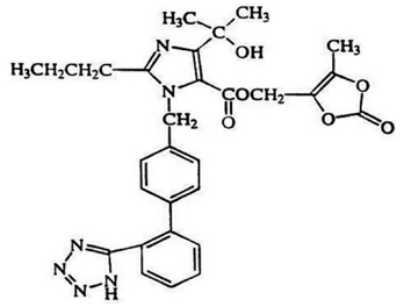


Fig 1. Chemical structures of Olmesartan medoxomil,

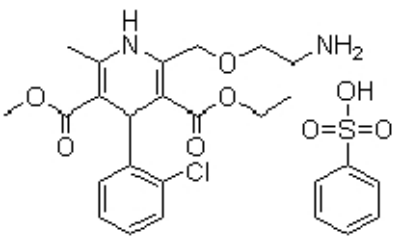


Fig 2. Chemical structures of amlodipine besylate

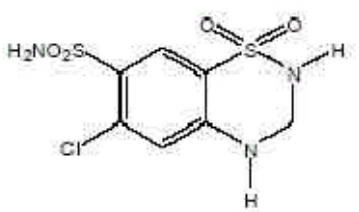


Fig 3. Chemical structures of hydrochlorthiazide.

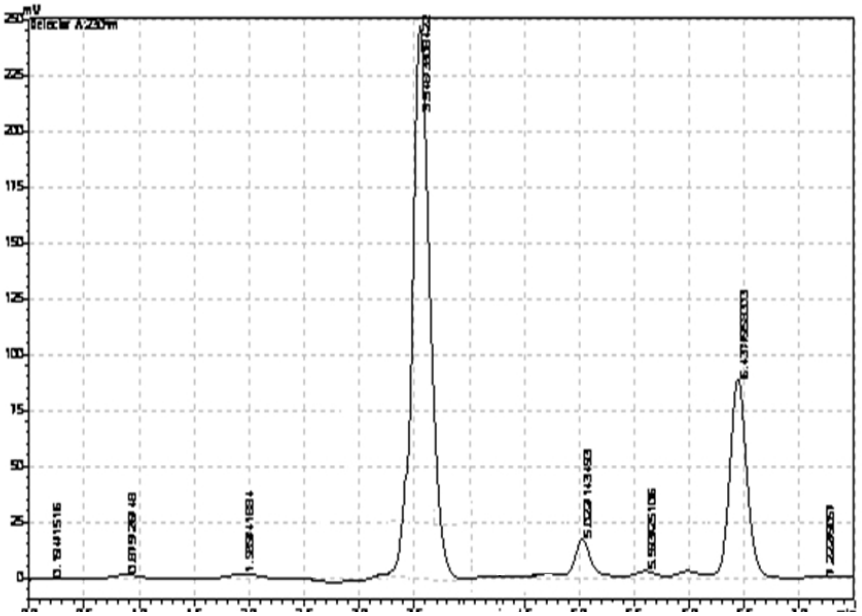


Fig 4. Chromatogram of OLM, AMLO and HTZ

TABLES

TABLE 1. SUMMARY OF CALIBRATION CURVE PARAMETERS OF OLM, AMLO AND HTZ.

Parameters	**Bioanalytical method**		
	OLM	**AML**	**HTZ**
Concentration range (ng/mL)	25-4000	10-4000	10-4000
Slope (n=6)	5.98±0.057	80.53±0.89	929.85±9.38
Intercept(n=6)	2416.67±80.36	56434.33±1005.36	96912.5± 972.24
Correlation coefficient(n=6)	0.994± 0.0016	0.997±0.00040	0.994±0.00081

n = number of determinations

TABLE 2. SPECIFICITY & SELECTIVITY OF BLANK HUMAN PLASMA OF OLM (25 ng/ml), AMLO (10 ng/ml) and HTZ (10 ng/ml)

Sr. no.	Sample Name	Area			% Of Area		
		OLM	AMLO	HTZ	OLM	AMLO	HTZ
1	**BLANK PLASMA (LOT NO :P106-06)**	111	0.00	0.00	0.92	0.00	0.00
2	**LLOQ (LOT NO :P106-06)**	11958	45784	846802			
3	**BLANK PLASMA (LOT NO :P106-07)**	0.00	0.00	145	0.00	0.00	0.02
4	**LLOQ (LOT NO :P106-07)**	11888	45564	845892			
5	**BLANK PLASMA (LOT NO :P106-08)**	0.00	135	578	0.00	0.0029	0.068
6	**LLOQ (LOT NO :P106-08)**	11678	45964	844882			

TABLE 3. SENSITIVITY OF OLM, AMLO AND HTZ AT LLOQ

Sr. No.	OLM			AMLO			HTZ		
	Conc. (ng/ml)	**Cal. Conc. (ng/ml)**	**% Nominal conc.**	**Conc. (ng/ml)**	**Cal. Conc. (ng/ml)**	**% Nominal conc.**	**Conc. (ng/ml)**	**Cal. Conc. (ng/ml)**	**% Nominal conc.**
1	25.00	22.17	88.69	10.00	8.60	86	10.00	9.32	93.2
2	25.00	20.55	82.22	10.00	8.55	85.5	10.00	9.13	91.3
3	25.00	23.46	93.84	10.00	8.96	89.6	10.00	8.92	89.2
4	25.00	22.56	90.25	10.00	9.01	90.1	10.00	9.02	90.2
5	25.00	22.17	88.69	10.00	9.22	92.2	10.00	8.53	85.3
Mean		22.18	88.738		8.86	88.68		8.98	89.84
SD		1.05	4.2073		0.28	2.85		0.29	2.93
%CV		4.74	4.74126		3.21	3.21		3.27	3.27

SD = standard deviation
CV = Coefficient of Variation

TABLE 4. ACCURACY OF OLM, AMLO AND HTZ

Sr. No.		OLM			AMLO			HTZ		
	Amount of Standard Added (ng/ml)	**Conc. Recovered (ng/ml)**	**% Nominal Conc.**	**%CV**	**Conc. Recovered (ng/ml)**	**% Nominal Conc.**	**%CV**	**Conc. Recovered (ng/ml)**	**% Nominal Conc.**	**%CV**
1	100	91.23	91.23		90.45	90.45	2.06	87.23	87.23	
	100	92.56	92.56	1.86	91.25	91.25		86.45	86.45	3.72
	100	89.23	89.23		89.47	89.47		85.17	85.17	
	100	88.25	88.25		93.56	93.56		90.46	90.46	
	100	90.23	90.23		88.72	88.72		93.28	93.28	
2	1500	1381.95	92.13		1318.95	87.93	1.67	1444.2	96.28	
	1500	1398.75	93.25		1328.4	88.56		1414.2	94.28	
	1500	1356.75	90.45	2.70	1308.6	87.24		1351.8	90.12	0.22
	1500	1323.6	88.24		1352.85	90.19		1371.9	91.46	
	1500	1313.4	87.56		1361.4	90.76		1326.3	88.42	
3	3000	2688	89.6		2738.4	91.28	1.95	2751.6	91.72	
	3000	2614.2	87.14	2.26	2771.1	92.37		2617.5	87.25	1.93
	3000	2782.2	92.74		2704.2	90.14		2697.9	89.93	
	3000	2705.1	90.17		2803.8	93.46		2712.3	90.41	
	3000	2668.8	88.96		2842.5	94.75		2737.5	91.25	

CV is Coefficient of Variation

TABLE 5. VARIABILITY ACROSS 3 DIFFERENT LEVELS OF OLM, AMLO AND HTZ

3 DIFF. LEVELS	**% Recovery**		
	OLM	**AMLO**	**HTZ**
HQC (100 ng/ml)	89.51	89.56	90.14
MQC (1500 ng/ml)	88.63	88.96	89.91
LQC (3000 ng/ml)	89.08	92.23	88.91
Mean	89.07	90.25	89.65
SD	0.44	1.74	0.65
%CV	0.49	1.92	0.72

SD = standard deviation
CV = Coefficient of Variation

TABLE 6. MATRIX EFFECT FOR OLM, AMLO AND HTZ

Sr. No.	**Sample ID**	**Nominal Conc. ng/ml)**	**% Nominal Conc for OLM**	**% Nominal Conc for AMLO)**	**% Nominal Conc for HTZ**
1	LOT NO :P106-06	100	88.24	91.28	87.99
2	LOT NO :P106-06	3000	88.95	92.43	90.73
3	LOT NO :P106-07	100	91.25	92.48	86.64
4	LOT NO :P106-07	3000	90.48	90.48	87.32
5	LOT NO :P106-08	100	91.25	92.48	86.64
6	LOT NO :P106-08	3000	90.75	89.16	89.14
7	Mean % Nominal Concentration (100 ng/ml)		90.24	92.08	87.09
8	Mean % Nominal Concentration (3000 ng/ml)		90.06	90.69	89.06

ACKNOWLEDGEMENT

The authors express their gratitude to Torrent Pharmaceutical Ltd, Gujarat (India) for providing gift sample of standard olmesartan medoxomil, amlodipine besylate and hydrochlorothiazide.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

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EVALUATION OF DIURETIC POTENTIAL OF EXTRACTS OF *HYGROPHILA SALICIFOLIA*, WHOLE HERB IN RATS

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ABSTRACT

Hygrophila salicifolia (Family: Acanthaceae) is an erect herb, nearly glabrous, mainly whole herb used as raw material in medicine. The present investigation was targeted to evaluate diuretic potential of different successive extracts of *Hygrophila salicifolia* (Family: Acanthaceae). Different extracts of *Hygrophila salicifolia* were given at doses 300mg/kg & 500mg/kg orally to albino rats. Furosemide (20mg/kg) was taken as reference standard. Diuretic index, Saliuretic index, Urine volume, Lipchitz value, sodium-potassium excretion and Na^+/k^+ ratio were used to evaluate diuretic potential of extracts. In methanolic extract, urine volume & sodium excretion were remarkably increased as compared to control group. We can deduce that *Hygrophila salicifolia* methanolic extract offered significant diuretic effect than other successive extracts when compared to standard drug. The present research imparts quantitative base to explain diuretic potential of methanolic extract of *Hygrophila salicifolia* than other extracts.

SUMMARY

Methanolic extract of *Hygrophila salicifolia* possesses the potential diuretic activity.

Keywords: *Hygrophila salicifolia,* Diuretic index, Lipchitz value, Saliuretic index

INTRODUCTION

Drug-induced diuresis is advantageous in many life frightening ailments such as hypertension, congestive cardiac failure, pregnancy, kidney failure and liver dysfunctioning (*1)*. Most of diuretic drugs show side effects such as weakness, impotence and fatigue (*2)*. Hence there is urgent necessity to search safe diuretic drug which is potassium sparing and causes excretion of sodium (*1-3*). Herbs are the important source of phytoconstituents with potential therapeutic action.WHO has estimated that 80% of the world population depends on herbal medicines (*4,5*). *Hygrophila salicifolia*, is an erect or ascending herb belongs to the family acantheceae. It is bestowed with many medicinal uses in traditional systems of medicine including ayurveda. Seeds and leaves used as poultice on inflammatory swellings. Leaves are strongly diuretic. The plant is found in moist and marshy places throughout the greater part of india. Stems up to 3 ft. long, more or less quadrangular, rooting at the lower nodes, leaves subsessile, linear-lanceolate; flowers pale purple in dense axillary whorls, capsule oblong, seeds many, ovoid, compressed, mucilaginous, hairy. The leaves are eaten as pot- herb. They contain 8% ash rich in potassium and are strongly diuretic. In Malaya, the leaves are used as poulticing swellings. The seeds swell into a gelatinous shining mass with water and used in java, in poultices for headaches and fevers. They yield25% of a fatty oil and contain traces of undentified alkaloid, a bitter substance and 4% ash consisting chiefly of calcium phosphate and potassium chloride (*6,7)*.The review of literature did not show any information on the diuretic investigation of this plant. So an effort was made in this research to evaluate the diuretic potential of different extracts of *Hygrophila salicifolia*.

MATERIALS AND METHODS

Procurement of chemicals:
Furosemide was obtained from khandelwal laboratories, Mumbai. The solvents used were of laboratory grade obtained from Emerck Ltd. Mumbai.

Animals:
Male albino rats weighing between (150-200 g) were selected for the work. They were kept in clean cages (made up of polypropylene with husk, bedding material) under controlled laboratory conditions such as 36-60% relative humidity, 25±3ºc temperature and 12h light/12h dark cycle. All the procedures were done accordingly CPCSEA guidelines and approved by the IAEC.

Plant extract:
Whole herb was obtained from the local areas in and around gujarat. The herb was identified and authenticated by Dr.reddy as *H. salicifolia* in the Bioscience department, Vallabh Vidyanagar, Gujarat. The plant was shade dried and ground to a fine powder in grinder to produce a coarse powder. Plant extracts were then obtained using soxhlet's apparatus using successive solvents. The extracts were concentrated using vacuum evaporator. It was dried in desiccators (*8-10*).

Acute toxicity study:
The test was carried out as given in OECD guideline. Wistar albino rats of either sex, weighing 200-300g were divided into six groups consisting of 6 animals each. Normal saline was given to control group (2 ml/kg, p.o.). 100, 200, 300, 600, 800, 1000, 2000, 3000 and 4000 mg/kg of the test extracts were given to other groups respectively. For the first 4 hours after dosing, the animals were observed continuously for any behavioral changes. They were kept under observation up to 14 days after drug administration to check out mortality, if any (*11*).

Evaluation of Diuretic activity:

Method:
Male wistar rats weighing 150-200g deprived of food and water for an overnight period. They were divided into four groups. Each consists of six rats. Lipchitz et al. method is used for the evaluation of diuretic activity. Before treatment, all animals were hydrated with (0.9% NaCl) physiological saline. Group-I (control) was received with normal saline solution (25ml/kg, p.o). Group-II (Standard, Positive control) was received Furosemide (20mg/kg, p.o) in saline. Group-III & Group-IV received the test extracts at doses 300mg/kg and 500mg/kg respectively. Doses of extract were based on acute toxicity studies. Immediately after oral administration, the rats were paired in metabolic cages. Urinary output was collected in graduated cylinder and recorded at the end of 5h. Evaluation parameters such as total urine volume and concentration of $Na^{+,}$ K^{+} and Cl^{-} in the urine were noted. Concentration of $Na^{+,}$ K^{+} were determined using flame photometer and Cl^{-} concentration was estimated titrimetrically using (N/50) $AgNo_3$ with few drops of 5% potassium chromate solution as indicator(*12-19*). (Diuretic index, Saliuretic index, Lipchitz's value, Na^{+}/K^{+} ratio)

Statistical Analysis:
The data were expressed as mean ± S.E.M and analyzed using one way ANOVA followed by Dunnett's multiple comparison test. $P< 0.05$ was considered as statistically significant.

RESULTS AND DISCUSSION

Acute toxicity study of test extracts displayed absence of mortality or no visible harmful effects in the animals during the experiment. No apparent changes were observed in the viscera of the rats. When as compared to control, the diuretic activity of methanolic extract of *Hygrophila salicifolia* was remarkable ($p<0.05$). The two test doses of the extract of *Hygrophila salicifolia* displayed remarkable increase in diuresis. As compared to control, the methanolic extract of *Hygrophila salicifolia* cause increase in $Na^{+,}$ K^{+} and Cl^{-}. Urine volume Methanolic extract of *Hygrophila salicifolia* at the dose of 300mg/kg body weight and 500mg/kg body weight was 8.38±0.25 and 13.15±0.12 ($p<0.05$) respectively, compared to the control group which was 3.02±0.20 (table 1&2). Methanolic extract of *Hygrophila salicifolia* at high dose shows equipotent diuretic activity as that of the standard drug (furosemide). As compared to control, the dose of 300mg/kg and 500mg/kg methanolic extract produced a remarkable increase in $Na^{+,}$ K^{+} and Cl^{-} excretion in a dose dependent manner. However urinary electrolyte excretions of both doses were less when compared with standard drug furosemide. When other successive extracts such as petroleum ether, toluene, chloroform, ethyl acetate,water were evaluated at same two doses 300mg/kg and 500mg/kg showed insignificant diuresis and electrolyte excretion when compared to control group. So it was revealed from the result that methanolic extract of the plant shows significant diuretic action compared to other successive extracts (table 1&2). The present research showed that methanolic extract of *Hygrophila salicifolia* remarkably increased the urinary output as well as urinary electrolytes at 500mg/kg and 300 mg/kg in dose dependent manner. The increase in urine volume was slight less compared to furosemide treated group. As emphasized diuretic property of methanolic extract of *Hygrophila salicifolia* may be due to its phytoconstituents such as alkaloid, flavanoid and phytosterol.

CONCLUSION

The results gained in this research impart a quantitative base to explain the traditional use of *Hygrophila salicifolia* as a potential diuretic agent.

TABLES

Table 1: Effect of different extracts of *Hygrophila salicifolia* on urine volume:

Sr. No.	Treatment	Mean urine volume (ml)	Lipchitz value	Diuretic index
1	Control	3.02 ± 0.20	---	---
2	Standard	14.63 ± 0.15	---	4.85
3	Pt. ether extract-300mg/kg	2.35 ± 0.13	0.16	0.80
4	Pt. ether extract-500mg/kg	3.28 ± 0.1	0.23	1.18
5	Toluene extract-300mg/kg	2.23 ± 0.15	0.16	0.63
6	Toluene extract-500mg/kg	2.4 ± 0.12	0.17	0.81
8	Chloroform extract-300mg/kg	3.2 ± 0.1	0.22	1.11
9	Chloroform extract-500mg/kg	3.5± 0.26	0.24	1.18
10	Ethyl acetate extract-300mg/kg	3.0±0.12	0.20	1.28
11	Ethyl acetate extract-500mg/kg	3.7 ± 0.15	0.25	1.52
12	Methanolic extract-300mg/kg	8.38 ± 0.25	0.57	2.86
13	Methanolic extract-500mg/kg	13.15 ± 0.12	0.89	4.47
14	Water extract-300mg/kg	3.0 ± 0.20	0.20	1.01
15	Water extract-500mg/kg	3.33 ± 0.12	0.23	1.13

Table 2: Effect of different extracts of *Hygrophila salicifolia* on electrolyte concentration:

Sr. No.	Treatment	Saliuretic index	Concentration Electrolyte Na^+	Concentration Electrolyte K^+	Na^+/K^+ ratio	Concentration Electrolyte Cl^-
1	Control	----	4.51± 0.17	9.68±0.16	0.47	13.57±0.17
2	Standard	1.65	12.70±0.11	15.54±0.08	0.82	18.54±0.01
3	Pt. ether extract-300mg/kg	0.98	4.29±0.90	9.26±0.01	0.46	13.33±0.12
4	Pt. ether extract-500mg/kg	1.0	4.46±012	9.55±0.09	0.47	13.49±0.18
5	Toluene extract-300mg/kg	0.97	4.32±0.06	9.37±0.12	0.46	13.39±0.08
6	Toluene extract-500mg/kg	0.99	4.96±0.13	9.53±0.10	0.52	13.47±0.02
7	Chloroform extract-300mg/kg	0.97	4.29±0.09	9.25±0.12	0.46	13.38±0.13
8	Chloroform extract-500mg/kg	0.98	4.48±0.10	8.78±0.14	0.51	13.50±0.13
9	Ethyl acetate extract-300mg/kg	0.94	4.19±0.04	8.82±0.28	0.48	13.32±0.09
10	Ethyl acetate extract-500mg/kg	0.96	4.39±0.15	8.88±0.17	0.49	13.45±0.14
11	Methanolic extract-300mg/kg	1.31	9.39±0.07	12.62±0.16	0.74	14.41±0.14
12	Methanolic extract-	1.55	11.45±0.12	13.90±0.21	0.82	17.63±0.34

	500mg/kg					
13	Water extract-300mg/kg	0.98	4.24±0.19	9.45±0.21	0.45	13.30±0.09
14	Water extract-500mg/kg	0.99	4.69±0.10	9.44±0.06	0.50	13.48±0.13

ACKNOWLEDGEMENT

The authors are grateful to the Pioneer Pharmacy Degree College, Baroda and R. K. University, Rajkot for their support and encouragement to conduct the research work.

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Existence and Extent of depression among college students

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ABSTRACT

Many college students who have depression aren't getting help they need sometimes they are unaware of their condition. They may not know where to go for help, or they may believe that treatment won't help. Others don't get help because they think their symptoms are just part of the typical stress of college, or they worry about being judged if they seek **mental health- care.** The main aim of this study was to find out the existence and extent of depression in students and to find out the leading causes behind that. After preparing the questioner form an in-depth counselling seasons was conducted with the students of different educational field. And with the collected data an in-depth analysis was carried out by applying various statistical tools. Based on the result it can be concluded that Depression in college student is a serious threat and required an immediate attention. The proper counselling is needed and so it indicates the rising opportunities for Start up of a chain counselling system in every College.

SUMMARY

To find out the current scenario of depression in college students and their possible causes to understand the need of student counselling and create awareness.

Keywords: Depression, Depression in college students, cause of depression , student counselling.

INTRODUCTION

Everyone occasionally feels blue or sad, but these feelings are usually short-lived and pass within a couple of days. When you have depression, it interferes with daily routine and thought process. Depression is a common but serious illness. Most of the people are unaware of the depression and they

ignore the symptoms which end up in severe case of depression. The term depression describes a wide range of emotional lows, from mere sadness to a pathological suicidal state. This is a common mental problem encountered in day to day stress filled life. Adolescence which is the transitional period from childhood to adulthood is a stage of emotional instability making them vulnerable to depression. The state of emotional instability results from difficulties in establishing self-identity and self-esteem leading to conflicts within family and peer groups. Due to these many reason the college students are more prone to have the mental stress which might lead them to depression.

MATERIALS AND METHODS

Objective:

- To understand the current scenario of Depression in college students.
- To find out the extent of depression in students.
- To find the most common reasons behind the depression in students.
- To make students aware about the depression and its symptoms.
- To find out the requirement of student counseling program in colleges.
- To understand the need of counseling in students.

Limitations:

The study is subject to the following limitations.

- 2 months period for conducting survey.
- The study was restricted to local Gandhinagar and Ahmadabad city only as it was a part of college project.
- The study was limited to the capabilities and willingness of the respondents to attend the survey form.
- Limited sample size up to 100 surveys as the survey was a part of college project.

Data Collection:

1) Primary data:-

Primary data is collected from the fieldwork. (By Questionnaire and counselling)

i. Duration of Study: - 2 months

ii. Target Population: - college students

iii. Sample Size: - 100

iv. Sampling Technique: - Convenience sampling

v. Sampling Unit: - Gandhinagar and Ahmadabad city

2) Secondary data:-

i. Secondary data is collected from Internet and television Advertisements

ii. It is valuable source of new ideas that can be explored later through primary research.

Calculation and graphical interpretation:

Statistical data, analysis and interpretation were done with the help of thorough study of the survey forms, arithmetical and graphical calculation, and also with computer based Microsoft excel sheet. A questionnaire was framed to analyze the above objectives. 100 students of different field were selected by convenient sampling technique. The results were analyzed with bar graphs and pie charts. The students are marked on the basis of their answers out of 60. Those who have scored between **1 to 6** are **Normal** those who have scored between **7 to 16** are under **Minor depression.** Those who have scored between **17 to 32** are under **Major depression** those who have scored between **33 to 60** are under **severe depression.**

Questionnaires: *(1-13)*

1. Have you felt sad or tearful for a majority of the day for at least the last two weeks?
2. I have felt down, depressed or hopeless.
3. I have had little interest or pleasure in doing the things I usually enjoy.
4. My sleep has been disturbed -- too little, too much, or broken sleep.
5. My energy level decreased to the point that normal daily activities seem too long?
6. I have had a poor appetite or have been eating more than usual. And that has resulted in a change in your weight.
7. I have felt bad about myself or felt like i am a failure or that I let myself or my family down.
8. I feel that I am a guilty person who deserves to be punished.
9. I have been moving or speaking so slowly that other people could have noticed.
   Or
   The opposite I have been fidgety and moving around a lot more than usual.
10. I feel lifeless -- more dead than alive.
11. I spend time thinking about HOW I might kill myself.
12. I feel trapped or caught.
13. I feel depressed even when good things happen to me.

If more than one of these symptoms is there, than possibly what could be the reason?

Study pressure
Family issue

Relationship problems
Health Problems
No freedom to express yourself
No appreciation for your work
Other.

RESULTS AND DISCUSSION

- 60% females and 38% male are under minor depression.
- 18% females and 20% male are under Major depression.
- 2% male are under severe depression.
- In males the reasons for depression are
  30% is Study pressure,
  16% is family issue,
  10% is Relationship problem ,
  2% is Health problem,
  18% Is No freedom to express yourself
  20% is no appreciation for your Work,
  4% is other reasons.

- In females the reason for depression are,
  26% is Study pressure,
  8% is Family issue ,
  26% is relationship problem,
  2% is Health problem,
  20% Is No freedom to express yourself,
  4% is no appreciation for your Work,
  14% is other reasons.

- 8 % of female felt sad or tearful for a majority of a day for more than 4 to 5 days a week
- 25% males and 21% female felt down depressed or hopeless for once or twice in a week without any fixed reason.

- 16% of female had little interest in doing the things they usually enjoy they felt this for 4 to 5 days a week.12% male felt this every day.

- 22% male have sleeping problem for 4 to 5 days a week and 16% male have it for almost every day

- 10% male and female felt their energy level decreased for 4 to 5 days a week.

- 15% male and 8% female have had a poor appetite or have been eating more than usual for 4 to 5 days a week.

- 22% male and 8% female felt bad about their selves or like a failure for once or twice in a week.

- 20% male and 15% female felt that I am a guilty person who deserves to be punished for Once or twice in a week.

- 24% male and 40% female felt that they have been moving or speaking so slowly for Once or twice in a week.

- 42% male and 30% female feel lifeless more dead than alive. for Once or twice in a week.

- 2% male and 4%female have spend time thinking about HOW I MIGHT KILL MY SELF. for 4 to 5 days a week.

- 4% male and 6%female feel trapped or caught. or have a strong feeling of Regret for 4 to 5 days a week. 8% feel trapped or caught. or have a strong feeling of Regret. .for almost every day.

- 8% male and 2% female have felt depressed even when good things happening to them for almost every day.

- 6% male and 2% female have uninterested in attending my favorite subject/lecture. For almost every day.

- 2% male and 8%female have all have misbehaved or showed some abnormal behavior with friends or family for 4 to 5 days a week.

Suggestions:

- Awareness about the depression should be created amongst college students.
- Proper counseling system should be formed in all the colleges and educational institutes.
- Lifestyle modification should be suggested by healthcare professionals.
- Positive atmosphere should be provided to students.
- Parent's awareness session should be conducted to help them understanding and identify the symptoms of depression.
- As youth is the future of nation, the youth counseling program is the emerging market for entrepreneurs to help the youth and nation.

CONCLUSION:

According to the survey almost 60% females and 38% males are under minor depression while 20% male and 18% females are under Major depression. This indicates a strong need of counseling and awareness on mental health. While 2% male are under severe depression who needs an immediate medical attention. So there is a huge scope and demand of Youth counseling program.

FIGURE:

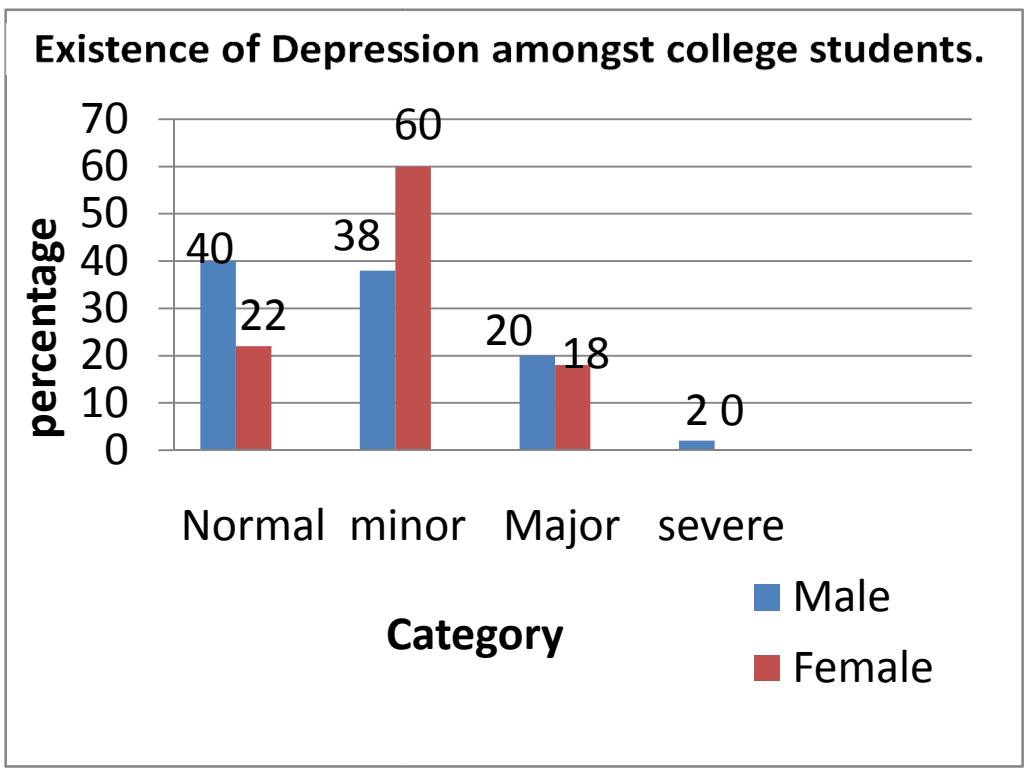


FIG: Existence and extent of Depression.

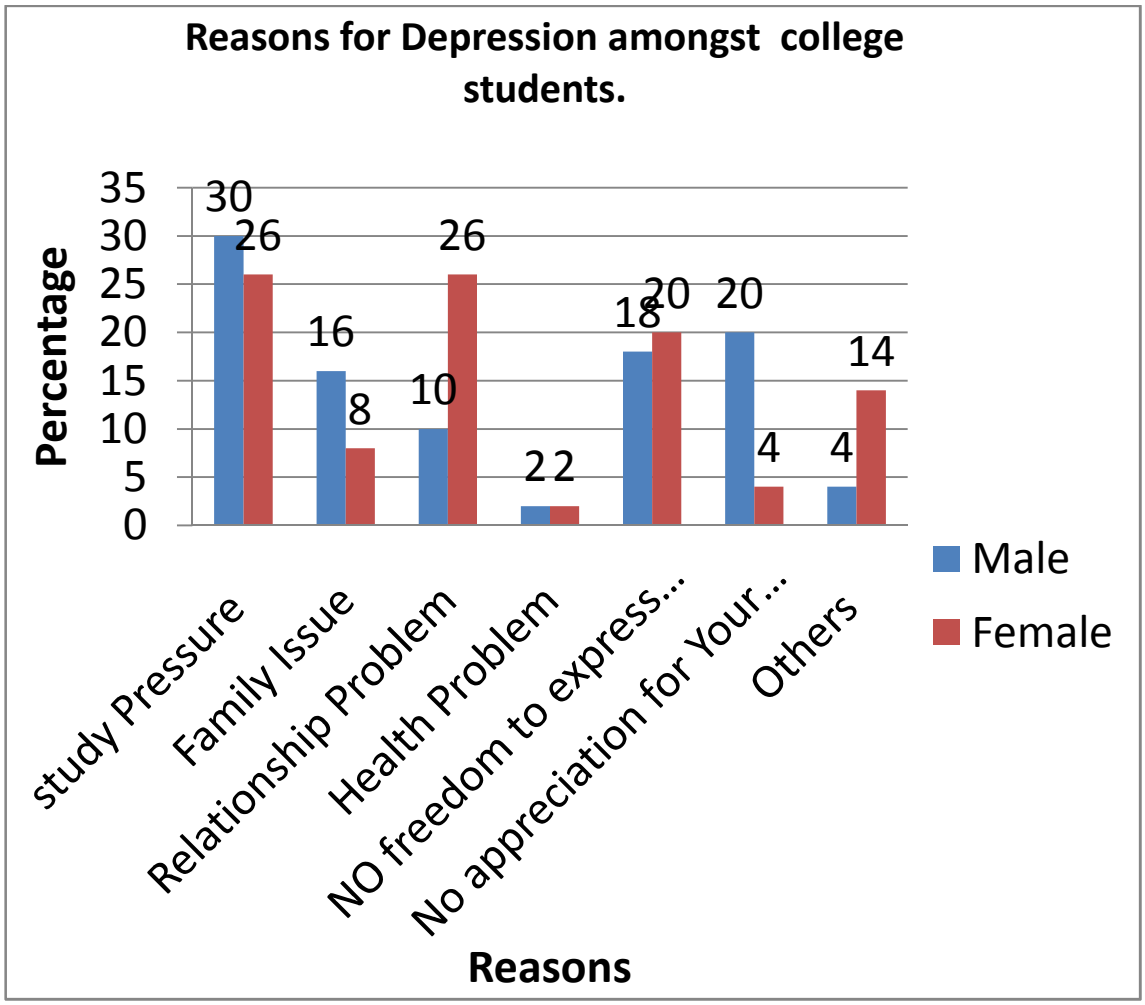
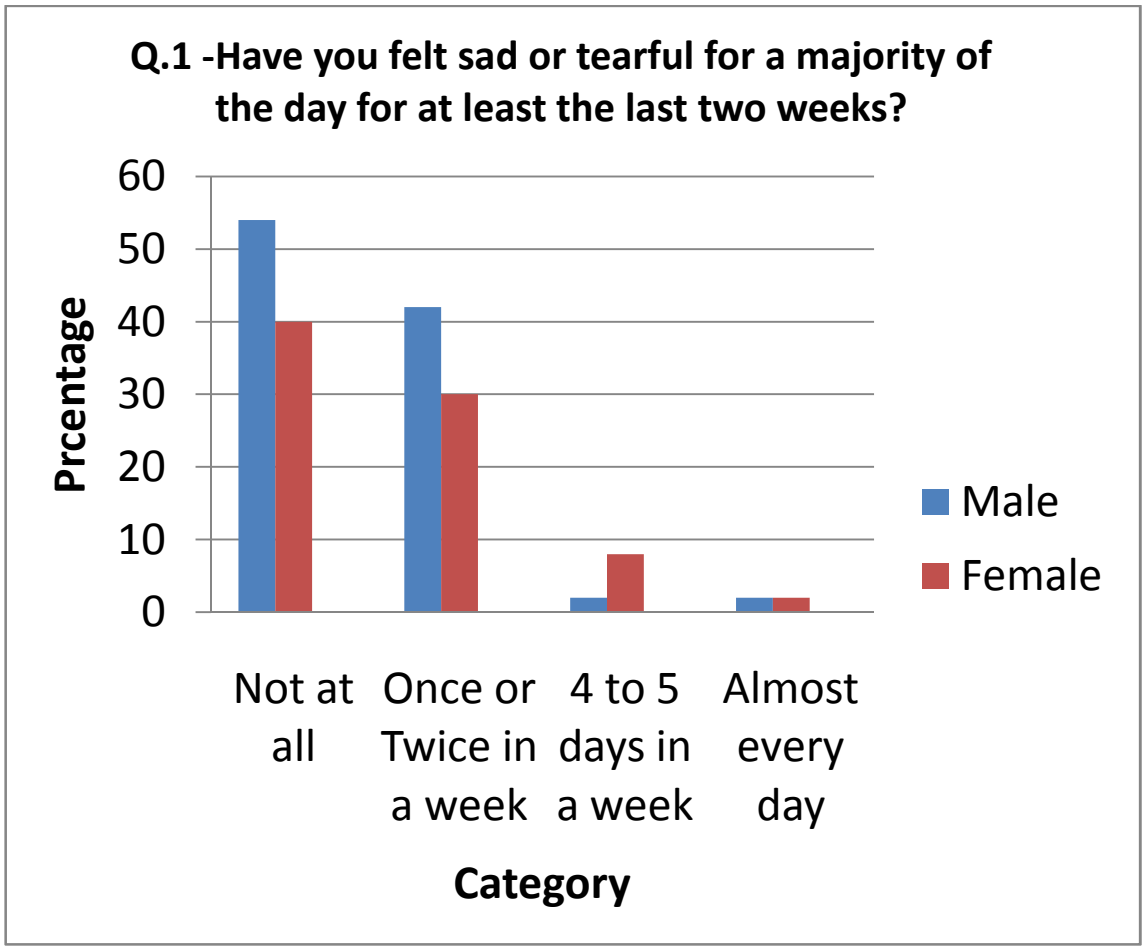
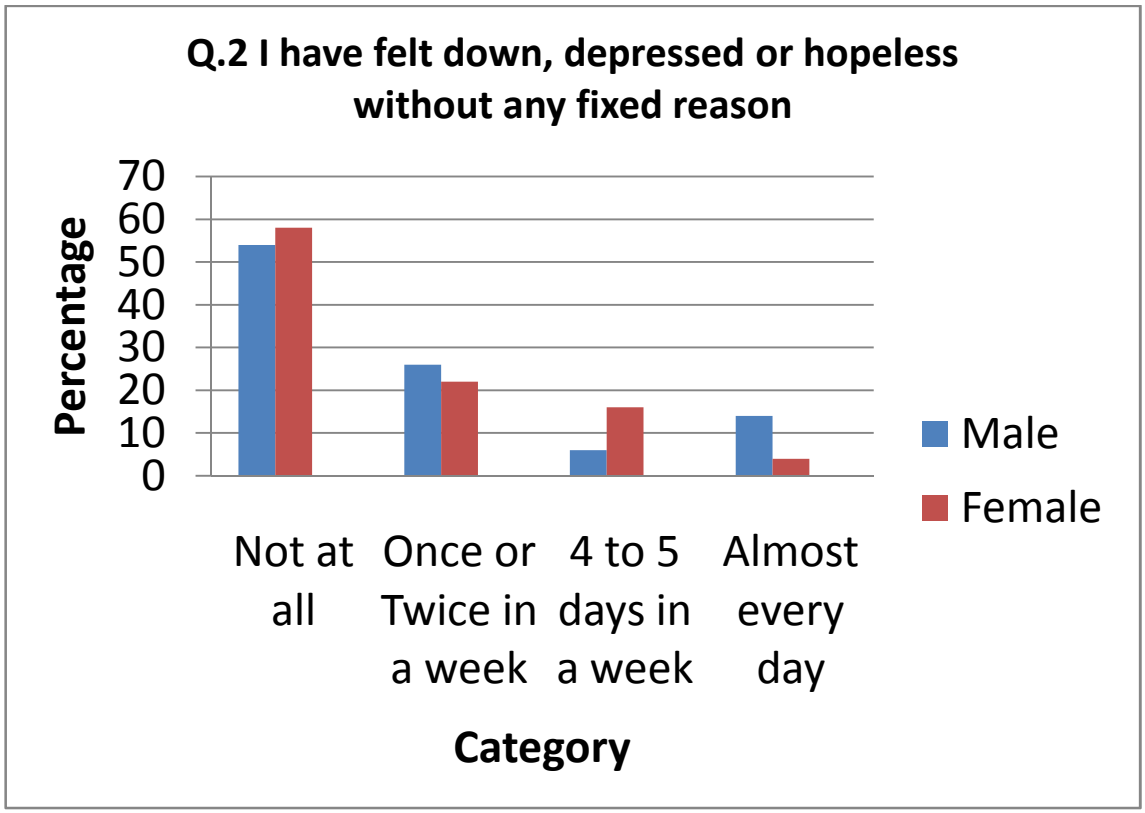
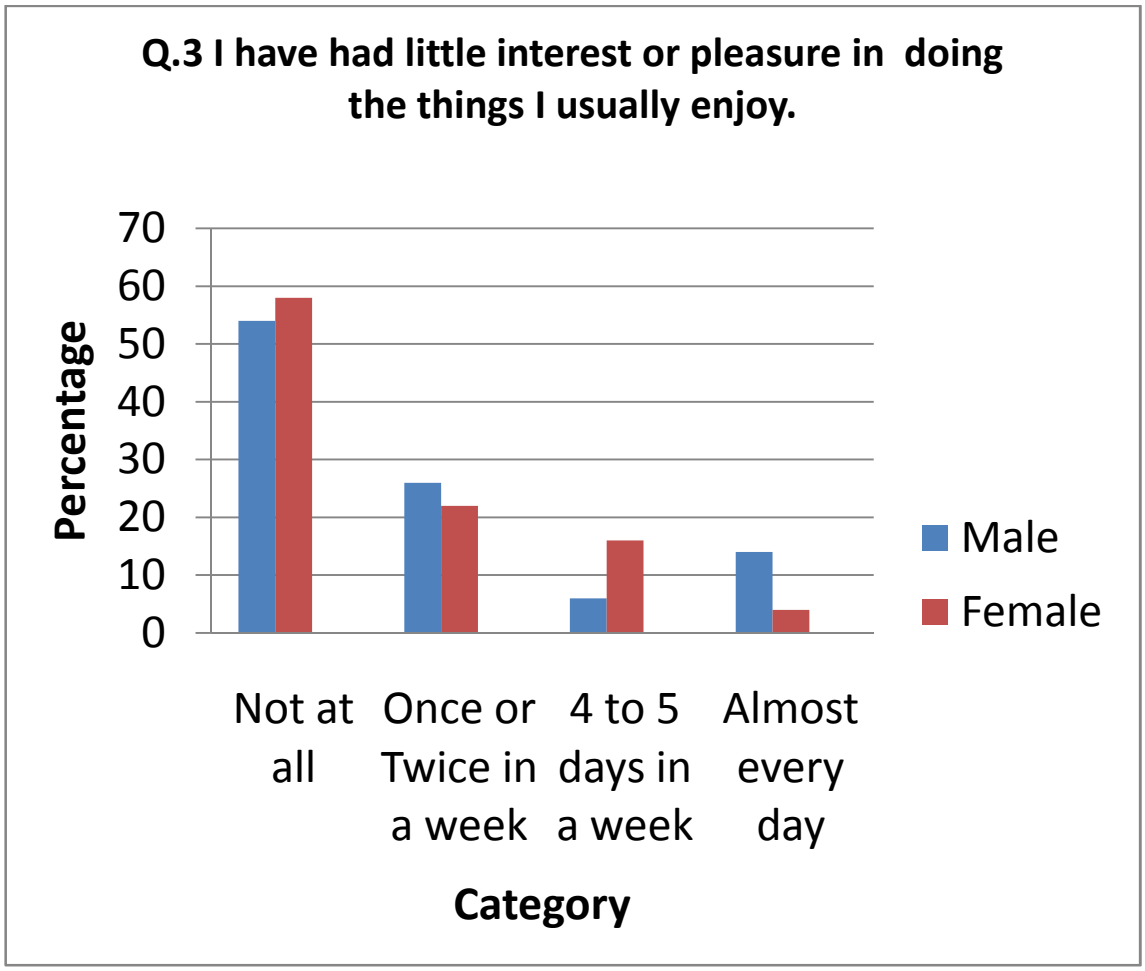
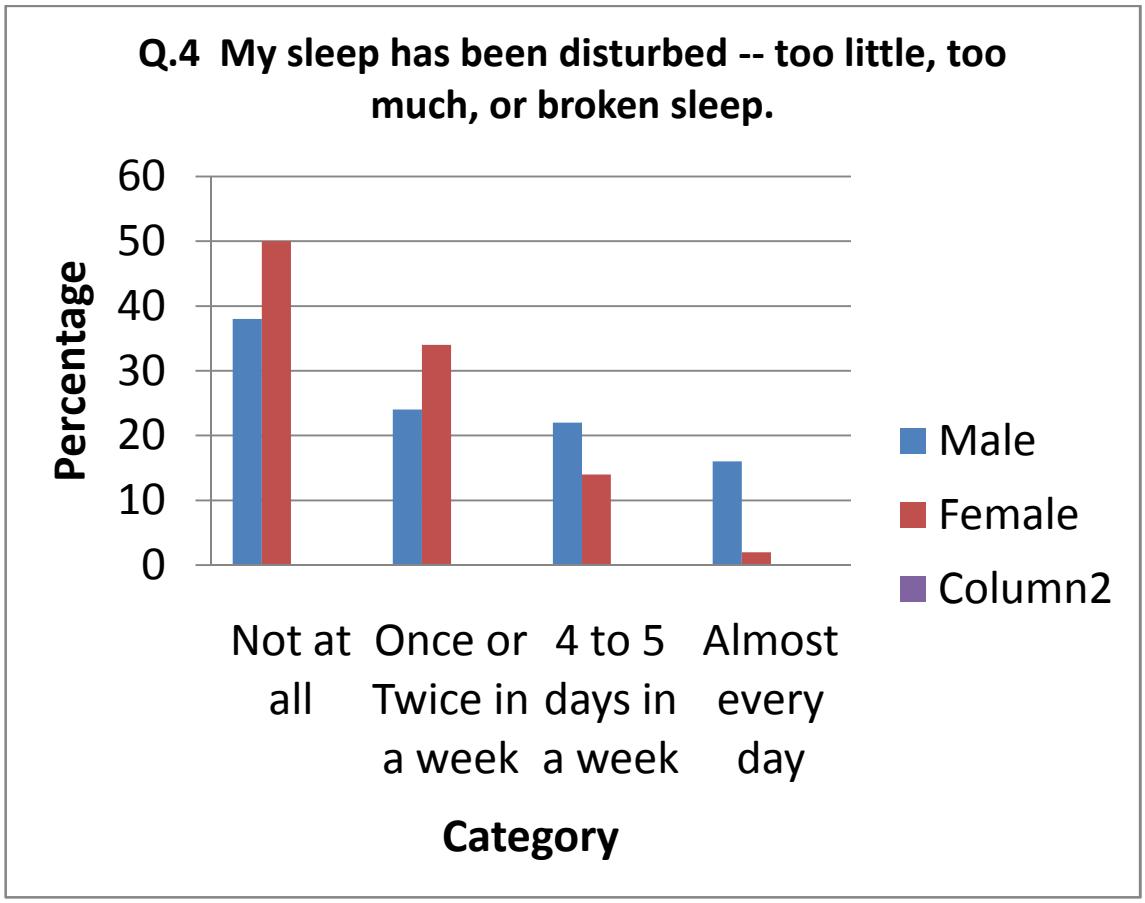


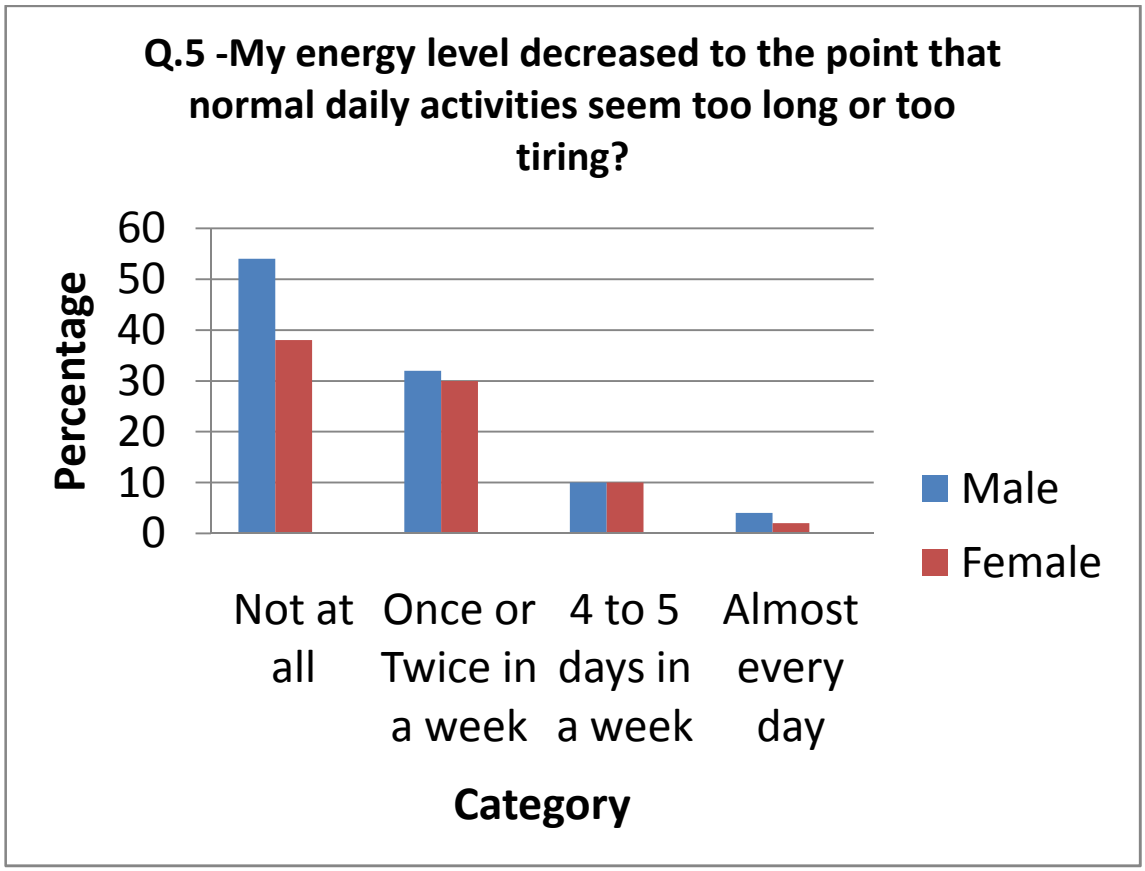
FIG: Leading causes of depression.

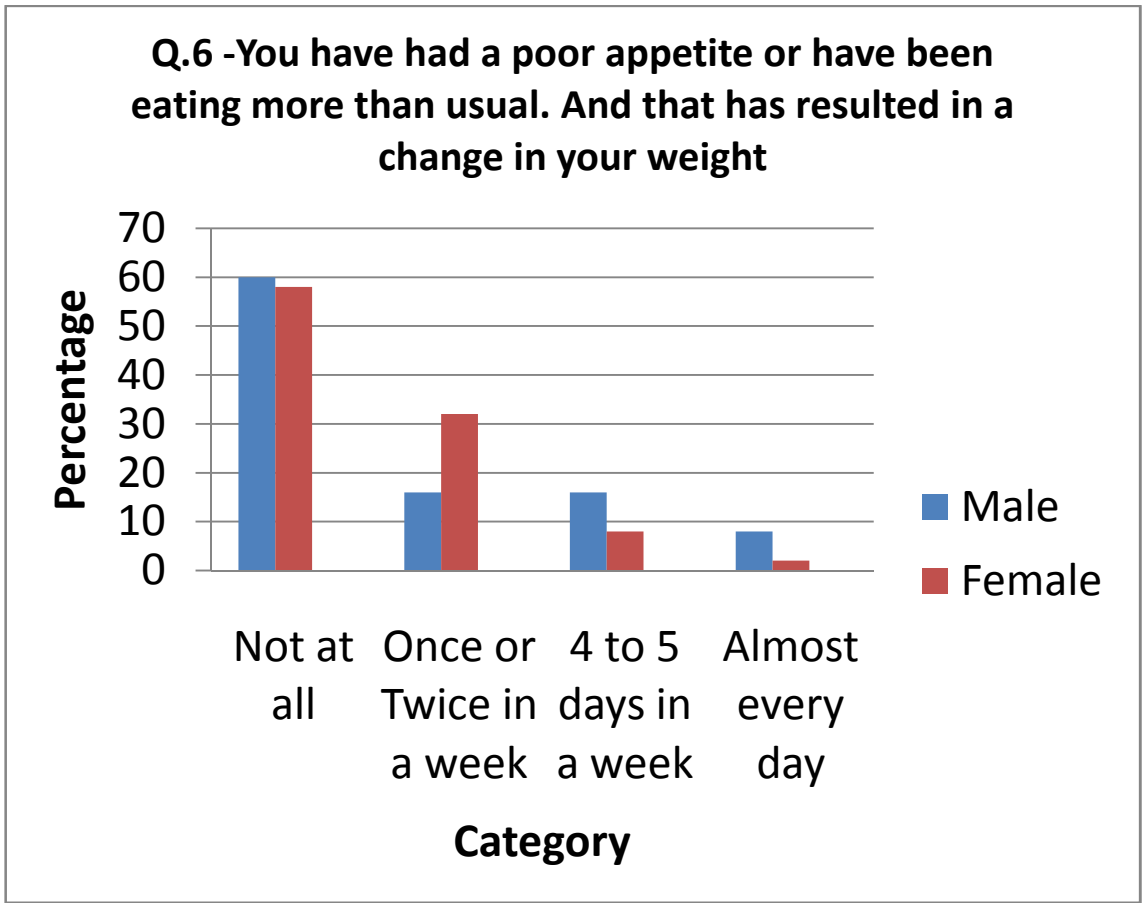

Q.1 -Have you felt sad or tearful for a majority of the day for at least the last two weeks?
Prcentage
60
50
40
30
20
10
0
Not at all
Once or Twice in a week
4 to 5 days in a week
Almost every day
Category
Male
Female

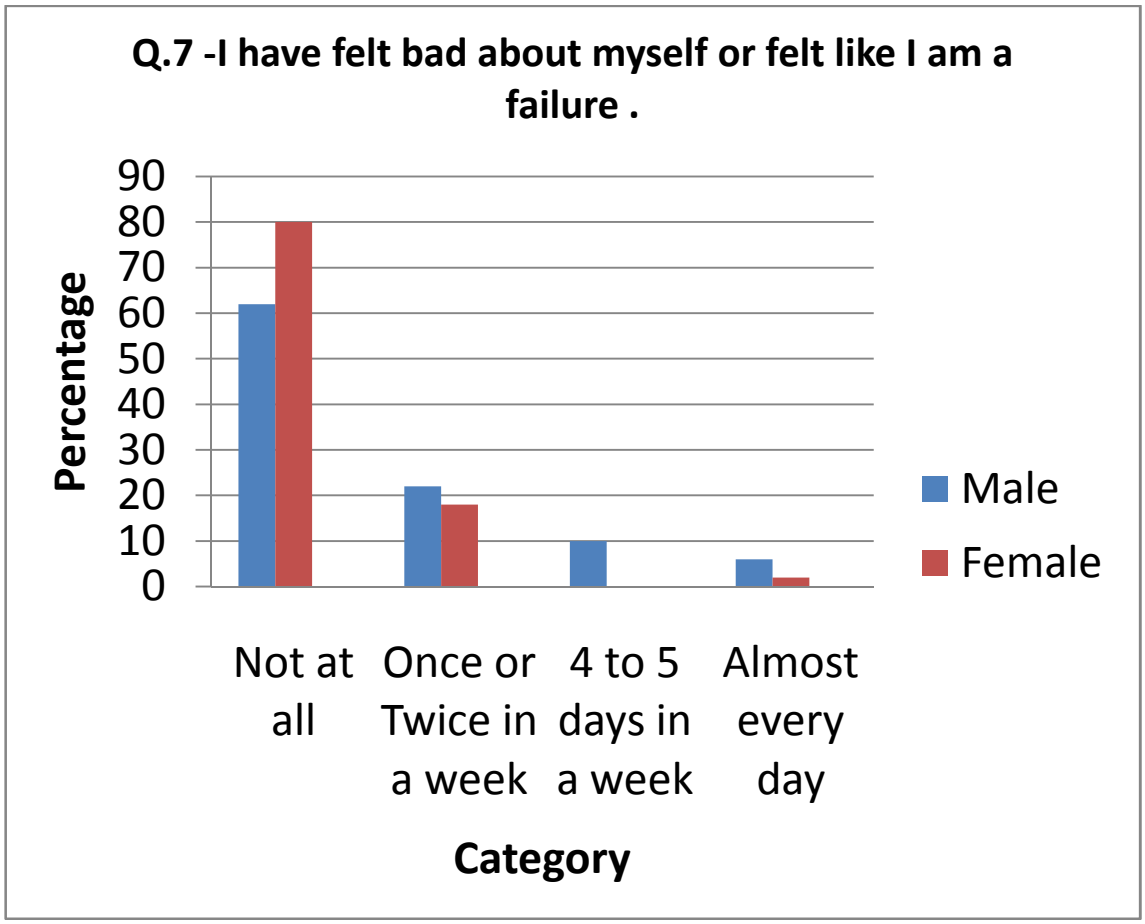

Q.2 I have felt down, depressed or hopeless without any fixed reason
Percentage
70
60
50
40
30
20
10
0
Not at all
Once or Twice in a week
4 to 5 days in a week
Almost every day
Category
Male
Female

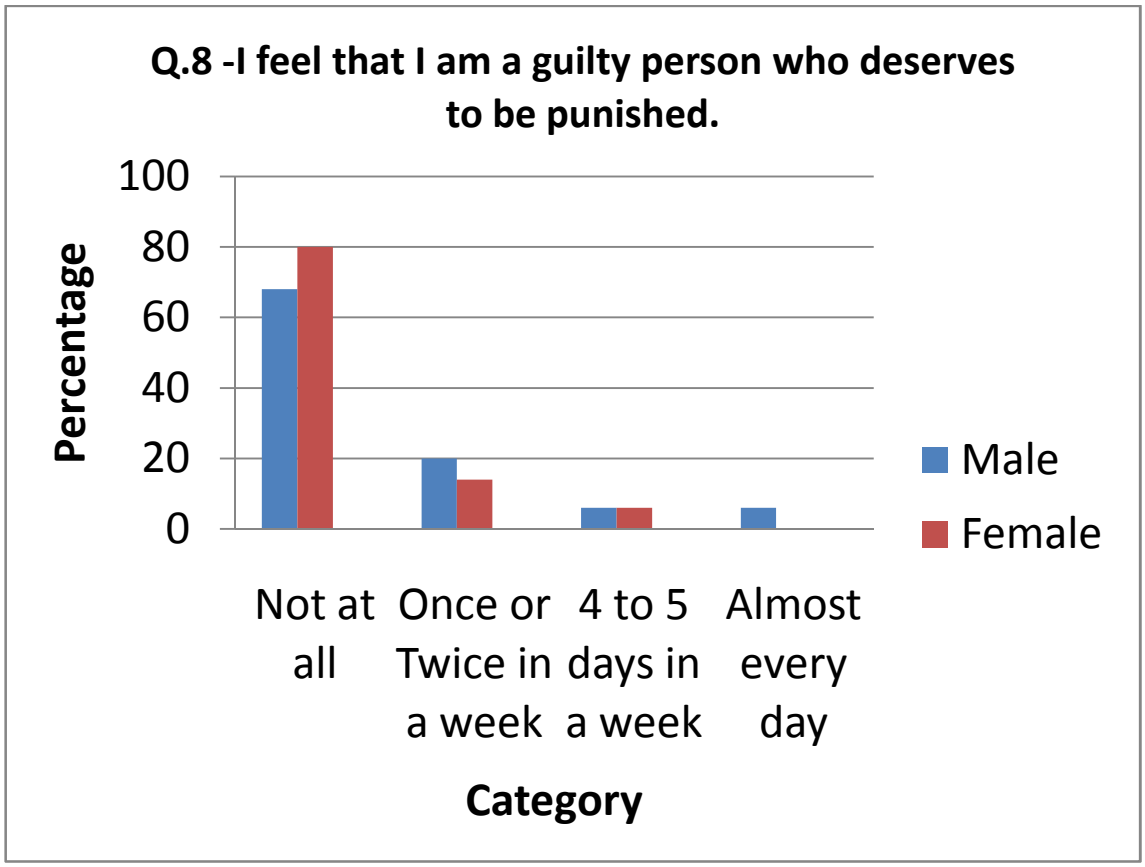

Q.3 I have had little interest or pleasure in doing the things I usually enjoy.
Percentage
70
60
50
40
30
20
10
0
Not at all
Once or Twice in a week
4 to 5 days in a week
Almost every day
Category
Male
Female

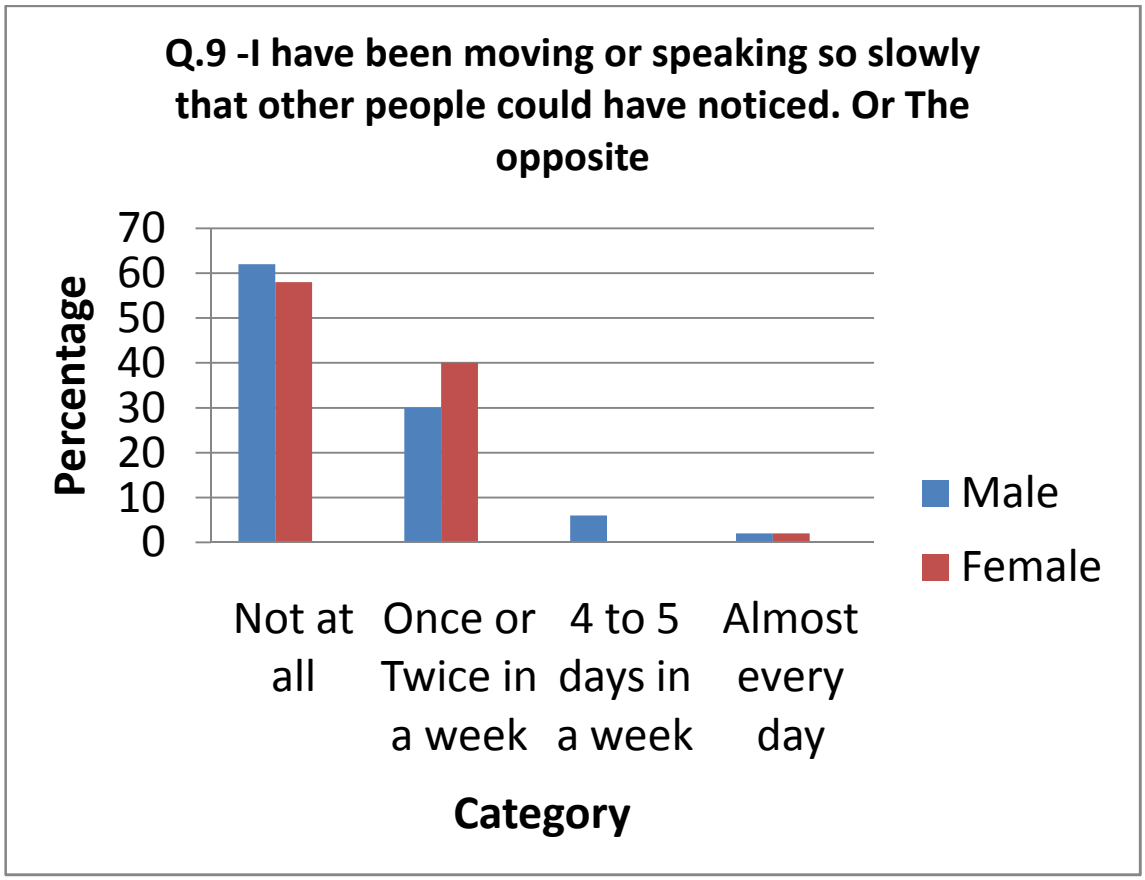

Q.4 My sleep has been disturbed -- too little, too much, or broken sleep.
Percentage
60
50
40
30
20
10
0
Not at all
Once or Twice in a week
4 to 5 days in a week
Almost every day
Category
Male
Female
Column2

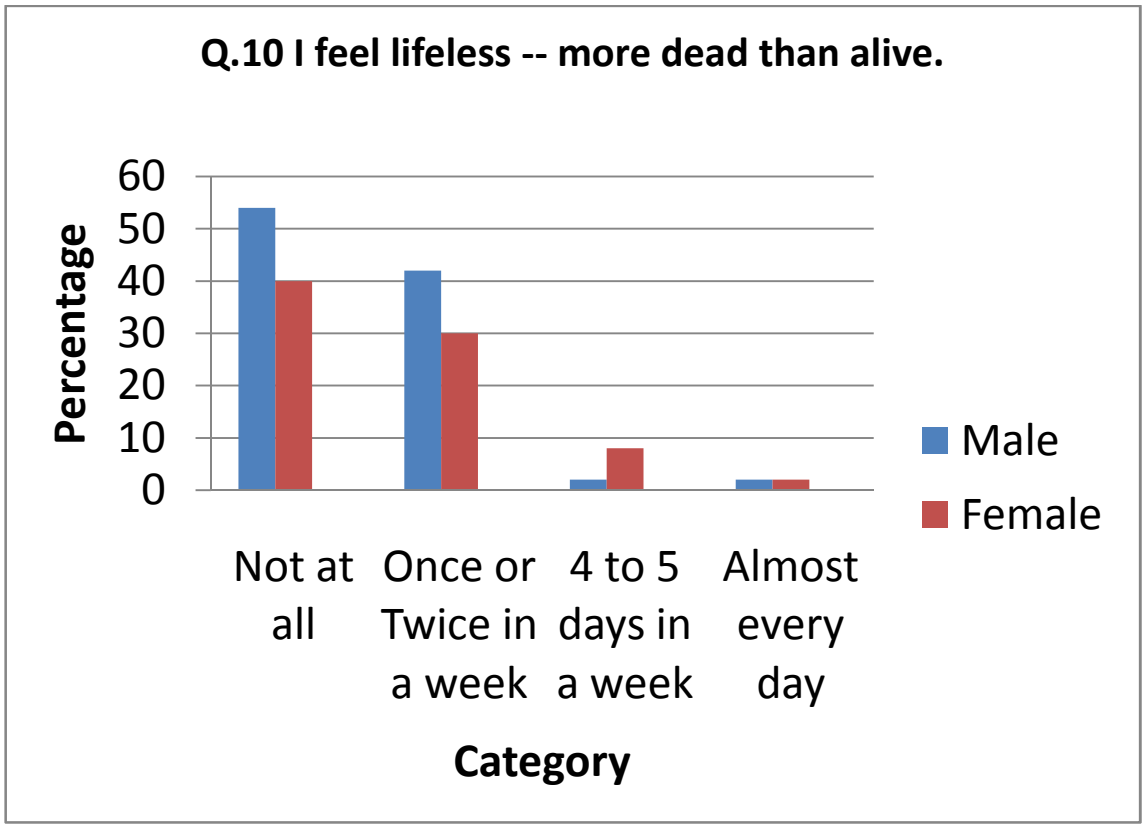

Q.5 -My energy level decreased to the point that normal daily activities seem too long or too tiring?
Percentage
60
50
40
30
20
10
0
Not at all
Once or Twice in a week
4 to 5 days in a week
Almost every day
Category
Male
Female

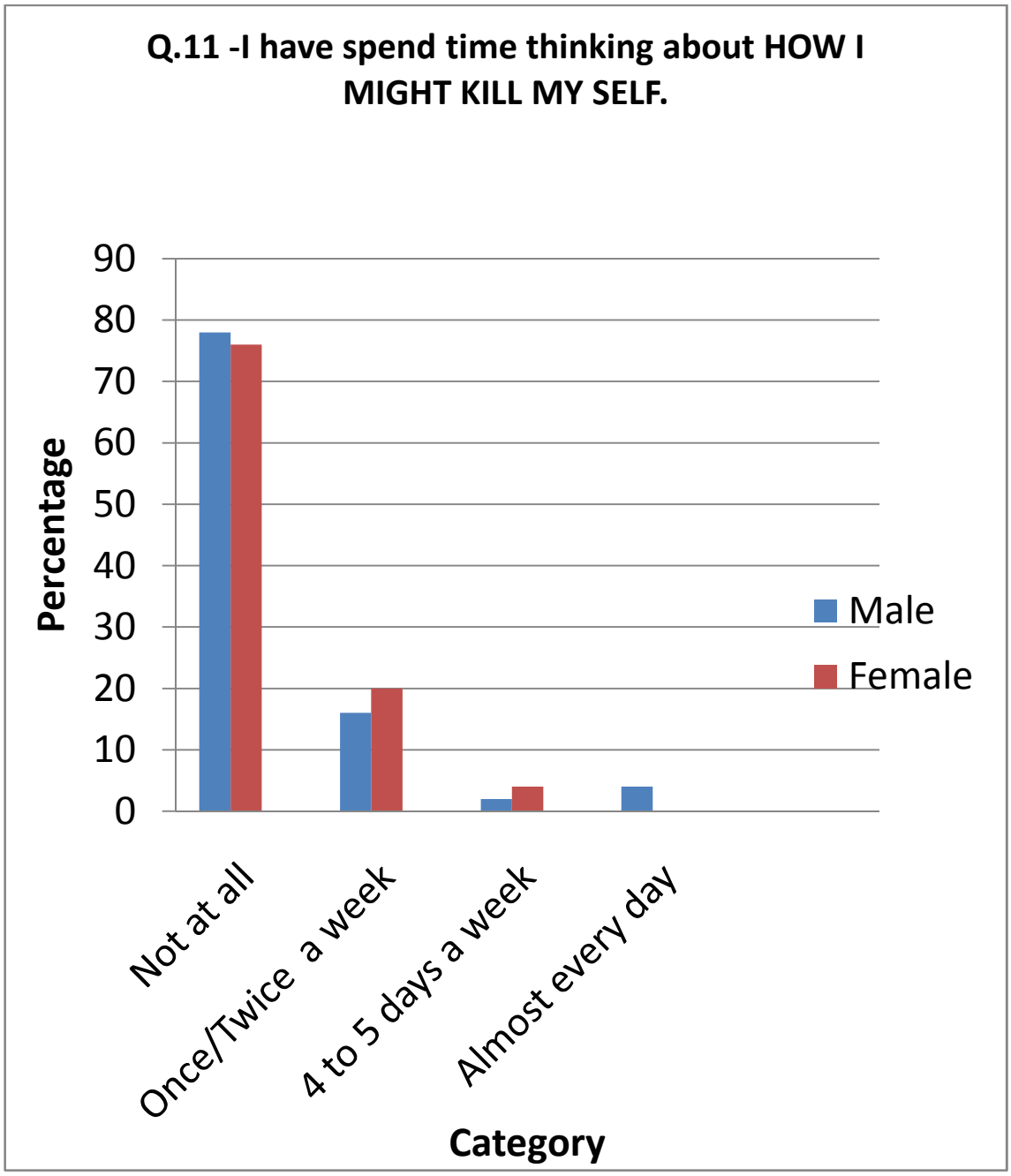

Q.6 -You have had a poor appetite or have been eating more than usual. And that has resulted in a change in your weight
Percentage
70
60
50
40
30
20
10
0
Not at all
Once or Twice in a week
4 to 5 days in a week
Almost every day
Category
Male
Female

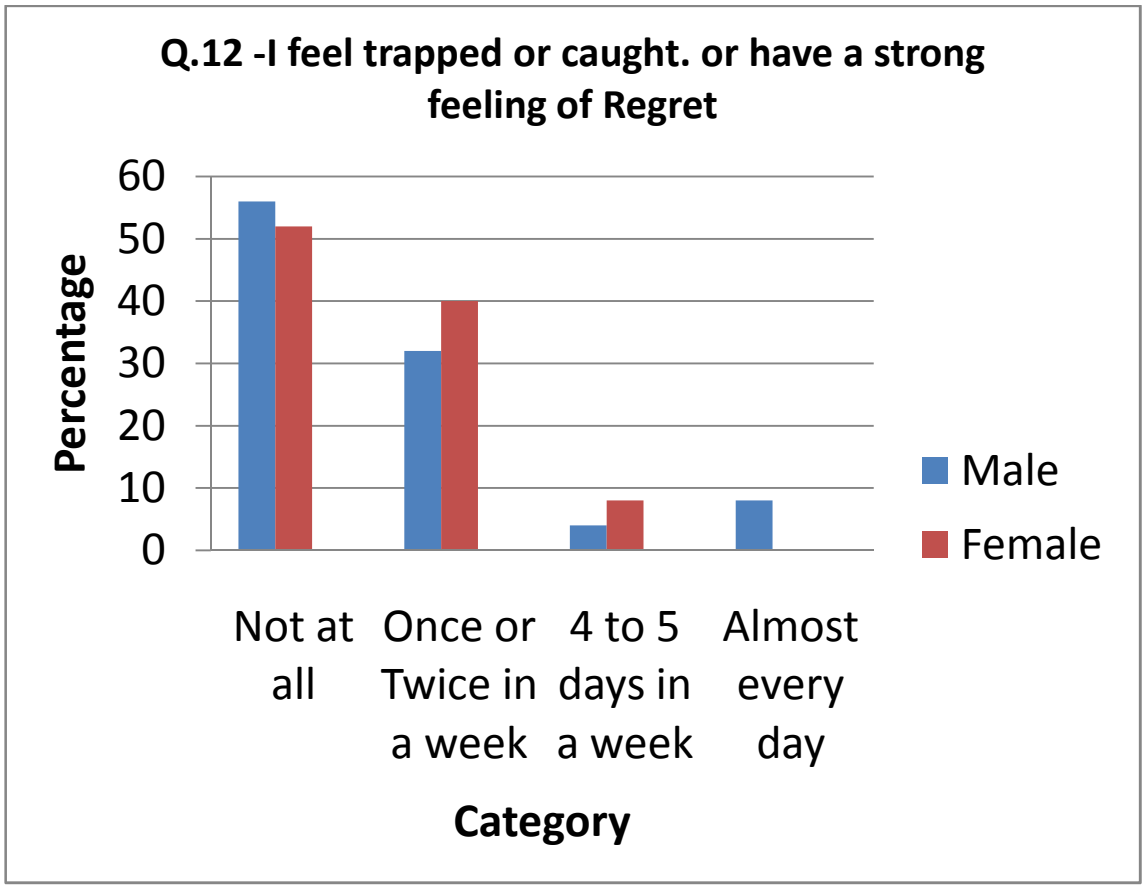

Q.7 -I have felt bad about myself or felt like I am a failure .
Percentage
90
80
70
60
50
40
30
20
10
0
Male
Female
Not at all
Once or Twice in a week
4 to 5 days in a week
Almost every day
Category

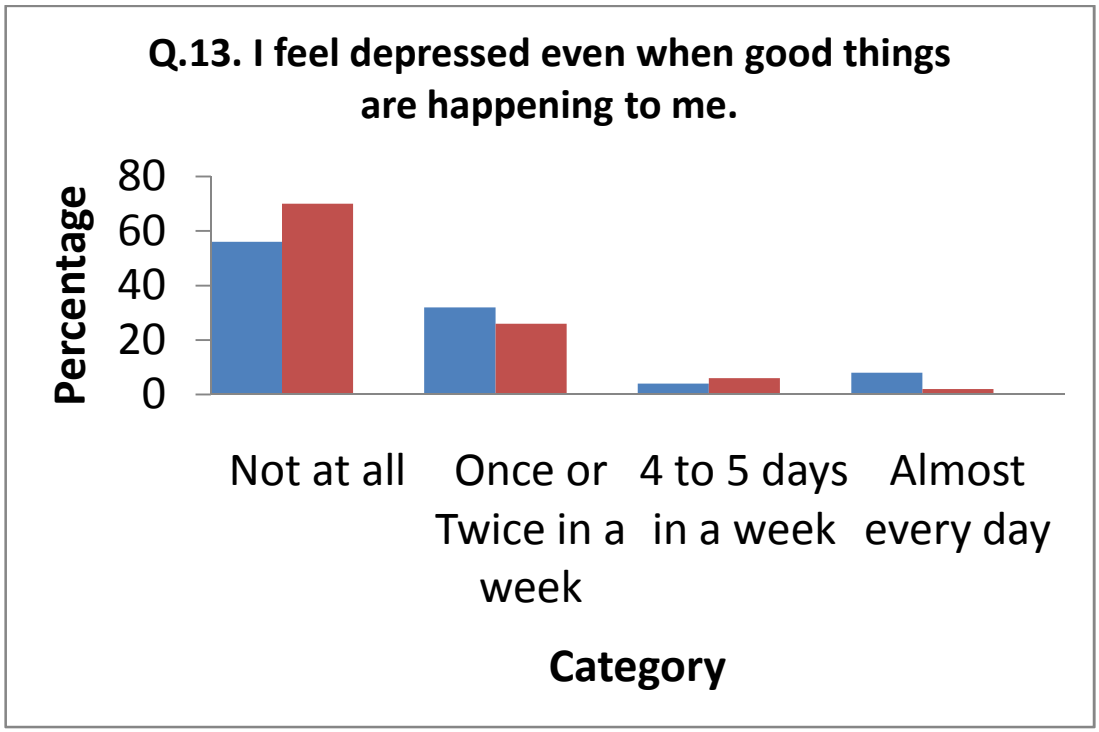

Q.8 -I feel that I am a guilty person who deserves to be punished.
Percentage
100
80
60
40
20
0
Not at all
Once or Twice in a week
4 to 5 days in a week
Almost every day
Category
Male
Female


Q.9 -I have been moving or speaking so slowly that other people could have noticed. Or The opposite
Percentage
70
60
50
40
30
20
10
0
Not at all
Once or Twice in a week
4 to 5 days in a week
Almost every day
Category
Male
Female


Q.10 I feel lifeless -- more dead than alive.
Percentage
60
50
40
30
20
10
0
Not at all
Once or Twice in a week
4 to 5 days in a week
Almost every day
Category
Male
Female


Q.11 -I have spend time thinking about HOW I MIGHT KILL MY SELF.
Percentage
90
80
70
60
50
40
30
20
10
0
Male
Female
Not at all
Once/Twice a week
4 to 5 days a week
Almost every day
Category


Q.12 -I feel trapped or caught. or have a strong feeling of Regret
Percentage
60
50
40
30
20
10
0
Not at all
Once or Twice in a week
4 to 5 days in a week
Almost every day
Category
Male
Female



ACKNOWLEDGEMENT

Authors are grateful to the K.S.V. University, Gandhinagar for supporting the interaction with students.

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(Proceedings available for download at rku.ac.in/icre)

RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

Consumer awareness and buying behavior towards mosquito repellent

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ABSTRACT

Mosquito belongs to the group of insects known as *diptera* or *flies* which spread many diseases like Dengue fever, Malaria, yellow fever, Chikungunya. Various market products are available acting as a mosquito repellant. The main aim of this study was to understand the mind set of people about the natural mosquito repellant and dangers of chemical mosquito repellants and to find out formulation strategy of new natural mosquito repellant. After collecting the required data, an in-depth analysis was carried out by applying various statistical tools. The survey was conducted by implying the personal interview with detailed questionnaire to elicit responses. Based on the results, it can be concluded that public awareness for hazards of chemical mosquito repellant products and substitution of the same by natural products should be done. Novel delivery system of natural mosquito repellent should be developed to attract public.

SUMMARY

Natural mosquito repellent formulation strategy by market and public survey

Keywords: Natural mosquito repellent, Market survey, formulation strategy

INTRODUCTION

A mosquito repellent is a substance applied to skin, clothing, or other surfaces which discourages mosquito from landing or climbing on that surface. Insect repellents help prevent and control the outbreak of mosquito diseases such as malaria, chikungunya, dengue fever, yellow fever and West Nile fever. Mosquito repellent are two type synthetic and natural anti mosquito product. Synthetic mosquito repellent suffers form drawbacks of allergic reaction if taken in breath in significant amount. Moreover, the smell of the same is also a characteristic one which is not acceptable by all. In nature there are many plants including Citronella, Horsemi, Marigolds, Ageratum, Catnip are available possessing moisquito repellent actions.

MATERIALS AND METHODS

Objective:

- To aware the people about danger of mosquito and to take care against mosquito bite.
- To find out which mosquito repellent are more used by people.
- To find out side effects caused while using mosquito repellent
- To aware the people for cleaning and preventive steps.

Limitations:

The study is subjected to the following limitations.

- Two months period for conducting survey.
- The study was restricted to local Gandhinagar and Ahmadabad city only.
- The study was limited to the capabilities and willingness of the respondents to approximately answering the questions.
- Limited sample size up to 50 surveys.

Data Collection:

1) Primary data:-

Primary data is collected from the fieldwork. (By Questionnaire)

i. Duration of Study: - 2 months

ii. Target Population: - General population

iii. Sample Size: - 50

iv. Sampling Technique: - Convenience sampling

v. Sampling Unit: - Gandhinagar and Ahmadabad city

2) Secondary data:-

i. Secondary data is collected from Internet and television Advertisements

ii. It is valuable source of new ideas that can be explored later through primary research.

Calculation and graphical interpretation:

Statistical data, analysis and interpretation were done with the help of thorough study of the survey forms, arithmetical and graphical calculation, and also with computer based Microsoft excel sheet.

Questionnaires: *(3-11)*

1. Are you aware of danger of mosquitoes?
2. Have you suffered from any disease by mosquito in past?
3. Which type of mosquito repellent do you use in routine?
4. How did you know about product?
5. From the following which mosquito repellent are you using currently?
6. What is the frequency of usage product?
7. From where do you buy the product?
8. If your brand is not available in store will you shift to other brand?
9. Do you agree that such products are harmful to health?

10. According to you which factor is most important for selection of mosquito repellent?
11. Do you refer label while purchasing product?
12. If yes what do you see on label?
13. Do you use any house hold remedies as mosquito repellent?
14. Do you know the availability of natural anti mosquito in market?
15. When the mosquito problem is high around your house?
16. Have you experience any problem while using repellent?
17. If yes then what types of problem have you getting?
18. What is the frequency of cleaning of your home surround?
19. Is your water tanks are cleaned regularly and mosquito proof?
20. Do you agree that mosquito repellent product is costly?

RESULTS AND DISCUSSION

As per the survey carried out on subj ect, following findings were drawn.

- Most of people are aware from danger of mosquito, only few people are not. Among them most of people take care against mosquito bite.
- Most of people suffered from mosquito disease in past, among that majority of people suffered from malaria, dengue & yellow fever and few people suffered from chikungunya.
- Almost people use synthetic product, remaining people use natural as well as synthetic both product.
- More than 50% people know about product from TV advertisement.
- Majority of people are using all out currently while less than 50% people are using good knight, mortein coil and odomos lotion.
- 40% people using product for 6hr, 30% for 12hr, 20% people for 18 hr and only 10 % people for 24hr.

- Medical store and mall are major source to buy mosquito repellent approached by people.
- The major parts of people want to follow their current brand but some people will shift to another brand if brand is not available in shop.
- More than 75% people agree that such product is harmful to health.
- Safety & health is most favourable opinion of people than quality, quantity or brand to select the product.
- 78% people refer the label while purchasing the product.
- Only 10% people see all on label like date of mfg & exp., price, company, and precaution. Majority of people see date of mfg & exp on label.
- Maximum people are not using any household remedies.
- Many people do not know about natural anti mosquito repellent and some people are aware about natural anti mosquito repellent.
- Some people say that mosquito is high in night and some say in evening.
- Majority of people are suffering from health problem while using mosquito repellent.
- The major problem of health while using mosquito repellent are headache and breathing problem. Also some people are suffering from eye irritation, skin rashes and cold.
- Almost people are cleaned their surrounding area after 4 days and very few people are cleaned their surrounding area after 1 month.
- More than 70% people are cleaning water tank and keep mosquito proof regularly.
- Most of people believe that product is not costly and some people believe product is costly.

CONCLUSION:-

- Most of people are more mosquito conscious and very few are not.

- Majority of population are using all out as mosquito repellent currently and most of them are informed by TV advertisement for mosquito repellent.
- 40% people use mosquito repellent for 6 hr, 30% people use mosquito repellent for 12 hr, while only 10% people use for 24 hr.
- Generally people buy product from medical store.
- Most of people prefer to use natural anti mosquito product.
- Most of people are suffered from headache and breathing problem due to smell of mosquito repellent, so should made odorless product.
- Maximum people kept their surrounding area of house clean regularly.
- Majority of people give first priority to safety and health so product should made more safe & beneficial to health.

FIGURES

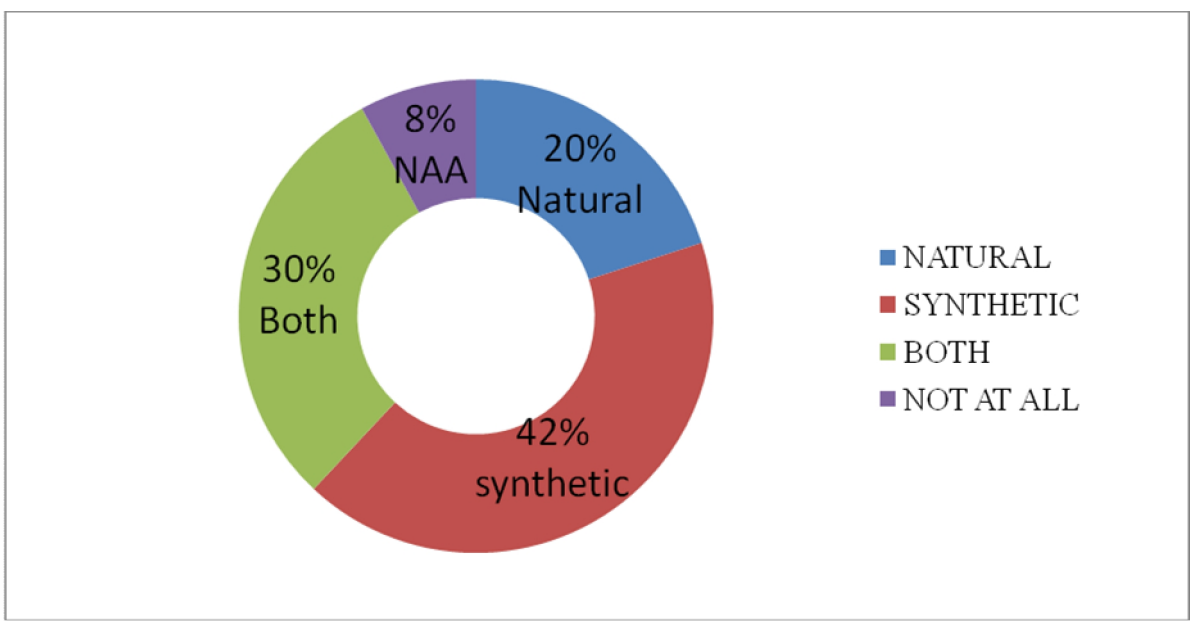


Fig1. Type of mosquito repellent used by people

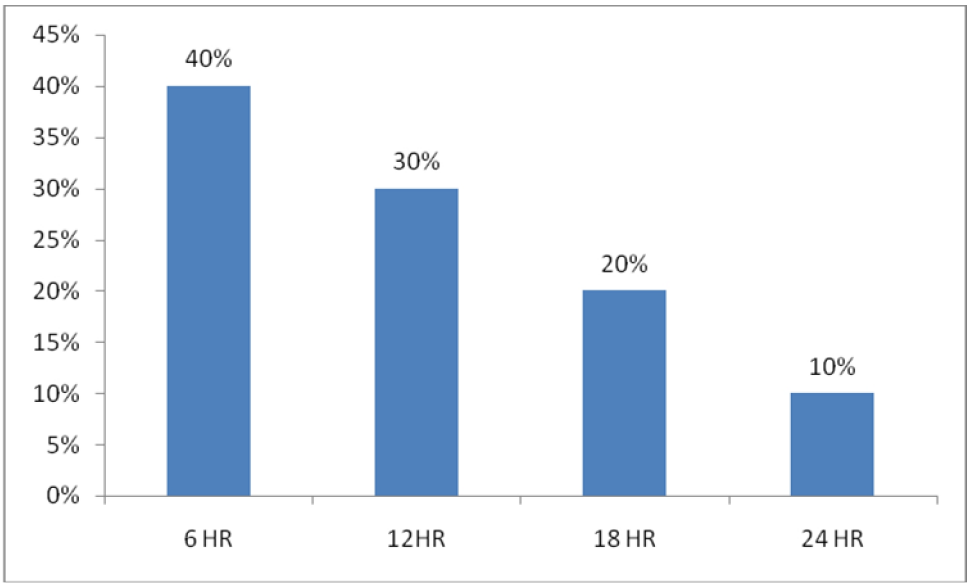


Fig2. Usage frequency of mosquito repellent

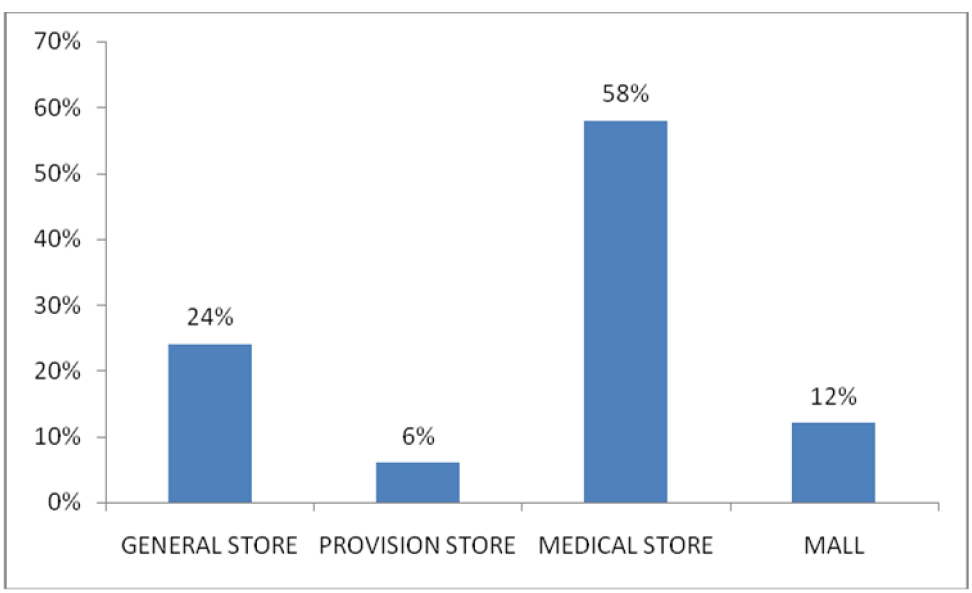


Fig3. Purchasing destination of mosquito repellent

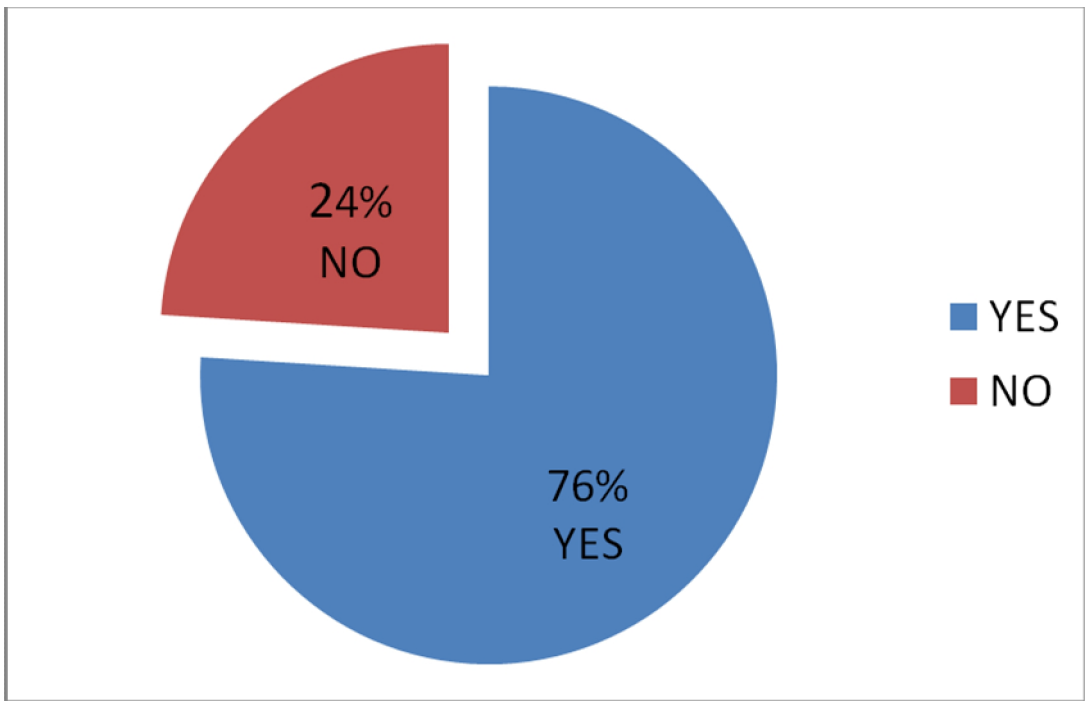


Fig4. Public opinion for danger of mosquito reppellent

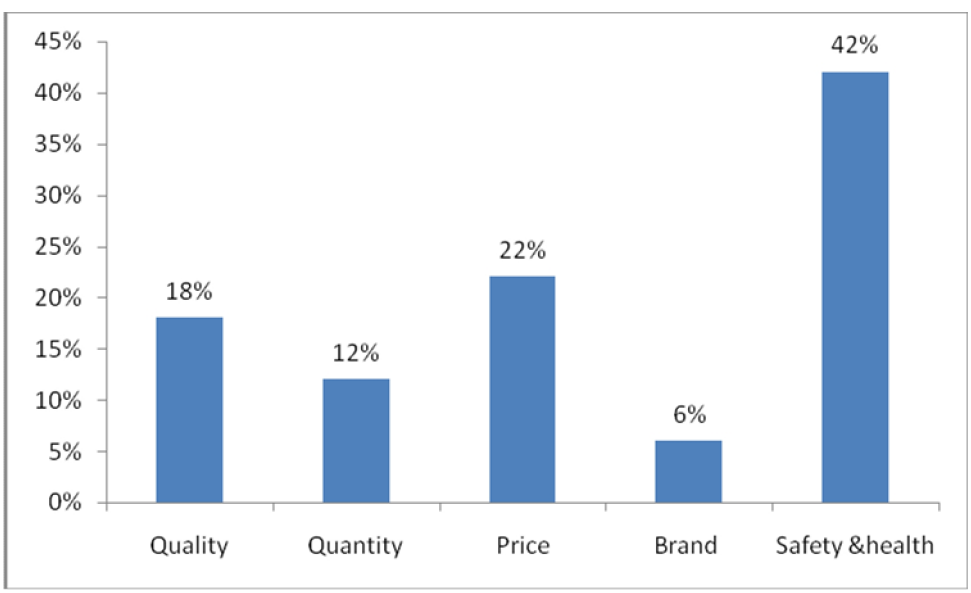


Fig5. Factor for selection of mosquito repellent

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ANNEXURE:

Format of Questionnaire:

1. Are you aware of danger of mosquitoes?

☐ yes ☐ No

If yes, then do you care against mosquito bite?

☐ yes ☐ No

2. Have you suffered from any disease by mosquito in past?

☐ No ☐Yes ☐ If yes how many times... ☐Name of disease……..

3. Amongst following which type of mosquito repellent do you use?

☐ All Out ☐ Hit spray ☐ Mortein Coil ☐Odomos Lotion ☐Electric mat ☐Any other

4. How did you know about product?

☐TV advertisement ☐News paper ☐By reference ☐Any other way……

5. From the following which mosquito repellent are you using currently?

☐ All out ☐Good knight ☐ Mortine ☐Any other……

6. What is the frequency of usage product?

☐ 0 – 6 hr ☐0 – 18 hr ☐0 – 12 hr ☐ 24hr

7. From where do you buy the product?

☐ General store ☐Provisional store ☐Medical store ☐Mall

8. If your brand is not available in store will you shift to other brand?

☐ Yes ☐No

9. Do you agree that such products are harmful to health?

☐Yes ☐No

10. According to you which factor is most important for selection of mosquito repellent?

☐Quality ☐Quantity ☐Brand ☐Price ☐ Safety & Healthy

11. Do you refer label while purchasing product?

☐Yes ☐ No

12. If yes what do you see on label?

☐Date of manufacture & expiry date ☐ Price

☐Company name ☐Precaution

13. Do you use any house hold remedies for mosquito repellent?

☐Yes ☐No ☐If yes then which remedies you use …….

14. Do you know the availability of natural anti mosquito in market?

☐ Yes ☐ No

15. When the mosquito problem is high around your house?

☐Morning ☐Afternoon ☐Evening ☐Night

16. Have you experience any problem while using repellent?

☐Yes ☐No

If yes then when…

☐Every time ☐Sometime ☐Rarely ☐Never

17. If yes then what types of problem have you getting?

☐Breathing ☐Sneezing

☐Eye irritation ☐Asthma

☐Headache ☐Skin irritation/ rashes

☐Cough & Cold ☐Other specific……

18. What is the frequency of cleaning of your home surround?

☐ 4 days ☐1 week ☐15 days ☐1 month

19. Is your water tanks are cleaned regularly and mosquito proof?

☐Yes ☐Sometimes ☐Rarely ☐No

20. Do you agree that mosquito repellent product is costly?

☐Yes ☐No

21. What is your opinion to improve the existing repellent in market?

……

……

THANK YOU

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RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

Development and Validation of Stability Indicating RP-HPLC Method for the Simultaneous Estimation of Nifedipine and Candesartan Cilexetil in Synthetic Mixture

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ABSTRACT

A novel stability indicating RP-HPLC method was developed and validated for simultaneous determination of Nifedipine (NIF-60 mg) and Candesartan Cilexetil (CAN-32 mg) in synthetic mixture. Effective chromatographic separation was achieved on a Phenomenexluna ODS C18 using a mobile phase containing Phosphate buffer (pH4): Methanol having ratio of 65:35 v/v. An isocratic HPLC system was used to pump mobile phase with flow rate of 1ml/min, injection volume 20µl and quantification was done at wavelength of 240 nm. Retention times were found, 4.313 min (NIF) and 8.116 min (CAN). Method was validated as per ICH guideline Q2 (R1). Calibration plots were linear for the concentration range of 6-18 µg/ml and 3.2¬-9.6 µg/ml with correlation coefficients 0.999 (NIF) and 0.9999 (CAN). Forced degradation studies were performed with good resolution between the degradants and analytes. The method was found sensitive and stability-indicating as degradation products revealed no interference with the detection.

SUMMARY

For the routine analysis and quantitative quality control of the any formulation of NIF and CAN; the developed HPLC method could be adopted.

Keywords: Nifedipine, Candesartan Cilexetil, Forced Degradation, RP-HPLC, Stability study

INTRODUCTION

Nifedipine is a dihydropyridineCa++ channel antagonist. It is also used for the treatment of vascular disorders such as Raynaud's phenomenon. Particularly nifedipine is used as a second line treatment in hypertension during pregnancy. Nifedipine is official in IP, BP and USP. Nifedipine is chemically known as [1,4-dihydro-2,6-dimethyl-4-(2- nitrophenyl)-3,5-pyridine dicarboxylic acid dimethyl ester *(1,2)*. Candesartan Cilexetil is potent, orally active and selective angiotensin II receptor antagonist, selective for AT1 receptors, with tight binding to and poor dissociation from the receptor and without agonist activity. Rapidly it can be converted to the active substance, Candesartan, due to ester hydrolysis during absorption from the gastrointestinal tract. It belongs to benzimidazole class and is used in treatment of angina and high blood pressure. Candesartan Cilexetil is chemically known [1-[[(cyclohexyloxy) carbonyl]oxy]ethyl2- ethoxy-1-[[2'-(1H-tetrazol-5-yl)[1,1'-biphenyl]-4-yl]methyl]-1H *(3,4)*. Both drugs are formulated together as a tablet for treatment of hypertension. Chemical structures of Nifedipine and Candesartan Cilexetil are shown in Figure 1.

Clinical trials for phase III studies for the selected combination has been completed with the dosage ratio of 60 mg of Nifedipine and 32 mg of candesartan Cilexetil*(5)*.

The detailed literature review revealed that, only few of the methods have been reported for the individual estimation of Nifedipine and Candesartan Cilexetil and for combination with other drugs in bulk and pharmaceutical formulations. Any stability indicating RP-HPLC method for Nifedipine and Candesartan Cilexetil in combined pharmaceutical dosage form has not been reported *(6–8, 9–11)*. Therefore, it was thought of interest to develop and validate accurate, precise and simple stability indicating assay method for simultaneous determination of NIF and CAN in synthetic mixture.

MATERIALS AND METHODS

Chemicals and Reagents

Pure Nifedipine drug sample was procured from Osaka Pharmaceuticals, Vadodara and Candesartan Cilexetil (CAN) drug sample was gifted by Alembic Pharma, Vadodara.

Methanol, Acetonitrile, Phosphoric acid, Sodium hydroxide, ammonium acetate, hydrochloric acid and hydrogen peroxide were purchased from Merck Specialities Pvt. Ltd. (Mumbai, India). All reagents used were of analytical grade except Methanol, HPLC water and Acetonitrile,, which were of HPLC grade.

Marketed formulation is not available for combination product, so synthetic mixture was prepared which has a composition of 60 mg of NIF and 32 mg of CAN.

Instrumentation

The isocratic high-pressure liquid chromatography system (SCHIMADZU LC-2010) with an autosampler and PDA detector was used for HPLC analysis. The analytical balance (BL-220H, Shimadzu Corporation, Japan) was used for precise and accurate weighing. The pH was determined using pH-meter (LI 610, Elico). Ultasonicator (FS 4, Frantline, India) was used to sonicate mobile phase and other reagents used for HPLC analysis.

Chromatographic Conditions

Optimization of experimental conditions of HPLC was done using Phenomenex Luna C18 (250 mm X 4.6 mm internal diameter, 5µm Particle size) analytical column. The drugs were subjected to separation using water, ortho Phosphoric acid, acetonitrile, and methanol at different compositions. An isocratic mobile phase consisting of Phosphate buffer pH-4: methanol in the ratio of 65:35 (v/v) at a temperature 25 ºC and flow rate of 1ml/minute, was the final optimized chromatographic conditions. The 0.45 µm microporous filter was used to filter the mobile phase and mobile phase was degassed by sonication before use. Both of the drugs showed satisfactory absorption at 240 nm wavelength, it was selected as

detection wavelength for the determination of NIF and CAN. The injection volume was 20 µl and the run time was 15 minutes.

Preparation of Mobile Phase

Phosphate buffer pH 4: Methanol (65:35)

Mobile phase was prepared by filtering and degassing the mixture of 65 ml of Phosphate buffer pH 4.0 and 35 ml of methanol.

Preparation of Standard Solution

60 mg of standard NIF and 32 mg of standard CAN were accurately weighed and transferred to a 100ml of dry and clean volumetric flask. 50 ml of methanol was used as a diluent, sonication was performed to dissolve, filtered through 0.45um membrane filter and the final volume was adjusted with diluent to give a solution having strength of 600µg/ml of NIF and 320µg/ml of CAN. From above solution, 25ml solution was taken and diluted upto 100 ml with same solvent to give solution of 150µg/ml and 80µg/ml NIF and CAN, respectively. Working standard solution of required strength was prepared by diluting stock solution.

Preparation of sample solution

20 tablets of prepared synthetic mixture were accurately weighed, powdered and taken quantity of powder, equivalent to 60 mg NIF and 32 mg CAN, transferred into 100ml volumetric flask and dissolved with 50 ml of diluent by sonication. 0.45µm membrane filter was used to filter the solution and diluents to wash the residues thoroughly. The washings and filtrate were mixed in a 100 ml volumetric flask and diluted upto mark with diluent. After filtering the solution, 25ml was pipette out into a 100ml volumetric flask and adjusted to 100 ml using diluents to give concentration 150µg/ml of NIF and 80µg/ml of CAN. From the above solution, 1ml was pipette out into 10 ml volumetric flask and made upto 10 ml with diluent to make final concentration 15µg/ml of NIF and 8µg/ml of CAN.

Forced Degradation Study *(10)*

Standard drug stock solutions

In order to establish stability-indicating nature of the method, standards of drugs, drug product and placebo were subjected to various stress conditions such as acid/ base hydrolysis, oxidative and thermal to conduct forced degradation studies. Stock solution of standard NIF (150µg/ml) and CAN (80µg/ml) were prepared separately using diluent. The same stock solutions were used after required dilutions for forced degradation studies to provide an indication of the stability indicating property and specificity of proposed method.

Acid degradation

An acidic media was used to perform forced degradation by taking 1ml of the standard stock solution of NIF and CAN, each in separate 10 ml of amber coloured volumetric flask. 1ml of 0.1 N HCl was added to that and solutions of NIF and CAN were kept separately for 12 hr at room temperature for degradation. The degradation samples were then neutralized using 0.1 N NaOH prior to HPLC analysis.

Alkali degradation

The basic media was used to perform forced degradation by taking 1ml of the standard stock solution of NIF and CAN, each in separate 10 ml of amber volumetric flask. 1ml of 0.1 N NaOH was added to that and solutions of NIF and CAN were kept separately for 12 hr at room temperature for degradation. The degradation samples were then neutralized using 0.1 N HCl prior to HPLC analysis.

Oxidative degradation

For Oxidative Degradation, 1 ml of the standard stock solution of NIF and CAN, each was taken in separate 10 ml of volumetric flask. Then 3ml of 3%H2O2 was added and the solutions of NIF and CAN were kept separately for 3 hr at room temperature for degradation.

Thermal Degradation

100 mg of synthetic mixture powder of NIF and CAN was taken in separate 10 ml of volumetric flask to perform thermal degradation and kept at controlled temperature in oven for 10 hrs at 105ºC. 5ml of diluent was added to that and sonicated for 5 minutes. The volume was made upto the mark.

Photodegradation

1ml of the standard stock solution of NIF and CAN, each was taken in separate volumetric flask to perform photo degradation. Later, the solutions of NIF and CAN were exposed to sun light for 3 hrs for degradation.

For the HPLC analysis, the degradation samples were diluted with diluent to get final concentration 15µg/ml and 8µg/ml of NIF and CAN, respectively. Besides, the solutions were prepared containing 15µg/ml NIF and 8.0µg/ml CAN separately, without performance of degradation of both of the drugs. 20µl solution was subjected to analyse in the HPLC system. Chromatograms were recorded and % degradation of drug was measured.

Validation of the Developed Method*(12)*

According to the ICH guideline Q2 (R1), the developed method was validated using various validation parameters.

Linearity and range

Linearity study was carried out at five different concentration levels by constructing calibration curves for the concentration range of 6-18µg/ml (NIF) and 3.2-9.6µg/ml (CAN).

Specificity

To check specificity of the method, excipients such as Lactose monohydrate and HPMC were spiked in an accurately weighed quantity of 60mg and 32mg of NIF and CAN respectively. The final solutions were prepared in the range of 6-18µg/ml and 3.2-9.6µg/ml of NIF and CAN, respectively. Then area was measured and a calculation was done to determine % interference.

Accuracy

Using recovery study from sample solution, the accuracy of the method was confirmed at three different levels of standard addition (80%, 100%, and 120%) of targeted solution (7.5µg/ml of NIF and 4.0µg/ml of CAN) in triplicate. Chromatogram was recorded and the % recovery of each aliquot was calculated.

Precision

Repeatability (n=6)

Standard mixture solution containing 15µg/ml of NIF and 8.0µg/ml of CAN were prepared and procedure was repeated six times. Percentage relatives of standard deviation was calculated for the recorded chromatograms.

Intraday precision (n=3)

Standard solutions containing 12, 15 and 18µg/ml of NIF and 6.4, 8.0 and 9.6µg/ml of CAN were prepared and analyzed within the day for three times. Standard deviation and percentage relatives of standard deviation was calculated for the recorded chromatograms.

Interday precision (n=3)

Standard solutions containing 12, 15 and 18µg/ml of NIF and 6.4, 8.0 and 9.6µg/ml of CAN were prepared and analyzed three times on the three different days. Standard deviation and percentage relatives of standard deviation was calculated for the recorded chromatograms.

Robustness

Few parameters were deliberately varied for evaluation of robustness. Different parameters were included like, change in mobile phase composition and variation of flow rate. The change was made at three levels and replicate for three times. Calculation was done for system suitability parameters of NIF and CAN.

Limit of Detection and Limit of Quantitation

Curve was calibrated for six times and the SD of the intercepts was calculated. LOD and LOQ were calculated using following formulas:

LOD= (3.3×SD)/Slope and LOQ= (10×SD)/Slope

RESULTS AND DISCUSSION

Method Development and Optimization

A simple and efficient stability indicating HPLC method for the assay of NIF and CAN in combination was developed and preliminary tests were performed for selection of the optimum and suitable conditions. HPLC parameters like, appropriate mobile phase, flow rate, their proportions, detection wavelength, and column temperature were carefully studied. To achieve optimum conditions, different mobile phases were examined. 240 nm wavelength was selected as wavelength of detection of NIF and CAN (100µg/ml) because at this wavelength, both drugs have significance absorbance.

Using a Phenomenex Luna C18 column and Phosphate buffer pH 4 adjusted with ortho phosphoric acid : Methanol (65:35) as optimized mobile phase, a symmetrical peak with good resolution and acceptable retention time was achieved. A typical placebo chromatogram for optimized conditions is shown in Figure 2. Chromatographic representation for standard mixture solution of NIF (15µg/ml) and CAN (8µg/ml) for the optimized condition is shown in Figure 3.

System Suitability

The system suitability parameters were performed by injecting the standard mixture solutions of NIF (15µg/ml) and CAN (8µg/ml) six times and analysing for various parameters like, retention time, theoretical plates, peak area, height equivalent to theoretical plate, and peak asymmetry. %RSD of less than 2% for all parameters was obtained from the system suitability results. Table 1 reveales that, the proposed method meets the accepted requirements.

Linearity

Calibration curves were prepared for the concentration range of 6-18µg/ml and 3.2-9.6µg/ml for NIF and CAN, respectively. The regression line was linear with R2 of 0.999 and 0.9999 for NIF and CAN, respectively.

Figure 4 and Figure 5 show calibration curves for NIF and CAN, respectively. Linearity data is shown in Table 2.

Specificity

Specificity was performed by calculating % interference before and after addition of excipients in the standard solutions of both the drugs. % interference of Nifedipine and Candesartan cilexetil was found within acceptance criteria as per ICH guidelines i.e. less than 1% shown in Table 3. So, the proposed method is specific in nature for estimation of Nifedipine and Candesartan cilexetil.

Accuracy

% Recovery of NIF and CAN was within acceptance criteria as per ICH Guidelines i.e. 98-102% and %RSD not more than 2.0%. Table 4 shows % Recovery data.

Precision

Result obtained from repeatability, Intraday precision and Interday precision reveals that SD and % RSD of Nifedipine and Candesartan cilexetil was within acceptancelimits as per ICH guidelines i.e. less than 1 and less than 2, respectively. So, the proposed method is precise in nature for estimation of Nifedipine and Candesartan cilexetil. Table 5, table 6 and table 7 represent Repeatability, Intraday precision and Interday precision data.

Robustness

Evaluation of robustness study was carried out by calculating % RSD of system suitability parameters such as area, tailing factor and resolution. Result reveals that, the selected factors were unaffected by slight variation of these parameters and %RSD was found less than 2, which demonstrates robustness of method. Table 8 summarizes variations imposed on the chromatographic method.

LOD and LOQ for NIF and CAN

Small amount of drugs can be detected and quantified precisely by the proposed method. Hence, sensitivity of proposed method was revealed. Table 9 shows LOD and LOQ data.

Forced Degradation Study

The various stress conditions studied were acid - base hydrolysis, oxidative degradation, thermal and photolysis. The sample stress solutions were analysed with reference to the standard mixture solutions of NIF and CAN. Table 10 summarizes % degradation of sample solutions and standards under stressed conditions. Table 11 enlists the degradation products.

Table 10 reveals that, CAN has shown maximum degradation for basic degradation condition. NIF has shown maximum degradation under photolytic and thermal degradation conditions. Resolution was found to be greater than 2.0 for both of the active ingredients. After exposure of the sample solution to different stress conditions, chromatograms of API and degradation products are shown in Figures (6-11).

Applicability of the Method to the Marketed tablet formulation

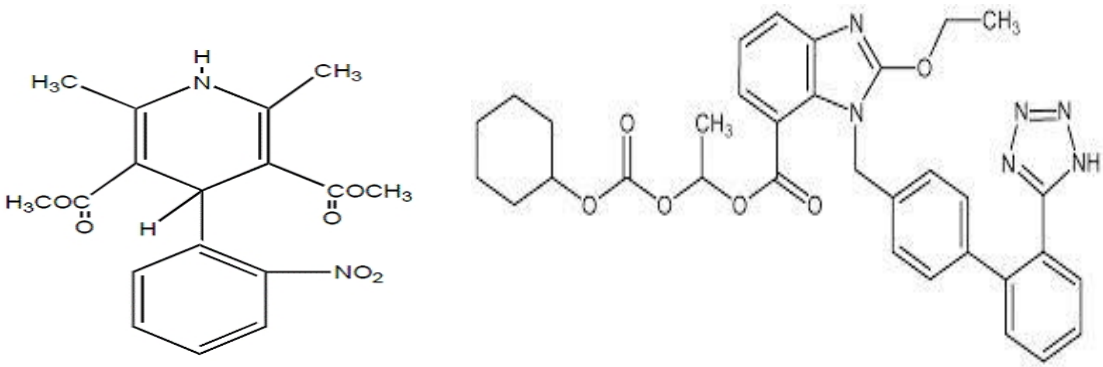
Satisfactory results were obtained for analysis of NIF and CAN by the developed stability indicating assay method. The validated method was used for marketed tablet formulation as shown in Table 12.

Acceptable values from the table 12 reveal that, the proposed method can be applicable for the routine analysis of the any dosage form of NIF and CAN, without any interference of the excipients.

CONCLUSION

A selective and validated stability-indicating RP-HPLC assay method for Nifedipine and Candesartan Cilexetil was developed for synthetic mixture on a C18 column, which could separate the drug and its degradation products formed under all stressed conditions. The developed method was evaluated for various validation parameters like, specificity, linearity and range, system suitability, accuracy (recovery), precision (intraday and interday precision, repeatability), and robustness. All the validation parameters were within the range given in the ICH guideline Q2 (R1). As a result, for the routine analysis and quantitative quality control of any formulation of NIF and CAN; the developed HPLC method could be adopted.

FIGURES



I **II**

Fig. 1. Chemical structure of (I) Nifedipine and (II) Candesartan Cilexetil

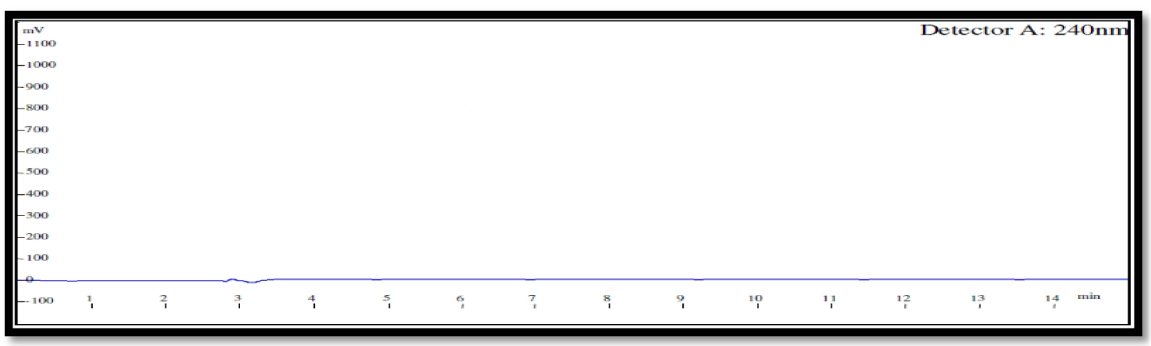


Fig. 2. Chromatogram of the Placebo

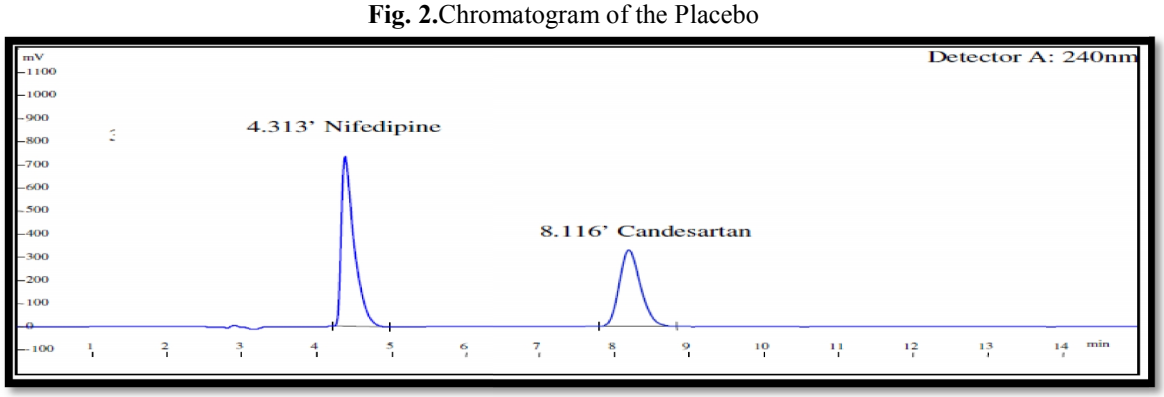


Fig. 3. Chromatographic representation of a standard mixture solution of 15µg/ml of NIF (4.313 min),8.0µg/ml of CAN (8.116 min)

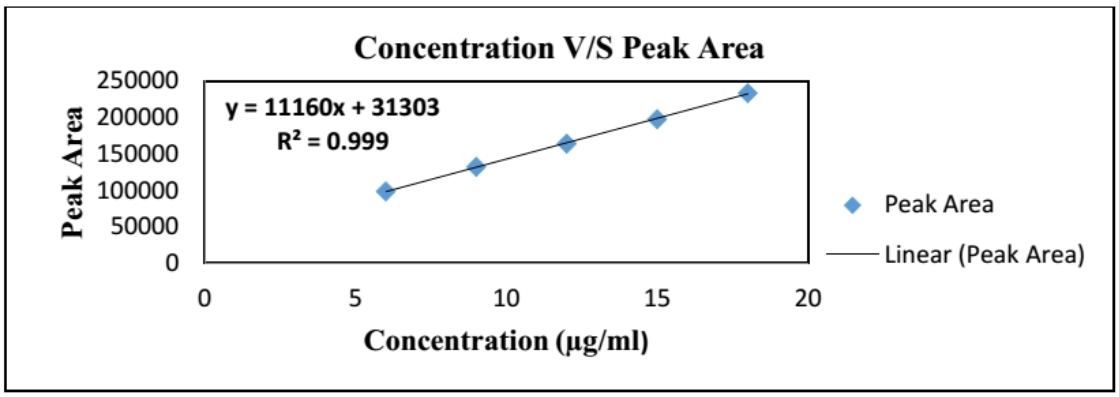


Fig. 4. Calibration curve for Nifedipine

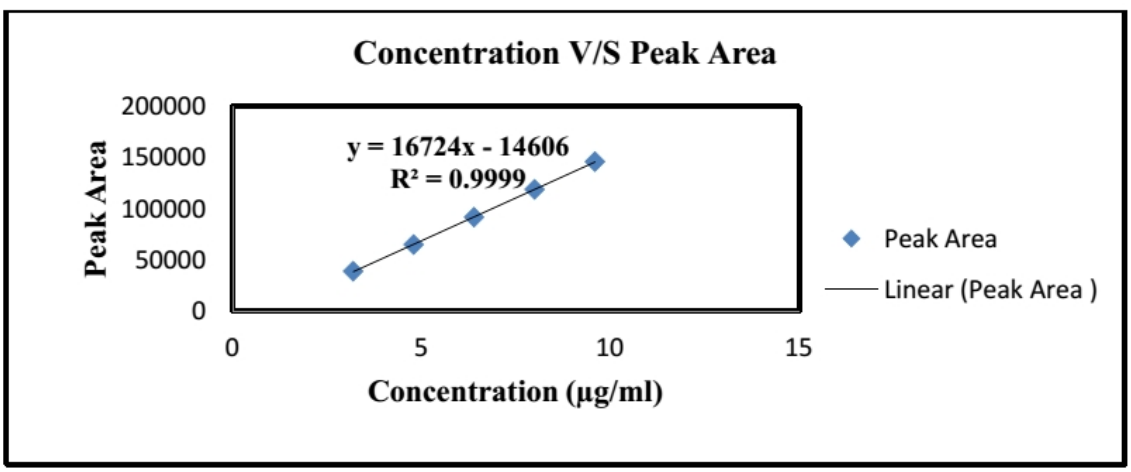


Fig. 5. Calibration curve for Candesartan Cilexetil

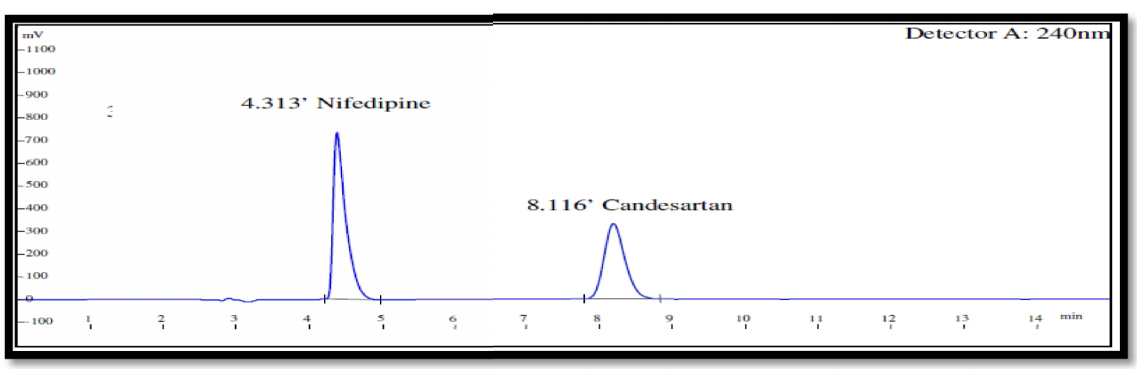


Fig. 6. Chromatographic representation of sample under normal chromatographic condition. NIF 15µg/ml and CAN 8.0µg/ml

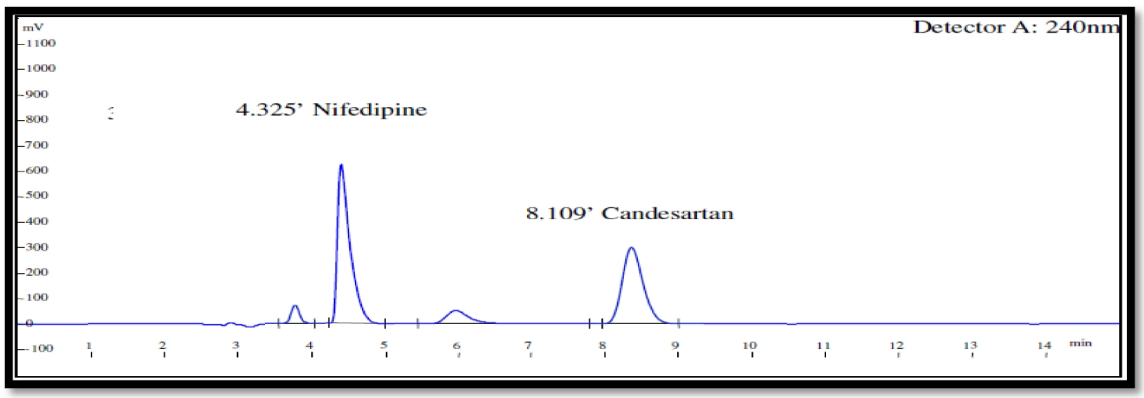


Fig. 7. Chromatographic representation of acidic hydrolysis of the synthetic mixture solution of NIF and CAN after reflux for 12 hr

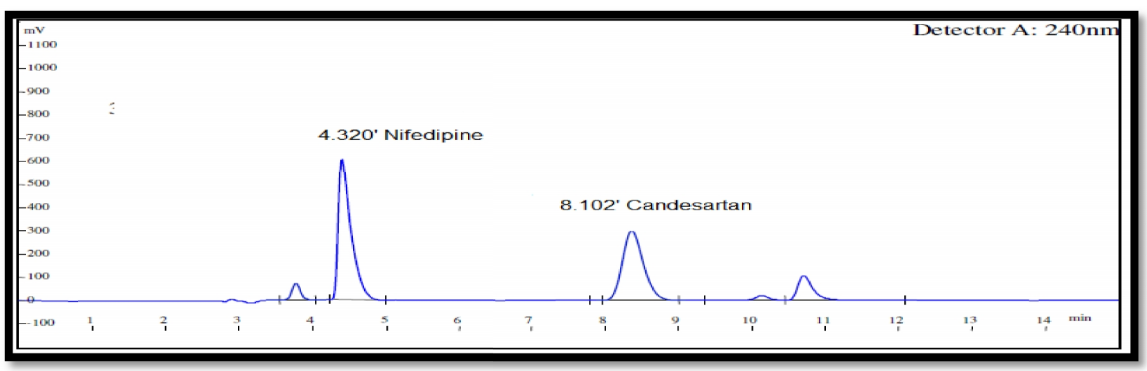


Fig. 8. Chromatographic representation of alkaline hydrolysis of the synthetic mixture solution of NIF and CAN after reflux for 12 hr

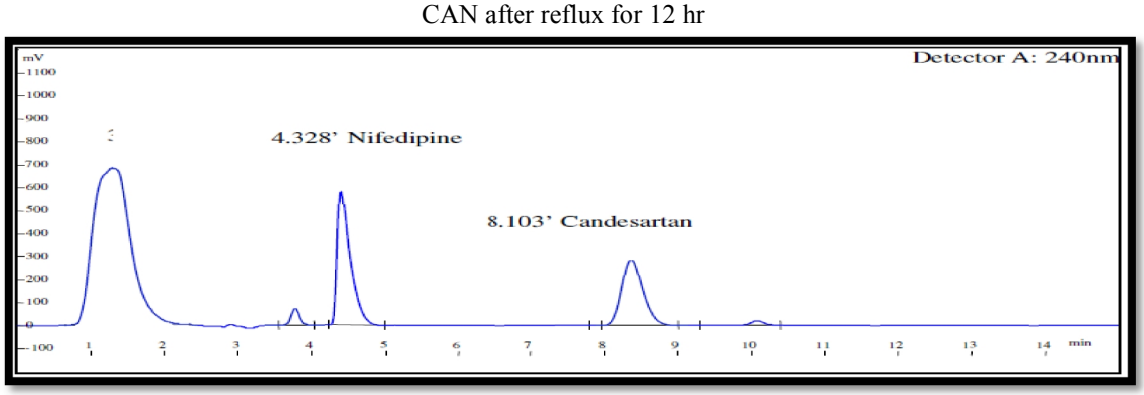


Fig. 9. Chromatographic representation of oxidative degradation (3% H_2O_2) of the synthetic mixture solution of NIF and CAN after reflux for 3 Hr

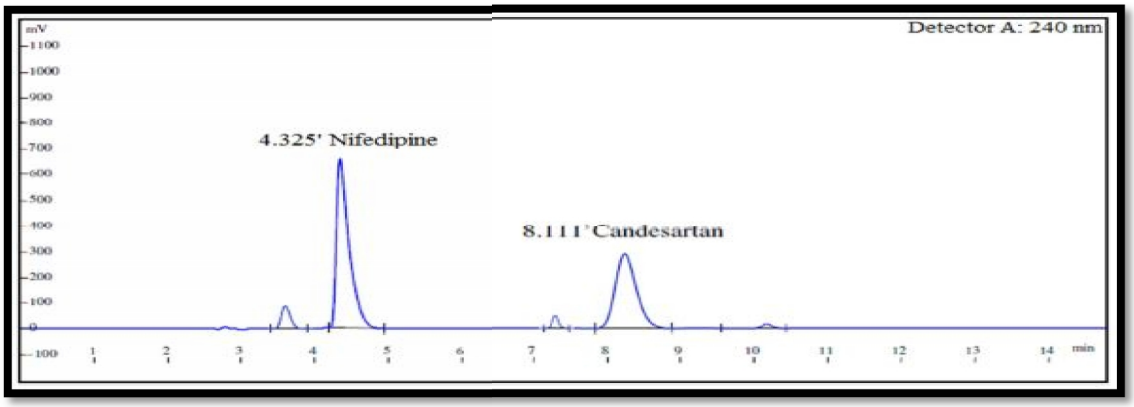


Fig. 10. Chromatographic representation of thermal degradation of the synthetic mixture solution of NIF and CAN after 10 hrs at 105 ºC

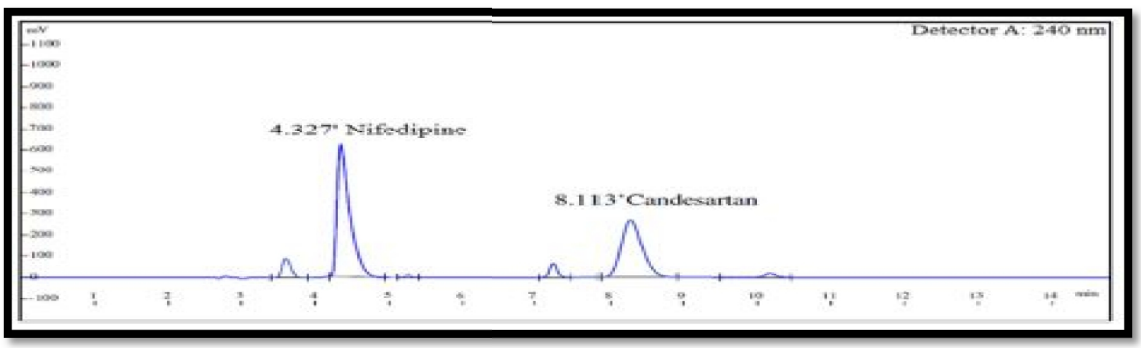


Fig. 11. Chromatographic representation of photolytic degradation of the synthetic mixture solution of NIF and CAN after 3 hrs at Sunlight

TABLES

Table 1. System suitability parameters

Parameters	NIF	CAN	Standard Value
Retention time (R_t)	4.313min	8.116 min	-
Resolution (R_s)	-	3.91	≥ 2.0
Theoretical plates (N)	7954	5894	≥ 2000
Tailing factor (T)	1.21	1.32	≤ 2.0

Table 2. Linearity data of Nifedipine and Candesartan Cilexetil

Sr. No	Concentration(µg/ml)		PeakArea ± SD (n=3)	
	NIF	CAN	NIF	CAN
1	6	3.2	98706 ± 129.93	39359 ± 42.85
2	9	4.8	132260 ± 796.49	65337 ± 83.11
3	12	6.4	164018 ± 1199.53	92066 ± 44.81
4	15	8.0	197746 ± 1016.98	119119 ± 4288.81
5	18	9.6	233356 ± 1004.17	146261 ± 599.32

Table 3. Specificity data for Nifedipine and Candesartan cilexetil

Drug	Concentration (µg/ml)	Area before addition of Excipients	Area after addition of Excipients	% Interference
Nifedipine	6	98706	98816	0.111
	9	132260	132415	0.117
	12	164018	163976	0.026
	15	197746	197786	0.020
	18	233356	233611	0.109
Candesartan cilexetil	3.2	39359	39639	0.711
	4.8	65337	65751	0.633
	6.4	92066	92234	0.182
	8.0	119119	119011	0.090
	9.6	146261	146024	0.162

Table 4. Accuracy data for Nifedipine and Candesartan cilexetil

Drug	Recovery Level	Conc. of standard solution (µg/ml)	Total conc. (µg/ml)	Sample Peak Area (n=3)	Conc. of Drug found (µg/ml) (n=3)	Mean % recovery ± SD (n=3)
NIF	80% [7.5]	6	13.5	181961	13.49	99.86 ± 0.15
	100% [7.5]	7.5	15	198565	14.98	99.73 ± 0.46
	120% [7.5]	9.0	16.5	215669	16.52	100.22 ±0.40
CAN	80% [4]	3.2	7.2	105695	7.18	99.56± 0.32
	100% [4]	4.0	8.0	118984	7.98	97.50± 0.63
	120% [4]	4.8	8.8	133125	8.84	100.83 ±0.32

Table 5. Repeatability results for NIF and CAN

Drug Name	Concentration (µg/ml) (n=6)	Peak Area (n=6)	Mean Concentration(µg/ml) ± SD	%RSD
NIF	15	197853.7	14.85±0.40	0.16
CAN	8.0	118722.2	7.93±5.22	0.40

Table 6. Intraday precision results for NIF and CAN

Drug Name	Sr. No.	Concentration (µg/ml) (n=3)	Peak Area (n=3)	Mean Concentration (µg/ml) ± SD	%RSD
Nifedipine	1	12	164219.7	12.13 ±0.38	0.15
	2	15	196992.7	14.98 ±0.68	0.41
	3	18	234650.3	18.03 ±0.14	0.78
Candesartan Cilexetil	1	6.4	92020.3	6.45 ± 0.47	0.11
	2	8.0	117982.7	8.03 ± 0.42	0.32
	3	9.6	145414.0	9.58 ± 0.60	0.57

Table 7. Interday precision results for NIF and CAN

Drug Name	Sr. No.	Concentration (µg/ml) (n=3)	Peak Area (n=3)	Mean Concentration (µg/ml)± SD	%RSD
Nifedipine	1	12	165478.3	12.11 ±0.39	0.37
	2	15	178991.6	14.95 ±0.73	0.52
	3	18	233772.4	18.11 ±0.15	1.00
Candesartan Cilexetil	1	6.4	92135.2	6.48 ± 0.51	0.36
	2	8.0	127872.6	8.07 ± 0.48	0.57
	3	9.6	143213.0	9.63 ± 0.81	0.77

Table 8. Robustness data of NIF and CAN

Parameters Varied		Drug name	Systemsuitability parameters (n=3)		
			Mean PeakArea ± %RSD	Tailingfactor ± %RSD	Resolution ± %RSD
As perproposed Method		NIF	197509 ± 0.1797	1.174±0.159	3.916 ± 0.0390
		CAN	117982 ± 0.3259	1.319±0.234	
Changein flowrate	0.8ml/ Min	NIF	196100 ± 0.0288	1.266 ±0.517	3.747 ± 1.5596
		CAN	120787 ± 0.0223	1.313±1.186	
	1.2ml/ Min	NIF	201168 ± 0.0168	1.226±0.517	3.849 ± 1.4409
		CAN	118588 ± 0.0330	1.321±1.186	
Changein Mobile phase composition	(63:37)	NIF	121587 ± 0.0223	1.237±0.080	3.917 ± 0.0390
		CAN	198968 ± 0.0168	1.323±0.269	
	(67:33)	NIF	118788 ± 0.0330	1.226±0.046	3.845 ± 1.4365
		CAN	123287 ± 0.0223	1.321±0.284	

Table 9. LOD and LOQ for NIF and CAN

Drug	LOD(µg/ml)	LOQ(µg/ml)
Nifedipine	0.106	0.324
Candesartan Cilexetil	0.086	1.98

Table 10. Forced degradation studies of NIF and CAN

Stress Conditions	% Degradation of API		% Degradation of formulation	
	NIF	CAN	NIF	CAN
0.1N Hydrochloric acid, reflux, 12 hr	15.0	16.4	15.6	16.1
0.1N Sodium Hydroxide, RT, 12 hr	19.8	21.4	19.5	21.1
3% w/v Hydrogen peroxide, 3 hr	18.6	16.0	18.3	16.4
Thermal, 10 hr at 105ºC	20.8	17.8	21.2	16.9
Photolytic, Sun light for 3 hr	21.6	16.2	21.1	16.5

Table 11. Forced degradation product of NIF and CAN Tablet solution

Forced Degradation Conditions	Nifedipine		Candesartan Cilexetil	
	RT of Drug	RT of Degradation Products	RT of Drug	RT of Degradation Products
0.1N Hydrochloric acid, reflux, 12 hr	4.325	3.800	8.109	6.001
0.1N Sodium Hydroxide, RT, 12 hr	4.320	3.805	8.102	10.227, 10.871
3% w/v Hydrogen peroxide, 3 hr	4.328	3.798	8.103	10.114
Thermal, 10hr at 105ºC	4.325	3.795	8.111	7.352, 10.124
Photolytic, Sunlight for 3 hrs	4.327	3.795, 5.210	8.113	7.356, 10.134

Table 12.% Assay of marketed tablet formulation

Drug	Conc. of Dosage Form	Conc. taken for assay (µg/ml)	Conc. Found(µg/ml) ± SD (n = 3)	%Assay± SD (n = 3)
NIF	60 mg	7.5	7.505 ± 0.15	100.07 ± 0.089
CAN	32 mg	4	3.99 ± 0.38	99.75 ± 0.028

ACKNOWLEDGEMENT

The authors would like to thank Alembic Pvt. Ltd. (Vadodara, India) for providing a gift sample of the standard Candesartan Cilexetil and Parul Institute of Pharmacy and research for providing the necessary facilities to carry out the research work.

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Formulation and Evaluation of Fast Disintegrating Metoprolol Succinate Sublingual Tablets

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ABSTRACT

In acute diseases, fast action of drug is required. Metoprolol succinate is used in hypertension and angina pectoris. Here we have prepared sublingual tablet of Metoprolol succinate using various superdisintegrants in concentration range of 1 to 10%. In vitro dissolution of formulation F7,8,25,26,27,28,31,32.35,36,37,38,39 and 40 showed more than 90 percent drug release within 3 minutes. Wetting time for all formulations was found to be 55 to 368 seconds. The water absorption ratios for all formulations were found to be 12.37 % to 97.06 %. The in vitro disintegration time for all formulation was found to be 10 second to 12 minute. Results shown that xanthan gum, cross povidone, colloidal silicon dioxide and alginic acid does not fulfill the requirement of disintegration time for sublingual tablet.

SUMMARY

This research work include formulation of fast disintegrating Metoprolol succinate sublingual tablet.

Keywords: Superdisintegrants, metoprolol succinate, sublingual, tablet, fast disintegrating

INTRODUCTION

The oral dosage form offers various advantages like ease of self administration, compactness and easy manufacturing. So it is considered as the most widely accepted and flexible route of drug administration. But common problem with oral dosage form is difficulty in swallowing for all age group. Especially

elderly, children, and patients who are mentally retarded, uncooperative or nauseated facing greater problem in swallowing oral dosage form *(1)*.

Fast disintegrating sublingual tablet is delivering the drug beneath the tongue and disintegrate rapidly within few minutes in presence of saliva. It offers various benefits like fast release of drug from the dosage form and bypasses the first pass hepatic metabolism of the Metoprolol succinate into the liver and it reaches the systemic circulation directly, so gives fast relief from the anginal pain and hypertension. Because bypassing of first pass hepatic metabolism, the bioavailability with drug is also increases. So, without need of swallowing, we can achieve fast release of drug. Also, the time required for onset of drug action for a conventional oral tablet is more which is generally not acceptable for acute disorders, but the sublingual drug delivery is most acceptable *(2)*.

Angina pectoris is acute disorder characterized by chest pain or discomfort due to coronary heart disease. In case of Hypertension (HTN), the blood pressure in the arteries is elevated *(3, 4)*. Metoprolol succinate is a cardio selective β1 adrenoreceptor blocker. It is mostly used in the treatment of acute disorders such as angina pectoris and also in chronic disease hypertension. It is a BCS (Biopharmaceutical Classification System) class-1 drug. It has high solubility and high permeability. Metoprolol succinate is freely soluble in water and methanol. The half life of Metoprolol succinate is approximately 3 to 4 hours. It undergoes extensive first pass hepatic metabolism resulting in 40% oral bioavailability. Hence the prepared sublingual tablet of Metoprolol succinate lead to enhance the bioavailability and avoidance of first pass hepatic metabolism *(5)*.

MATERIALS AND METHODS *(6-52)*:

Type here Metoprolol succinate (API), mannitol (Directly compressible material), microcrystalline cellulose (diluent and disintegrating agent), crosspovidone (super disintegrating agent), cross carmellose sodium (super disintegrating agent), xanthan gum (super disintegrating agent), chitosan (super disintegrating agent), colloidal silicon dioxide (super disintegrating agent), alginic acid (super disintegrating agent), agar (super disintegrating agent), guar gum (super disintegrating agent), karaya gum (super disintegrating agent), gellan gum (super disintegrating agent), polyvinyl-pyrrolidone K-30 (binder), sodium saccharine (sweetener) and talc (lubricant).

Formulation of Metoprolol Succinate Fast Disintegrating Sublingual Tablets:

Direct compression technique was used for formulation of sublingual tablets of Metoprolol succinate. All ingredients were first passed through 40# sieve. An accurate amount of drug and all excipients were homogenously blended using geometric dilution method. Here talc was added at last for lubrication and mix thoroughly. The tablets were prepared with the help of 7 mm flat punch by rotary tablet punching machine *(53, 54)*. (Table 1 to 5)

Pre-formulation study:

The drug-excipients compatibility study was performed by FT-IR spectroscopy *(53, 54)*.

Micromeritic properties of tablet powder mixture:

The flow properties were evaluated by various parameters like bulk density, tapped density, angle of repose, carr's index and hausner's ratio *(55, 56)*.

Evaluation of Tablets:

Appearance:

Tablets were evaluated for shape, colour, odor, taste etc *(55)*.

Thickness and Diameter:
The size of tablets was evaluated by Vernier calipers *(55)*.

Hardness:
The hardness was measured using Monsanto hardness tester. Three tablets were randomly selected from each formulation and the average hardness was noted *(54)*.

Friability:
This test was performed to determine the effects of friction and shock. Pre-weighed tablets was placed in the friabilator (roche friabilator) and rotated at 25 rpm (rotation per minute) for 4 minutes. The tablets were dedusted and reweighed, and the percentage friability was calculated using standard equation *(53, 54, 56, 58)*.

Weight variation:
Twenty tablets were selected randomly from each formulation, weighed individually and the average weight and % variation of weight was calculated *(53)*.

Wetting Time:
A piece of tissue paper was cut circularly (6.5 cm diameter) and placed on a petridish containing 6 ml of water at room temperature *(53, 54, 59)*. A tablet was placed on the surface of the tissue paper and the time required for the complete wetting of the tablet was noted.

Water Absorption Ratio:
A piece of tissue paper folded twice was kept in a petridish containing 6 ml of purified water. The tablet was placed on the tissue paper and allowed to wet completely. The wetted tablet was removed and reweighed *(56)*.
Water absorption ratio (R) is calculated using below equation;

$$R = 100 (W_a - W_b) / W_b$$

Where, W_b =Weight of tablet before absorption, W_a =Weight of tablet after absorption.

Disintegration time:
Official method as per USP:
In vitro disintegration time was determined using a modified disintegration method (n=5) by using disintegration tester at 37±0.5°C in distilled water. The tablet was carefully kept in a basket without covering plastic disks and 2 minutes is specified as the acceptable time limit for tablet disintegration *(54, 60)*.

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RESULTS AND DISCUSSION

Type The use of superdisintegrant for the preparation of fast dissolving sublingual tablet is highly effective and easily available. Here various superdisintegrants were used for preparation of Metoprolol succinate tablet.

The pre-formulation study showed that there was no any interaction between Metoprolol succinate and excipients. Various properties of prepared tablet and preformulation parameters were evaluated. The results obtained are shown in a table. (Table 6 to 8)

Wetting time for all formulations was found to be 55 to 368 seconds. The water absorption ratios for all formulations were found to be 12.37 % to 97.06 %. The *in vitro* disintegration time for all formulation was found to be 10 second to 12 minute.

In vitro dissolution of formulation F7,8,25,26,27,28,31,32.35,36,37,38,39 and 40 showed more than 90 percent drug release within 3 minutes.

CONCLUSION

The fast disintegrating metoprolol sublingual tablets were prepared using various superdisintegrants. We found that cross carmellose sodium, agar, guar gum, karaya gum and gellan gum showed fast disintegration. So, these superdisintegrants can be successfully used for preparation of marketable formulation with good release rate.

TABLES

Table 1. Formulation of batch F1 to F8.

Ingredients	**Formulation Code (Quantity in mg per tablet)**							
	F1	**F2**	**F3**	**F4**	**F5**	**F6**	**F7**	**F8**
Metoprolol succinate	12.5	12.5	12.5	12.5	12.5	12.5	12.5	12.5
Mannitol	64	64	64	64	64	64	64	64
MCC	16	14	12	10	18	16	15	14
Xanthan gum	4	6	8	10	-	-	-	-
CCS	-	-	-	-	2	4	5	6
PVP K-30	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
Sodium saccharine	1	1	1	1	1	1	1	1
Talc	1	1	1	1	1	1	1	1

Final weight of tablet (mg)	100	100	100	100	100	100	100	100

Table 2. Formulation of batch F9 to F16 by Direct Compression Method

Ingredients	**Formulation Code (Quantity in mg per tablet)**							
	F9	**F10**	**F11**	**F12**	**F13**	**F14**	**F15**	**F16**
Metoprolol succinate	12.5	12.5	12.5	12.5	12.5	12.5	12.5	12.5
Mannitol	64	64	64	64	64	64	64	64
MCC	18	16	15	14	16	14	12	10
Chitosan	-	-	-	-	4	6	8	10
Cross povidone	2	4	5	6	-	-	-	-
PVP K-30	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
Sodium saccharine	1	1	1	1	1	1	1	1
Talc	1	1	1	1	1	1	1	1
Final weight of tablet (mg)	100	100	100	100	100	100	100	100

Table 3. Formulation of batch F17 to F24 by Direct Compression Method

Ingredients	**Formulation code (Quantity in mg per tablet)**							
	F17	**F18**	**F19**	**F20**	**F21**	**F22**	**F23**	**F24**
Metoprolol succinate	12.5	12.5	12.5	12.5	12.5	12.5	12.5	12.5
Mannitol	64	64	64	64	64	64	64	64
MCC	16	14	12	10	16	14	12	10
Colloiodal silicon dioxide	4	6	8	10	-	-	-	-
Alginic acid	-	-	-	-	4	6	8	10
PVP K-30	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
Sodium saccharine	1	1	1	1	1	1	1	1
Talc	1	1	1	1	1	1	1	1
Final weight of tablet (mg)	100	100	100	100	100	100	100	100

Table 4. Formulation of batch F25 to F32 by Direct Compression Method

	Formulation code

Ingredients	(Quantity in mg per tablet)							
	F25	**F26**	**F27**	**F28**	**F29**	**F30**	**F31**	**F32**
Metoprolol succinate	12.5	12.5	12.5	12.5	12.5	12.5	12.5	12.5
Mannitol	64	64	64	64	64	64	64	64
MCC	16	14	12	10	16	14	12	10
Agar	4	6	8	10	-	-	-	-
Guar gum	-	-	-	-	4	6	8	10
PVP K-30	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
Sodium saccharine	1	1	1	1	1	1	1	1
Talc	1	1	1	1	1	1	1	1
Final weight of tablet (mg)	100	100	100	100	100	100	100	100

Table 5. Formulation of batch F33 to F40 by Direct Compression Method

Ingredients	Formulation code (Quantity in mg per tablet)							
	F33	**F34**	**F35**	**F36**	**F37**	**F38**	**F39**	**F40**
Metoprolol succinate	12.5	12.5	12.5	12.5	12.5	12.5	12.5	12.5
Mannitol	64	64	64	64	64	64	64	64
MCC	16	14	12	10	16	14	12	10
Karaya gum	4	6	8	10	-	-	-	-
Gellan gum	-	-	-	-	4	6	8	10
PVP K-30	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
Sodium saccharine	1	1	1	1	1	1	1	1
Talc	1	1	1	1	1	1	1	1
Final weight of tablet (mg)	100	100	100	100	100	100	100	100

MCC - Microcrystalline cellulose, CCS - Cross carmellose sodium, PVP- Polyvinyl-pyrrolidone.

Table 6. Bulk density, tapped density, angle of repose, carr's compressibility index and hausner's Ratio

Sr. No.	Formulation code	Bulk density	Tapped density	Angle of repose	Carr's compressibility index	Hausner's ratio
1	F1	0.521	0.623	32.8	16.37	1.196
2	F2	0.524	0.626	29.5	16.29	1.195
3	F3	0.528	0.629	30.9	16.06	1.191
4	F4	0.529	0.632	28.7	16.30	1.195
5	F5	0.521	0.623	30.9	16.37	1.196
6	F6	0.523	0.62	30.4	15.64	1.185
7	F7	0.520	0.60	29.6	13.33	1.154
8	F8	0.521	0.62	29.7	15.97	1.190
9	F9	0.521	0.623	31.9	16.37	1.196
10	F10	0.523	0.62	30.3	15.65	1.184
11	F11	0.520	0.60	30.6	13.33	1.154
12	F12	0.521	0.62	29.1	15.97	1.190
13	F13	0.519	0.620	29.9	16.29	1.194
14	F14	0.513	0.619	31.3	17.12	1.207
15	F15	0.519	0.603	29.6	13.93	1.162
16	F16	0.521	0.62	30.1	15.97	1.190
17	F17	0.501	0.603	34.9	16.92	1.204
18	F18	0.513	0.589	35.3	12.90	1.148
19	F19	0.505	0.597	35.6	15.41	1.182
20	F20	0.507	0.595	34.1	14.79	1.174
21	F21	0.518	0.613	31.9	15.50	1.183
22	F22	0.522	0.619	30.3	15.67	1.186
23	F23	0.519	0.621	28.5	16.43	1.197
24	F24	0.515	0.617	26.9	16.53	1.198
25	F25	0.516	0.609	30.3	15.27	1.180
26	F26	0.520	0.614	30.2	15.31	1.181
27	F27	0.514	0.628	28.3	18.15	1.222
28	F28	0.516	0.620	28.9	16.77	1.202
29	F29	0.534	0.617	30.6	13.45	1.156
30	F30	0.530	0.624	30.8	15.06	1.177
31	F31	0.534	0.627	28.3	14.83	1.174
32	F32	0.529	0.624	28.9	15.22	1.180
33	F33	0.525	0.643	32.9	18.35	1.225
34	F34	0.523	0.625	31.5	16.32	1.195
35	F35	0.524	0.627	32.4	16.43	1.197
36	F36	0.531	0.629	29.1	15.58	1.185
37	F37	0.548	0.633	28.9	13.43	1.155
38	F38	0.542	0.639	30.4	15.18	1.179
39	F39	0.549	0.631	28.6	13.00	1.149
40	F40	0.535	0.647	26.9	17.31	1.209

Table 7. Hardness, thickness, diameter, percentage friability and weight variation of formulation F1 to F40

Formulation	Hardness (kg/cm^2)	Thickness (mm)	Diameter (mm)	% Friability	Average Weight (mg)
F1	3.4± 0.15	1.85±0.02	6.79±0.03	0.88±0.02	102.5±0.52
F2	3.3± 0.20	1.89±0.03	6.83±0.01	0.81±0.01	103.4±0.45
F3	3.7± 0.26	1.87±0.02	6.84±0.03	0.77±0.03	101.2±0.38
F4	3.2± 0.29	1.89±0.01	6.85±0.02	0.84±0.02	104.3±0.36
F5	3.6± 0.16	1.85±0.01	6.84±0.02	0.79±0.01	103.3±0.51
F6	3.7± 0.26	1.88±0.03	6.81±0.02	0.73±0.02	104.6±0.43
F7	3.5± 0.21	1.90±0.02	6.83±0.03	0.81±0.03	102.4±0.55
F8	3.6± 0.22	1.87±0.03	6.87±0.02	0.84±0.01	104.6±0.51
F9	3.5± 0.17	1.86±0.01	6.86±0.03	0.84±0.01	104.7±0.51
F10	3.8± 0.29	1.87±0.03	6.84±0.03	0.77±0.03	102.1±0.65
F11	3.6± 0.19	1.89±0.01	6.86±0.02	0.84±0.02	105.3±0.45
F12	3.7± 0.25	1.90±0.02	6.88±0.03	0.88 ± 0.02	104.5±0.58
F13	3.4 ± 0.18	1.84±0.01	6.89±0.03	0.85 ± 0.03	103.7±0.51
F14	3.3 ± 0.21	1.83±0.03	6.74±0.03	0.87 ± 0.04	101.1±0.28
F15	3.5 ± 0.11	1.85±0.01	6.86±0.02	0.81± 0.04	103.3±0.56
F16	3.6 ± 0.20	1.91±0.02	6.98±0.03	0.89 ± 0.03	102.5±0.36
F17	3.8± 0.27	1.89±0.03	6.89±0.02	0.86±0.02	103.6±0.54
F18	3.9± 0.32	1.76±0.03	6.74±0.04	0.79±0.03	102.2±0.63
F19	3.7± 0.21	1.87±0.02	6.76±0.02	0.82±0.02	103.3±0.45
F20	3.6± 0.24	1.93±0.02	6.78±0.03	0.87 ± 0.02	103.5±0.54
F21	3.5± 0.22	1.79±0.03	6.79±0.03	0.85±0.02	102.6±0.53
F22	3.7± 0.22	1.74±0.03	6.73±0.04	0.76±0.03	103.2±0.64
F23	3.8± 0.23	1.85±0.03	6.82±0.02	0.89±0.02	102.3±0.45
F24	3.5± 0.24	1.90±0.02	6.71±0.03	0.83 ± 0.02	101.5±0.51
F25	3.6± 0.23	1.84±0.03	6.84±0.02	0.84±0.04	102.6±0.51
F26	3.7± 0.32	1.78±0.03	6.71±0.04	0.89±0.03	103.2±0.63
F27	3.6± 0.21	1.85±0.02	6.74±0.02	0.88±0.02	102.3±0.51
F28	3.5± 0.24	1.92±0.04	6.79±0.03	0.91 ± 0.02	101.5±0.54
F29	3.3± 0.18	1.83±0.02	6.77±0.03	0.89±0.02	101.5±0.52
F30	3.4± 0.20	1.79±0.03	6.81±0.01	0.84±0.01	101.4±0.45
F31	3.5± 0.26	1.88±0.02	6.74±0.03	0.79±0.03	102.2±0.38
F32	3.4± 0.29	1.91±0.01	6.84±0.02	0.86±0.02	103.3±0.36
F33	3.6± 0.18	1.86±0.02	6.75±0.03	0.92±0.02	101.5±0.52
F34	3.4± 0.23	1.92±0.03	6.91±0.01	0.88±0.01	102.4±0.48
F35	3.3± 0.26	1.85±0.02	6.72±0.03	0.91±0.03	104.2±0.36
F36	3.6± 0.23	1.94±0.01	6.86±0.02	0.88±0.02	101.3±0.36
F37	3.5± 0.21	1.96±0.03	6.71±0.01	0.94±0.02	102.4±0.51
F38	3.6± 0.26	1.95±0.04	6.94±0.02	0.78±0.02	101.4±0.49
F39	3.4± 0.21	1.85±0.03	6.78±0.02	0.97±0.03	102.3±0.32
F40	3.3± 0.22	1.92±0.03	6.96±0.02	0.89±0.03	103.3±0.35

Table 8. Disintegration time, wetting time, water absorption ratio and drug content uniformity of formulation F1 to F

Formulation	Wetting time (sec)	Water absorption ratio	Disinteg-ration time	% Drug content	Cumulative % drug release in 3 min (%CCR)
F1	290 sec	63.76	> 10 min	99.28	20.3 ± 0.9
F2	270 sec	60.34	> 12 min	101.42	19.3 ± 0.7
F3	280 sec	72.41	> 10 min	102.85	20.1 ± 0.5
F4	260 sec	59.64	> 11 min	99.64	19.3 ± 0.4
F5	73 sec	69.78	57 sec	102.5	74.9 ± 0.9
F6	70 sec	70.87	36 sec	98.92	86.6 ± 0.9
F7	78 sec	76.37	24 sec	101.78	102.4 ± 1.3
F8	68 sec	78.76	33 sec	99.28	90.6 ± 0.8
F9	266 sec	37.13	> 8 min	101.45	21.3 ± 0.5
F10	260 sec	45.54	> 8 min	99.76	19.4 ± 0.7
F11	245 sec	38.98	> 8 min	100.65	22.5 ± 0.5
F12	250 sec	42.86	> 8 min	102.54	21.3 ± 0.4
F13	110 sec	44.74	7 min	101.65	24.3 ± 0.6
F14	100 sec	53.76	4 min	99.45	53.3 ± 0.8
F15	130 sec	64.95	120 sec	98.56	78.1 ± 0.5
F16	120 sec	58.74	3 min	101.34	59.3 ± 0.4
F17	260 sec	18.75	> 7 min	102.43	27.3 ± 0.4
F18	100 sec	24.04	> 7 min	99.65	29.4 ± 0.6
F19	96 sec	22.54	> 7 min	98.67	32.5 ± 0.3
F20	62 sec	18.51	> 7 min	101.76	31.3 ± 0.7
F21	368 sec	18.08	6 min	102.55	37.3 ± 0.4
F22	340 sec	16.83	6 min	101.44	38.9 ± 0.7
F23	240 sec	20.59	7 min	99.87	42.4 ± 0.5
F24	200 sec	12.37	8 min	100.87	41.3 ± 0.4
F25	120 sec	78.00	74 sec	101.54	97.3 ± 0.4
F26	100 sec	89.04	65 sec	100.56	98.9 ± 0.7
F27	78 sec	97.06	16 sec	99.87	102.4 ± 0.5
F28	56 sec	93.16	14 sec	100.87	101.3 ± 0.4
F29	145 sec	19.69	50 sec	99.76	85.7 ± 0.3
F30	150 sec	21.98	45 sec	101.67	88.9 ± 0.7
F31	130 sec	16.47	40 sec	99.76	92.4 ± 0.4
F32	134 sec	20.96	36 sec	100.54	101.3 ± 0.3
F33	101 sec	16.51	185 sec	101.43	83.5 ± 0.5
F34	200 sec	88.57	160 sec	99.73	88.9 ± 0.7
F35	160 sec	84.54	150 sec	101.55	91.2 ± 0.2
F36	180 sec	75.76	145 sec	99.98	97.1 ± 0.3
F37	70 sec	13.98	43 sec	100.76	93.8 ± 0.3
F38	65 sec	12.54	36 sec	98.87	94.9 ± 0.6

F39	55 sec	14.43	15 sec	101.23	97.5 ± 0.3
F40	60 sec	12.65	10 sec	100.56	99.1 ± 0.5

ACKNOWLEDGEMENT

The author thanks Alembic Pharma, Vadodara for providing gift samples of Metoprolol succinate to carry out this work. The author also thankful to management of School of Pharmacy, RK University, Rajkot for providing infrastructure and facilities for carried out this research work. Author also thanks GUJCOST, Gandhinagar for providing financial assistance for this work under minor research project grant.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

(Proceedings available for download at rku.ac.in/icre)

RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

STANDARDISATION AND PHYTOCHEMICAL INVESTIGATION OF SOME ANTIDIABETIC MEDICINAL PLANTS

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ABSTRACT

Diabetes is a metabolic disease now categorized under lifestyle disorder. Diabetes prevalence is rising everywhere in the world by frightening rate. Currently available oral hypoglycemic agents have good control on sugar level but, they are costly and also implicate with long term side effects. Herbs serve best alternative as they have lesser or no side effects and cheaper, but lacuna is with their standardization. Hence an attempt was made to explore standardization of some claimed antidiabetic plants. Standardization was performed with proximate analysis using physicochemical investigation (extractive value, ash value and loss on drying), preliminary phytochemical investigation as well as assay of important principle viz. estimation of polyphenols in *Cinnamomum zeylanicum*, bitters in *Momordica charantia* and *Tinospora cordifolia*, gymnemic acid in *Gymnema sylvestre*and Withanolides in *Withania somnifera*.

SUMMARY

Moisture content for all extracts was found within limit, which implicated material can prevent the microbial growth and sticking problem may not be observed, if the solid dosage form would be developed using these extracts.

Keywords: Standardization 1, Phytochemical investigation 2, Polyphenols 3, Bitters 4, Gymnemic acid 5, Withanolides6.

INTRODUCTION

Diabetes is metabolic disorder, was recognized in India in 6th century, Sushruta has accurately described diabetes around 1,000 B.C.*(1)* Clinically it is mainly characterized by hyperglycaemia, which occurs due to relative or absolute deficiency of insulin or resistance of insulin at the cellular level *(2)*.

Disease is categorized as; Type I (IDDM/ juvenile)where body does not produce insulin. It seen often in early adulthood and patient has to depend on exogenous insulin for the rest of their life*(3)*. Body does not produce enough insulin for proper function, or the cells in the body do not react to insulin (insulin resistance) in Type II (NIDDM/ maturity). Incidence of type II is much higher than type 1*(4)*. Gestational diabetes affects females during pregnancy and can be controlled by exercise and diet.

Prevalence of Diabetes

Now a day diabetes is categorized under lifestyle disorder, it's prevalence of is rapidly increasing all over the world at alarming rate *(5)*. According to American Diabetes Association at every 21 seconds, someone is diagnosed with diabetes *(6)***.** This make diabetes the seventh most common reason of death in the developed world. Increase in prevalence of type 2 diabetes – more than 90%.India leads the world with highest subjects of diabetic and known as "DIABETES CAPITAL OF THE WORLD"*(7)*. In India Gujarati people are very prone to develop diabetes due to genetic sedentary lifestyle, susceptibility, dietary preference for sweet and oily foods *(8)*. WHO show that 32 million people diagnosed for diabetes in year 2000. This is further set to rise 80 million in 2030*(9)*. Surprisingly, the shifting in age of onset of diabetes to a younger age. This could have adverse effects on nation's health and hence, economy *(10)*.

Treatment of diabetes is generally done with oral anti hypoglycemic drugs many times use of it is restricted by their pharmacokinetic properties, secondary failure rates, costly and chances of side effects are high on the other hand herbal drugs are cheap, locally available and free from side effects *(11)*. Still 80% population of developing country relay on natural medicines for primary health care. Even population of developed countries turned to natural remedies due to increasing side effects of synthetic medicines *(12)*. Ayurveda and other traditional medicinal system for the treatment of diabetes stated number of plants can be used as herbal drugs. Despite the fact that herbal products have become increasingly popular throughout the world, one of the challenge lies in their acceptance due to lack of standard quality control profile *(13)*. Standardization means analyzing the amount and the potency of active ingredient meant to be present in each herb claimed in formulation *(14)***.** Herbal medicines are prepared from plant origin materials; hence relative proportion of active constituents can vary from plant to plant of the same species as well as in different plant parts. Method of cultivation, collection, drying, transportation and processing critically affect on the composition of constituents. As well material is prone to contamination, deterioration during storage; which is the main reason for inferior quality of herbal products with less or no therapeutic efficacy. The quality of herbal medicine, mainly the profile of the constituents in the final product has implication in safety and efficacy *(15)*. The quality control of crude drugs and herbal formulations is of extreme importance for their acceptance in modern system of medicine. But non-availability of rigid quality control profile for herbal material and their formulations is one of the major concerns faced by the herbal drug industry. WHO has emphasized the need to ensure quality control of medicinal plant products by applying suitable parameters, using modern technique and standards *(16, 17)*. Accurate standardization methodologies are required to ensure its efficacy for human use. Here we have standardized some antidiabetic herbs for their acceptance in formulation preparation.

MATERIALS AND METHODS

Selected plant material

For the present study we have selected five different antidiabetic plants *Momordica charantia*, *Gymnema sylvestre*, *Withania somnifera*, *Tinospora cordifolia*, *Cinnamomum zylenicum*.

Collection and authentication of plant

Momordica charantia, *Tinospora cordifoliai*, *Cinnamomum zylenicum*are collected from local market vadodara and *Gymnema sylvestre*, *Withania somnifera*were collected from medicinal garden of Parul Arogya Seva Mandal. All plants were authentified by Dr. Darshika shah..

Macroscopical studies

Macroscopic evaluation of the fresh plant was carried out by comparing their morphological characters mentioned in the literature.

Preparation of plant material

All plants were dried under shade and required part of plant was separated from the other parts of plant, powdered by size reduction, passed from 40# sieve and stored in air tight container individually.

Proximate analysis*(18)*

Ash Value

It is important measure to determining quality and purity of drug. Total ash is remained material after complete ignition. The value was determined by taking accurate weight and evenly spreading 2 gm of sample into pre ignited tarred silica crucible. Crucible is subjected to ignition by keeping in muffle furnace at 600°C for 3 h. Later, it was allowed to cool and resultant ash was weighed. Percentage total ash value was determined with reference to air dried crude drug. Boil the obtained ash formed from above procedure with 2N HCl (25 ml) for 5 minutes respectively for quantitative estimation of acid insoluble ash. Recovery of the insoluble ash was done on an ash less filter paper and washing in hot water later. Insoluble sample was transferred into a crucible and again allowed to burn for 20 minutes and weighed. The acid insoluble ashes percentages were determined with reference to the air-dried drug. Determination of water soluble ash was carried out by using the recovered ash during the estimation for total ash was taken and allowed to boil with 25 ml water for 5 minutes. Recovery of the insoluble ash was done on an ash less filter paper and washing in hot water later. Insoluble sample was transferred into a crucible and again allowed to burn for 20 minutes and weighed. The water insoluble ashes percentages were determined with reference to the air-dried drug.

Extractive Values

The nature of chemical constituents present in crude drug can be known by determination of extractive value.

Alcohol soluble extractive

Maceration of 4 gm of the air dried powdered material was done with 100 ml of alcohol (90%) in a closed conical flask for 24 hours, shaken periodically at an interval of 6 hours, and then kept undisturbed for 18 hours. After 18 hours filtration was done rapidly to prevent any loss during evaporation. Porcelain dish was used to evaporate 25ml of the filtrate to dryness at 105°C to get constant weight. Alcohol soluble extractive percentage was calculated with reference to air-dried drug.

Water soluble extractive

100 ml of water was used to soak 4 gm of air dried powder material in a closed conical flask for 1hour with periodical shaking and boiled gently for one hour on water bath; allowed to cool, weighed and readjusted the weight. Porcelain dish was used to evaporate 25ml of the filtrate to dryness at 105°C to get constant weight. Water soluble extractive percentage was calculated with reference to air-dried drug.

Determination of Moisture content

100 gm of accurately weighted drug was placed in a tarred evaporating dish where, drying was done at 105°C for 5 hours in an oven, and weighed. Drying was continued and weighed at 1 hour interval until

difference between two successive weighing corresponds not more than 0.25%. When two consecutive weighing after drying for 50 minutes, showed not more than 0.01gm difference, constant weight was reached. Percentage moisture content was calculated with reference to air dried crude drug.

Preliminary phytochemical analysis*(19, 20)*

All five plants extracts were subjected to following preliminary phytochemical tests for the identifying various active constituents like, glycosides, carbohydrate, flavonoids, alkaloids, amino acids, tannins, fixed oil, gum and mucilage, phytosterols etc. according to the standard procedure.

Assay of important of Phytoconstituent

Estimation of polyphenol in *Cinnamomum Zeylanicum(21)*

Preparation of sample: Ethanol was used to dissolve100 mg of extract and sonicated for 20 minutes; volume was made up 100ml with ethanol. Filtration was done through Whatman filter paper. 10ml of filtrate was transferred to 25mlvolumetric flask and volume was made up with water. From the above solution 1 ml was taken and mixed with 15 ml water, 1mlFolin reagent, 3 ml Na_2CO_3saturated solution. Resultant mixture was heated on water bath for 20 minutes.

Preparation of standard

Same as sample take Gallic acid at place of sample. (Purity-97%)

Measure optical density at λ_{max} 750nm for both using water as a blank.

$$\text{Polyphenol present (\%)} = \frac{\text{Absorbance of sample}}{\text{Absorbance of Standard}} \text{ X } \frac{\text{Weight of Standard}}{\text{Weight of Sample}} \text{ X } \text{Purity of Standard}$$

Estimation of bitters in *Momordica charantia*and *Tinospora Cordifolia(22)*

1 gm of extract was refluxed using 25 ml of alcohol on water bath for one hour and filtered, process was repeated two times for complete extraction of bitters. Resultant filtrate was subjected to solvent evaporation and finally obtained residue was shaken with 25 ml,15 ml, 15 ml of water and then ethyl acetate with same quantities of solvent. Ethyl Acetate was collected, evaporated to dryness and weighed.

$$\text{\% Bitters} = \frac{\text{Wt. of residue}}{\text{Wt. of sample}} \text{ X } 100$$

Estimation of Gymnemic acid in *Gymnema sylvestre(23)*

1 gm of extract was dissolved in 30 ml distilled water, filtered and 10% of HClwas added to get pH 1.5. It was allowed to stand for 30 minutes at room temperature and filteredusing Whatmanfilter paper.Precipitates were washed with 20 ml distilled water and dissolvedin 20 ml of 80% methanol.Methanol was evaporated, residue was weighed and total % of Gymnemic acid was calculated.

$$\text{\% of Gymnemic acid} = \frac{\text{Weight of Residue}}{\text{Weight of Sample}} \text{ X100}$$

Estimation of Withanolides in *Withania somnifera(24)*

1 gm of extract was dissolved in 50 ml of 50 % methanol. Defat aqueous methanolic fraction with hexane and then with ether(5×25 ml). Ether extract was combined and washed twice with water. Evaporate ether fraction was evaporated, residue was weighed and total % of Withanolides was calculated.

$$\text{\% of Withanolides} = \frac{\text{Weight of Residue}}{\text{Weight of Sample}} \text{ X 100}$$

RESULTS AND DISCUSSION

Since ages, plants and other natural products are being used for health and maintenance of life. An ancient Indian scripture and the Vedic literature which are most authentic, gives the reference of number of plants for different diseases and their cure.

Momordica charantia, *Gymnema sylvestre*, *Cinnamomum zeylanicum*, *Tinospora cordifolia*, and *Withania somnifera,* are traditionally used and claimed antidiabetic plants. Plants may act as hypoglycemic by different mechanism of action. Chirantin in *Momordica charantia* works as homologous to insulin, *Gymnema sylvestre*can regenerate pancreatic beta cells as well stimulate beta cells for insulin secretion. *Cinnamomum zeylanicum* increases peripheral glucose uptake.

Table 1 shows % yield of water and alcoholic extracts of each plant. As shown in Table 2, ash values of all these five plants were within pharmacopoeial limits which indicate low degree of contamination during collection and processing. All Plants showed good water extractive values as shown in Table 3. Though, it can be inferred that amount of constituents extracted more by water than alcohol. Table 4 shows moisture content of plants lied between 2.5% to 4.8%, less than pharmacopoeial limit 5% w/w.

Results of qualitative phytochemical tests are shown in Table 5. Phytochemical tests revealed that, the fruits of *Momordica charantia* were rich in glycosides, steroids, triterpenoids, flavonoids, saponins and alkaloids. Leaves of *Gymnema sylvestre* were rich in glycosides and phenols. *Tinospora cordifolia* stem was rich in alkaloids, flavanoids, phenols and saponins. *Cinnamomum zeylanicum* bark was rich in flavanoids, tannins and polyphenols. Roots of *Withania somnifera* were rich in alkaloids, glycosides, steroids triterprnoids, saponins and flavanoids. Amount of polyphenol present in *Cinnamomum zeylanicum* water and alcoholic extract was found to be 12% and 6.7% respectively as shown in Table 6. Table 7 shows that, water and alcoholic extracts of *Momordica charantia* contained percentage of bitters as 5.1% and 4.8% respectively. While, *Tinospora cordifolia* contained the amount of bitters in water and alcoholic extracts 5.6% and 4.2% respectively. Gymnemic acid in *Gymnema sylvestre* water extract was found to be 75% as per Table 8. Estimated withanolides in *Withania somnifera* root was 5.4% and 2.8%in water and alcohol extracts respectively, and are shown in Table 9.

CONCLUSION

All the five herbs complied with standard organoleptic characters. Ash value showed that organic matters were totally removed. Moisture content was found within limit, which implicated material can prevent the microbial growth and sticking problem may not be observed, if the solid dosage form would be developed using these extracts. The high water soluble extractive value and moisture content also suggested its usefulness in the preparation of solid dosage form.

TABLES

Table 1. % Yield of water an alcoholic extract

Sr. No.	Plant (Part) Name of plants	%% Yield	
		Water	Ethanol
1	*Momordica charantia* (Fruit)	11.2	6.5
2	*Gymnema sylvestre* (Leaf)	10.3	6.6
3	*Tinospora cordifolia* (Stem)	15.1	10.8
4	*Cinnamomum zeylanicum* (Bark)	6.4	3.03
5	*Withania somnifera* (Root)	9.3	4.8

Table 2. Ash value

Sr No.	Name of Plants	Total ash (% w/w)	Acid insoluble ash (% w/w)	Water soluble ash (% w/w)
1	*Momordica charantia*	7.2	2.56	3.2
2	*Gymnema sylvestr*	8.9	1.90	3.6
3	*Tinospora cordifolia*	8.3	1.9	3.8
4	*Cinnamomum zeylanicum*	6.3	1.9	5.4
5	*Withania somnifera*	6.2	1.8	4.9

Table 3. Extractive value

Sr No.	Name of plants	Water soluble (%w/w)	Alcohol soluble (% w/w)
1	*Momordica charantia*	35.30	23.33
2	*Gymnema sylvestr*	32.45	19.20
3	*Tinospora cordifolia*	34.21	15.20
4	*Cinnamomum zeylanicum*	22.23	18.44
5	*Withania somnifera*	38.23	22.23

Table 4. Moisture content

Sr No.	Name of plants	Moisture content(% w/w)
1	*Momordica charantia*	4.8
2	*Gymnema sylvestr*	2.5
3	*Tinospora cordifolia*	3.3
4	*Cinnamomum zeylanicum*	3.6
5	*Withania somnifera*	4.6

Table 5. Phytoconstituents by preliminary phytochemical screening

Sr. No.	Phytoconstituents	*M.c*		*G.s.*		*T.c.*		*C.z*		*W.s*	
		Aq.	Al.	Aq.	Al.	Aq.	Al.	Aq.	Al.	Aq	Al.
1	Alkaloids	+	+	+	-	+	+	-	+	+	+
2	Glycosides	+	+	+	+	+	-	-	-	+	+
3	Carbohydrates	+	+	+	+	+	-	+	+	+	+
4	Steroids	+	+	-	-	+	-	-	-	+	+
5	Triterpenoids	+	-	-	-	-	+	+	+	+	+
6	Flavonoids	+	-	-	+	+	+	+	+	+	+
7	Tannins	-	-	+		+	+	-	+	+	-
8	Phenoles	-	-	+	+	+	+	+	+	-	+
9	Saponins	+	-	+		+	+	-	-	+	+
10	Proteins	+	+	-	-	+	+	-	-	-	-

Table 6. Estimation of polyphenol in *Cinnamomum zeylanicum*

Parameter	Water extract	Alcoholic extract	Standard
Weight (mg)	100	100	50
Absorbance	0.252	0.135	0.976
% of Polyphenol	12	6.7	-

Table 7.Estimation of bitters

Parameter	*Momordica charantia*		*Tinospora cordifolia*	
	Water extract	Alcoholic extract	Water extract	Alcoholic extract
Weight of sample (gm)	0.5	0.5	0.5	0.5
Weight of residue (gm)	0.025	0.024	0.028	0.021
% of Bitters	5.1	4.8	5.6	4.2

Table 8.Estimation of Gymnemic acid in *Gymnema sylvester*

Parameter	Water extract
Weight of sample (gm)	1.3
Weight of residue (gm)	0.97
% of Gymnemic acid	75

Table 9.Estimation of Withanolides in *Withania somnifera*

Parameter	Water extract	Alcohol extract
Weight of sample (gm)	2.3	2
Weight of residue (gm)	0.12	0.05
% of Withanolides	5.4	2.8

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

(Proceedings available for download at rku.ac.in/icre)

RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

Development and validation of a bioanalytical method for the quantification of Diclofenac sodium and Chlorzoxazone in human plasma using an RP-HPLC method

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ABSTRACT

A selective, sensitive and rapid RP-HPLC method was developed and validated according to USFDA guideline for quantitation of diclofenac sodium (DFS) and chlorzoxazone (CZX) in human plasma. Sample preparation method was protein precipitation having extracting solvent 600 µl acetonitrile:methanol(2:1). Separation was carried out by RP-HPLC on a Phenomenex Luna-C_{18}(250 x 4.60 mm, 5 µm) column using isocratic mobile phase comprising of methanol: acetonitrile:10mM potassium dihydrogen orthophosphate pH 4 adjusted with 10% OPA(45:20:35 %v/v/v) at 1.0 ml/min flow rate for 14 min using naproxen as internal standard. Calibration curves were linear in range of 0.05–20 µg/ml for DFS and 0.1–40 µg/ml for CZX with $r^2 > 0.9990$ in human plasma. The intraday and interday precision and accuracy study of QC samples were within acceptance criteria (% CV < 15%). The proposed method was suitable for routine determination of DFS and CZX in plasma and successfully applied for pharmacokinetic study.

SUMMARY

Newly developed and validated RP-HPLC bioanalytical method was applied for quantification of DFS and CZX in rat plasma for pharmacokinetic study.

Key words: Diclofenac sodium, Chlorzoxazone, RP-HPLC, Protein precipitation, validation

1. INTRODUCTION

Musculoskeletal complaints account for more than 315 millions patient visits per year. *(1)* Currently, Centers for Disease Control and Prevention suggest that 33% (69.9 millions) people of United States distress from arthritis disorder. *(1)* Few of them are self-aggrandizing situation require minimal reckoning and only symptomatic relief. *(1)* Muscle spasm is treated with a combination of physical therapy, centrally acting muscle relaxants, and anti-inflammatory agents. *(1)* Diclofenac sodium (DFS, 2 - (2, 6 - dichloroanilino) phenyl] acetate) is NSAIDs used in the rheumatic disorders whereas Chlorzoxazone (CZX, 5 – Chloro – 2 - (3H) - benzoxazolone) is spasmolytics used in skeletal muscle spasm (Figure 1). (*1*) Combination of these two drugs is used in effective treatment of acute inflammation associated with the spasm. (*1*)

Prior art and through literature review reveals that various bioanalytical method were available for estimation of DFS and CZX in different biological fluids individually. The available methods for DFS include RP-HPLC/UV in rat plasma (*2, 3*), RP-HPLC/UV in human plasma (*4-8*), LC-MS/MS in human plasma (*9*), RP-HPLC/ECD in aqueous humor fluid (*10*). For CZX, RP-HPLC-UV in human plasma (*11*), fluorimetry in plasma and urine (*12*) and LC-ESI/MS/MS in rat plasma were reported. (*13*)

Till date, no bioanalytical method is available for the estimation of both drugs in human plasma simultaneously. In present study, we have used UV detector because it is convenient, economical and suitable for routine analysis of biological samples. In this notion, it is thought of interest to develop and validate bioanalytical RP-HPLC method with UV detector for simultaneous estimation of DFS and CZX and its pharmacokinetic application in newly developed solid dispersion and marketed formulation.

2. MATERIALS AND METHODS

2.1 Materials

Diclofenac sodium (DFS) and Chlorzoxazone (CZX) was supplied as a gratis sample by Baroque Pharmaceutical Ltd., Khambhat, Gujarat, India and Bimal Pharmaceutical Ltd., Mumbai, Maharashtra, India respectively. Marketed formulation, Difisal Relax tablets (Kopran Pharma Ltd., India) that contains 50 mg Diclofenac Sodium and 250 mg Chlorzoxazone was procured from local market. Methanol (Lichrosolv grade) and acetonitrile (Lichrosolv grade) were obtained from Merck, Mumbai. KH_2PO_4

(HPLC grade) and orthophosphoric acid (HPLC grade) was obtained from Merck Ltd., Mumbai. Double distilled water was prepared in-situ. Drug-free human plasma was supplied as gratis sample from Red Cross Society, Anand and stored in polypropylene bottle at -20 °C until analysis.

2.2 Instruments

A High Performance Liquid Chromatography system, with LC solutions-1.25 data management system (Shimadzu-LC2010-CHT), with Photo diode array detector and an auto sampler was used for analysis. LC 2010 solution software was used to record the data. Shimadzu AUW220 balance, Japan, Eltek centrifuge TC 450 D, Remi motors Cyclo Mixer CM101, Vacuum filtration assembly TID 15, and AXIVA Nylon membrane filters 0.2 µm were used in study.

2.3 Optimization of chromatographic conditions

Various solvents in different ratios such as methanol, acetonitrile, double distilled water along with potassium dihydrogen orthophosphate buffer were tried. The mobile phase was cleaned using 0.45 µm membrane filter and degassed in sonicator for 10 min prior to use. Flow rate was set at 1.0 ml/min and the elution was monitored at 279 nm. The injection volume to carry out chromatography was set at 20 µl.

2.4 Standards and quality control preparation

A standard stock solutions of DFS (1,000 µg/ml) and CZX (1,000 µg/ml) were prepared by dissolving 10 mg of DFS and CZX in 10 ml of methanol respectively and was stored at -20°C in a clear glass volumetric flasks, and protected with aluminium foil. From above stock solutions, eight working standard solutions have concentration of DFS:CZX with ratio of 40:20, 20:10, 8:4, 4:2, 2:1, 0.8:0.4, 0.2:0.1 and 0.1:0.05 µg/ml were prepared when necessary. Additionally, standard stock solution of internal standard (1,000 µg/ml) was prepared by dissolving accurately weighed 10 mg of Naproxen (NPX) in 10 ml of methanol and was stored. Above solution was suitably diluted with methanol to obtain 3 µg/ml of naproxen working solution. In a 2 ml graduated ependroff, DFS and CZX working standard solutions were spiked in appropriate volume to drug free plasma to achieve calibration standards of 0.05-20 µg/ml DFS, 0.1-40 µg/ml CZX. Similarly, quality control samples were prepared by spiking and mixing the working solution to control blood plasma for obtaining three levels namely higher quality control (HQC), middle quality control (MQC) and lower quality control (LQC) (10, 2 and 0.4 µg/ml for DFS and 20, 4 and 0.8 µg/ml for CZX). All the above solutions were prepared in bulk and stored in a deep freezer at -20 ± 2°C.

2.5 Sample preparation

The sample preparation for the drugs was done by protein precipitation technique. Various precipitating solvents like methanol, acetonitrile were tried individually and in different ratio for obtaining maximum

extraction recovery. Sample was prepared by transferring 200 µl of plasma in 2 ml ependroff tube followed by spiking 100 µl of mixed standard solution and 100 µl of ISTD (3 µg/ml) and extremely vortexed to ensure complete mixing of contents for 2 min. The samples were extracted by addition of 600 µl extracting solvent i.e. acetonitrile: methanol (2:1) and again vortex mixed for 5 min to ensure uniform mixing and tubes were centrifuged at 5000 rpm for 15 min at -20 ± 2°C in cooling centrifuge (Eltek centrifuge TC 450 D). After centrifugation, supernatant was collected and directly injected into HPLC column.

2.6 Method validation

Developed bioanalytical method was validated as per USFDA guidelines, which includes various parameters like linearity, range, accuracy, precision, selectivity, sensitivity, extraction recovery and stability. (*14, 15*) Method linearity for DFS and CZX was accessed in the range of 0.05 - 20 and 0.1 - 40 µg/ml respectively by taking five replicates. The linearity obtained by plotting graph of peak area ratio of drug to ISTD was plotted against the specified concentration of drugs and corresponding regression equation was derived. In the present study, the relevance of ordinary regression model was evaluated by performing test for homoscedasticity. (*16, 17*) The accuracy study was performed in five replicates on three QC samples (10, 2 and 0.4 µg/ml for DFS and 20, 4 and 0.8 µg/ml for CZX) and area ratio was measured. Intraday precision was performed on the same day whereas interday precision was performed on different day on three QC samples (10, 2, 0.4 µg/ml for DFS and 20, 4, 0.8 µg/ml for CZX) in five replicates. Extraction recoveries of protein precipitation technique from plasma were estimated by comparing the mean peak area ratio of extracted plasma samples spiked with known amount of DFS (10, 2, 0.4 µg/ml, n = 5) and CZX (20, 4, 0.8 µg/ml, n = 5) before extraction and after extraction at the same nominal concentration prior to injection. The extraction recovery at each concentration was calculated using following equation.

$$\% \text{ Extraction recovery} = \left[\frac{\text{Mean peak area ratio of extracted sample}}{\text{Mean peak area ratio of non-extracted sample}}\right] \times 100$$

Sensitivity was calculated from the background noise or response from five lowest limit of quantification (LLOQ) samples. The five replicates should have a precision of ≤ 20% and an accuracy of ± 20%. Selectivity, analysis of blank samples of different lots of plasma was done. Each blank sample should be tested for interference, and selectivity study performed at LLOQ (0.05 µg/ml for DFS and 0.1 µg/ml for CZX). Stability of DFS and CZX under various storage conditions was investigated. Long term, short term and freeze/thaw stability studies were assessed using two quality control samples. Freeze/thaw stability study was determined by at least two aliquots at LQC and HQC levels stored at the -22°C temperature for 24 hours and thawed unassisted at room temperature. The freeze/thaw cycle was

repeated three times and compared with freshly prepared QC samples. The short-term temperature stability study was also determined at -22°C temperature for 12 hours and compared with freshly prepared QC samples. In addition, the long-term stability study for a period of about 30 days was evaluated at LQC and HQC level stored at -22°C by comparing against the freshly prepared quality samples.

2.7 Method applicability for pharmacokinetic study

Male Sprague dawley rats (150-250 g) were utilized for in-vivo experiments. The animals were maintained in animal house under standard temperature of 20 ± 2°C, relative humidity (55 ± 5 %), and 12/12 h light/dark cycle. Animals received standard pellet diet and water ad libitum. The experimental protocol (Protocol No-1506, Approved date -16/03/2015) was approved by Institutional Animal Ethics Committee of Anand Pharmacy College as per the guidelines of CPCSEA, Ministry of Social Justice and Empowerment, Government of India. Rats were randomly divided into four groups (n = 6) based on the time of blood sampling. The first group received distilled water, second group received untreated DFS and CZX, third group received marketed formulation and fourth group received prepared solid dispersion of DFS and CZX. The blood samples (~ 500 µl) were taken from the retro orbital vein in sodium citrate tubes at 0, 1, 2, 3, 4, 5 h after the administration. Samples were immediately centrifuged at 4000 rpm for 10 min to separate the plasma, which was then frozen at -20°C until analysis.

The estimation of DFS and CZX in all the samples was undertaken within 6 - 7 h of blood collection by the method as described above. Various pharmacokinetic (PK) parameters namely area under the curve from zero time to 5 h (AUC_{0-5}), area under the curve for zero to infinite time ($AUC_{0-\infty}$), peak plasma concentration (C_{max}), time to reach maximum plasma concentration (t_{max}), elimination rate constant (K_{el}), terminal elimination half life ($t_{1/2}$), absorption rate constant (K_{ab}) and absorption half life ($t_{1/2}$) were determined for untreated DFS and CZX, marketed formulation (Difisal Relax Tablet) and developed solid dispersion of DFS and CZX using a non-compartmental approach.

3. RESULTS AND DISCUSSION

3.1 Optimized chromatographic conditions

From literature review, it is revealed that HPLC bioanalytical method for quantification of DFS and CZX alone or with other drug in biological fluid had been reported, where selected mobile phase comprised of methanol, acetonitrile, water and phosphate buffer (*5, 11*). Hence, various combinations of methanol, acetonitrile and water in different proportions were tried at 279 nm. However, no proper resolution of both drugs from internal standard and appropriate peak shape of drugs were observed. To solve aforementioned problem, water was replaced with buffer (10 mM potassium dihydrogen orthophosphate)

that gave well resolved and sharp peak shape with drastic change in retention time of drugs. Hence, further development was carried out with methanol: acetonitrile: 10 mM potassium dihydrogen orthophosphate buffer with different pH that gave very sharp, resolved and reproducible peaks. The mobile phase finally optimized for study was methanol: acetonitrile: 10 mM KH_2PO_4 pH 4 adjusted with 10% orthophosphoric acid (OPA) (45:20:35, %v/v/v) which gave retention time; 10.17 min for DFS, 4.26 min for CZX and 6.15 min for ISTD (Naproxen) (Figure 2).

3.2 Optimized sample preparation

Several extraction techniques like protein precipitation, liquid-liquid extraction and solid phase extraction were available for extraction of DFS and CZX from plasma individually (*2-13*). Till date, sample preparation using protein precipitation for simultaneous estimation of DFS and CZX from human plasma has not yet been reported. Hence in the present research work, attempt was made to extract the DFS and CZX from human plasma using protein precipitation method. Various precipitating solvents like methanol, acetonitrile were tried individually and in different ratio for obtaining maximum extraction recovery. Here, extraction recovery with methanol and acetonitrile alone was found to be 86.84 - 89.63 %. However, the % extraction recovery using acetonitrile: methanol (2:1) was found to be higher than methanol and acetonitrile alone and gave about 98 % extraction recovery of both drugs hence, selected as extracting solvent.

3.3 Method validation

The linearity of the method was determined using ordinary linear regression analysis of standard plots associated with mentioned calibration standards with five replicates with acceptable coefficient of determination (r^2) (Table 1). Test for homoscedasticity was confirmed by Bartlett's test and the response of peak area ratio for DFS and CZX to ISTD (NPX) showed homogenous variance that was exemplified by the χ^2 value less than the tabulated value $\chi^2(0.05, 7) = 14.07$ at 95% confidence interval level. Thus, from the obtained results, there was further no need of weighting and transformation approach. (*16, 17)* The accuracy was calculated, for each spiked quality control samples as five replicate by comparing the theoretical concentration with the assayed concentration (Table 2). The %CV value was found to be within 15% of the actual value indicating that the proposed method provides acceptable accuracy for determination of DFS and CZX. The results of precision study reveals that all % CV values were less than 5.0% which is acceptable according to specification in USFDA bioanalytical validation guidelines (*14, 15*), confirming that the method is precise (Table 2). Extraction recovery study performed at three concentration levels showed 81.41 - 99.00% recovery of spiked drugs as shown in Table 3. The results of extraction recovery study reveals that all % CV value found was less than 15% which shows high efficiency of extraction procedure and sensitivity of proposed method (Table 3). The lowest limit of

quantification for the analyte was set 0.1 µg/ml for CZX and 0.05 µg/ml for DFS (Fig. 3). The selectivity of method was assured in presence of endogenous substance present in different plasma samples at LLOQ sample (0.1 µg/ml for CZX and 0.05 µg/ml for DFS), in triplicate manner. From Figure 5, it is seen that there is no interference observed between plasma and the spiked drugs. % CV of long-term, freeze/thaw, short-term stability studies at two concentration levels was found to be less than 15 that indicate that drug had remained stable after stability cycles (Table 4).

3.4 Method applicability for pharmacokinetic study

Based on the results of in-vitro dissolution study, optimized solid dispersions for DFS and CZX were screened for in-vivo study. The untreated DFS and CZX, optimized solid dispersion of DFS and CZX and marketed formulation was administered orally in rats and concentration of DFS and CZX in plasma was estimated by proposed RP-HPLC method. Results of pharmacokinetic parameters of optimized solid dispersion and comparison with untreated API of both DFS and CZX and marketed formulation are illustrated in Figure 6. It clearly appears from the obtained results that absorption of DFS and CZX from solid dispersion is increased as compared to untreated DFS and CZX, marketed formulation. This is due to enhancement in solubility of solid dispersion in gastrointestinal tract as compared to untreated DFS and CZX and marketed formulation. Generally, such faster absorption was linked to the better wettability of the solid dispersion. Incorporating the water soluble polymer like Soluplus into the poorly soluble crystals improved the dissolution in gastrointestinal fluid followed by increase in solubility and bioavailability in rat. The results of present study revealed that for transformed data AUC_{0-5} values for the untreated DFS and CZX product was found to be 13.99 ± 2.32 and 38.57 ± 2.02µg.h/ml that of marketed formulation was found to be 18.05 ± 0.02 and 57.95 ± 1.02 µg.h/ml and for solid dispersion 22.37 ± 1.05 and 72.00 ± 2.08 µg.h/ml. Absorption profile of solid dispersion was more as compared to untreated DFS and CZX and marketed formulation as shown in Figure 6. C_{max} provides indication that the drug is sufficiently systemically absorbed to achieve a therapeutic response. After the oral administration of untreated DFS and CZX, marketed formulation and solid dispersion C_{max} values were found to be 5.70 ± 2.04 and 17.98 ± 2.01 µg/ml, 8.09 ± 3.21 and 18.53 ± 2.93 µg/ml and 9.87 ± 2.13 and 24.03 ± 3.45 µg/ml respectively. From the C_{max} value, it is evident that solid dispersion of DFS produces higher response at 3 h and CZX at 1 h compared to untreated DFS and CZX and marketed formulation, indicating that the solubility of solid dispersion is more resulting in more bioavailability in blood.

4. CONCLUSION

A rapid, sensitive, accurate, precise and reproducible isocratic RP-HPLC with UV detection method for simultaneous estimation of DFS and CZX in plasma using protein precipitation sample preparation

technique was developed and validated according to USFDA guideline. The developed method offered a numerous advantages in term of good quantitative ability, high recovery, simple in nature, rapid, inexpensive and environmental benignity. The proposed validated RP-HPLC method has been applied for the evaluation of pharmacokinetic parameters of solid dispersion of DFS and CZX in rat with acceptable precision, accuracy and recovery values. As the developed method could effectively detect drug in plasma with good recovery value, it can be applicable in BA/BE study.

FIGURES

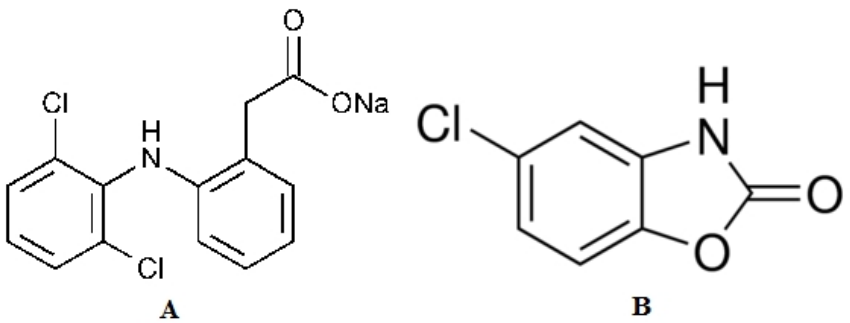


Fig. 1. Chemical structure of A: diclofenac sodium and B: chlorzoxazone

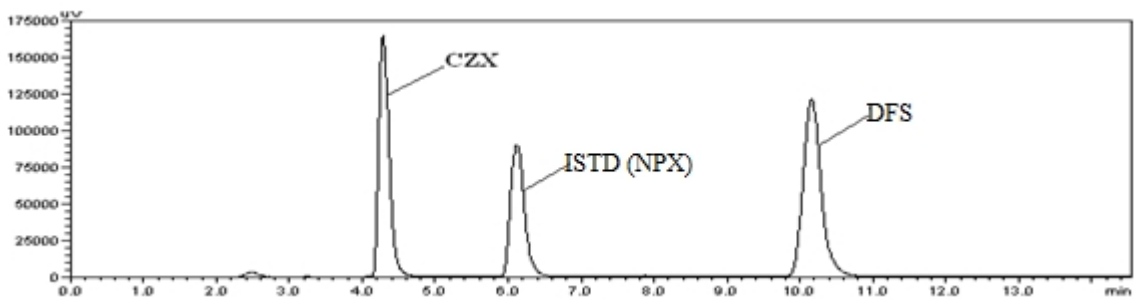


Fig. 2. Chromatogram of 50 µg/ml of CZX, ISTD (NPX) and DFS

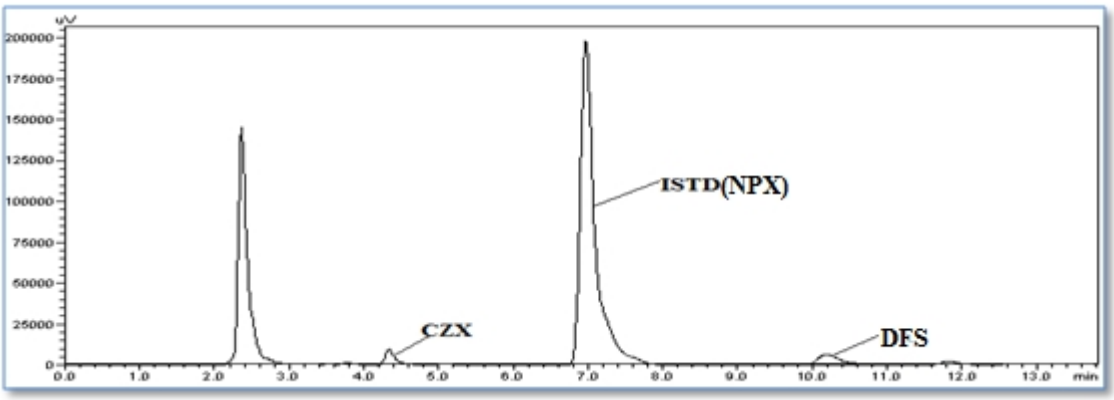


Fig. 3. Chromatogram at lower limit of quantification (LLOQ) containing DFS and CZX

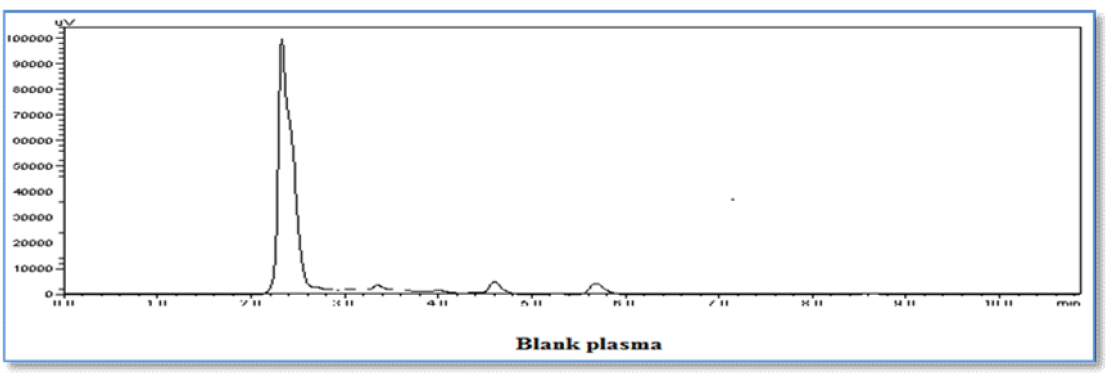


Fig. 4. Chromatogram of blank human plasma

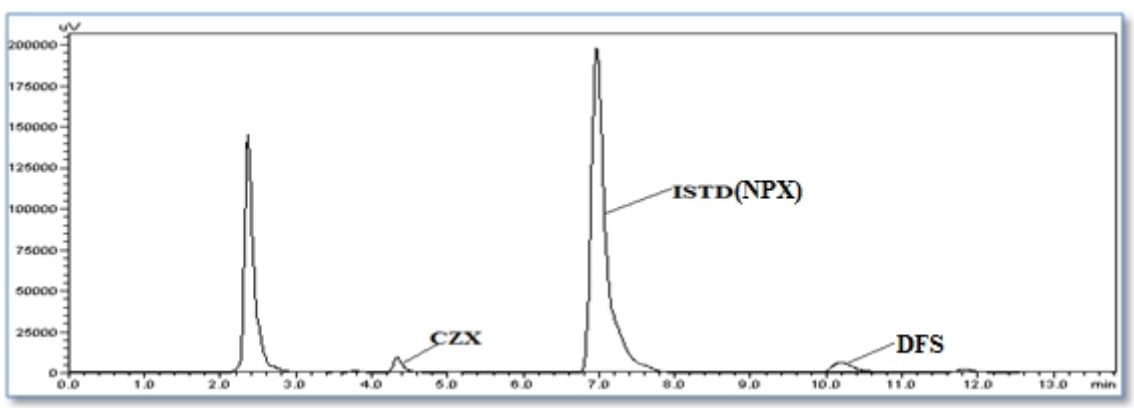
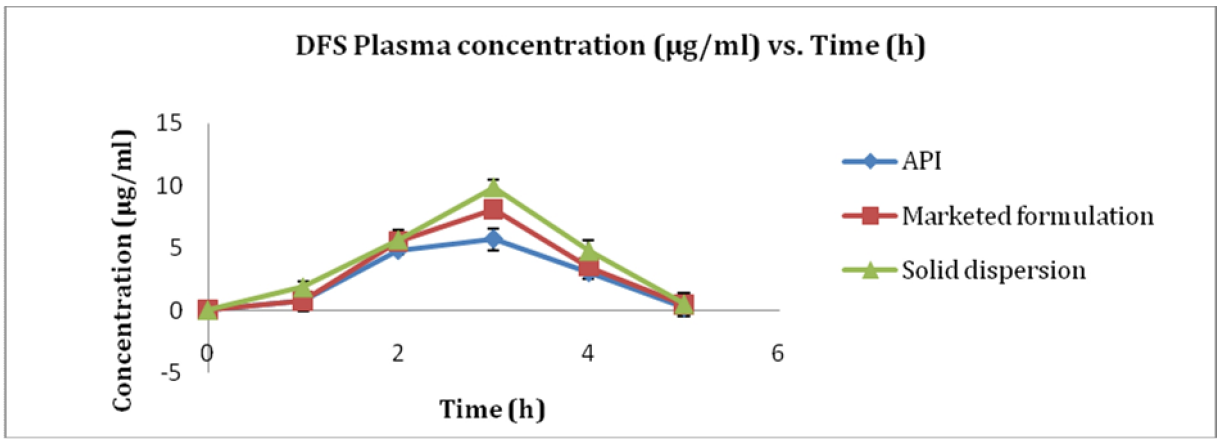
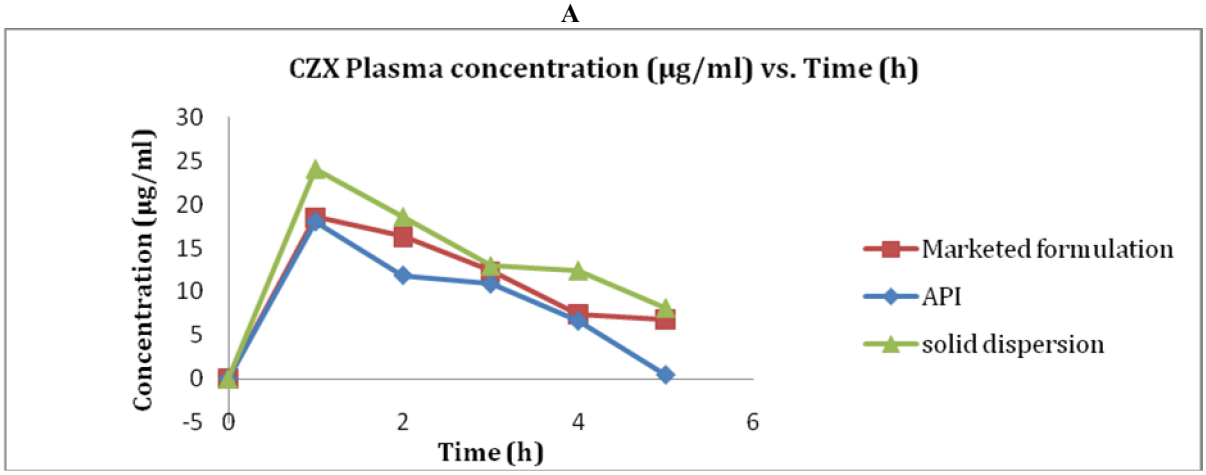


Fig. 5. Chromatogram shows plasma spiked with DFS and CZX and ISTD at LLOQ level



A



B

Fig. 6. Absorption profile of API, solid dispersion and Marketed formulation in rat plasma A: DFS B: CZX

TABLES

Table 1. Linear regression parameters of DFS and CZX

Drug code	**DFS**		**CZX**	
Linear regression parameters	**Mean** [a]	**%CV**	**Mean** [a]	**%CV**
Slope	0.197	46.20	0.150	12.31
Intercept	0.049	12.17	0.126	45.01
Correlation coefficient (r^2)	0.9991	4.466	0.9998	7.582
Linearity range (µg/ml)	0.05 - 20		0.1 - 40	
Bartlett's test (χ^2)	0.307		0.288	

[a] mean of five replicates, % CV: % coefficient of variance, χ^2 (0.05, 7) = 14.07 at 95% confidence interval level

Table 2. Results of accuracy and precision study of DFS and CZX

Drug Code	Level	Nominal Concentration (µg/ml)	Accuracy			Precision			
						Intra-day		Inter-day	
			MCF [a] (µg/ml)	% CV	% accuracy	MCF [a] (µg/ml)	% CV	MCF [a] (µg/ml)	% CV
	LQC	0.4	0.38	0.97	96.32	0.39	0.49	0.39	2.02
DFS	MQC	2	1.99	7.02	99.59	1.98	3.73	1.93	2.50
	HQC	10	8.72	1.13	87.21	0.31	0.85	8.95	1.83
	LQC	0.8	0.73	1.06	92.80	0.75	1.75	0.74	2.87
CZX	MQC	4	3.70	2.04	93.44	3.88	4.85	3.99	1.60
	HQC	20	19.9	0.28	99.98	19.4	2.04	19.6	2.40

[a] average of five determinations, % CV: % coefficient of variance, MCF: Mean concentration found

Table 3. Extraction recovery study of DFS and CZX

Drug code	LQC		MQC		HQC	
	%Mean recovery [a]	%CV	%Mean recovery [a]	%CV	%Mean recovery [a]	%CV
DFS	81.41	2.52	86.34	2.89	98.88	0.51
CZX	88.81	1.65	97.38	0.49	99.00	0.32

[a] average of five determinations, % CV: % coefficient of variance

Table 4. Stability studies of DFS and CZX

Drug code	Level	Freeze/Thaw stability		Short term stability		Long term stability	
		%Mean recovery [a]	% CV	%Mean recovery [a]	% CV	%Mean recovery [a]	% CV
DFS	LQC	71.82	0.007	99.18	0.568	84.02	0.786
	HQC	81.01	0.03	85.83	0.251	89.17	0.162
CZX	LQC	79.82	0.69	90.14	0.677	80.69	1.58
	HQC	76.90	0.18	97.42	0.814	78.08	1.724

[a] average of three determinations, % CV: % coefficient of variance

ACKNOWLEDGEMENT

The authors are grateful to Baroque Pharmaceutical Ltd., Khambhat, Gujarat and Bimal Pharmaceutical Ltd., Mumbai, providing for gift sample of DFS and CZX respectively.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

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RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

Formulation and Evaluation of Extended-Release Multiparticulate delivery system for Repaglinide

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ABSTRACT

Diabetes mellitus arising as a result of a relative or absolute deficiency of Insulin secretion, resistance to insulin action, or both. Repaglinide is an oral blood glucose-lowering drug of the meglitinide class used in the management of type 2 diabetes mellitus (also known as non-insulin dependent diabetes mellitusorNIDDM). After oral administration repaglinide is rapidly and completely absorbed from the gastrointestinal track, and has a plasma half-life of approximately 1 hour. An immediate release tablet of repaglinde needs to take 3-4 times in a day. Hence in the present study work an attempt has been made to develop Extended-Release Multiparticulate delivery system for Repaglinide that release drug at predetermine time interval to give extended release profile for 12 hr. Drug embedded matrix system, Drug with polymer coated system and reservoir system has been evaluated. Suitable multiparticulate system further optimized for formulation component and manufacturing process parameter using design of experiments.

SUMMARY

In the present study work an attempt has been made to develop Extended-Release Multiparticulate delivery system for Repaglinide that release drug at predetermine time interval to give extended release profile for 12 hr. Different multiparticulate system has been evaluated and suitable multiparticulate system further optimized for formulation component and manufacturing process parameter using design of experiments.

Keywords: Repaglinide, Multi-particulate system, Design of experiments, Extended-Release, Anti-diabetic drug

INTRODUCTION

Diabetes mellitus is a group of metabolic diseases characterized by hyperglycemia arising as a result of a relative or absolute deficiency of Insulin secretion, resistance to insulin action, or both. There are different classes of anti-diabetic drugs, and their selection depends on the nature of the diabetes, age and situation of the person, as well as other factors (*1,2*).

Repaglinide is an oral blood glucose-lowering drug of the meglitinide class used in the management of type 2 diabetes mellitus (also known as non-insulin dependent diabetes mellitus or NIDDM). Repaglinide, S(+)2-ethoxy-4(2((3-methyl-1-(2-(1-piperidinyl) phenyl)butyl) amino)-2-oxoethyl) benzoic acid, is chemically unrelated to the oral sulfonylurea insulin secretagogues.(*3,4*)

Repaglinide lowers blood glucose levels by stimulating the release of insulin from the pancreas. This action is dependent upon functioning beta (ß) cells in the pancreatic islets. Insulin release is glucose-dependent and diminishes at low glucose concentrations. (*3,4*)

After intravenous (IV) dosing in healthy subjects, the volume of distribution at steady state (Vss) was 31 L, and the total body clearance (CL) was 38 L/h. Protein binding and binding to human serum albumin was greater than 98%.(*3,4*)

Repaglinide is completely metabolized by oxidative biotransformation and direct conjugation with glucuronic acid after either an IV or oral dose. As repaglinide is very short acting (half life 1 hr) it require multiple dosage during a day (3 to 4 times in a day).(*3,4*)

Hence in the present study work an attempt has been made to develop Extended-Release Multiparticulate delivery system for Repaglinide. Drug embedded matrix system, Drug with polymer coated system and reservoir system has been evaluated and suitable multiparticulate system. Suitable multiparticulate system further optimized for formulation component and manufacturing process parameter using design of experiments.

Theoretical target dissolution profile is calculated using equation of loading and maintenance dose with available pharmacokinetic data of repaglinide.

Table 1. shows pharmacokinetic parameter of repaglinide and target dissolution profile for 12 hr.

MATERIALS AND METHODS

Repaglinide (Biocon Ltd.), HPMC K4M premium CR (Dow Chemicals), HPMC K15M premium CR (Dow Chemicals), Sugar Sphere (Hanns G. Werner) Celesphere (Signet), Eudragit RSPO and RLPO (Evonic), Surelease 19010 (Colorcon) Isopropyl alcohol (Merck), Dichloromethane (merck).

Surelease® is a aqueous amoniacal ethylcellulose dispersion. Sugarsphere is spherical particle prepared from sugar using extrusion spherodization. Celesphere is spherical particle prepared from microcrystalline cellulose using extrusion spherodization

Selection of Suitable Multi particulate system for Extended-Release dosage for Repaglinide

Multiparticulate system will be formulated using three approaches as described in figure1.

Formulation and Evaluation for Drug Embedded Matrix system for Repaglinide Extended-Release Multiparticulate system.

Drug embedded matrix pellets of Repaglinide were prepared using Hypromellose (HPMC K4M) as Extended-Release Polymer and lactose and Microcrystalline cellulose as water soluble and insoluble diluents. Repaglinde were mixed with Lactose monohydrate and Microcrystalline cellulose in Rapid Mixture granulator and granulated with Purified water to prepare dough mass that can be extruded. Wet mass were extruded using extruder having 0.5 mm screen, extruded mass were spherodised using spheronizer with rpm of 1000. Wet pellets were died using fluidized bed dryer at inlet temperature of 60°C to get loss on drying not more than 2% w/w at 105°C measured using Halogen moisture analyzer.
Prepared pellets were filled in hard gelatin capsule of size 3 using automatic capsule filling machine.
Repaglinide Extended-Release capsules filled with extended release pellets of repaglinde were studied for dissolution profile and compare with theoretical target dissolution profile using similarity factor (F2).
Table 1 contains qualitative and quantitative composition for drug embedded matrix system and Table 2 summarises comparative dissolution profile with target dissolution profile, fig. 2 provides graphical presentation of comparative dissolution profile.

Formulation and Evaluation for Drug with Polymer in coating system for Repaglinide Extended-Release Multiparticulate system.

Drug with polymer coating on pellets of Repaglinide were prepared using coating of repaglinide and Eudragit RSPO and RLPO on non pareil seeds of Sugar sphere or celeSphere.
Repaglinide along with Eudragit RSPO and RLPO and Hypromellose E15 were dissolved in mixture of Isopropyl alcohol and Dichloromethan in ratio of 60:40. Drug layering solution was sprayed on sugar sphere or celeSphere using fluidized bed processor GPCG 1.1. Coated pellets were filled in size 3 hard gelatin capsules.
Table 4 contains qualitative and quantitative composition for Drug with Polymer in coating system and Table 5 summarises comparative dissolution profile with target dissolution profile, fig. 3 provides graphical presentation of comparative dissolution profile.
Repaglinide Extended-Relase capsules filled with extended release pellets of repaglinde were studied for dissolution profile and compare with theoretical target dissolution profile using similarity factor (F2)

Formulation and Evaluation for Reservoir System for Extended-Release multiparticulate system.

Reservoir type multiparticulate system for extended release of repaglinide was prepared using sugar Sphere coated with drug along with binder and coated with controlled release coating for extended-Release up to 12 hr.
Repaglinide was dissolved in Mixture of Isopropyl alcohol and Dichloromethan in ratio of 60:40 with HPMC 6 cps as binder. Drug layer pellets were coated with control release film using surelase with HPMC 6 cps as pore former.

Drug Layering:

Repaglinde was dissolved in mixture of Isopropyl alcohlol and dichloromethane in ratio of 60:40 with HPMC 6 cps as binder. Drug layering solution were spared on non pearile seeds of sugar Sphere and celeSphere using fluidized bed processor GPCG 1.1

Controlled Release Coating:

Ethyl cellulose based aqueous coating Ready to use control release coating system (surelease 19010) was used as control release coating system with HPMC 6 cps as pore former. Contolled release coating solution were spared on drug layered pellets using fluidized bed processor GPCG 1.1

Table 6 contains qualitative and quantitative composition for reservoir system and Table 7 summarises comparative dissolution profile with target dissolution profile, fig. 4 provides graphical presentation of comparative dissolution profile.

Optimization of Drug layering process for reservoir system using design of Experiment:

Drug layering process using fluidized bed process involved multi factor and that may interact to each other. It is require optimizing processing parameter to achieve desired assay and meets criteria for fines and agglomerates with reasonable processing time. Table 8 shows Qualitative and Quantitative composition for Drug layering process

Table 9 shows List of fixed and Variable drug layering process parameter using GPCG 1.1 Based on this parameter variable parameter was optimized using 2^{4-1} factorial design using DX9.1 software of Stat easy. Table 10 presents Identification of factor and level for design of experiment and response

Based on 2^{4-1}factorial design 11 trial of different combination were run and evaluated for % fines, % agglomerates and Assay Table 11 shows Experimental run for Drug layering process optimization using 2^{4-1} factorial design. Experimental design were analysed statistically and ANOVA, Half normal plot, effect analysis, pareto chart, surface chart and conter plot were calculated and presented.

Optimization of control release coating composition for reservoir system using design of Experiment:

Ethyl cellulosed based aquous dispersion of Surelase 19010 from colorcon were used as controlled release polymer and HPMC 6 cps was used as pore former. Ratio of surelease a9010 to HPMC 6 cps were optimized using response surface method with 6 centre point and axial points. Table 15 represents qualitative and quantitative composition for control release coating. Table 16 summarises the Identification of factor and level for design of experiment and response. Table 17 shows Experimental run for control release coating optimization using response surface method. Experimental design were analysed statistically and ANOVA, FDS chart, surface chart and conter plot were calculated and presented.

RESULTS AND DISCUSSION

Formulation and Evaluation for Drug Embedded Matrix system for Repaglinide Extended-Release Multiparticulate system.

Table 2 summarises comparative dissolution profile with target dissolution profile, fig. 2 provides graphical presentation of comparative dissolution profile.

Dissolution profile achieved with drug embedded matrix system is not similar with target dissolution profile with respect to similarity factor

Formulation and Evaluation for Drug with Polymer in coating system for Repaglinide Extended-Release Multiparticulate system.

Table 5 summarises comparative dissolution profile with target dissolution profile, fig. 3 provides graphical presentation of comparative dissolution profile.

Dissolution profile achieved with drug with polymer in coating system is not similar with target dissolution profile with respect to similarity factor

Formulation and Evaluation for Reservoir System for Extended-Release multiparticulate system.

Table 7 summarises comparative dissolution profile with target dissolution profile, fig. 4 provides graphical presentation of comparative dissolution profile. Dissolution profile of MUMPS 11 batch is similar with target dissolution profile and matching profile can be achieved by further optimization

Optimization of Drug layering process for reservoir system using design of Experiment:

ANOVA table shows model is significant and lack of fit is non significant as desired and can be proceed.

1. **% Fines**

   Half normal chart and pareto chat indicates that main contribution in fines generation is effect of product temperature, Air volume and interaction of product temperature and air volume have significant impact on % fines generation. There is very little interaction of product temperature and air volume

2. **% Agglomerates**

   Half normal chart and pareto chat indicates that main contribution in agglomerate generation is effect of product temperature, Air volume and spray rate have significant impact on % fines generation. There is not any significant interaction.

3. **% Assay**

   Half normal chart and pareto chat indicates that main contribution in % assay is effect of product temperature, Air volume and interaction of product temperature and air volume have significant impact on % fines generation. There is very little interaction of product temperature and air volume.

Optimization of control release coating composition for reservoir system using design of Experiment:

ANOVA table for all three response shows model is significant and lack of fit is non significant as desired to proceed.

DISCUSSION:

- Formulation of repaglinide extended-Release dosage form using drug embedded pellets technique does not give desire release and dissolution profile is not similar with target dissolution profile. Dissolution profile at initial hours was fast compare to target dissolution profile. Also manufacturing of drug embedded pellets involves complex manufacturing process of extrusion and spherodisation.
- Formulation of repaglinide extended-Release dosage form using polymer in drug coating system also not give desire release and dissolution profile is not similar with target dissolution profile. Also there is limitation of use of polymer as all polymer does not offer easy of spraying in fluidized bed processor.
- Formulation of repaglinide extended-Release dosage form of multi-particulate system using reservoir system offers easy of manufacturing process and initial experiments give promising results and target dissolution profile can be achieved by further optimization.
- Starter pellets of sugar sphere and celesphere were evaluated and concluded that sugar sphere will help to achieve extend of release at end our compare to celesphere
- Further drug layering process will be optimized using of factorial design by identifying the high risk factor that affect % fines generation, % agglomerates generation and % assay.

- Optimized process parameter was determined using design space and these parameter were used in further trial of control release coating optimization.
- Aqueous dispersion of Ethylcellulose (surelease 19010) from colorcon was used as control release membrane and HPMC 6 cps were used as pore former.
- Suitable ratio of Surelease and HPMC 6 cps were determine using Response surface methodology using design of experiments.
- Optimized trial was confirm by executing trial and compared with target dissolution profile and fount satisfactory.

CONCLUSION

Conclusion for Drug with Polymer in coating system for Repaglinide Extended-Release Multiparticulate system.: from above experiment it can concluded that target dissolution profile cannot be achieved using drug embedded matrix pellets and required further controlled release at initial hrs.

Conclusion for Drug with Polymer in coating system for Repaglinide Extended-Release Multiparticulate system: from above experiment it can concluded that target dissolution profile cannot be achieved using drug with polymer in coating pellets and required further controlled release at initial hrs.

Conclusion for reservoir system: From above experiment it can conclude that target dissolution profile can be achieved using reservoir system. Further experiments will be carried out to optimize drug layering process and control release composition using design of Experiment.

Optimization of Drug layering process for reservoir system using design of Experiment:

Based on analysis of factorial design experiment design space were created and optimized drug layering parameter were derived as presented in Table 14.

Optimization of control release coating composition for reservoir system using design of Experiment:

Based on analysis of response surface model, optimised composition was derived for target dissolution profile and experimental trial was conducted as per composition provided in tablets 18. Dissolution profile achieved using optimised formulation is matching with target dissolution profile with F2 of 72.32

Multt-particulates drug delivery system provides various advantages like dose dumping and reduction in sea saw fluctuation. Repaglinide can be formulated using multi-particulate system having reservoir system. Optimized drug layering process offers identified range of process parameter and optimized controlled release coating composition of suitable ration of ethyl cellulose and HPMC 6 cps provide target dissolution profile.

FIGURES

Fig. 1: Different techniques for Multiparticulate drug delivery system

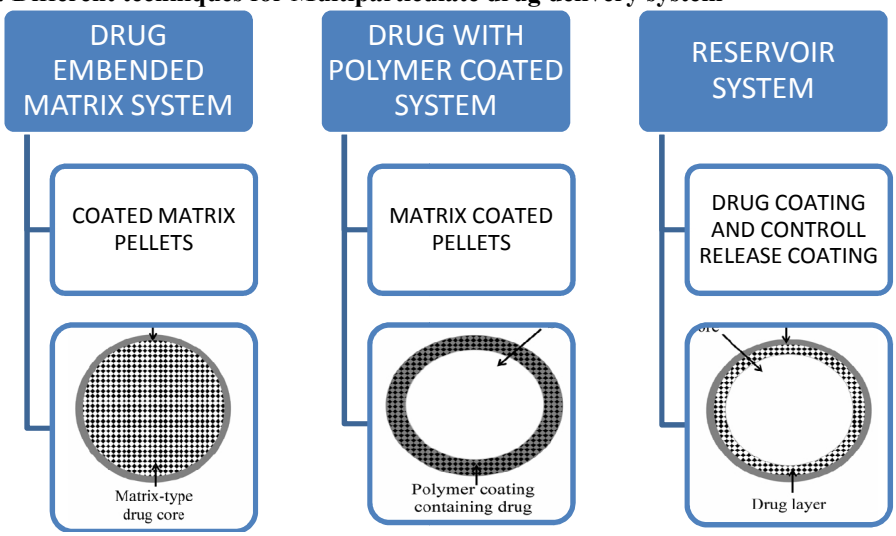


Fig. 2: Graphical presentation of comparison of dissolution Profile

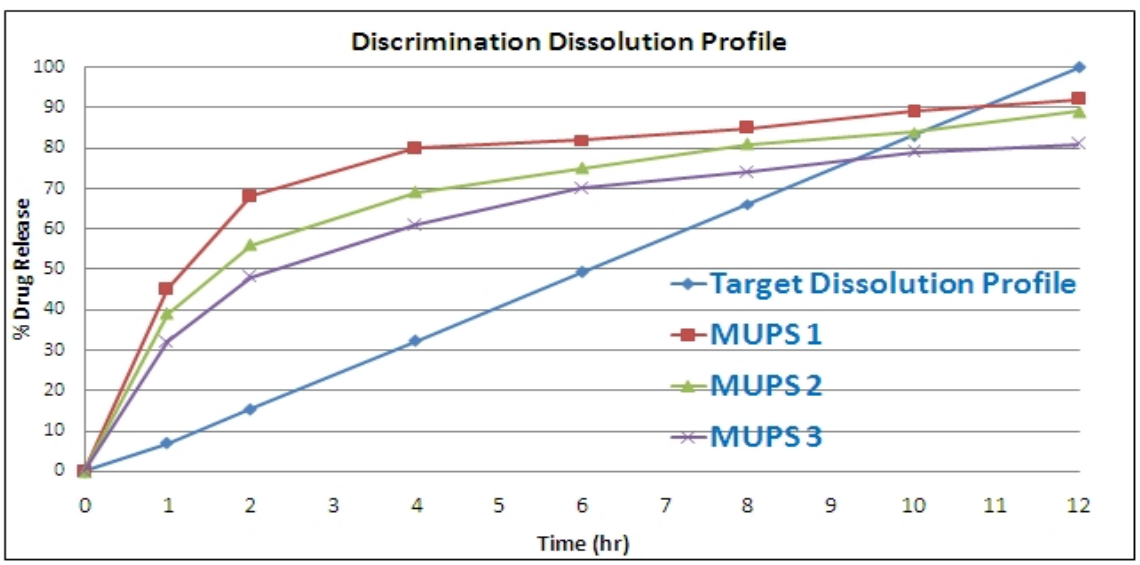


Fig. 3 Graphical presentation of comparison of dissolution Profile

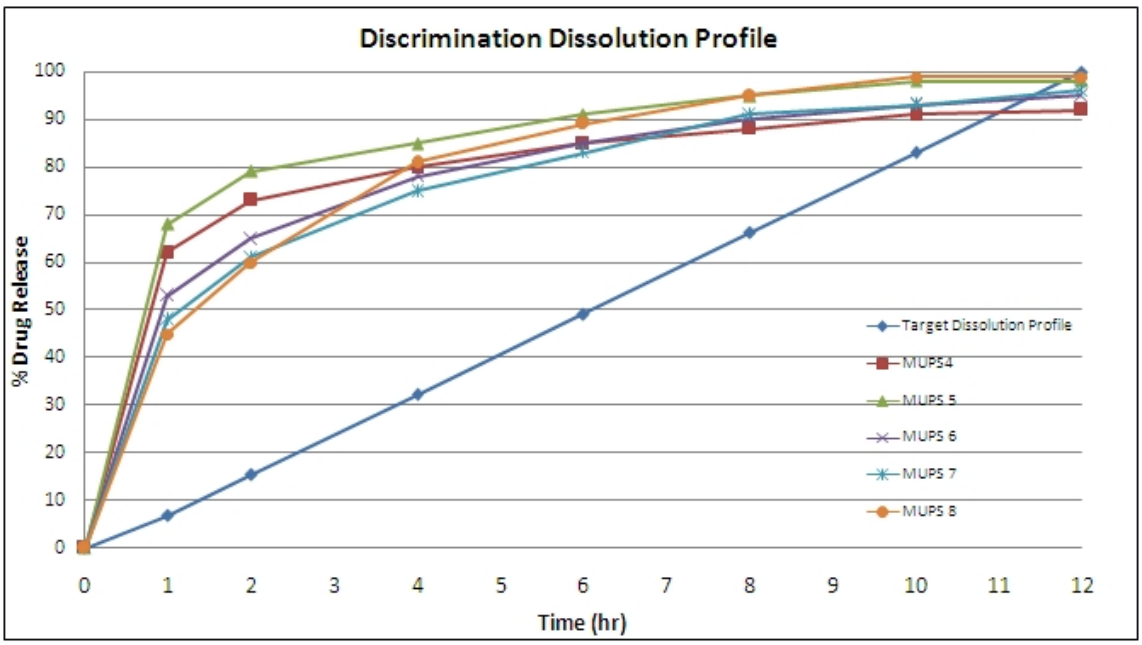


Fig. 4: Graphical presentation of comparison of dissolution Profile

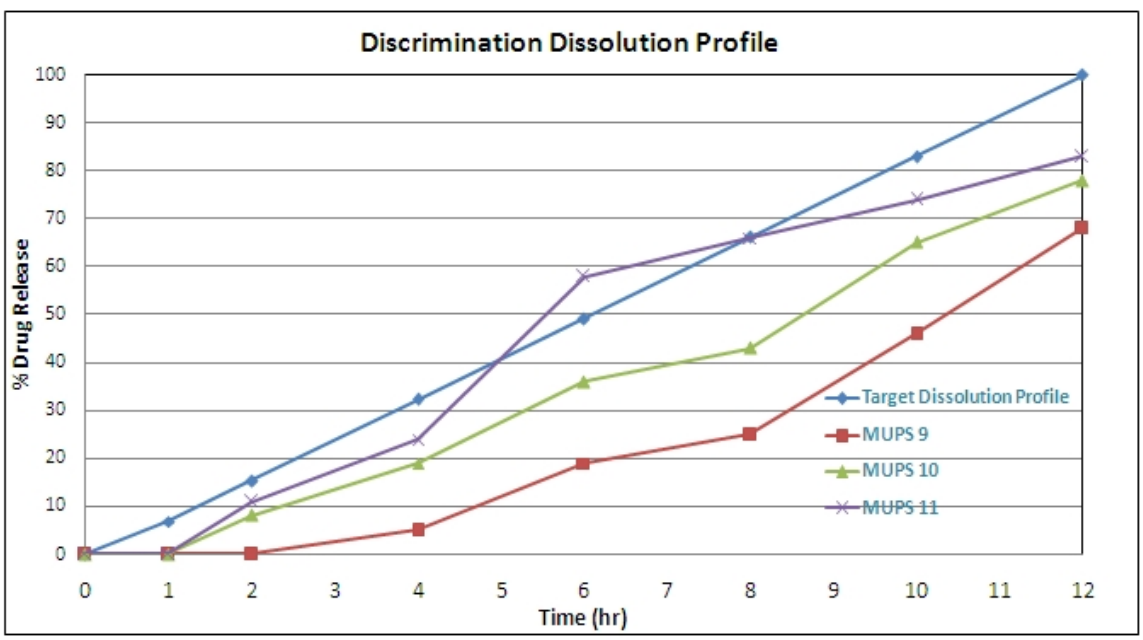


Fig. 5: schematic of Fluidized bed processor

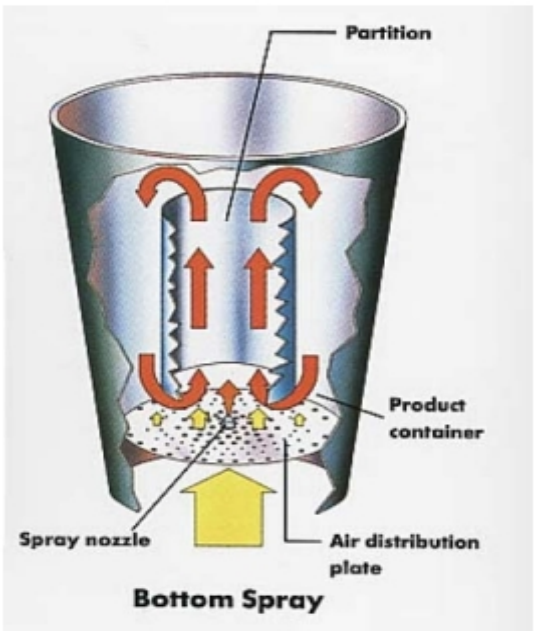


Fig. 6. Half normal chart for % fines

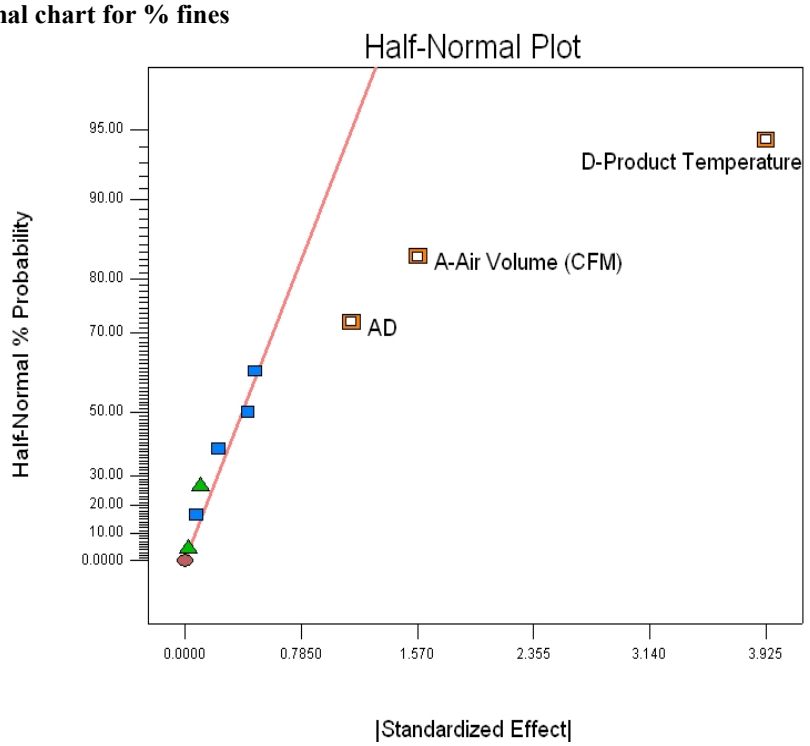


Fig. 7 Pareto chart for % fines

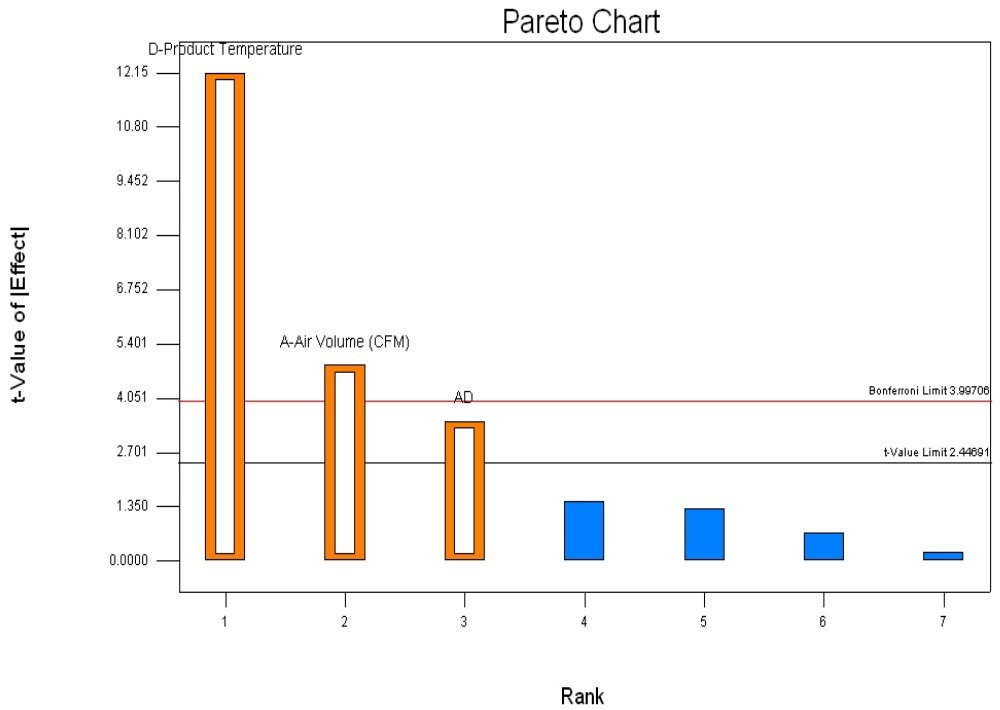


Fig. 8: Interaction effect chart for % fines

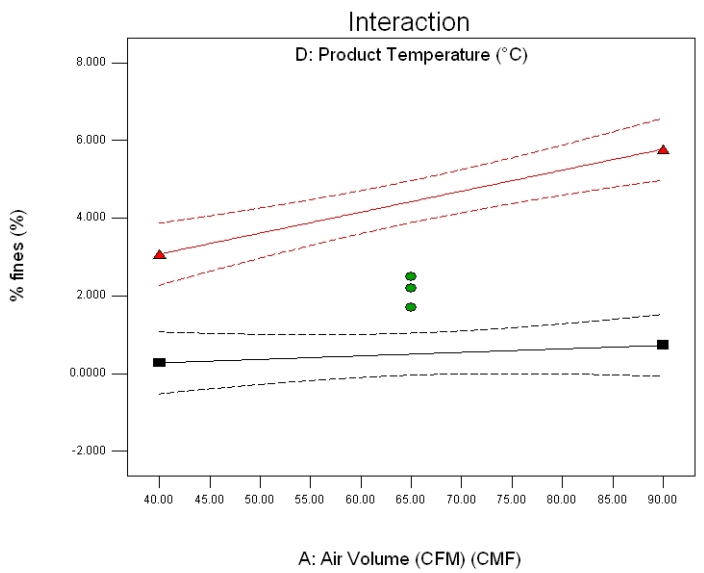


Fig. 9. Half normal plot for % agglomerates

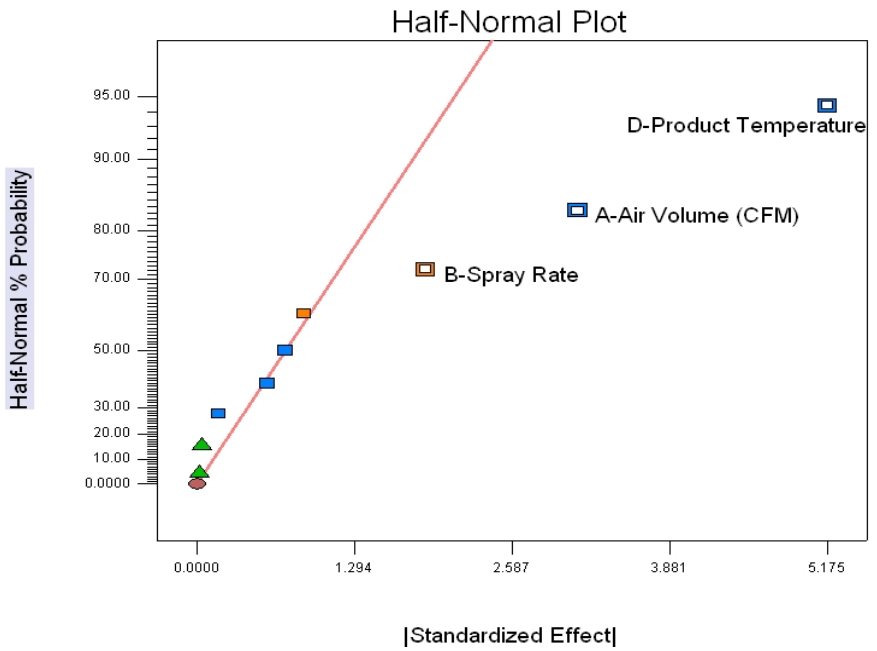


Fig. 10: Pareto chart for % agglomerates

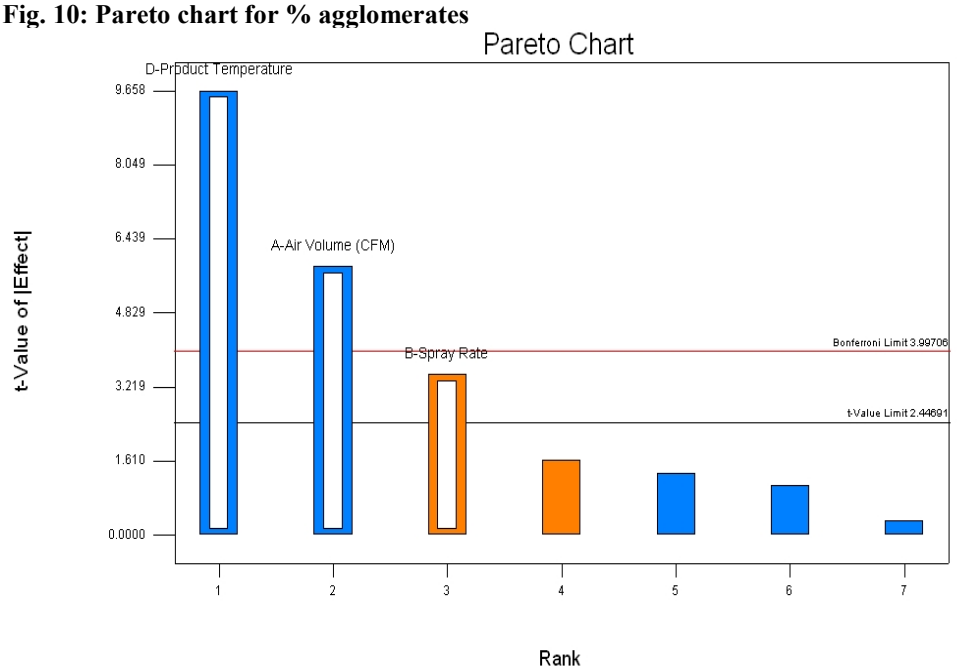


Fig. 11: Interaction effect for % agglomerates

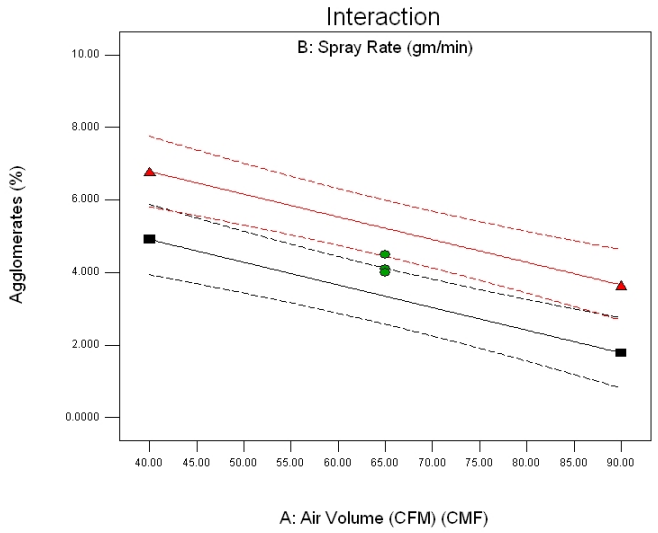


Fig.12. Half normal plot for % assay

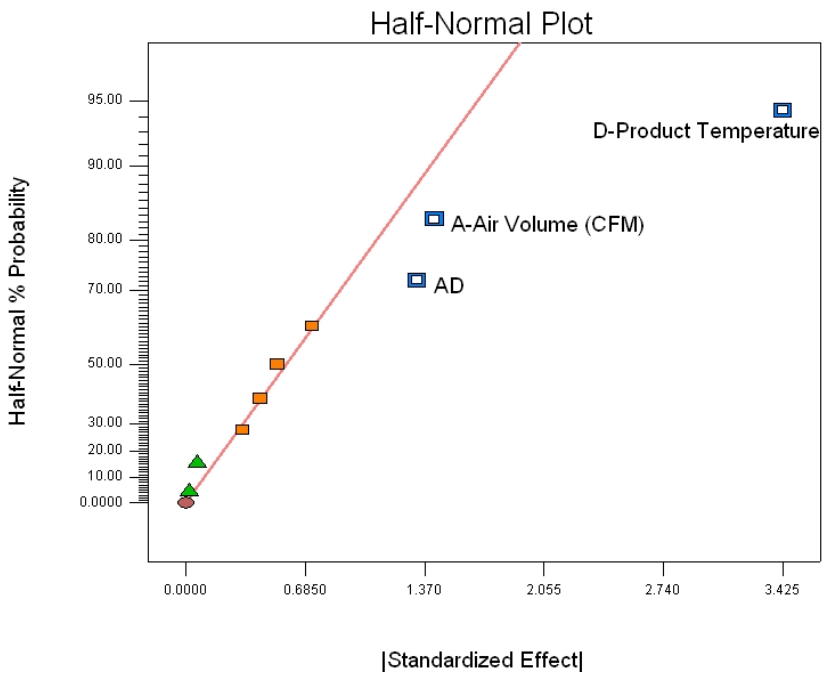


Fig. 13. Pareto chart for % assay

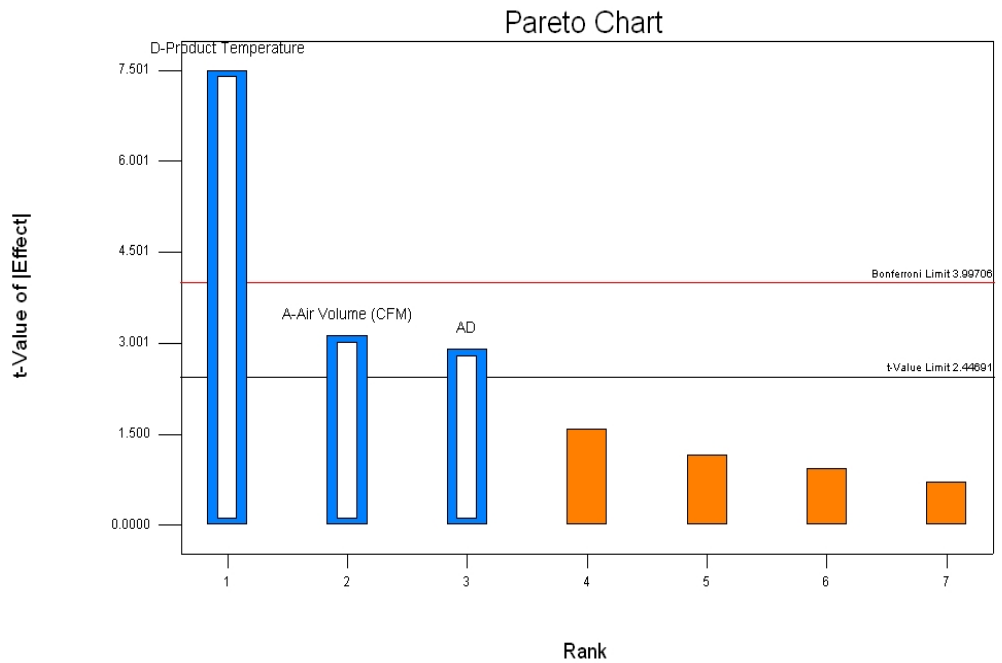


Fig. 14. Interaction Plot for % assay

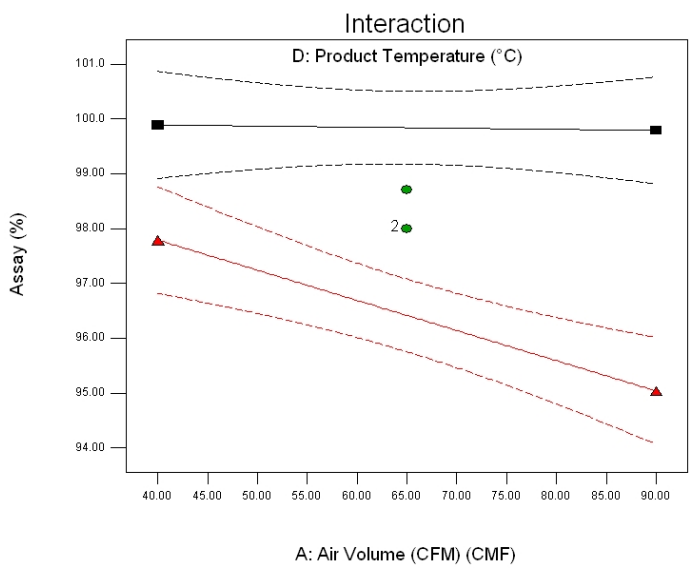


Fig. 15. Counter plot for Assay

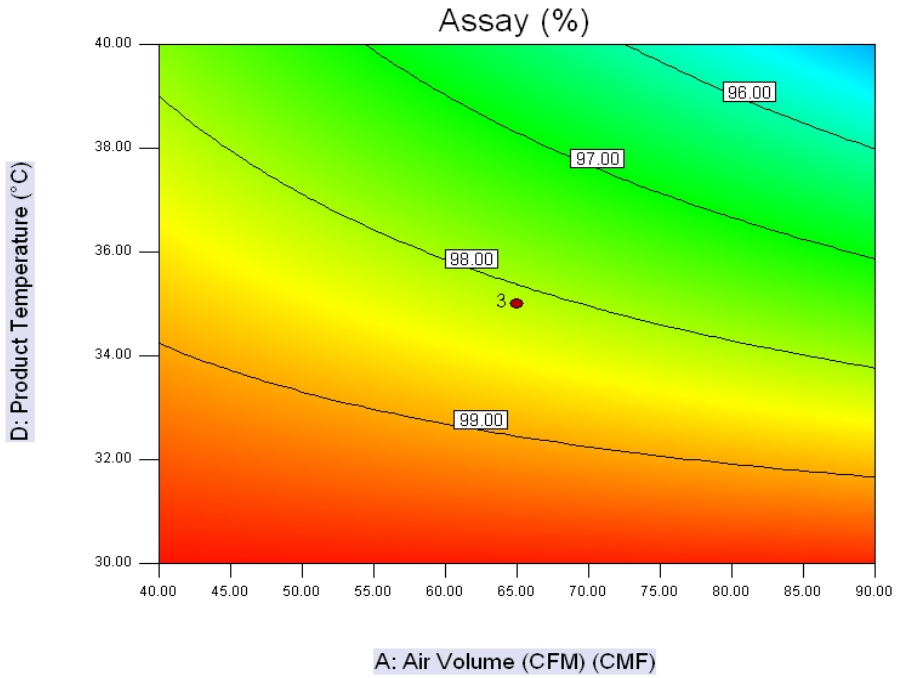


Fig. 16. Surface plot for Assay

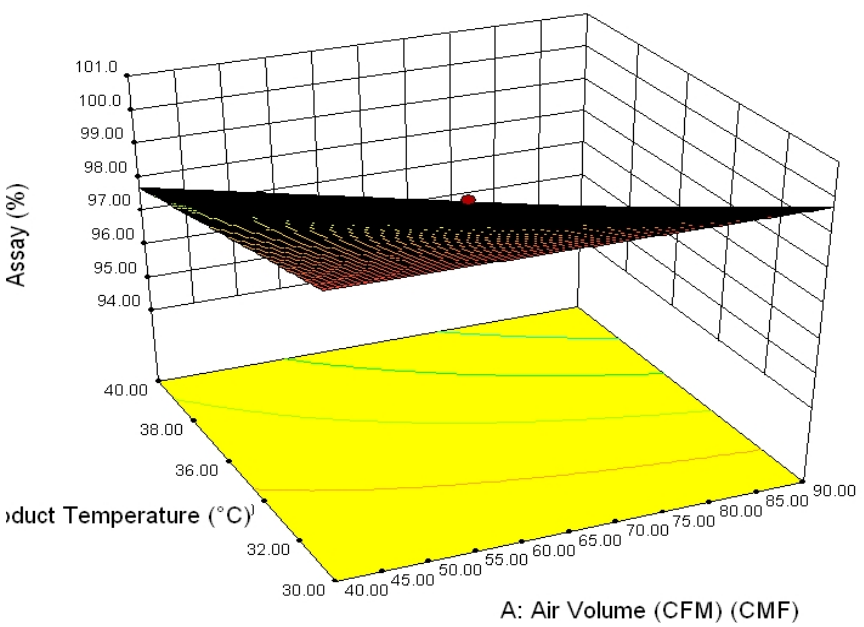


Fig. 17: design space for drug layering process

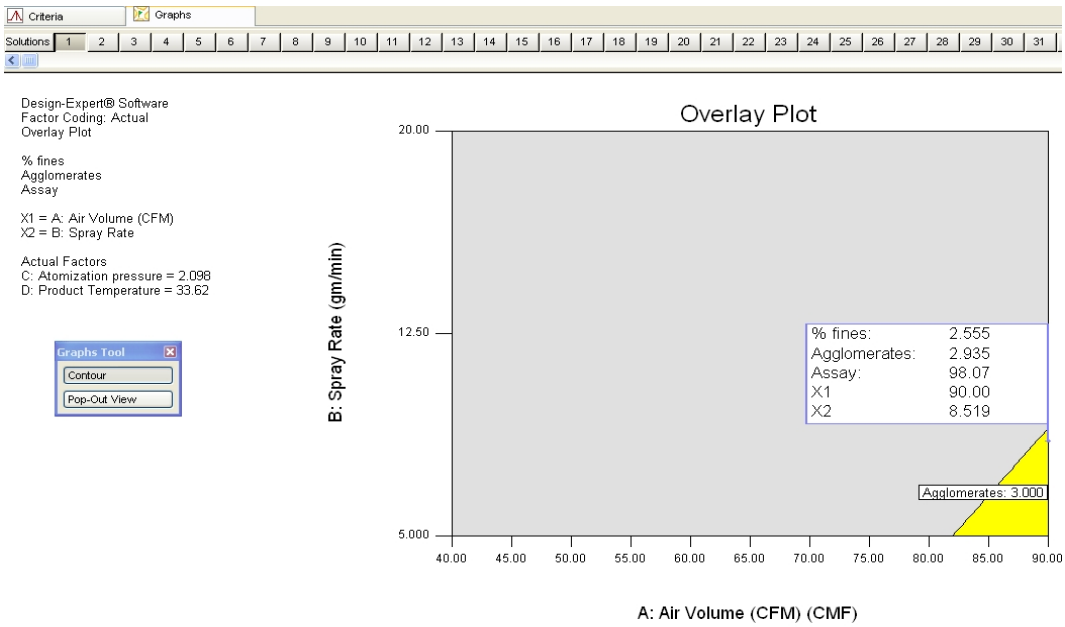


Fig. 18: Fraction of design space graph for response surface method

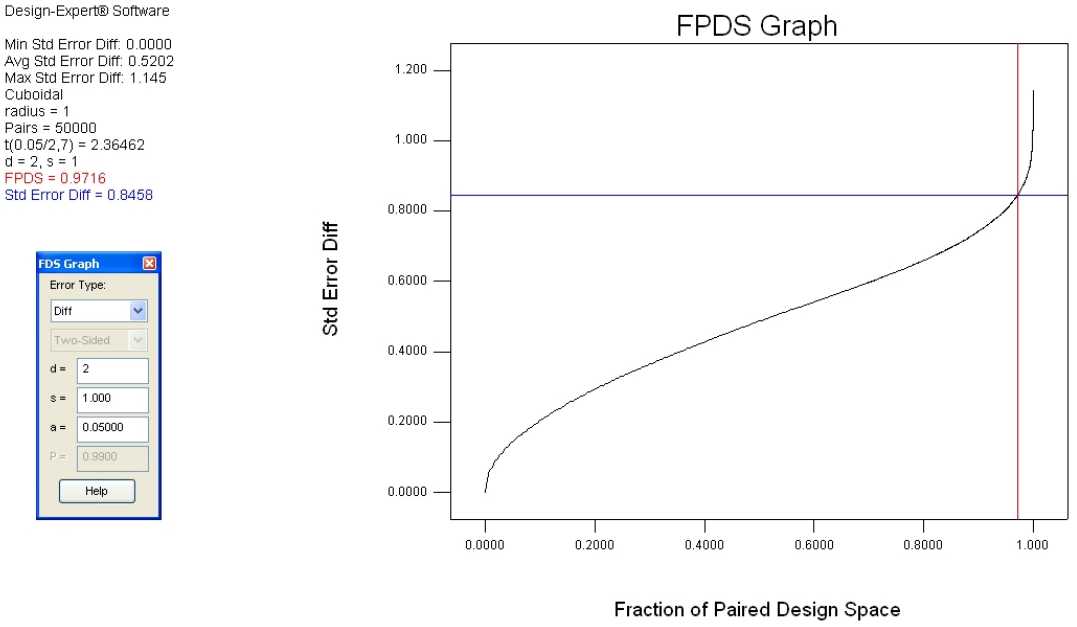


Fig. 19: ANOVA table

Response 1 Release at 1hr

ANOVA for Response Surface Reduced Quadratic model

Analysis of variance table [Partial sum of squares - Type III]

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Model	1018.	4	254.5	229.4	< 0.0001	significant
A-Surelase	*783.8*	*1*	*783.8*	*706.4*	*< 0.0001*	
B-HPMC 6 cp	*96.10*	*1*	*96.10*	*86.62*	*< 0.0001*	
AB	*6.250*	*1*	*6.250*	*5.633*	*0.04500*	
A²	*131.8*	*1*	*131.8*	*118.8*	*< 0.0001*	
Residual	8.876	8	1.109			
Lack of Fit	*3.676*	*4*	*0.9190*	*0.7069*	*0.6275*	*not significant*
Pure Error	*5.200*	*4*	*1.300*			
Cor Total	1027.	12				

Fig. 20: Counter Plot for release at 1 hr

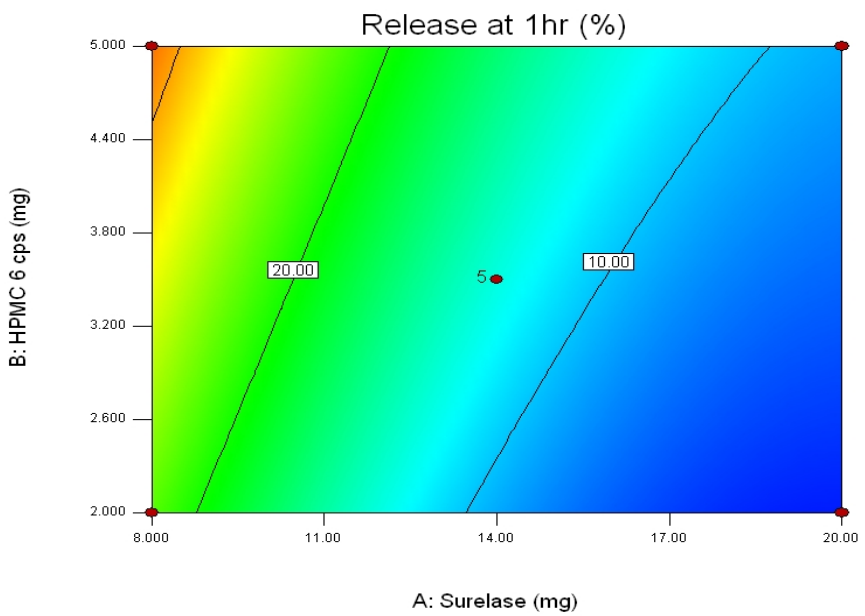


Fig. 21: surface plot for release at 1 hr

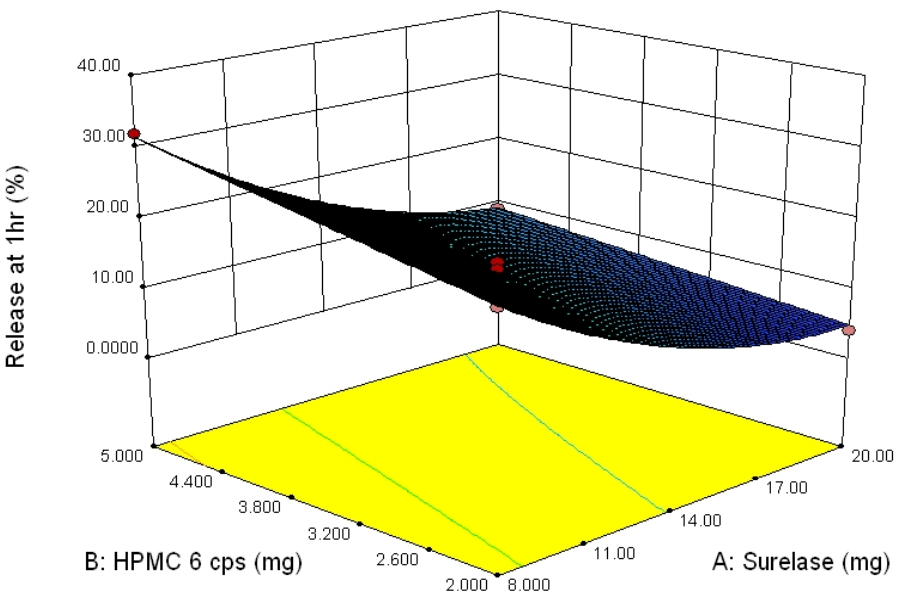


Fig. 22: interaction Plot for release at 1 hr

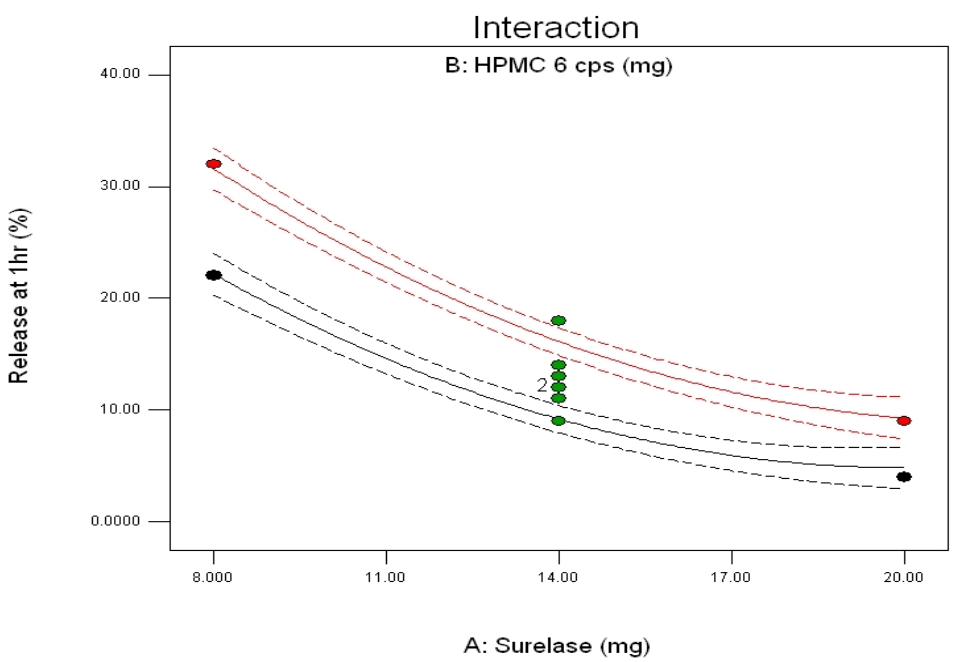


Fig 23: Counter Plot for release at 6 hr

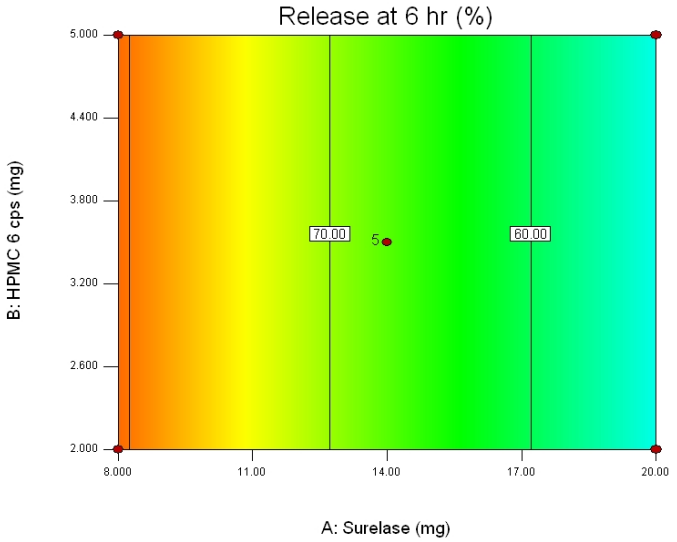


Fig. 24: surface plot for release at 6 hr

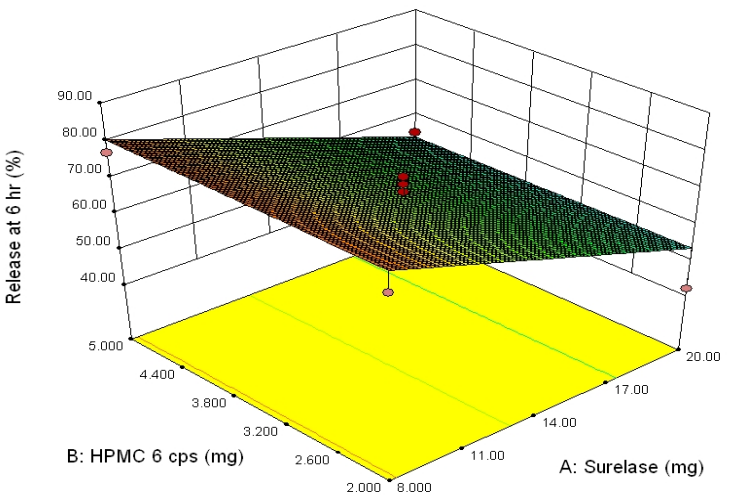


Fig. 25: ANOVA for release at 6 hr

Response 2 Release at 6 hr

ANOVA for Response Surface Reduced Linear model

Analysis of variance table [Partial sum of squares - Type III]

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Model	1440.	1	1440.	55.80	< 0.0001	significant
A-Surelase	*1440.*	*1*	*1440.*	*55.80*	*< 0.0001*	
Residual	283.8	11	25.80			
Lack of Fit	*256.6*	*7*	*36.66*	*5.392*	*0.06138*	*not significant*
Pure Error	*27.20*	*4*	*6.800*			
Cor Total	1724.	12				

Fig. 26: Counter Plot for release at 12 hr

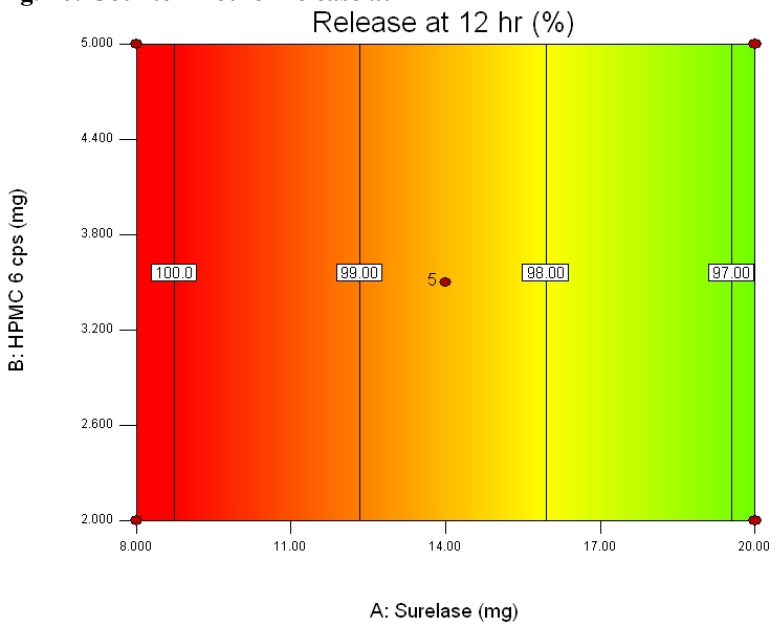


Fig. 27: surface plot for release at 12 hr

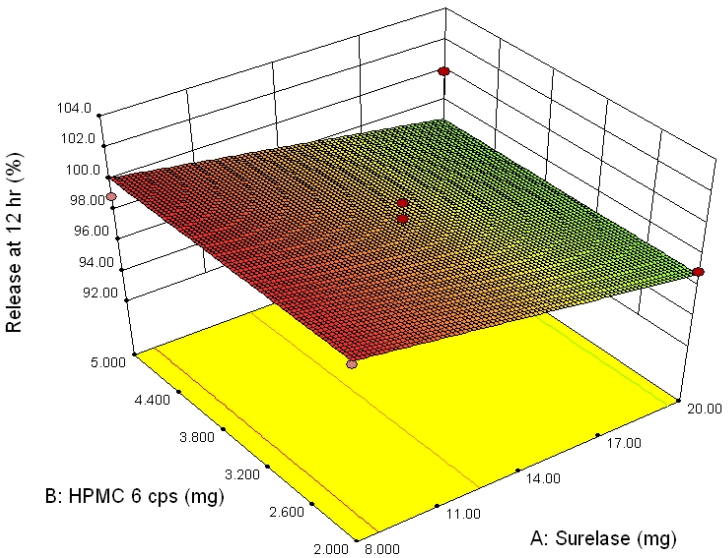


Fig. 28: ANOVA for release at 12 hr

Response 3 Release at 12 hr

ANOVA for Response Surface Reduced Linear model

Analysis of variance table [Partial sum of squares - Type III]

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Model	22.16	1	22.16	6.238	0.02964	significant
A-Surelase	*22.16*	*1*	*22.16*	*6.238*	*0.02964*	
Residual	39.07	11	3.552			
Lack of Fit	*33.07*	*7*	*4.725*	*3.150*	*0.1422*	*not significant*
Pure Error	*6.000*	*4*	*1.500*			
Cor Total	61.23	12				

Fig. 29: Optimization trial from surface response method

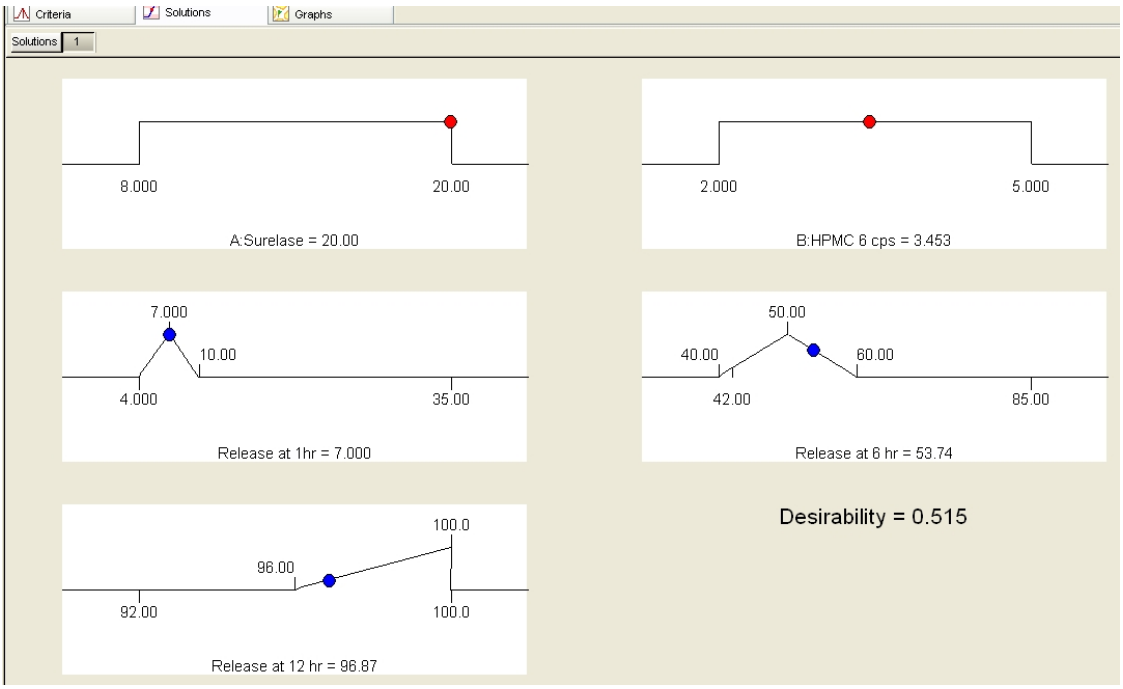
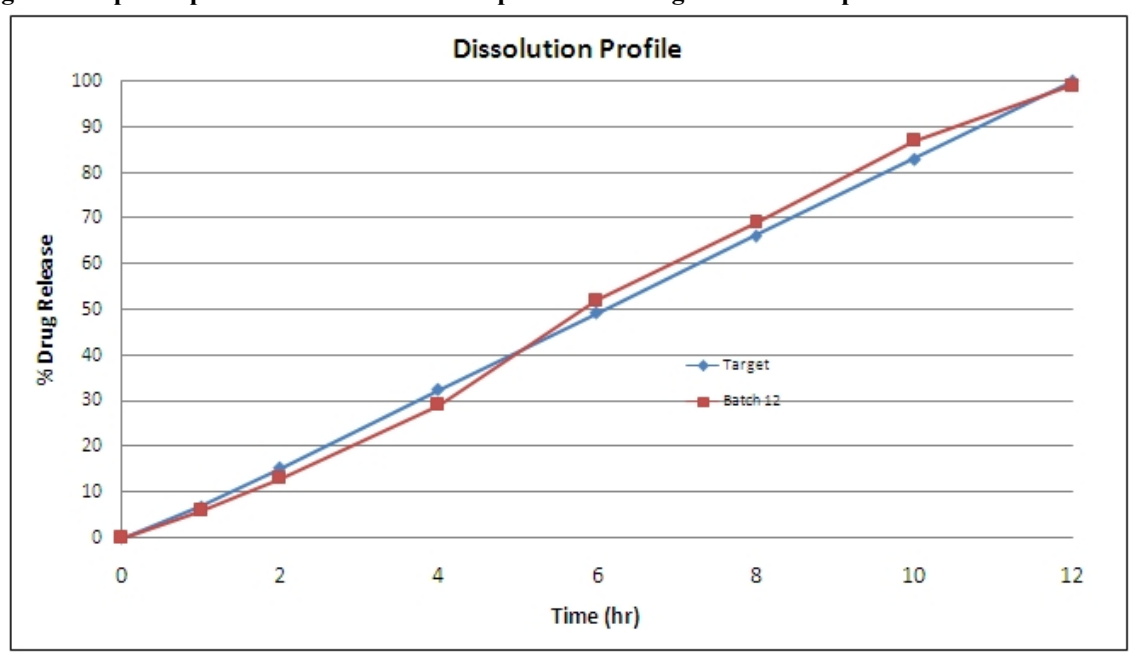


Fig. 30. Graphical presentation of dissolution profile with target dissolution profile



TABLES

Table 1. Pharmacokinetic parameter and Theoretical target profile of Repaglinide for 12 hr

PHARMACOKINETIC PARAMETER(*3,10*)	
Cp desired	4.00 ng/ml
Half Life	Half-life of approximately 1 hour.
Bioavailability (F)	The mean absolute bioavailability is 56%.
Distribution (Vd)	31 L at steady state
Total body clearance (CL)	38 L/h.

Time (hr)	Amount of Drug in mg	Desired % Drug Release
1	0.2214	6.90
2	0.4982	15.37
3	0.7642	23.83
4	1.0356	32.29
5	1.3070	40.76
6	1.5784	49.22
7	1.8498	57.68
8	2.1212	66.15
9	2.3926	74.61
10	2.6640	83.07
11	2.9354	91.54
12	3.2068	100.00

Table 2. Qualitative and Quantitative composition for drug embedded matrix system.

Sr. No	Ingredients	MUPS 1	MUPS 2	MUPS 3
Dry Mixing				
1	Repaglinide	3.0	3.0	3.0
2	Microcrystalline cellulose (Avicel pH 101)	100	100.0	100.0
3	Lactose Monohydrate	67.0	57.0	47.0
4	HPMC K4M CR	10.0	20.0	30.0

Granulation				
5	Purified Water	Qs	qs	Qs
	Total	180.0	180.0	180.0

Table 3. Dissolution profile comparison with Target dissolution Profile

Dissolution Method: 900 ml of 0.1 N HCl pH 2.0, Basket, 100 rpm				
Batch No **Time (hr)**	**Target Dissolution** **% Release**	**MUPS 1** **% Release**	**MUPS 2** **% Release**	**MUPS 3** **% Release**
1	6.9	45	39	32
2	15.37	68	56	48
4	32.29	80	69	61
6	49.22	82	75	70
8	66.15	85	81	74
10	83.07	89	84	79
12	100	92	89	81
F2 (similarity Factor)		23.25	28.56	32.85

Table 4. Qualitative and Quantitative composition for Drug with Polymer in coating system

	Batch No	**MUPS4**	**MUPS5**	**MUPS6**	**MUPS7**	**MUPS8**
Sr.	**Ingredients**	**Mg/tab**	**Mg/tab**	**Mg/tab**	**Mg/tab**	**Mg/tab**
Starter Core						
1	**Cele Sphere (20# -25#)**	**137.0**	**127.0**	**117.0**	**102.0**	**--**
2	**Sugar Sphere (20#-25#)**	**--**	**--**	**--**	**--**	**102.0**
Drug Layering						
3	**Repaglinide**	**3.0**	**3.0**	**3.0**	**3.0**	**3.0**
4	**Eudragit RS PO**	**30.0**	**30.0**	**40.0**	**50.0**	**50.0**
5	**Eudragit RL PO**	**--**	**10.0**	**10.0**	**15.0**	**15.0**
6	**HPMC 15 cps**	**10.0**	**10.0**	**10.0**	**10.0**	**10.0**
7	**Isopropyl alcohol**	**Qs**	**Qs**	**Qs**	**Qs**	**Qs**
8	**Dichloromethane**	**Qs**	**Qs**	**Qs**	**Qs**	**Qs**
	Total	**180.0**	**180.0**	**180.0**	**180.0**	**180.0**

Table 5. Dissolution Profile Comparison with Target dissolution Profile

Batch No	Target Dissolution	MUPS 4	MUPS 5	MUPS 6	MUPS 7	MUPS 8
Method	0.1 N HCL pH 2.0, 900 ml, 100 rpm, Basket					
Time (hr)	% Release	% Release	% Release	% Release	% Release	% Release
1	6.9	62	68	53	48	45
2	15.37	73	79	65	61	60
4	32.29	80	85	78	75	81
6	49.22	85	91	85	83	89
8	66.15	88	95	90	91	95
10	83.07	91	98	93	93	99
12	100	92	98	95	96	99
F2 (similarity Factor)		20.57	17.97	22.69	24.29	22.90

Table 6. Qualitative and Quantitative Composition for reservoir system

	Batch No	MUPS9	MUPS10	MUPS11
Sr.	Ingredients	Mg/tab	Mg/tab	Mg/tab
Starter Core				
1	Cele Sphere (20# -25#)	140	140	--
2	Sugar Sphere (20#-25#)	--	--	140
Drug Layering				
3	Repaglinide	3.0	3.0	3.0
6	HPMC 6 cps	1.5	1.5	1.5
7	Isopropyl alcohol	Qs	Qs	Qs
8	Dichloromethane	Qs	Qs	Qs
Control Release Coating				
9	Su-release Clear 19040	14.45	11.56	11.56
10	HPMC 6 cps	--	2.89	2.89
	Total	158.95	158.95	158.95

Table 7: Dissolution Profile comparison with target dissolution Profile

Batch No	Target Dissolution	MUPS 9	MUPS 10	MUPS 11
Method	0.1 N HCL pH 2.0, 900 ml, 100 rpm, Basket			
Time (hr)	% Release	% Release	% Release	% Release
1	6.9	0	0	0
2	15.37	0	8	11
4	32.29	5	19	24
6	49.22	19	36	58
8	66.15	25	43	66
10	83.07	46	65	74
12	100	68	78	83
Similarity factor (F2)		26.60	39.70	51.84

Table 8: Qualitative and Quantitative composition for Drug layering process

Sr.	Ingredients	Mg/tab
Starter Core		
1	Suagar Sphere (20# -25#)	125
Seal Coating		
2	Surelease 19040	10.0
Drug Layering		
3	Repaglinide	3.0
6	HPMC 6 cps	1.5
7	Isopropyl alcohol	Qs
8	Dichloromethane	Qs

Table 9. List of fixed and Variable drug layering process parameter using GPCG 1.1

Drug layering process Parameter		Variable/Fixed
Equipment Model	Glatt GPCG 1.1	Fixed
Batch Size	500 gm	Fixed
Partition column Height	12 cm	Fixed
Distribution Plate	C plate	Fixed
Nozzle tip diameter	1.0 mm	Fixed
Dew Point	10 $\pm 2^0$C	Fixed

Inlet temperature	40-60^{0}C	Variable
Product temperature	35^{0}C	Variable
Spray rate	5-20 gm/min	Variable
Atomization pressure	1-2.5 bar	Variable
Air volume	40-90 CFM	Variable

Table 10: Identification of factor and level for design of experiment and response

Factor: Process Parameter		**Levels**	
		-1	+1
A	Air volume (cfm)	40	90
B	Spray rate (g/min)	5	20
C	Atomization pressure (bar)	1	2.5
D	Product temperature (°C)	30	40
Three centre point were selected to evaluate curvature effect.			
Response		Target Range	
Y1	Fines < 250 µm (%)	≤ 5	
Y2	Agglomerates > 420 µm (%)	≤ 5	
Y3	Assay (HPLC method) (% w/w)	95	105

Table 11. Experimental run for Drug layering process optimization using 2^{4-1} factorial design

Std	Run	Factor 1 A:Air Volum... CMF	Factor 2 B:Spray Rate gm/min	Factor 3 C:Atomizatio... bar	Factor 4 D:Product Te... °C	Response 1 % fines %	Response 2 Agglomerates %	Response 3 Assay %
2	1	90.00	5.000	1.000	40.00	6.500	0.4000	94.00
8	2	90.00	20.00	2.500	40.00	5.300	0.8000	96.00
7	3	40.00	20.00	2.500	30.00	0.5000	10.00	99.80
3	4	40.00	20.00	1.000	40.00	2.900	3.800	97.60
1	5	40.00	5.000	1.000	30.00	0.3000	7.800	99.90
5	6	40.00	5.000	2.500	40.00	3.500	1.900	97.90
9	7	65.00	12.50	1.750	35.00	2.200	4.100	98.00
10	8	65.00	12.50	1.750	35.00	1.700	4.000	98.70
4	9	90.00	20.00	1.000	30.00	0.8000	6.400	100.0
6	10	90.00	5.000	2.500	30.00	0.9000	3.400	99.50
11	11	65.00	12.50	1.750	35.00	2.500	4.500	98.00

Analysis of Experiments for DOE:

1. **ANOVA Table:**

Table 12 : ANOVA table for 2^{4-1} design of experiment

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Model	31.03	3	10.34	24.81	0.0008823	significant
A-Air Volume	*4.061*	*1*	*4.061*	*9.741*	*0.02056*	
D-Product Te	*23.46*	*1*	*23.46*	*56.27*	*0.0002903*	
AD	*3.511*	*1*	*3.511*	*8.421*	*0.02726*	
Curvature	0.04640	1	0.04640	0.1113	0.7500	
Residual	2.502	6	0.4169			
Lack of Fit	*2.175*	*4*	*0.5438*	*3.329*	*0.2441*	*not significant*
Pure Error	*0.3267*	*2*	*0.1633*			
Cor Total	33.58	10				

The following ANOVA is for a model that does not adjust for curvature.
This is the default model used for prediction and model plots.

ANOVA for selected factorial model
Analysis of variance table [Partial sum of squares - Type III]

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Model	31.03	3	10.34	28.42	0.0002718	significant
A-Air Volume	*4.061*	*1*	*4.061*	*11.16*	*0.01242*	
D-Product Te	*23.46*	*1*	*23.46*	*64.45*	*< 0.0001*	
AD	*3.511*	*1*	*3.511*	*9.646*	*0.01718*	
Residual	2.548	7	0.3640			
Lack of Fit	*2.221*	*5*	*0.4443*	*2.720*	*0.2904*	*not significant*
Pure Error	*0.3267*	*2*	*0.1633*			
Cor Total	33.58	10				

Table 13: process parameter, level and goals for optimization of drug layering process.

Factor: Process Parameter		Levels		Goals
		-1	+1	
A	Air volume (cfm)	40	90	Suggest
B	Spray rate (g/min)	5	20	Maximize
C	Atomization pressure (bar)	1	2.5	Suggest
D	Product temperature (°C)	30	40	Suggest
Response		Target Range		
Y1	Fines < 250 µm (%)	≤ 5		Minimize
Y2	Agglomerates > 420 µm (%)	≤ 5		Minimize
Y3	Assay (HPLC method) (% w/w)	95	105	Maximize

Table : 14: Optimized drug layering process parameter

Optimized Drug Layering Process Parameter	
Equipment Model	Glatt GPCG 1.1
Batch Size	500 gm
Partition column Height	12 cm
Distribution Plate	C plate
Nozzle tip diameter	1.0 mm
Dew Point	10 ±2^{o}C
Inlet temperature	40-60^{o}C
Product temperature	32^{o}C
Spray rate	5-20 gm/min
Atomization pressure	2.1bar
Air volume	90 CFM

Table 15: qualitative and quantitative composition for control release coating

	Batch No	MUPS9
Sr.	Ingredients	Mg/tab
Starter Core		
1	Suagar Sphere (20# -25#)	125

Seal Coating		
2	**Surelease 19040**	**10.0**
Drug Layering		
3	**Repaglinide**	**3.0**
6	**HPMC 6 cps**	**1.5**
7	**Isopropyl alcohol**	**Qs**
8	**Dichloromethane**	**Qs**
Control Release Coating		
9	**Su-release Clear 19040**	**8 to 20**
10	**HPMC 6 cps**	**2 to 5**

Table 16: Identification of factor and level for design of experiment and response

Factor: Process Parameter		**Axial Points**	**Levels**		**Axial Points**
			-1	**+1**	
A	**Surelease 18040**	**5.515**	**8**	**20**	**22.49**
B	**HPMC 6 Cps**	**1.379**	**2**	**5**	**5.621**
Response		**Target Range**			
Y1	**% Release at 1 hr**	**7%**			
Y2	**% Release at 6 hr**	**50%**			
Y3	**% Release at 12 hr**	**100%**			

Table 17. Experimental run for control release coating optimization using response surface method

Std	Run	Factor 1 A:Surelase mg	Factor 2 B:HPMC 6 cps mg	Response 1 Release at 1hr %	Response 2 Release at 6 ... %	Response 3 Release at 1 ... %
1	1	8.000	2.000	22.00	75.00	100.0
3	2	8.000	5.000	32.00	77.00	99.00
11	3	14.00	3.500	12.00	72.00	100.0
13	4	14.00	3.500	13.00	70.00	99.00
7	5	14.00	1.379	9.000	70.00	100.0
12	6	14.00	3.500	11.00	66.00	97.00
5	7	5.515	3.500	35.00	85.00	100.0
2	8	20.00	2.000	4.000	42.00	97.00
4	9	20.00	5.000	9.000	55.00	100.0
8	10	14.00	5.621	18.00	73.00	98.00
6	11	22.49	3.500	8.000	48.00	92.00
10	12	14.00	3.500	12.00	68.00	99.00
9	13	14.00	3.500	14.00	72.00	100.0

Table 18: qualitative and quantitative composition for Optimized Reservoir system for repaglinide

	Batch No	MUPS9
Sr.	Ingredients	Mg/tab
Starter Core		
1	Suagar Sphere (20# -25#)	125
Seal Coating		
2	Surelease 19040	10.0
Drug Layering		
3	Repaglinide	3.0
6	HPMC 6 cps	1.5
7	Isopropyl alcohol	Qs
8	Dichloromethane	Qs
Control Release Coating		
9	Su-release Clear 19040	20.0
10	HPMC 6 cps	3.45
	Total	

Table 19 Comparison of Dissolution Profile:

Batch No	Target Dissolution	MUPS
Method	0.1 N HCL pH 2.0, 900 ml, 100 rpm, Basket	
Time (hr)	% Release	% Release
1	6.9	6
2	15.37	13
4	32.29	29
6	49.22	52
8	66.15	69
10	83.07	87
12	100	99
Similarity Factor		77.32

ACKNOWLEDGEMENT

The author would like to thank Dr. H.M. Tank, Principal, Matushree V. B. Manvar College of Pharmacy, Dumiyani, India for his help in preparing this paper.

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Structural and Electrical Transport Properties of

$(1-x)La_{0.7}Sr_{0.3}Mn_{0.94}Co_{0.06}O_3 + x$ (micro Al_2O_3) composite (x = 0%, 4%)

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ABSTRACT

The structural and temperature dependence of electrical resistivity of $(1-x)La_{0.7}Sr_{0.3}Mn_{0.94}Co_{0.06}O_3$ (LSMCO)+x micro Al_2O_3 (x = 0%, 4%) composites were investigated systematically. XRD, SEM and EDX data analysis indicates that micro Al_2O_3 is not substituted into the main LSMCO phase; it is manly distributed at the grain boundaries. XRD result of LSMCO shows rhombohedral structure with the space group of R-3c. We report the resistivity (ρ) – temperature (T) curves for LSMCO+x micro Al_2O_3 (x = 0%, 4%) composite over temperature regime of 5-300 K. Al_2O_3 addition has increased the resistivity of this composite. The metal (M) – insulator (I) transition temperature (T_{MI}) decreases with adding micro Al_2O_3content. The metallic part of the temperature dependent resistivity curve, ρ(T) (below T_{MI}) is well fitted with $\rho = \rho_0 + \rho_2 T^2 + \rho_{4.5} T^{4.5}$ indicating the importance of electron-magnon interaction for explaining low-temperature ($T<T_{MI}$) transport data of the LSMCO/micro Al_2O_3 composite.

SUMMARY

Include a brief summary of your research work in exactly one sentence.

Keywords: Manganite, Composite, Electrical resistivity

INTRODUCTION

Recently, perovskite manganites with the general formula as $Ln_{1-x}R_xMnO_3$ (Ln = Rare earth and R = Ca, Pb, Sr, Ba) have been the subject of considerable attention due to their colossal magnetoresistance (CMR) properties. The mixed valence manganites have been an area under discussion of intensive research from both the scientific and technological points of view due to their good properties and their practical applications in recording devices, magnetic sensors and switching etc. *(1)*. The colossal magnetoresistance effect occurred due to the "double exchange" mechanism. Two types of CMR effect observed, mostly explain the intrinsic CMR effect within the grains and extrinsic CMR effect is largely dependent on the grain boundary properties.

To enhance the CMR effects, many research groups have tried to synthesize manganite based composites by adding some insulating materials, such as LSMO/CeO_2 *(2)*, LSMO/NiO *(3)*, LBMO/YSZ *(4)*. There are several reported papers deal with CMR composite, but studies related to the electrical transport behaviour of the (1-x)LSMCO+x micro Al_2O_3 (x = 0%, 4%) composite are rare. In this investigation, we have mainly focused on the study of structural changes and grain boundaries contribution in the LSMCO+x micro Al_2O_3 (x = 0%, 4%) through XRD, SEM and electrical transport measurement. So we synthesized the LSMCO+x micro Al_2O_3 (x = 0%, 4%) composite by modified inexpensive sol-gel method.

MATERIALS AND METHODS

The (1-x)LSMCO+x micro Al_2O_3 (x = 0%, 4%) composite samples were synthesized by two steps: 1st step, the $La_{0.7}Sr_{0.3}Mn_{1-x}Co_xO_3$ (x = 0.06) powder was prepared by modified inexpensive sol-gel reaction method *(5)*. High Purity (> 99.9%) acetates were mixed in stochiometry proportion and heated at 200 ^oC with ethylene glycol & dried. It was ground and calcined at 800 ^oC for 12 h. After regrinding, it was divided into two parts and one part was palletized & sintered at 1200 ^oC for 24 h. Finally got pure LSMCO. In 2nd step, the second part of calcined LSMCO and 4% micro Al_2O_3 were mixed, ground pelletized and sintered at 1200 °C for 24 h and finally got LSMCO + 4% micro Al_2O_3 composite. The structure and phase purity of the samples were analyzed through the X-ray diffraction (XRD) with 2 theta (2θ) range (20-80°) and the scan was performed with a 0.02 step size using CuKα (λ=1.5406 Å). The resistivity measurement with (5T) and without magnetic field were carried out using a four-probe techniques in the temperature range from 5-300 K for both the samples.

RESULTS AND DISCUSSION

The XRD patterns at RT show that LSMCO is a single phase in nature with no detectable secondary phases and LSMCO has a rhombohedral structure in hexagonal lattice with the space group R-3c. XRD pattern of LSMCO+4% micro Al_2O_3 powder indicates the clear presence of Al_2O_3 phase in the composite. X-ray diffraction analysis shows that the reflection lines of LSMCO do not shift even after addition of secondary phase; the major intense reflection peaks corresponding to Al_2O_3 are also observed in composite. This indicates that no reaction between the two phases occurred, which suggests that micro Al_2O_3 is mostly distributed at the grain boundaries or on the surface of LSMCO. The indication of two phases was observed through SEM. The SEM of LSMCO+x micro Al_2O_3 composite with x = 0% and 4% are shown in Fig. 2, in that order. The interfaces between LSMCO and micro Al_2O_3 can be distinguished. Moreover, EDX spectra (Fig. 2) of the composites for x = 0% and 4% show the Al peak along with La, Sr, Mn and O peaks, which shows the presence of Al_2O_3 in the composite.

The temperature dependence of resistivity (ρ-T) plots of both LSMCO & LSMCO+4% micro Al_2O_3 composite samples in the range of 5-300 K at H = 0, 5T are shown in Fig. 3 (A & B). Both the samples are exhibiting metal-insulator transition (T_{MI}). In the available temperature range (5-300K) resistivity in composite is suppressed due to the applied magnetic field (5T). Due to the application of magnetic field T_{MI} shift towards higher temperature, T_{MI} values are shown in table-1. This may be due to the fact that the application of magnetic field delocalizes the charge carriers suppressing the resistivity causing local ordering of the electron spins. Further, T_{min} is found shifting towards low temperature side with applied field 5T. This shifting indicates that the strong dependence of resistivity minimum on applied magnetic field. In The ρ-T curves slightly upturn below T_{min} is also found. Keeping these observations in mind, it is concluded that the T_{min} might be originated from the spin dependent scattering.

For both LSMCO & LSMCO+4% micro Al_2O_3 composite samples at the temperature ($T<T_{MI}$), the metallic behaviour of each sample can be explained in terms of electron magnon scattering of the carriers as shown in inset Fig. 3 (a & b). In this temperature region, the resistivity data are fitted quite well with the following equation,

$$\rho = \rho_0 + \rho_2 T^2 + \rho_{4.5} T^{4.5} \quad \ldots\ldots\ldots (1)$$

where, ρ_0 is the temperature independent part which arises due to grain and domain boundaries. The $\rho_2 T^2$ comes as a result of electron-electron (e-e) and electron-phonon scattering process and the $\rho_{4.5} T^{4.5}$ term is a combination of electron-electron, electron-magnon and electron-phonon scattering process *(6)*. The best fitted parameters values are shown in table-1.

CONCLUSION

The LSMCO/x micro Al_2O_3 (x = 0%, 4%) composite was synthesized and their structural & electrical resistivity were investigated. It is concluded through XRD, SEM and EDX data analysis that micro Al_2O_3 does not reach to with LSMCO lattice and is observed to remain at grain boundaries with not disturbing the stoichiometry of LSMCO phase. The resistivity measurement shows that metal insulator transition (T_{MI}) for both the samples. For the sample with additional micro Al_2O_3 content, T_{MI} shifts towards lower temperature. The resistivity ($T<T_{MI}$) data gives an excellent quantitative fit to $\rho(T) = \rho_0 + \rho_2 T^2 + \rho_{4.5} T^{4.5}$.

FIGURES

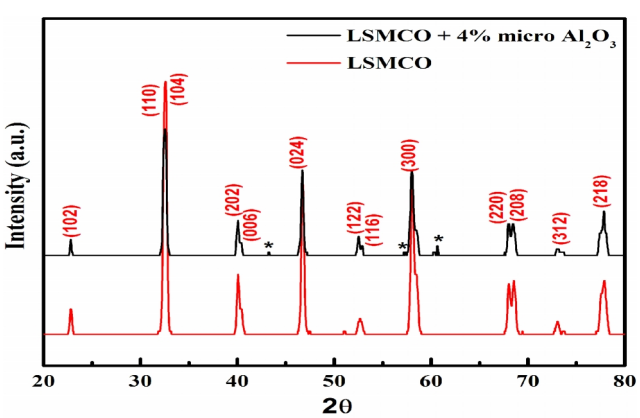


Fig. 1. XRD patterns of LSMCO/x micro Al_2O_3 composite samples with x = 0% & 4%.

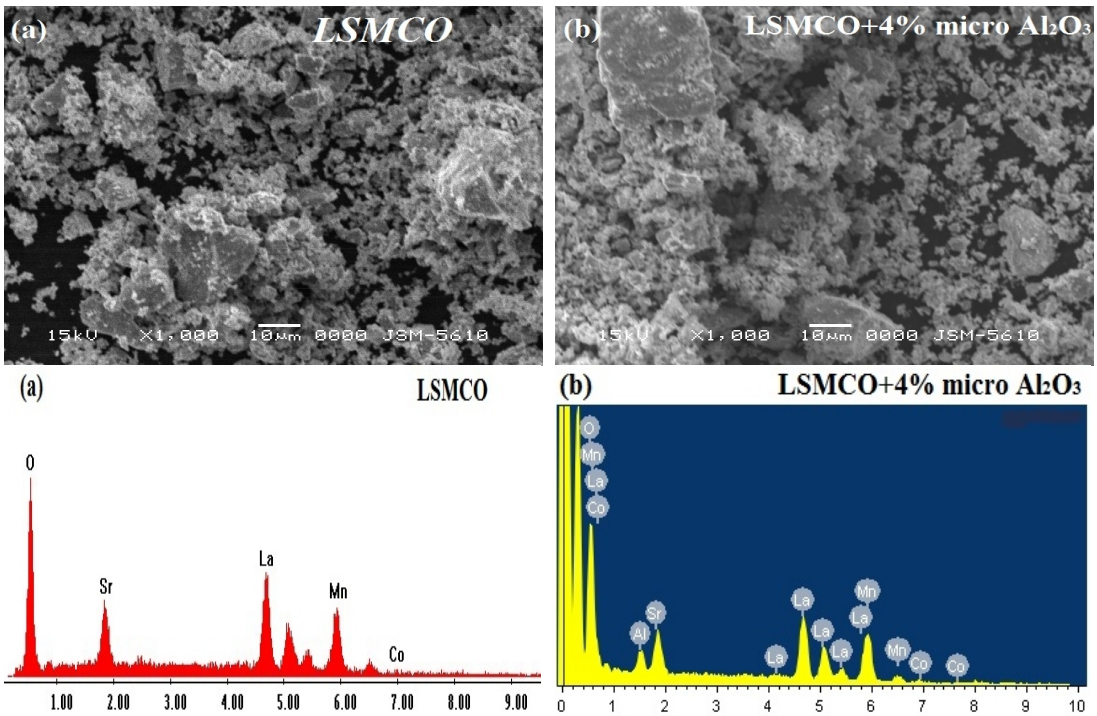


Fig. 2. SEM and EDX spectra of LSMCO/x micro Al_2O_3 composite samples with x = 0% & 4%.

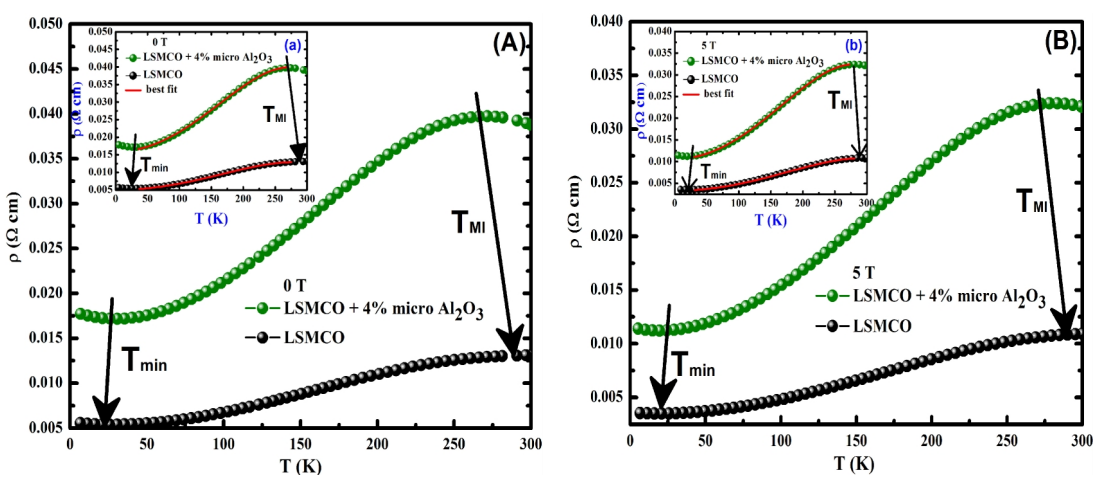


Fig. 3. Temperature (T) dependence of resistivity (ρ) for LSMCO/x micro Al_2O_3 composite samples with x = 0% & 4%. (A) H = 0T and (B) H = 5T. Inset (a & b) show fitting of data using equation-(1) under 0T and 5T magnetic field at low temperature ($T<T_{MI}$).

TABLES

Table 1. Values of all coefficients in equation-1, T_{min} and T_{MI}

Parameters	LSMCO		LSMCO+4% micro Al_2O_3	
	0 T	**5 T**	**0 T**	**5 T**
ρ_0 (Ω cm)	0.00503	0.0033	0.01588	0.01059
ρ_2 (Ω cmK^{-2})	1.83E-07	1.58E-07	5.87E-07	5.07E-07
$\rho_{4.5}$ (Ω cmK$^{-4.5}$)	-6.37E-14	-4.90E-14	-2.15E-13	-1.74E-13
R^2	0.99974	0.99991	0.99962	0.99992
T_{min} (K)	24.57	22.20	29.33	27.03
T_{MI} (K)	290.701	292.503	268.500	272.104

ACKNOWLEDGEMENT

Authors are thankful to UGC-DAE Consortium for Scientific Research, Indore for providing some experimental facilities. Help from Dr. R. Rawat, UGC-DAE CSR, Indore is thankfully acknowledged.

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Effect of physiological conditions on the production of Indole acetic acid produced by plant growth promoting rhizobacterial strains

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ABSTRACT

Plant Growth Promoting Rhizobacteria are the prominent source to bypass the use of Polluting chemical fertilizers, for that this study focuses on the characterization of plant hormones produced by the selected bacterial isolates. IAA (Indole3-acetic acid) producing ability of isolates were checked at varying pH (4-8), different incubation time (day1-6), varying temperature (30-45 c), with presence of tryptophan. All the test isolate had produced IAA at stressed conditions and all the above parameters were checked for Gibberellin production also. None of Bacterial isolate were showed haemolysis on Blood Agar plate and all the test isolate were tolerated UV light exposure and heavy metal concentration and showed Plant Growth Promoting Phenotypes evaluated by seed germination assay, these isolate may be useful to cultivate crops facing Physiological stress conditions.

SUMMARY

Characterization of plant hormones produced by the selected bacterial isolates.

Keywords: *Indole3-acetic acid, Plant Growth Promoting Rhizobacteria, Heavy Metal, UV light.*

INTRODUCTION

PGPR are free living soil born bacteria which when applied to plant promotes the growth of plant by direct and indirect mechanism. The Direct Promotion of plant growth by PGPR entails either providing the plant with a compound that is synthesized by the bacterium, for example phytohormones, or facilitating the uptake of certain nutrients from the environment. The indirect promotion of plant growth occurs when PGPR prevent the deleterious effects of one or more phytopathogenic organisms. This can happen by inducing resistance to plant pathogens.

MATERIALS AND METHODS

Microbial strains:

Bacterial isolates were previously isolated by the Bhatt PV et al 2014, were grown in the Nutrient broth for the 24 h at 30°C on a rotary shaker (120 rpm), the culture broth were centrifuged at 10,000 rpm for 15 mins and washed with phosphate buffer and resuspended pallets were used as the microbial inoculants for the all experiments.

Indole 3- acetic acid optimization:

The bacterial isolates were grown in the NBRIP broth at varying incubation period (1-6 day), different pH (4-8), different temperature (30-45 °C) and in presence of various carbon source as a sole source of energy. The growth of bacterial strains were measured in terms of turbidity as compared with controls. IAA produced were measured by the method developed by Solon et al 1950, briefly Grown cultures of bacterial isolates were centrifuged and the supernatant was mixed with 70 µl of the ortho phosphoric acid and 2 ml of Salkowaski's reagent allowed to incubate in dark for an hour and A_{535} was recorded. The value is compared with the standard plot prepared from the known concentration of IAA.

Determination of gibberellic acid:

The culture supernatant was centrifuged at 10,000 rpm for 15 mins and acidified with 5N HCl, Extracted gibberellic acid mixed with equal volume of ethyl acetate and evaporated. The precipitated content was measured spectrophotometrically and compared with the standard curve of gibberellic acid.
Rotary shaker is of Today-Tech SCIENTIFIC SOLUTIONS Company.

Pathogenicity Testing:

Test isolates were spotted over the blood agar and incubated at 30°C for the 72 h and observed for the zone of haemolysis, No zone of inhibition indicates microbes are not degrading RBCs.

UV light tolerance of bacterial isolates:

The known numbers of microbes were exposed to the UV light and after the intervals of 15,30,45 and 60 min, number of microbes were evaluated with the total plate count method.

Heavy metal tolerance of bacterial isolates:

Isolates were grown with N-broth in the presence of the heavy metals like (Co, Cr, Cu, Hg, Pb and Zn). And growth of microbes were evaluated after incubation period.

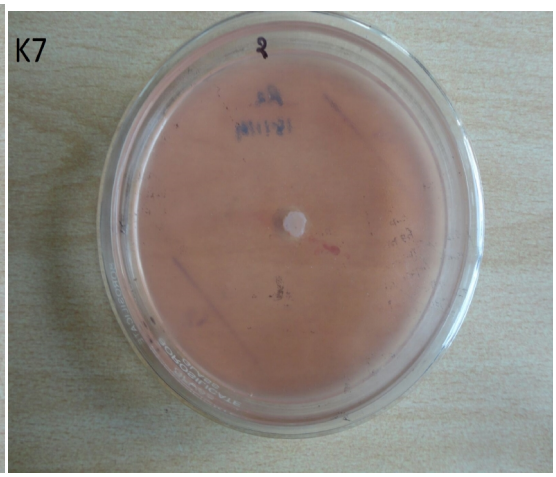
DISCUSSION

PGPR are commonly used to improve the crop yield, however the need is to explore the indigenous microbial strains for the optimized plant growth promoting activity. This study focuses on the characterization of biopolymer produced by the PGPR isolate having multiple plant growth promoting activities such a bacterial strains were previously isolated and identified. It has been observed that isolates produced IAA in more quantity at day 3 as compare to other incubation times. All the bacterial isolated produced IAA at varying pH ranges but isolate K7 produced IAA at pH 8 as compare with other isolates. At temperature 30-45 °C isolates survived and produced IAA may be exploited as bio-inoculants where temperature is not constant. Isolates were tested for their Pathogenicity and did not showed zone of lysis on the blood agar plates. Also all the isolates were tolerated varying amount of heavy metal concentration and showed plant growth promotion in seed germination assay by improving plant growth morphological parameters.

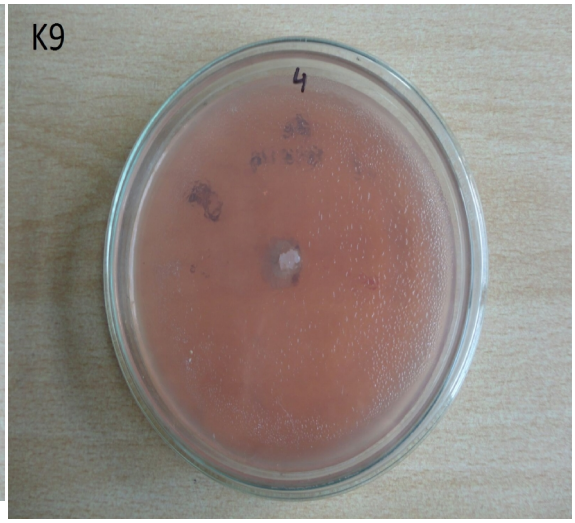
FIGURES

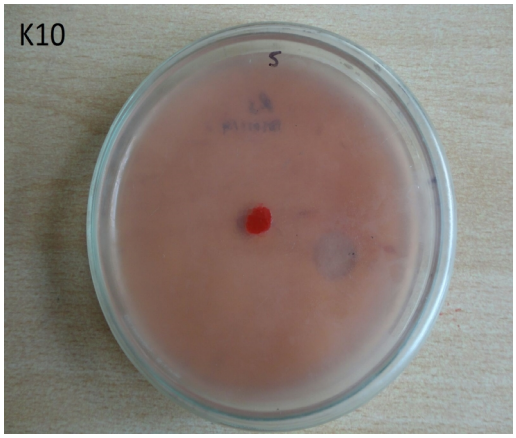
Pathogenicity test of PGPR on Blood Agar


K6
1

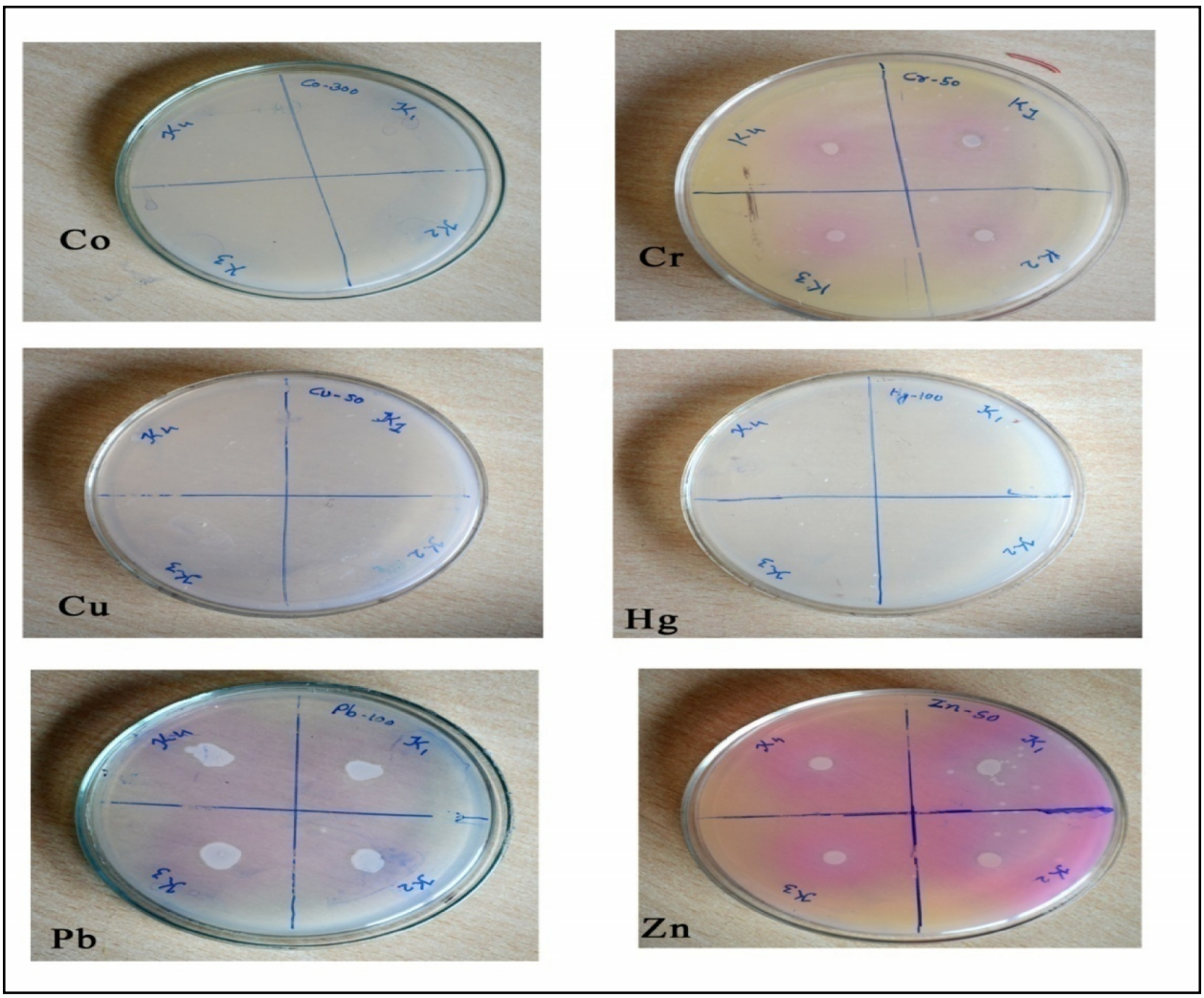

K7
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K8
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K9
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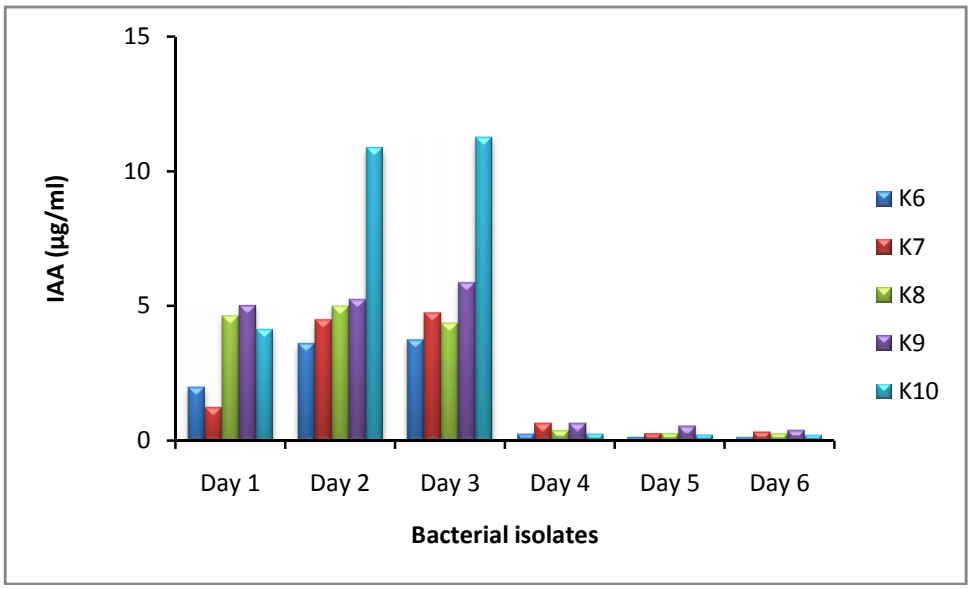

K10
5

Phosphate Solubilzation zone under heavy metals stress condition

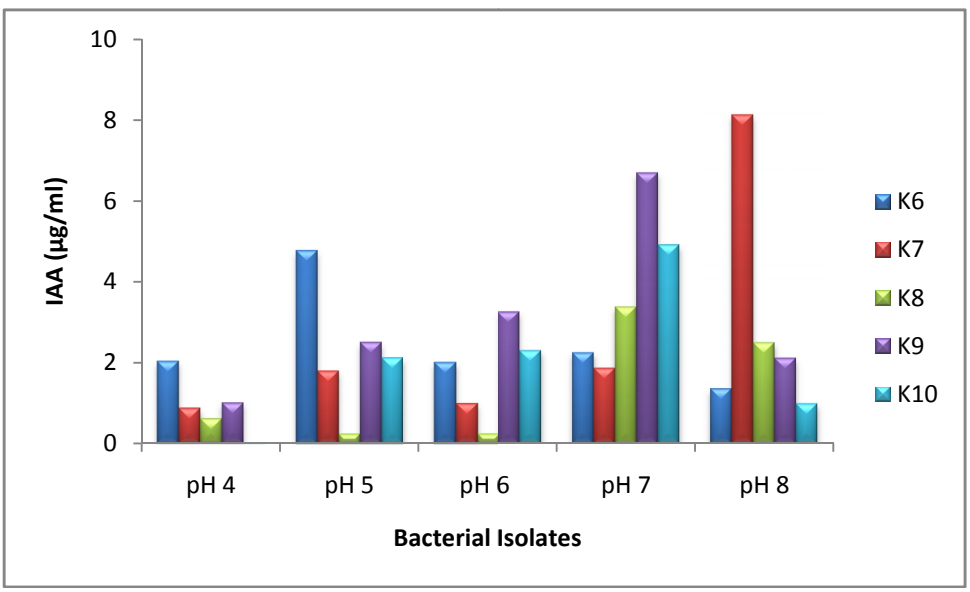


TABLES

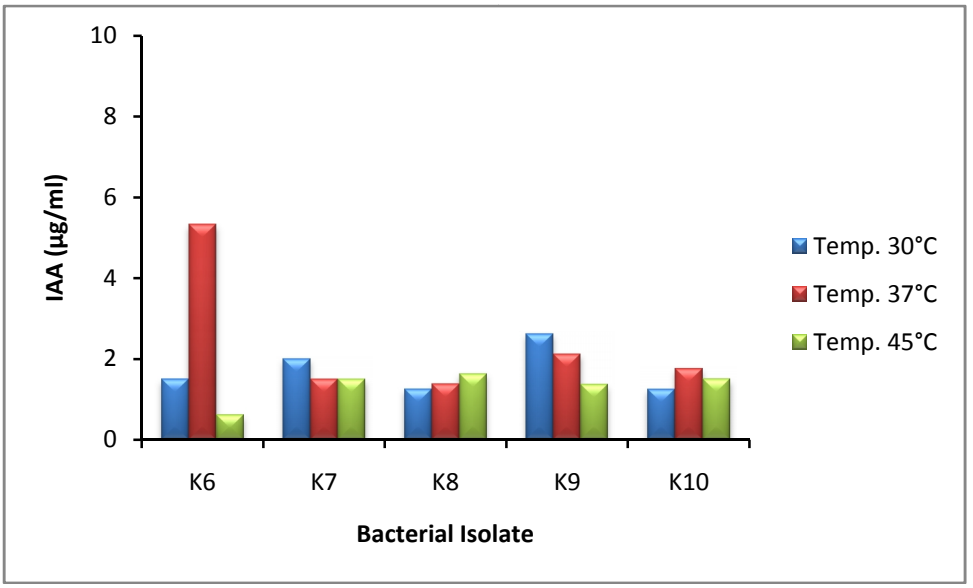
IAA Produced in NBRIP Media in the presence of Tryptophan.



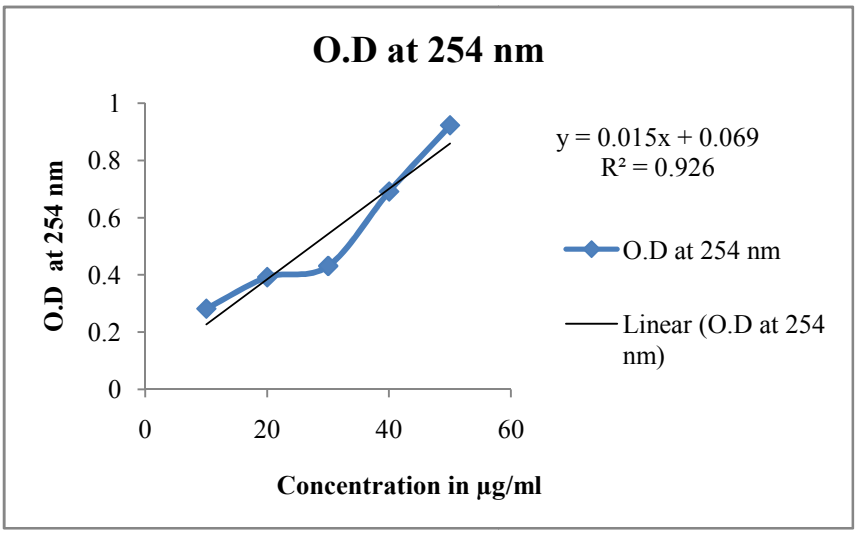
Effect of Different pH in Auxin Production by NBRIP Medium:



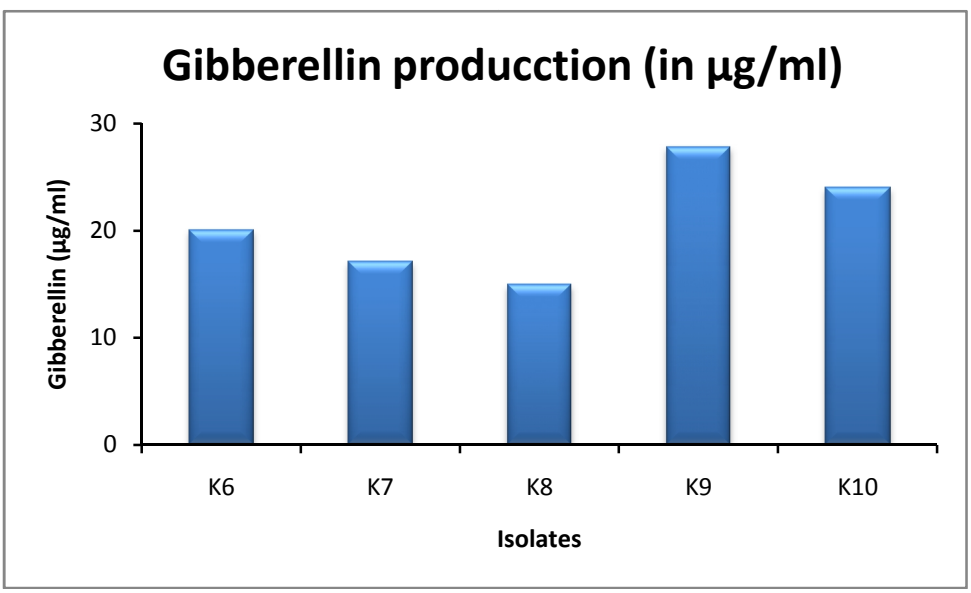
Effect of Different temperature in Auxin Production by NBRIP Medium:



Gibberellin Production:



Giberellin Produced in Nutrient broth medium:



ACKNOWLEDGEMENT

Authors are acknowledged to RK University for providing all the experimental facilities and instruments.

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Ferroelectric and Ferromagnetic Study of $BiFe_{0.95}Co_{0.05}O_3$ Powder Synthesized at lower Sintering Temperature

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ABSTRACT

Multiferroics are very interesting because of their potential to show various ferroic properties simultaneously. Bismuth ferrite ($BiFeO_3$) is a multiferroic material which shows ferromagnetism and ferroelectricity simultaneously in a single phase. $BiFeO_3$ (BFO) shows these properties at room temperature but because of spin canting its ferromagnetic component is negligible. Literature study shows that to enhance the ferromagnetic component in BFO, transition-metal element such as Co can be substituted at Fe. In the present work Co doped $BiFeO_3$ was prepared using sol-gel method. To study the crystal structure, X-Ray Diffraction (XRD) was used. The analysis of X-Ray diffraction data was done using Rietveld Refignment which reveals that synthesized material has Rhombohedraly distorted Perovskite structure with space group R3c. FTIR data confirms the absence of organic impurities. TG measurements of precursor showed the various weight loss regions corresponding to the removal of starting materials. Ferromagnetic and ferroelectric natureswere confirmed through the magnetic and electrical analysis.

SUMMARY

Present paper is mainly focused on synthesis and characterization of cobalt doped BFO.

Keywords: Multiferroic, Bismuth Ferrite, $BiFe_{0.95}Co_{0.05}O_3$, Sol-gel

INTRODUCTION

Multiferroic is now concept of greater interest because of their unusual nature of showing ferromagnetic and ferroelectric properties simultaneously in a single phase. Using the concept of magnetoelectric coupling multiferroic materials can be used for possible applications in spintronic, data storage and microelectronic device and the opportunity of magnetization (M) controlled by an applied electric field (E) or vice versa. Bismuth ferrite ($BiFeO_3$) is one of the member of multiferroic materials which simultaneously shows spontaneous polarization and spontaneous magnetization with ferroelectric Curie temperature (T_C)~ 820 °C - 830 °C and antiferromagnetic Neel temperature a (T_N) = 370 °C. Single phase $BiFeO_3$ (BFO) is little bit difficult to synthesize because of very thin temperature consistency range of the phase. Formation of impurity phases during preparation is the main drawback with $BiFeO_3$ system if synthesized by solid state reaction (SSR) method. Bulk $BiFeO_3$ synthesized through SSR, exhibits weak antiferromagnetic properties at room temperature. $BiFeO_3$ shows anti-ferromagnetic nature with a G-type magnetic structure (*1*), but because of the presence of canted spin structure it shows residual magnetic moment which leads to weak ferromagnetism. However, by replacing Fe by transition metal elements like Co and Mn, it is possible to develop spontaneous magnetic moment in the solid solutions, which transform antiferromagnetic material into ferromagnetic. There are many reports on the doping of transition metal ions (Co, Ru, Cr, Ti, Mn, Zn etc.) at Fe-sites in BFO and manganites with the enhanced magnetoelectric and magnetoresistive properties (*2-5*). Literature survey shows that the $BiFeO_3$ is mostly prepared by physical methods. Nevertheless, chemical methods like sol-gel or wet chemical methods are relatively cheaper as compared to physical methods (*1*). In the present work, we have synthesized Cobalt doped $BiFeO_3$ with doping concentration of 5% ($BiFe_{0.95}Co_{0.05}O_3$) using sol-gel technique.

MATERIALS AND METHODS

All the solvents used in the synthesis process were highly pure (>98% purity). $BiFe_{0.95}Co_{0.05}O_3$ (BFCO) powder was prepared by sol-gel technique. The precursor solution was prepared by mixing appropriate amounts of $(CH_3COO)Bi$, $(CH_3COO)Co$ and $(CH_3COO)Fe$ (molar ratio of Bi, Fe and Co = 1:0.95:0.05) in water + acetic acid solution under constant magnetic stirring. The mixture was stirred for 4 hours continuously at 80 ºC temperature to make a gel form of the mixture. Then, the gel was dried at 130 ºC and grinded into powder. After that, pallets were prepared from the powder and sintered for 6 h in air atmosphere at 750 ºC.

The crystallinity and phase analysis of the synthesized sample was determined by the XRD. XRD was performed with a diffractometer using Cu-Kα (λ = 0.154056 nm) radiation at 40 kV and 30 mA. The data were obtained between 20° to 80° (2θ) by step scanning of 0.02°. To study the thermal decomposition behavior of dried powder, TG analysis was carried out under static air atmosphere at rate of 15 °C/min from 24 to 900 °C. The infrared spectrum was recorded using Thermo Scientific Nicolat 6700 Spectrometer with the scanning range; 400 to 4000 cm^{-1}. Magnetization was measured by changing magnetic field (M-H) using Vibrating Sample Magnetometer (Lakeshore VSM 7410at S.A.I.F., IIT Madras) up to the applied field of 15000 Oe at room temperature. P-E loop data were obtained with P-E loop tracer up to the applied electric field 14 kV/cm.

RESULTS AND DISCUSSION

As shown in Fig. 1, TGA of the precursor demonstrates that the precursor powder is completely decomposed at around 700 ºC when heated with the rate of 15 ºC/min. The major weight loss between temperature 150 and 300 ºC was observed because of the breakdown of gel network and ignitions of most of the carbon-based materials. A small weight loss was observed in the graph between 300 and 400 ºC which is due to the release of carbon dioxide. Weight loss was observed in the temperature range from

500 to 700 ºC correspond to the phase-crystallization step. The precursor/crystal has been found not to melt up to temperature 900 °C. Fig. 2 shows the infrared spectrum of thermally treated powder. Peaks observed in the region between 700 to 400 $cm^{-1}$are due to metal oxides bonds. The presence of the peak at 560 cm^{-1} leads to the presence of stretching vibrations along the Fe-O axis and 440 cm^{-1} peak leads to the presence of the Fe-O bending vibration.

Analysis of the X-Ray powder diffraction pattern for $BiFe_{0.95}Co_{0.05}O_3$ was done by Rietveld refinement program "Full-Prof". Refined XRD patterns of $BiFe_{0.95}Co_{0.05}O_3$ is shown in Fig.3. The diffraction peaks observed for the samples were strong and sharp, suggesting good crystallization of the samples. A structural model which is closer to the actual structure is initial requirement for the Rietveld refinement. Bismuth Ferrite shows rhombohedral structure with space group R3c. Hence, space group R3c was used for Rietveld refinement of the XRD pattern of the doped sample. Pseudo-Voigt function was used to describe the peak shapes and cosine Fourier series was used to model the background of the XRD pattern. The peak profile parameters including the peak symmetry were refined. The peak positions were achieved after a good match. Fig. 3 shows the observed, calculated, and difference in X-ray diffraction pattern obtained from the refinement. Agreement between the observed and refined XRD pattern was confirmed. The parameters viz. half width profile parameters (u, v, w), isotropic thermal parameters, scale factor, lattice parameters, background parameters and atomic functional positions were refined. Bond angles & bond lengths obtained from the refinement, Brag R-factor, refined structural parameters and the goodness of fit χ^2 are reported in Table 1. The formation of $BiFe_{0.95}Co_{0.05}O_3$ phase and the formation of $Bi_{25}FeO_{39}$ impurity phases were observed with ratio of 0.77:0.23. Noticeable amount of impurity phase was observed which is not removed even after increasing calcination time and which lead to more volatization of Bi_2O_3. The Fe-O bond length is associated with the buckling of the octahedron and the Fe-O-Fe bond angle is an extent of the tilting of the octahedron. The angles of Fe/Co-O-Fe/Co differ from the ideal value for perovskite (180^o). Co doping is suggested to reduce the grain size of BFCO ceramics, which can be interpreted as a result of the suppression of oxygen vacancies due to charge compensation mechanism, resulting in slower oxygen ion motion and a consequently lower grain growth rate. Debye Scherrer equation $D=K\lambda/\beta\cos\theta$ was used to calculate the average crystallite size from XRD peak broadening, where K (shape factor) = 0.89, β is FWHM, λ is X-ray wavelength used to obtain diffraction pattern and θ is the Bragg angle. The value was found to be 32 nm for BFCO sample.

Fig. 4 shows the P-E hysteresis loop of Cobalt doped $BiFeO_3$ ceramic at room temperature. The hysteresis loop is lossy and exhibits an unsaturated behavior. The P-E loop could not achieve saturation polarization manifest a conducting behavior of ceramics due to leakage current and presence of impurity. However, a Coercive Field (E_c) of 12.09 kV cm^{-1} and Remanent Polarization (P_r) of 0.29 μC cm^{-2} were observed in case of the $BiFe_{0.95}Co_{0.05}O_3$ sample.

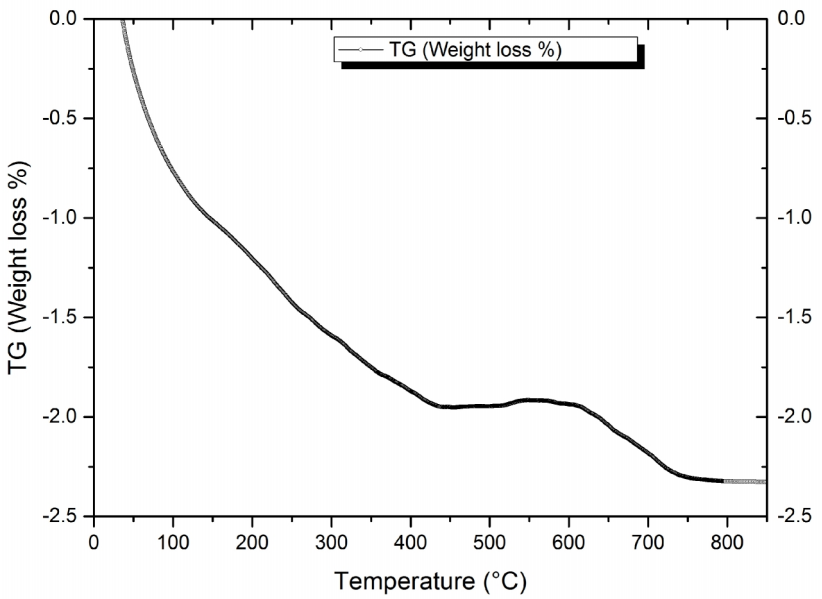
Fig. 5 shows the magnetization hysteresis (M–H) loop of Cobalt doped $BiFeO_3$ sample. The measurement was carried out on the sample at room temperature. Pure BFO behaves as antiferromagnetic (*1*). M-H data clearly states that synthesized Cobalt doped samples shows weak ferromagnetic behavior. A saturated magnetization of 67.24 mile emu and remanent magnetization of 23.64 mile emu were observed at 300 K. Enhancement in the magnetic property of Cobalt doped BFO was observed compare to the undoped BiFe0.95Co0.05O3 (*1*). Either the canting of the anti-ferromagnetically ordered spins (by a structural distortion) or breakdown of the equilibrium between the anti-parallel sub-lattice magnetization of Fe^{3+} (due to ion substitution with a different valence) appears reason behindthe appearance of ferromagnetism in solid solution systems (*6*). Structural distortion due to the difference in ionic radii of Fe^{3+} and Co^{3+} may contribute to the higher magnetization in the Co substituted BFO (*7*).

CONCLUSION

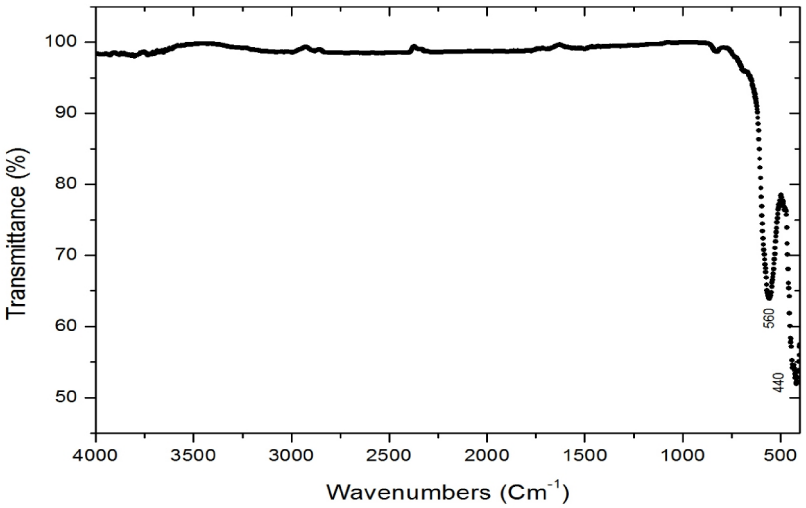
We have successfully synthesized $BiFe_{0.95}Fe_{0.05}O_3$ powder at the sintering temperature of 750 °C using sol-gel technique. The Rietveld refinement of XRD data confirms that sol-gel technique produces

perovskite $BiFe_{0.95}Co_{0.05}O_3$ oxides with noticeable amount of impurity phase $Bi_{25}FeO_{39}$ at sintering temperature of 750 °C. Presence of impurity phase has been observed due to lower sintering temperature during synthesis process. The synthesized BFCO has nearly single perovskite phase with a ratio of Bismuth, Iron and Cobalt close to 1:0.95:0.05 and smaller crystallite size (32 nm). FTIR analysis proves the absence of the organic impurities and presence of Fe-O and Bi-O bonds in the sample. VSM analysis (M-H Loop) confirms the weak ferromagnetic nature of the BFCO powder. A ferroelectric measurement proves weak ferroelectric behavior of the synthesized BFCO sample.

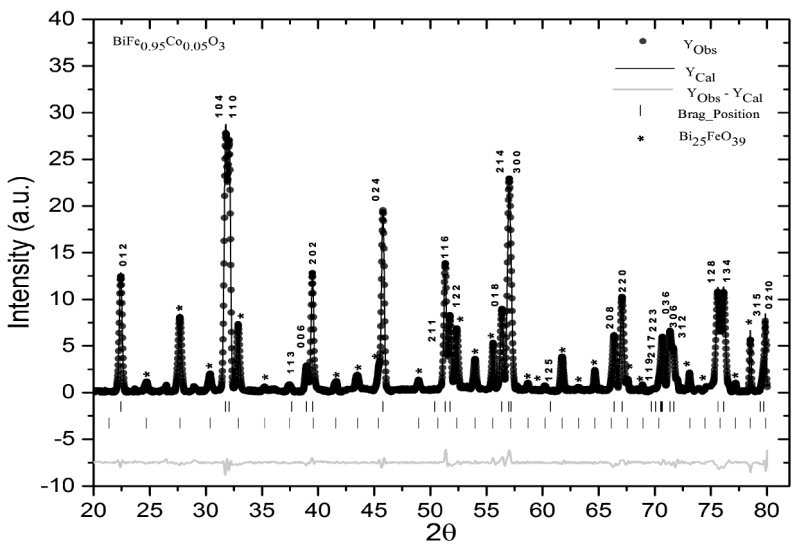
FIGURES



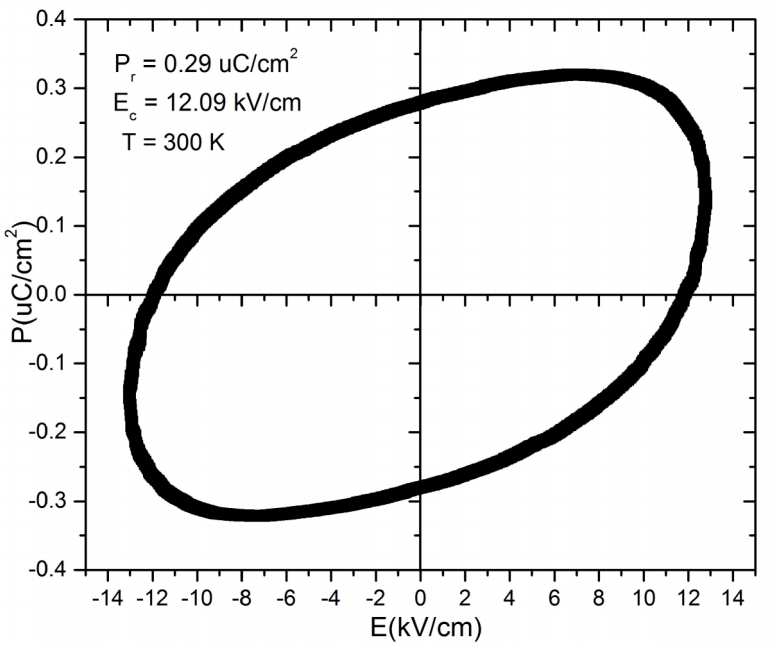
"**Fig.1**: TGA Curve for $BiFe_{0.95}Co_{0.05}O_3$."



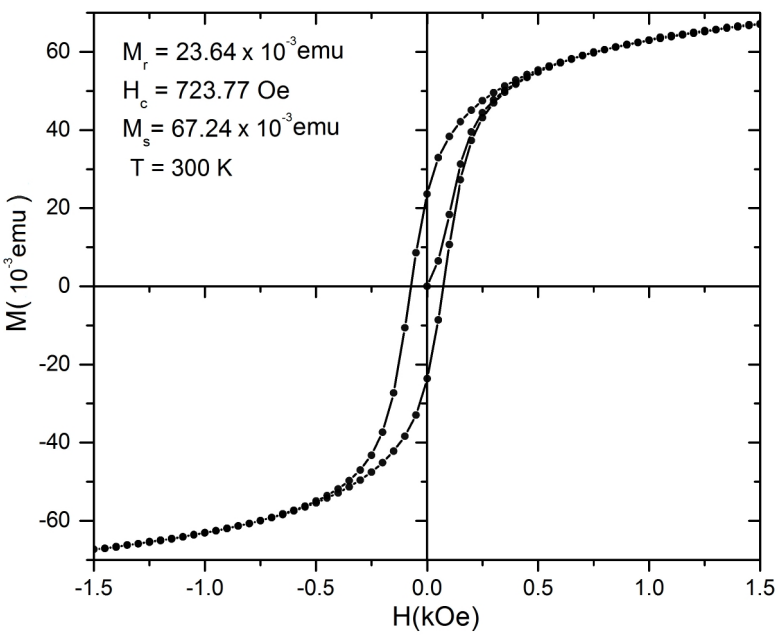
"**Fig.2**: FTIR Spectrum of $BiFe_{0.95}Co_{0.05}O_3$."



"**Fig.3**: Rietveldrefined XRD pattern of the $BiFe_{0.95}Co_{0.05}O_3$."



"**Fig.4**: P-E Loop of $BiFe_{0.95}Co_{0.05}O_3$."



"**Fig.5**: M-H curve of $BiFe_{0.95}Co_{0.05}O_3$."

TABLES

Table 1: Lattice parameters, R-factors and Interatomic angle of both phase after Refignment.

	$BiFe_{0.95}Co_{0.05}O_3$	$Bi_{25}FeO_{39}$
Lattice parameters		
Lattice parameter a (Å)	5.57589	10.1841
Lattice parameter b (Å)	5.57589	10.1841
Lattice parameter c (Å)	13.8590	10.1841
Lattice parameter α (^{o})	90	90
Lattice parameter β (^{o})	90	90
Lattice parameter γ (^{o})	120	90
Cell Volume	373.156	1056.253
Fract (%)	77.28	22.72
R-factors (%)		
Bragg R-factor	4.41	6.83
χ^2 (Goodness of fit)	0.48	0.48
Interatomic angle (^{o})		
Fe/Co-O-Fe/Co	169.2958(0)	-

ACKNOWLEDGEMENT

Authors from Department of Physics, Saurashtra University are thankful to the departmental UGC DRS-SAP & DST-FIST programmes for providing basic facilities.

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Structural, Transport and Magnetoresistive Properties of Ti-Doped $La_{0.7}Sr_{0.3}Mn_{1-x}Ti_xO_3$ Manganites

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ABSTRACT

In the present study polycrystalline $La_{0.7}Sr_{0.3}Mn_{1-x}Ti_xO_3$ (x = 0.00 and 0.09) manganite samples were synthesized using auto-combustion technique. The Rietveld refinement confirms that both the samples are in single phase with rhombohedral structure in hexagonal lattice having a space group $R\bar{3}c$. Vibrational assignments of samples were examined by Fourier transform infrared spectroscopy. Thermogravimetric analysis for the $Ti_{0.09}$ sample confirms that the precursor does not melt up to 1050 °C. The energy dispersive spectroscopy data indicates that the observed concentration of elements is very close to the calculated values from its chemical formula. Scanning electron microscopy shows that each sample has fine and clear grain boundaries (GBs). The electrical resistivity was measured by standard four probe resistance technique which shows that the applied field suppresses the resistivity peak at T_{MI} significantly. The maximum MR % was observed around 89 % for $Ti_{0.09}$ at 5 T.

SUMMARY

This paper correspond to the synthesis of $La_{0.7}Sr_{0.3}Mn_{1-x}Ti_xO_3$ (x = 0.00 and 0.09) and their structural, morfological and magnetic behaviour.

Keywords: CMR, Manganite, Transport Properties, Elctrical Conductivity

INTRODUCTION

In current years, one of the most concentrated studied materials are hole doped rare-earth manganites because of their related physics and potential applications. The ability of an external magnetic field H to alter the electrical resistivity ρ of a sample has long been known and the term magnetoresistance (MR) has been adopted to quantify the relative change in the resistivity. Attempts to increase the size of the MR effect have largely been driven by the needs of the technology industry to keep up with the demand for increased magnetic storage density. When an external magnetic field is applied to a material, there is change in the value of its electrical resistance that is called magnetoresistive behaviour of the material. Without doping $LaMnO_3$ is an insulator with antiferromagnetic order. If rare-earth element is partially replaced by alkaline earth metals like Ba, Sr or Ca, it results in conversion of an appropriate number of Mn^{+3} to Mn^{+4}. This gives rise to ferromagnetic Mn^{+3}-O-Mn^{+4} double exchange interactions. In this interaction the e_g electron from Mn^{+3} is transferred to Mn^{+4} with a parallel spin configuration (*1*).

In order to distinguish the effect of Ti^{+4} occupying both Mn^{+3} and Mn^{+4} sites two sets of compounds having compositions, $La_{0.7}Sr_{0.3}Mn_{1-x}Ti_xO_3$ with x = 0.00 and 0.09 were prepared using auto-combustion technique. Ti doped CMR manganite put forward such an opportunity. Ionic radius of Ti^{+4} ions is known to be in between the ionic radii of Mn^{+4} and Mn^{+3}. Therefore there exists a distinct possibility that some Ti^{+4} ions can occupy Mn^{+3} sites in addition to the Mn^{+4} sites. The existing literature signifies that Ti^{+4} ions also occupy the isovalentMn sites in these kinds of materials (*2*). Current information has revealed that it has a preference to occupy Mn^{+4} sites. However, in case of higher doping levels it moreover occupies Mn^{+3} sites.

Experimental

Polycrystalline $La_{0.7}Sr_{0.3}Mn_{1-x}Ti_xO_3$ (x = 0.00 and 0.09) (LSMTO) manganite samples were synthesized using auto-combustion technique (*3-5*). The high purity starting compounds of $La(CH_3CO_2)_3.H_2O$ (Lanthanum Acetate), $Sr(CH_3CO_2)_2$ (Strontium Acetate) and $Mn(CH_3CO_2)_2.4H_2O$ (Manganese Acetate) were taken in stoichiometric ratio. All the three compounds were breaked up into acetic acid and double distilled water having 1:1 volume ratio. To obtain 0.6 M solution of precursors, continuous stirring is required and to achieve a sol state of the material the precursor solution was dehydrated at 80 ^oC. Further heat treatment at slightly higher temperature ~150 ^oC for 4 hours resulted into the gel form through the gelation process. The gel was dried to obtain the black powder of LSMO mixture. High purity TiO_2 (nano powder) was added as per the concentration of Ti into mixture of LSMO and the whole mixture was ground. The Ti doped black powder was heated at 400 ^oC for 6 hours to obtain the black powder of LSMTO manganite. Further, the calcined black powder of LSMTO manganite was heated at 400 ^oC for 8 hours to obtain the well calcined black powder of LSMTO manganite. Finally the powder was pressed into pellets under 2 ton pressures for 4 minutes using the hydraulic press. Final product in the form of pellets was sintered at 1100 ^oC for 24 hours. The microstructure polycrystalline LSMTO manganiteshereafter will be referred, $La_{0.7}Sr_{0.3}MnO_3$ as a$Ti_{0.00}$ and $La_{0.7}Sr_{0.3}Mn_{0.91}Ti_{0.09}O_3$ as a $Ti_{0.09}$.

RESULTS AND DISCUSSION

The phase formation in $Ti_{0.00}$ and $Ti_{0.09}$ samples was studied by powder X-ray diffractometer (Philips Xpert MPD) with Cu-Kα radiation at room temperature. Both the samples were scanned in an invariable mode from 20^o – 80^o with 0.02 degree step size. The Rietveld analysis is a refinement method for powder diffraction patterns to study the crystallographic structures. The refinements of the XRD of the samples were made using FULLPROF program. Fig. 1 shows Rietveld refinement of XRD data for both the samples. XRD graphs confirm that both the samples are in single phase with no detectable secondary

phase or phases and the compounds in the entire substitution range have the structure of rhombohedral with $R\bar{3}c$ space group. Table 1 shows the achieved values of lattice parameters. With the substitution of Mn^{+4} by Ti^{+4}, the structure does not undergo any transformation, but there is an increase in lattice parameters with doping of Ti. As a and c cell parameters increase, cell volume also increases with Ti content. It can be associated to the bigger ionic radii of Ti^{+4} (r_{Ti}^{+4} = 0.605 Å) ion as compared to that of Mn^{+4} (r_{Mn}^{+4} = 0.540 Å) ion.

The FT-IR spectroscopy of both the samples was carried out in the range of 400-4000 cm^{-1} by Nicolet 6700 FT-IR. To check the influence of Ti doping on the J-T distortion, both the samples were measured by the infrared transmission spectrometer. For the undoped sample (shown in Fig. 2), three large absorption peaks were obviously observed at 418.57 cm^{-1}, 611.21 cm^{-1} and 2349.23 cm^{-1}. The peak at 418.57 cm^{-1} is recognized to Mn-O-Mn bending mode, the next peak is due to Mn-O stretching mode of vibration at 611.21 cm^{-1}, and the moisture contain at third peak at 2344.23 cm^{-1}. In IR transmission spectra of the $Ti_{0.09}$ sample only one additional peak is observed at 576.74 cm^{-1} due to Ti-O Stretching modes. These results prove the Ti substitution at Mn site (*6- 7*).

The Thermogravimetric analysis involves measurement of a change in weight of a sample as the temperature is increased at pre-determined rate (10^{o}C/min). The thermogravimetric analysis of Ti0.09 was carried out using PC Controlled LineseisSimulteneous Thermal Analyzer (STA) with 10 ^{o}C/min heating rate. TGA graph of the $Ti_{0.09}$ precursor is shown in Fig. 4. The weight loss profile of TGA could be divided into three phases. The first phase of the TGA graph, with the weight loss of about 8% to 9% of its original mass observed in the range of 40 ^{o}C to 120 ^{o}C, is because of the loss of water and low boiling organic materials. At this temperature rang the precursor was not dried enough and it might have absorbed water. A fast weight loss phase of about 11% to 12 % in the range of 120 ^{o}C to 380 ^{o}C in second phase, which is due to the decomposition of the connected coordination species, acetate and oxide components. The third stage of TGA shows a weight loss of about 18 % in the range of 380 ^{o}C to 620 ^{o}C which is attributed to the decomposition of ethylene glycol and at last above 700 ºC there is no mass change and it is the stable state of precursor. The precursor has been found not to melt up to temperature 1050 °C (*8- 9*).

Mapping of the distribution of the different chemical elements constituting the specimen can be obtained. For verifying the stoichiometric composition in $Ti_{0.09}$ sample (shown in Fig. 5), the EDS mesurments were carried out. A quantitative analysis of the energy-dispersive spectroscopy data specifies that the observed concentration of elements is very close to the calculated values from its chemical formula. Name of each element, observed and calculated values of weight % are tabulated in Table 2. These results of EDS patterns confirm that, there is no impurity element present in the sample (*10*). Fig. 6 & Fig. 7 depict the representative images elucidating the surface morphology of $Ti_{0.00}$ and $Ti_{0.09}$ micrographs obtained using Scanning Electron Microscopy (JEOL JSM-5610LV Scanning Electron Microscope). Typical SEM micrographs for both the samples show that each sample possesses fine and clear grain boundaries (GBs). Average grain size about 800 nm was observed in entire doping range. These results confirm that Ti doping at Mn site has no obvious effect on the grain size of the samples.

The electrical resistivity measurements of $Ti_{0.00}$ and $Ti_{0.09}$ samples were carried out using standard four probe resistance measurement technique with and without applied magnetic field. Fig. 8 & Fig. 9 show the temperature dependence of the resistivity for the $Ti_{0.00}$ and $Ti_{0.09}$ samples measured within a temperature range of 5 K to 300 K without magnetic field and at the external magnetic field of 1 T, 5 T and 8 T respectively. As shown in Fig. 8, $Ti_{0.00}$ exhibits metallic behaviour in the entire temperature range of 5 K to 300 K under a field of 0 T and 8 T. As shown in Fig. 9, the resistivity increases rapidly at low temperature (5 K) and it exhibits insulating behaviour throughout the measured temperature range of 5 K

to 300 K for all values of applied magnetic field in the case of $Ti_{0.09}$. An increase in resistivity and a decrease in metal to insulator transition temperature (T_{MI}) were observed by doping of Ti for Mn. It is ascribed to the replacement of some Mn^{+3}-O-Mn^{+4} bonds by the Mn^{+3}-O-Ti^{+4} bonds. An increase of the average (Mn,Ti)-O distance due to the higher radius of Ti, suppresses double exchange interaction between Mn^{+3}/Mn^{+4} accordingly decreases the ferromagnetic coupling between adjacent Mn ions (*11–14*). It is also found that replacement of Mn^{+3}/Mn^{+4} by Ti^{+4} slows down the transfer rate of electron through the Mn^{+3}-O^{-2}-Mn^{+4}.

For manganite material, MR is most important characteristic which decides the applications of material. Both of the $Ti_{0.00}$ and $Ti_{0.09}$ samples show the resistivity suppression under applied magnetic field of 1 T, 5 T and 8 T leading to a negative CMR effect. The temperature dependent magnetoresistance (MR) for the $Ti_{0.00}$ and $Ti_{0.09}$ samples measured within a temperature range of 5 K to 300 K in magnetic field of 1 T, 5 T and 8 T is shown in Fig. 10 and Fig. 11 respectively. Magnetoresistance (MR) was calculated by the formula:

$$MR\ \% = [(\rho_0 - \rho_H)/\rho_0] \times 100$$

Where, ρ_0 and ρ_H represent the resistivity without and applied magnetic field respectively. The maximum MR (89 %) is observed for $Ti_{0.09}$ at 5 T, which is comparatively larger than $Ti_{0.00}$. This result indicates that if we substitute Ti at Mn site up to 9 %, we can improve the room temperature magnetoresistance, which is important for application point of view. This result suggests that magnetoresistance behaviour is improved by addition of Ti at Mn site.

CONCLUSION

In the present study polycrystalline $La_{0.7}Sr_{0.3}Mn_{1-x}Ti_xO_3$ (x = 0.00 and 0.09) (LSMTO) manganite samples were synthesized using auto-combustion technique. XRD patterns show that both the samples are single phase with no detectable secondary phases and the compound in the shown substitution range have rhombohedral $R\bar{3}c$ space group. By doping of Ti the structure does not undergo any transformation, but there is an increase in lattice parameters with doping of Ti. In IR transmission spectra of the $Ti_{0.09}$ sample only one additional peak is observed at 576.74 cm^{-1} due to Ti-O Stretching modes. These results prove the Ti substitution at Mn site. The thermogravimetric analysis represents that there is no mass change in precursor above 700 ºC and it does not melt up to the temperature of 1050 °C. The result of EDS pattern confirms that, there is no impurity element present in the $Ti_{0.09}$ sample, because, the observed concentration of elements is very close to the calculated values from its chemical formula. Typical SEM micrographs for both samples show that, each sample possesses fine and clear grain boundaries (GBs) and found that the average grain size about 800 nm in present doping range. The resistivity increases rapidly at low temperature (5 K) and it exhibits insulating behaviour throughout the measured temperature range 5 K to 300 K for all values of applied magnetic field in the case of $Ti_{0.09}$. An increase in resistivity and a decrease in metal to insulator transition temperature (T_{MI}) were observed by doping of Ti for Mn. The maximum value of the temperature dependent magnetoresistance (MR) for $Ti_{0.09}$ was observed around 89 % for $Ti_{0.09}$ at 5 T, which is comparatively larger than $Ti_{0.00}$. This result indicates that magnetoresistance property is improved by substitution of Ti at Mn site in LSMO.

FIGURES

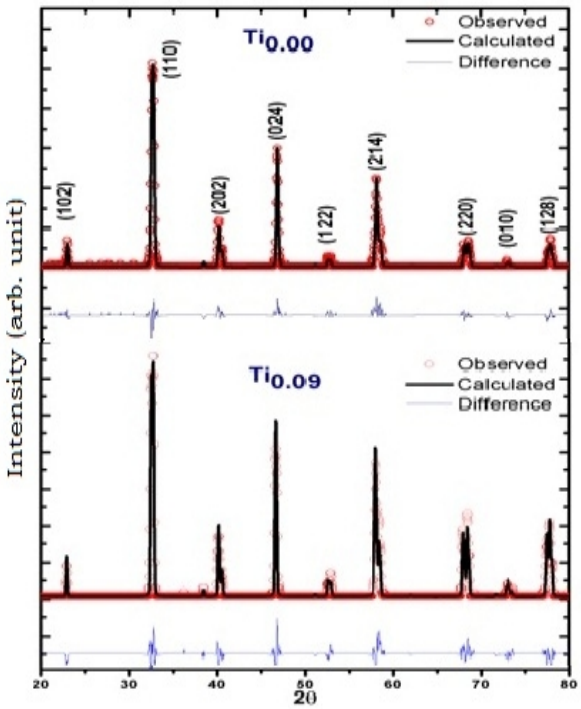


Fig. 1. Comparisation of Rietveld graph of Ti0.00 and Ti0.09.

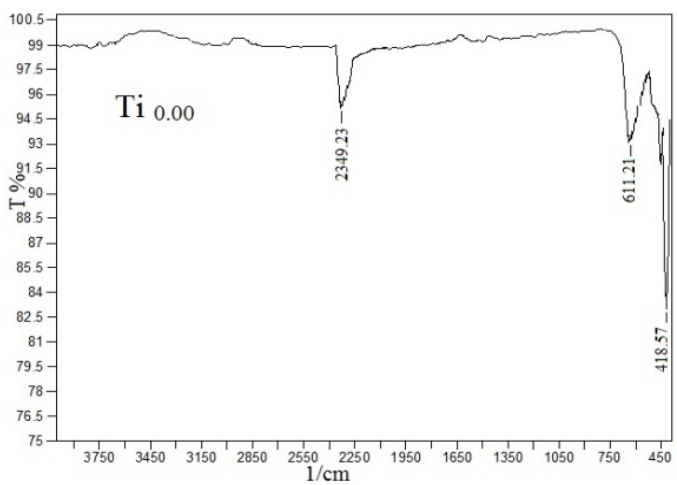


Fig. 2.Fourier Transform IR spectrum of $Ti_{0.00}$ sample.

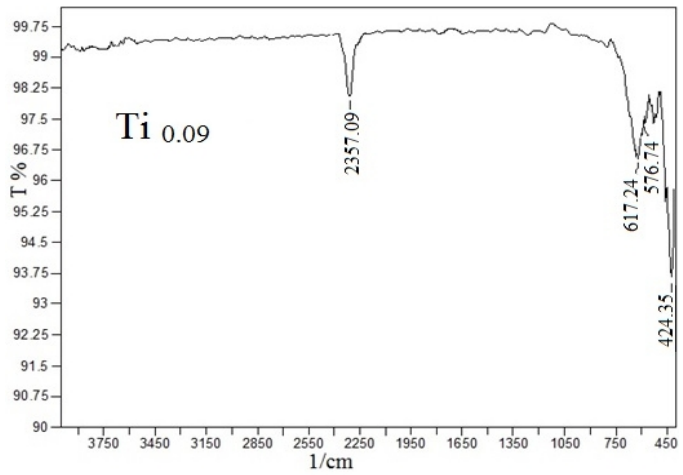


Fig. 3. Fourier Transform IR spectrum $Ti_{0.09}$ Sample.

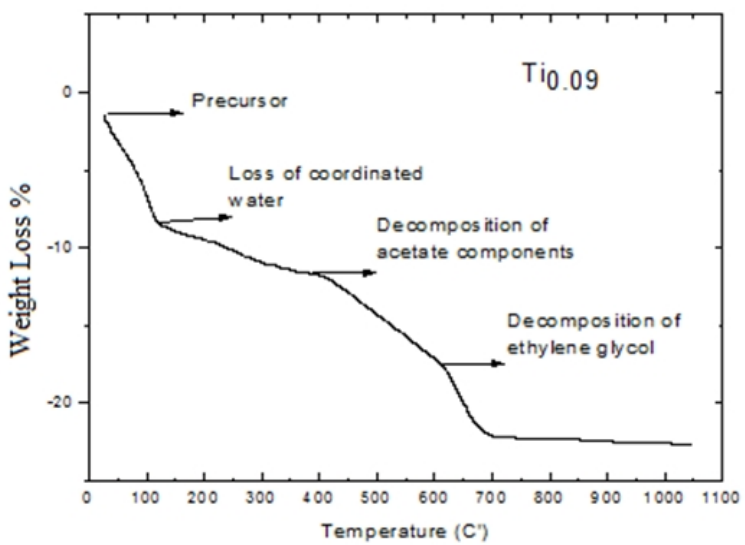


Fig. 4. The thermogram of $Ti_{0.09}$ (precursor).

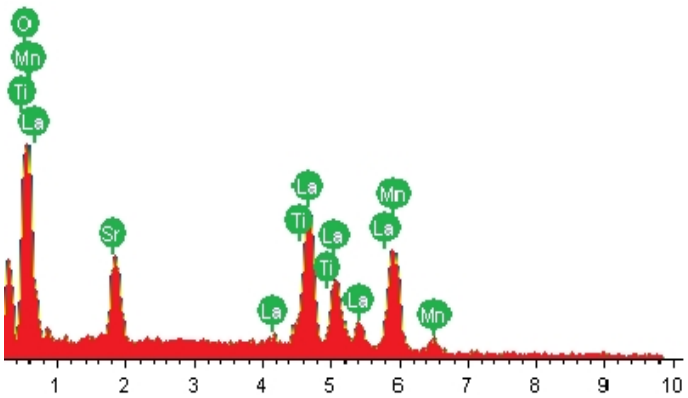


Fig. 5. EDS spectra of $Ti_{0.09}$.

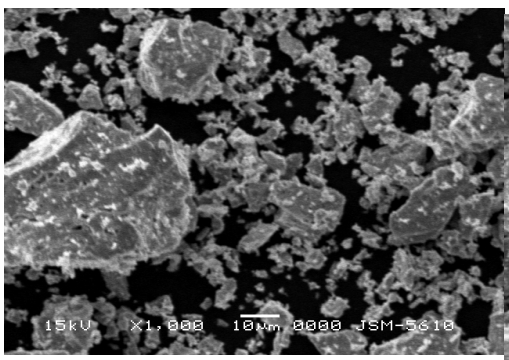


Fig. 6.SEM micrographs of $Ti_{0.00}$.

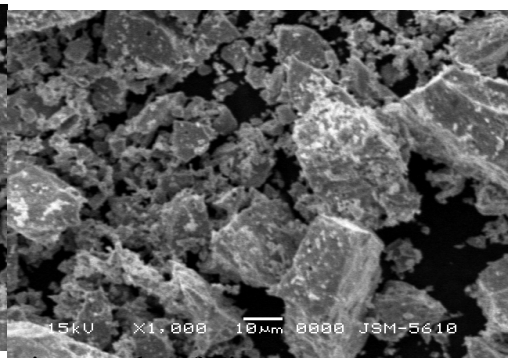


Fig. 7.SEM micrographs of $Ti_{0.09}$.

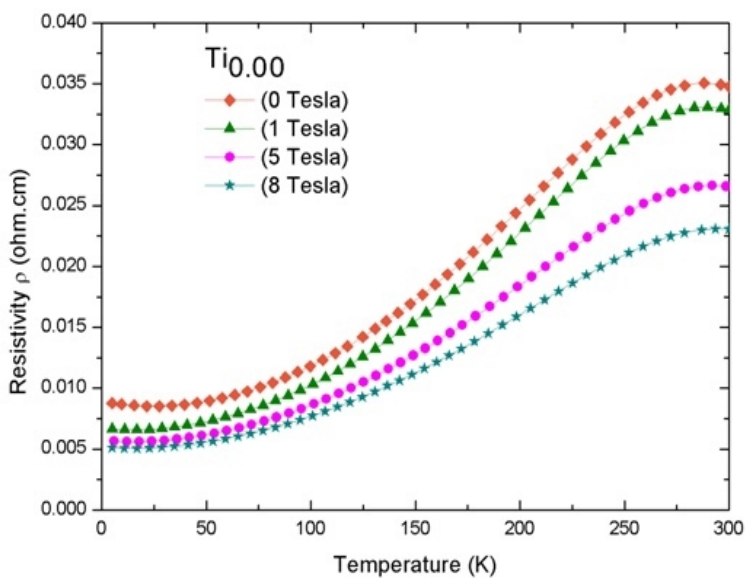


Fig. 8. Electrical resistivity as a function of temperature for $Ti_{0.00}$ at 0 T, 1T, 5 T and 8 T.

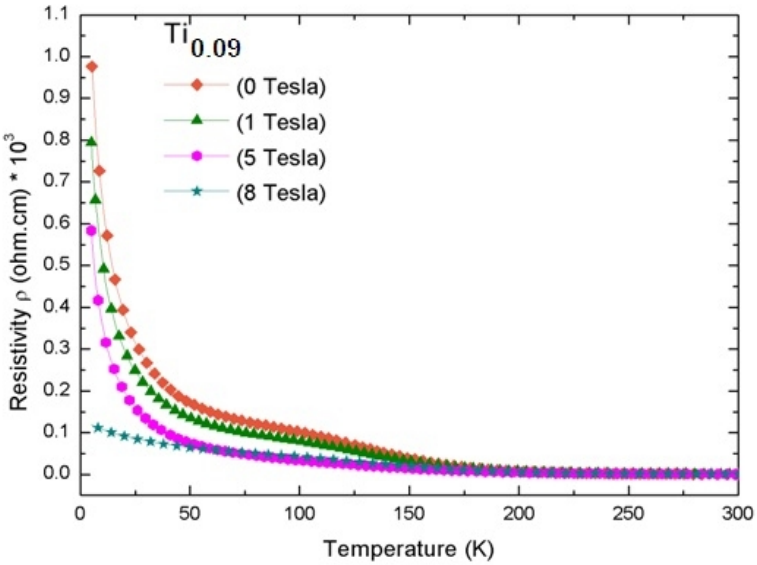


Fig. 9. Electrical resistivity as a function of temperature for $Ti_{0.09}$ at 0 T, 1 T, 5 T and 8 T.

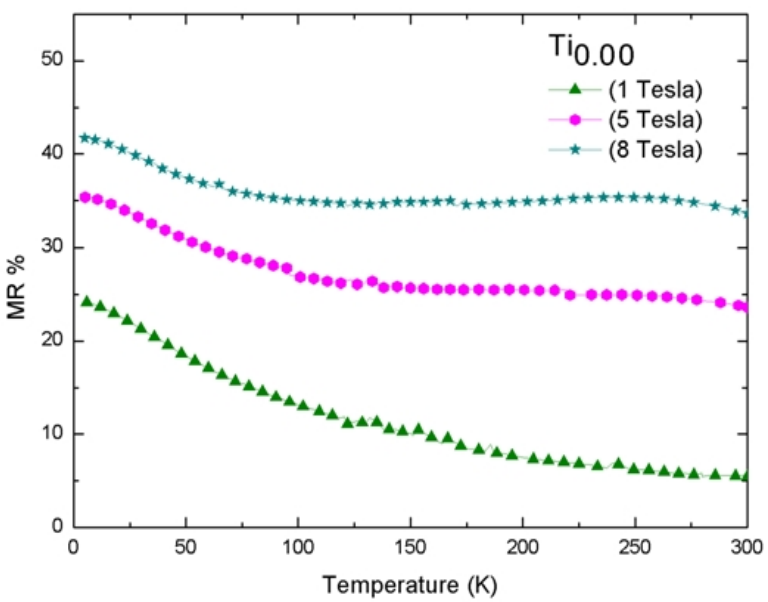


Fig. 10. Variation in MR % with temperature under 1 T, 5 T and 8 T applied magnetic field for $Ti_{0.00}$.

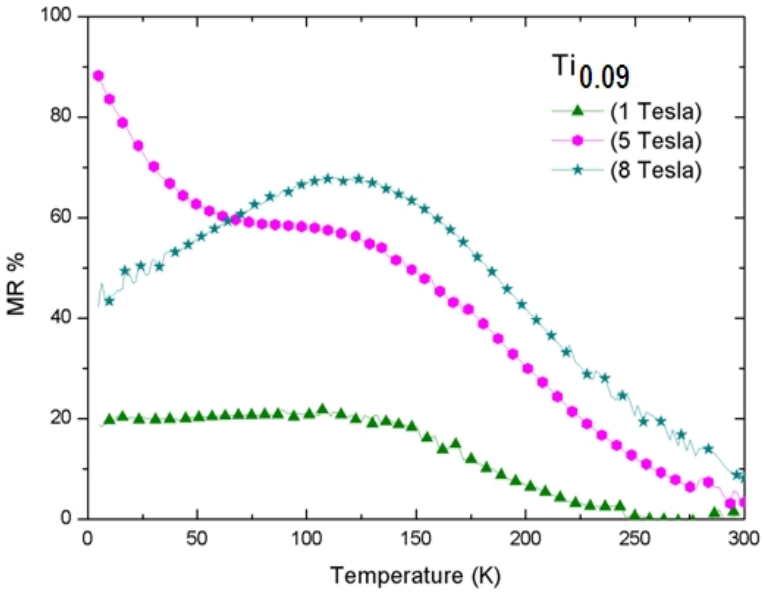


Fig. 11. Variation in MR % with temperature under 1 T, 5 T and 8 T applied magnetic fieldfor $Ti_{0.09}$.

TABLES

Table 1: Structural parameters of $Ti_{0.00}$ and $Ti_{0.09}$ determined from FULLPROF refinement to XRD pattern performed at room temperature.

Parameters	**Name of samples**	
	Ti 0.00	**Ti 0.09**
a (A°)	5.5024	5.5189
b (A°)	5.5024	5.5189
c (A°)	13.3560	13.3804
Unit cell volumeV(A°)^3	350.19	352.94
Structure	Rhombohedral	Rhombohedral
Space group	$R\bar{3}c$	$R\bar{3}c$
χ^2	1.79	2.12
FWHM	0.3203	0.4331
Partical size (nm)	25.83	19.11
X-ray density gm/cm^3	6.45	6.37

Table 2: Measured & calculated elemental weight % of $Ti_{0.09}$.

Element	Measured weight %	Calculated weight %
La	45.08	43.06
Sr	11.92	11.64
Mn	21.92	22.14
Ti	2.79	1.91
O	18.29	21.25
Total	100	100

Table 3. MR% for $Ti_{0.00}$ and $Ti_{0.09}$ at different temperature and different applied magnetic field.

T (K)	$Ti_{0.00}$			$Ti_{0.09}$		
	MR%			MR%		
	1 T	5 T	8 T	1 T	5 T	8 T
5	24	35	42	24	89	44
100	12	27	35	20	58	67
200	7	25	35	7	30	42
300	6	23	33	1	3	8

ACKNOWLEDGEMENT

Authors from Departmnet of Physics, Saurashtra University are thankful to the departmental UGC DRS-SAP & DST-FIST programmes for providing basic facilities.

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Synthesis, Spectral Studies and Anti-microbial screening of New Pyrazoline Derivatives

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ABSTRACT

A series of new 3-{4-[3-(methyl)-4-(2, 2, 2-trifluoroethoxy) pyridin-2-yl]methylamino phenyl}-4,5-dihydro-5-aryl 1-H-pyrazol) (4a-4k) have been articulated by the reaction of 3-aryl-{4-[3-(methyl)-4-(2, 2, 2-trifluoroethoxy) pyridin-2-yl]methylamino phenyl}prop-2-en-1-one(3a-3k) with hydrazine hydrate. The structural elucidation has been made by using Mass Spectrometry, Infrared Spectroscopy and ^{1}H Nuclear Magnetic Resonance Spectroscopy. All lately synthesized derivatives were recognized for their anti-bacterial and anti- fungal activities against two Gram positive, two Gram negative bacteria and one fungi. Anti-microbial evolution of above compounds compared with known standard drugs.

SUMMARY

Synthesis of heterocyclic compound to target the micro-organism.

Keywords: Pyrazol ; Pyridine; Anti- bacterial ; Anti-fungal ; Trifluoroethoxy pyridine

INTRODUCTION

The pyridine ring is outstanding heterocyclic scaffold in large number of bioactive molecule[1]. Over and above variety of heterocyclic compound having pyridine ring are allied with varied pharmacological properties like antimicrobial[2], anticonvulsant[3], anti-HlV[4], anticancer[5], antiviral[6], antimycobacterial and antifungal activities [7].

In addition the pyrazoline containing molecules have been potentially emerging to reveal activities such as antimicrobial[8], anti-inflammatory [9-10] and antihypertensive [11].

Prompted by modern prose interpretation, several new pyridine derivatives were synthesized, leading to appealing heterocyclic scaffolds that are particularly useful for the formation of miscellaneous chemical libraries of drug-like molecules for microbial screening.

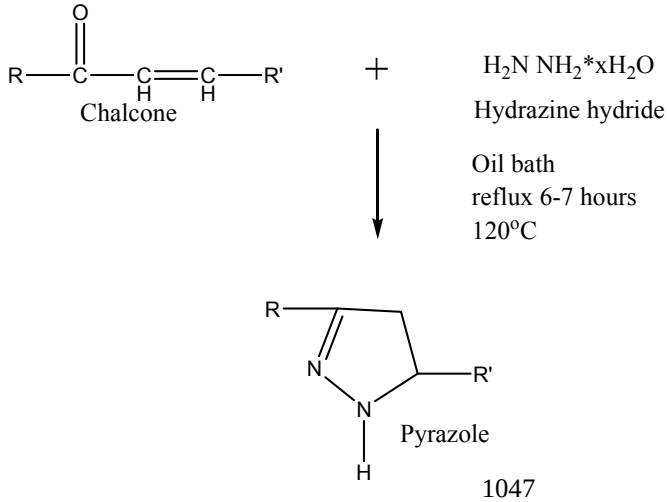
In this scientific study we intended to integrate the two bioactive entities Pyridine and pyrazoline into one condensed structure and assess their biological effectiveness through anti-bacterial and anti-fungal studies.

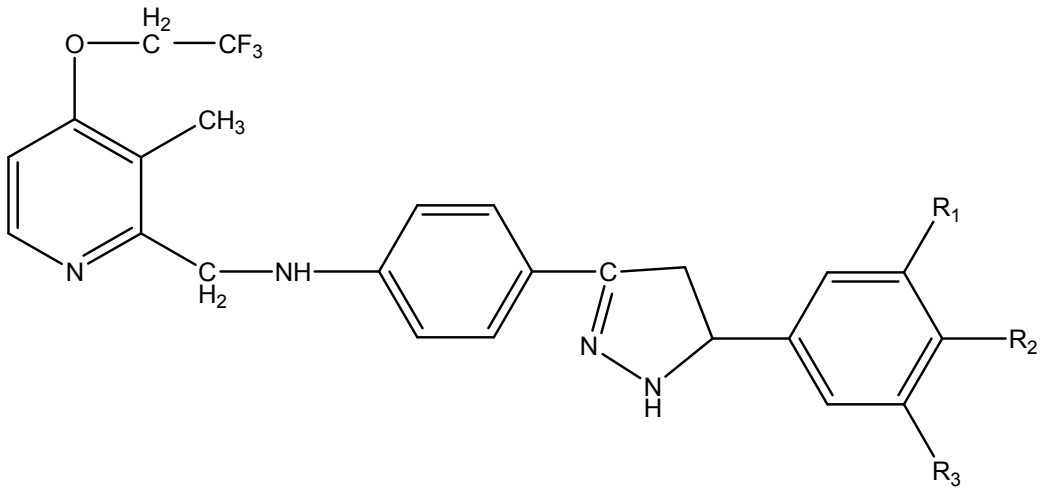
RESULTS AND DISCUSSION

1. Chemistry

The purpose of this study was to carry some pyridine bearing different pyrazolinyl moieties . Since the direct introduction of some specific substances for the construction of 2, 2, 2-trifluoroethoxy pyridine derivative and 4,5-dihydro-5-aryl 1-H-pyrazol nucleus are not always easy, new methods for the direct synthesis of these compounds from Chalcones[14] have been of great interest. The synthetic course to locate the 3-{4-[3-(methyl)-4-(2, 2, 2-trifluoroethoxy) pyridin-2-yl]methylamino phenyl}-4,5-dihydro-5-aryl 1-H-pyrazol) (4a-4k) is given in Scheme 1. The necessary input aldehyde intermediate 1 was obtained from market. Furthermore Chalcones 3a-k were synthesized by conventional Claisen-Schmidt condensation reaction. Followed by recrystallization with the help of ethanol all analogous Chalcones were over come in 76-95% yield. The 3-{4-[3-(methyl)-4-(2, 2, 2-trifluoroethoxy) pyridin-2-yl]methylamino phenyl}-4,5-dihydro-5-aryl 1-H-pyrazol) (4a-4k) were synthesized from Chalcones 3a-k by reacting with 3-aryl-{4-[3-(methyl)-4-(2, 2, 2-trifluoroethoxy) pyridin-2-yl]methylamino phenyl}prop-2-en-1-one(3a-3k) and hydrazine hydrate at 110 ^{O}C, correspondingly. Each of the isolated products will carry for washing with diethyl ether to get meet the expense of title products in 55-89% yield. The structural characterizations of all newly synthesized compounds were elucidated on the basis of spectral data such as IR, ^{1}H-NMR, Mass and elemental analysis.

Scheme 1 : The synthetic path projection for **4a-k** compounds.





3-{4-[3-(methyl)-4-(2, 2, 2-trifluoroethoxy) pyridin-2yl]methylamino phenyl}-4,5-dihydro-5-aryl 1-H-pyrazol) (4a-4k)

Experimental

Laboratory chemicals were supplied by Oxford India Ltd., and Angel Scientific Ltd. The melting points of the recently prepared derivetives were precisely recorded in open-glass capillaries supported to Stuart-SMP10 assembly. The thin layer chromatography deployed to determine the purity of the compounds. Silica gel plates kiesel gel having dimension of 0.25 mm, 60G F254 where percolated sheets supplied by Merck, utilized for TLC. The solvent system of n-hexane: methanol (7:3) was found suitable, over and above the visualization of identical spots was by ultraviolet light (254nm). IR spectra (KBr disc) were recorded on Instrument: SHIMADZU-FT-IR-8400 having frequency of range 4000-400 cm-1 (consist of KBr disc). 1H NMR spectra were scanned by Internal standard: TMS; solvent : $CDCl_3$: Instrument : BRUKER (300 MHz) . Elemental analysis of the all newly synthesized derivatives were determine by Elementar Vario EL III Carlo Erba 1108 and Antimicrobial activity was done at Department of microbiology, School of science RK University.

General procedure for synthesis of 3-{4-[3-(methyl)-4-(2, 2, 2-trifluoroethoxy) pyridin-2-yl]methylamino phenyl}-4,5-dihydro-5-aryl 1-H-pyrazol) (4a-4k)

3-{4-[3-(methyl)-4-(2, 2, 2-trifluoroethoxy) pyridin-2-yl]methylamino phenyl}-4,5-dihydro-5-aryl 1-H-pyrazol) (4a-4k) was synthesized by carry out reaction between 0.01 M(4.75 gm) of 2-methoxy-4-(3-(4-((3-methyl-4-(2,2,2-rifluoroethoxy)pyridin-2-yl)methylamino)phenyl)-4,5-dihydro-1H-pyrazol-5-yl)phenol with 0.015 M (1.09 ml) of Hydrazine hydride in methanol (20ml) at 110^o C in oil bath up to 7 hours. At the end of 6.4 hours reaction mixture was settling at room temperature and pointy dropped in to 40 gm of cursed ice. The product obtained was collected, dried and Re-crystallized carried out in methanol. m.p found 165^{o}C. IR (KBr) 2962, 1452, 3010; 1504; 1335; 1139-1188 3417; ^{1}H-NMR ($CdCl_3$, δ ppm): 2.0 (2H), 2.32 (d, H), 3.73 (d, 3H), 3.9 (t), 4.46(m, 2H), 4.0 (S, 1H), 4.46 (d, 2H), 5 (s, 1H), 6.54-6.56(M, 4H), 6.67(s,1H), 7.0 (s, 1H) 7.62 (s,1H) MS : (m/z) 470.19 ; Anal. Calcd. $C_{25}H_{25}O_3N_4F_3$: C: 61.72%, H: 5.18%, N: 11.72%; F, 11.72%; O, 9.87%. Found: C:

61.58%, H: 5.20%, N: 11.62%.; F, 11.70%; O, 9.80%. Similerly other compounds are synthesized as mention in table-1

Biological assay

Compounds and cells

A series of newly articulated compound were taken in to solvent DMSO to prepare 1280 μg/ml as an initial concentration and followed by culture medium for serial dilution. Bacterial strains were provided by the American Type Culture Collection.

Antibacterial assay

The newly articulated compounds were tested for their antibacterial activity against two gram-positive and two gram-negative bacteria as mention in table -2, by the guidelines in NCCLS-approved standard document M7-A4, using the micro dilution broth procedure[15]. Ampicillin trihydrate was considered as the reference antibacterial agent. The MIC of each newly synthesized derivative was noted as the lowest concentration for series of derivatives filled in the tubes where growth is completely disappear (no turbidity observed) .

Antifungal assay

For the antifungal activities of the newly articulated pyrazoline derivatives were tested against 1 yeast strain according to the course of action mention in NCCLS, using the micro-dilution broth procedure[16]. Fluconazole was consider as the reference antifungal agent. The MIC of each chemical compound was noted as the lowest concentration of each newly synthesized derivatives in the tubes where growth is completely disappear of inoculated yeast. The MIC values of the compound screened are given in below table.

CONCLUSION

In digest, we have scaffold unique pyrazoline base pyridine by synthesizing a series of novel 3-{4-[3-(methyl)-4-(2, 2, 2-trifluoroethoxy) pyridin-2-yl]methylamino phenyl}-4,5-dihydro-5-aryl 1-H-pyrazol) (4a-4k). each of the newly synthesized derivatives was recognized precisely by spectroscopic data like ^{1}H-NMR, Mass, IR Spectra and elemental examination in addition new derivatives were screened for antibacterial and antifungal activity. Anti-microbial assay shows that 4c and 4g have moderate activity with MICs between 20 and 40 µg/mL, the significance of this work comes in the prospect that the new-fangled derivatives might be more effectual drugs against bacteria, which could be accommodating for designing stronger antibacterial agent for therapeutic use. While compound 4j and 4k shows optimistic antifungal activity lies at 10 µg/mL. It may lead to overcome as moderate antifungal agent.

TABLES

Sr. No	Code	M.F	R 1	R2	R3	M.W	M.P/^{o}C
1	4a	$C_{24}H_{20}N_4F_3$	H	H	H	421	187
2	4b	$C_{25}H_{22}O_2N_4F_3$	OCH_3	H	H	451	143

3	4 c	$C_{26}H_{24}O_3N_4F_3$	OCH_3	OCH_3	H	481	122-126
4	4 d	$C_{27}H_{26}O_4N_4F_3$	OCH_3	OCH_3	OCH_3	511	179
5	4 e	$C_{25}H_{25}O_3N_4F_3$	OCH_3	OH	H	470	165
6	4 f	$C_{24}H_{23}O_2N_4F_3$	OH	H	H	440	180
7	4 g	$C_{24}H_{23}O_2N_4F_3$	H	OH	H	440	174
8	4 h	$C_{24}H_{22}O_3N_5F_3$	NO_2	H	H	469	96
9	4 i	$C_{24}H_{22}O_3N_5F_3$	H	NO_2	H	469	113
10	4 j	$C_{24}H_{22}O_3N_5F_3$	H	H	NO_2	469	99
11	4 k	$C_{24}H_{21}O_3N_4F_3Cl$	H	Cl	H	489.5	137

Table:-1

Antibacterial assay Compound (µg/ml)												
Microbes	**4a**	**4b**	**4c**	**4d**	**4e**	**4f**	**4g**	**4h**	**4i**	**4j**	**4k**	**Amp**
Escherichia coli ATCC 4230	160	640	20	80	20	40	40	40	40	80	160	20
Micrococcus luteus ATCC 9345	320	640	40	40	80	40	10	320	320	160	320	10
Salmonella typhi ATCC 14028	160	160	10	160	10	80	10	80	160	160	640	10
Staphylococcus aureus ATCC 6538	320	160	20	40	10	40	20	640	320	320	320	10
Microbes	**4a**	**4b**	**4c**	**4d**	**4e**	**4f**	**4g**	**4h**	**4i**	**4j**	**4k**	**Fluconazole**
Candida albicans ATCC 14053	160	320	160	160	640	160	40	80	640	10	10	5

Table-2

ACKNOWLEDGEMENT

We are thankful to the management of RK University, Rajkot to provide advance laboratory condition and lab wear, Thanks are also due to Sophisticated Instrument Facility, NFDD Saurashatra University Rajkot. Mr. Parth Bhatt senior microbiologist at School of science RK University who performed antimicrobial screening and authors are truly appreciates his efforts.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

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RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

Infrared Spectroscopy Study Of Mn^{2+}-Doped $CoFe_2O_4$ Nano-Ferrites

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ABSTRACT

Fourier Transform Infrared Transmission Spectroscopy (FTIR) is used to study the elastic properties at 300K for Manganese doped Cobalt ferrite ($Mn_xCo_{1-x}Fe_2O_4$). The nano-ferrites particles of $Mn_xCo_{1-x}Fe_2O_4$ (here, x = 0.0 to 1.0, step 0.2) system are prepared by the chemical co-precipitation method. The X-ray diffractometry was employed to confirm the single phase spinel structure formation and to determine the cell-edge parameter. The force constants for tetrahedral (A) and octahedral (B) sites of the spinel lattice were determined by infrared spectral analysis. The elastic constants like bulk modulus, rigidity modulus, Young's modulus, Poisson's ratio are determined with incorporation of zero porosity correction. The force constant and elastic moduli for all the samples are found to be nearly of same value as observed the constancy in vibritional frequency for both the interstitial sites.

SUMMARY

Include a brief summary of your research work in exactly one sentence.

Keywords: Spinel Ferrite, FTIR, XRD, elastic moduli

INTRODUCTION

The nano-ferrites are scientifically vital materials with the crystal structure of mineral spinel of a FCC lattice which consists oxygen ions, having unit cell of 8 formula units show AB_2X_4 type structure. The tetrahedral (A) and octahedral [B] sites accommodate the metallic cations *(1)*. The spinel structure is one

of the most frequently encountered with the compounds. X represents oxygen or some chalcogenic bivalent anion (S^{2+}, Se^{2+}, Te^{2-}) which may be partly substituted by proper monovalent anions (F^{-}, I^{-}, Br^{-}) *(2)*.

The general chemical composition of spinel ferrites is $MO{\bullet}Fe_2O_3$ and can be derived from magnetite $Fe^{2+}O{\bullet}Fe_2{}^{3+}O_3$ by the replacement of the Fe^{2+} ion by other divalent ions like Ni, Co, Cu, Zn, Cd, etc. It is also possible to replace Fe^{3+} by ions like Al^{3+}, Ga^{3+}, Cr^{3+}, etc. A monovalent ion Li^{1+} can be introduced in the spinel structure provided a trivalent ion ($M=Li^{+}_{0.5}\ Fe^{+}_{0.5}$) is simultaneously introduced for charge balance. The structural and magnetic properties of spinel ferrites is highly depends on synthesis method *(3)*. There are various methods to prepare nano-structured ferrites namely co-precipitation, citrate precursor method, auto-combustion, flash combustion, polymeric precursor and sol-gel techniques. Each of these methods has been standardized depending upon the composition of final ferrite product. In this work, the co-precipitation method is used to prepare nano-structured MnxCo1-xFe2O4 ferrite system with compositions x = 0.0, 0.2, 0.4, 0.6, 0.8 and 1.0.

The study of elastic constants reveals the nature of biding forces and elastic moduli for ferrite materials. The elastic moduli give an idea of mechanical hardness of material which is an important characteristic of ferrite for specific technological application. The conventional technique like ultrasonic pulse transmission for the determination of elastic constants and Debye temperature requires large sample dimensions. The yield of non-conventional chemical routes of ferrite-materials is too small to be adequate for the sample preparation for ultra-sonic pulse transmission technique. The solution lies in the infra-red spectroscopy technique where very small quantity of powder is sufficient for the characterization. FTIR spectroscopic method is important investigative tool which provide the best results of structural characterization. The technique depends on fundamental principal that every chemical substance exhibits distinct signature of infra-red absorption. It is well known that more IR bands may appear if the symmetry of the spinel structure is lower than cubic or supplementary ordering of cation exists. This work is to study IR-spectroscopic signature as far as the vibrations of tetrahedral (A) and octahedral (B) molecules are concerned and also to calculate elastic properties of Mn doped cobalt ferrite using cation distribution deduce through X-ray diffraction patterns.

EXPERIMENTAL DETAIL

The final products of nano-sized $Mn_xCo_{1-x}Fe_2O_4$ (x = 0.0 to 0.1, Step: 0.2) ferrites prepared by co-precipitation (wet-chemical) method. The stating materials were weighted according to the stoichiometric proportion. The chemicals of analytical grades i.e. Ferrous sulfate and Manganese/Cobalt sulfate were used in the proportion 2:1, added to double distilled water and stirring to complete dissolution. The 1M sodium hydroxide solution was used to adjust the pH 11 of solution and kept at 65°C for 5 hour. After 5 hour of reaction, the precipitated particles are washed and filtered six times and then dried at 180°C for 3 hour. The final products all specimens were characterized by powder X-ray diffractometry to check purity and surety of the compounds using Philips X'pert MPD X-ray diffractometer with CuKα radiation of wavelength λ=1.5481Å. X-ray powder diffraction data were obtained in a scanning range from 10 to 80 degree with a step size of 0.02 with 1 second counting time at each step. It is always a good practice to ascertain the presence of single crystalline phase and the absence of any unreacted ingredient in the final ferrite product. It will be a futile exercise to interpret the physical properties of a ferrite product if it has multi-crystalline phases or residue of unreacted ingredients (Typical XRD patterns x=0.4 is presented here). The FTIR spectral study gives information about structure and elastic properties. The FTIR spectra were recorded at 300 K using Nicolet Thermo Fisher make IR spectrometer having optical resolution of 0.04 cm^{-1}, in the range from 400 cm^{-1} to 800 cm^{-1} in KBr disc medium.

RESULTS AND DISCUSSION

All the nano-sized powder samples are found to be single phase fcc spinels (space group: Fd3m). The compositional variation of lattice constant (a) is shown in Table 1. The lattice constants of two end-members, $CoFe_2O_4$ is found to be 8.379 Å and $MnFe_2O_4$ is 8.503 Å. It shows linear increase with increase in Mn-concentration (x). This is attributable to the large difference in the Paulling ionic radius of Co^{2+} (0.79 Å) and Mn^{2+} (0.80 Å) cations occupying the interstitial voids of spinel lattice. In fact, according to the Paulling rules, the ionic radius is very much affected by its coordination number i.e. 4 for tetrahedral (A) site and 6 for octahedral (B) site. But looking to the marked preference of Co^{2+} for B-site and Mn^{2+} for A-site, the above mentioned values are adopted. The linear variation in lattice constant (Fig. 2) can be explained on the basis of the Vegard's law *(4)*. The Vegard's law predicts linear change in cell edge parameter for spinel system with substitution of cations. The X-ray density decreases with Mn-content (x) as it is expected.

The study of changes in chemical and molecular structure of ferrites due to some foreign atom doping is extract through FTIR spectroscopy. For ferrites, generally two assigned absorption bands appear ν_1 (around 600 cm^{-1}) and ν_2 (around 400 cm^{-1}). The two strong spinel ferrite bands are found; around 400 cm^{-1} is due tetrahedrally coordinated metal ions and at 600 cm^{-1} is due to octahedrally coordinated metal ions *(5, 6)*. In present case also the IR-absorption bands that appear within 400-470 cm^{-1} is due to tetrahedral stretching vibrational mode ($O–M_{Tet}$) while second within the range 560-585 cm^{-1} is due to octahedral stretching vibration mode($O–M_{Oct}$). The appearance of these standard IR-absorption bands immediately confirms and supports the formation monophase spinel ferrites. The FTIR spectra of all specimens in the spectral range 400-800 cm^{-1} are depicted in Figs. 3-5. These show essentially two absorption bands near 405 cm^{-1} due to tetrahedral sites and near 565 cm^{-1}. These absorption bands remain almost fixed and uninfluenced by Mn content because all the cations in the system have nearly equal atomic weights (Table 1). One additional band is observed near 470 cm^{-1} for $x \geqslant 0.8$. Generally in spinel ferrites having significant difference in atomic weights of substituting cations exhibit large variation in vibrational frequencies and therefore the force constants. It is known that in $Zn_xNi_{1-x}Fe_2O_4$ spinel ferrite system, Zn^{2+} replaces the Fe^{3+} ions at tetrahedral sites therefore it decreases vibrational force constant of the A-site cause decrease in absorption frequency. In our case, almost equal atomic weights of the constituent cations keep the restoring force constant, giving constancy in force constants and vibritional frequency for both the interstitial sites. The force constants and elastic constants calculated *(7, 8)* from FTIR spectra are given in Table 2.

CONCLUSION

The co-precipitation method for the production of ferrite found to be simple and economic. The Manganese doped Cobalt ferrite nanoparticles were prepared by chemical co-precipitation method. The XRD revealed single phase spinel structure of present ferrite system. Infrared studies indicated the presence of two strong absorption bands ν_1 and ν_2 which endorsed to the tetrahedral and octahedral sites respectively. The force constants and elastic constants were determined by infrared spectroscopy.

FIGURES

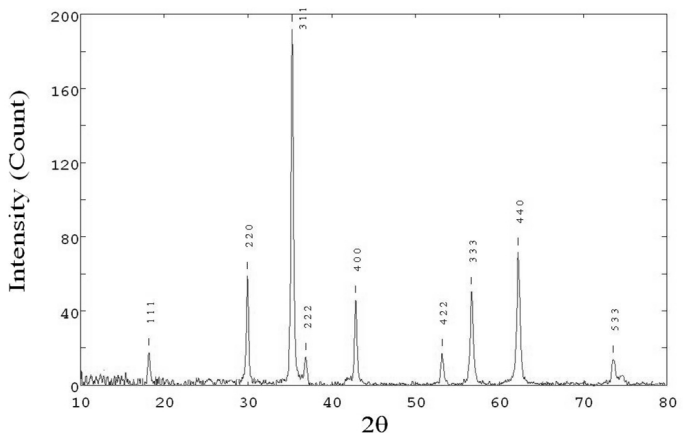


Fig. 1. Typical XRD pattern for x =0.4 of spinel ferrite system $Mn_xCo_{1-x}Fe_2O_4$.

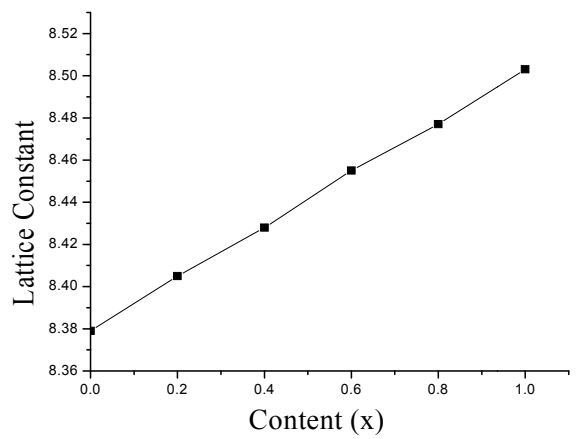


Fig. 2. Compositional variation of lattice constant for spinel ferrite system $Mn_xCo_{1-x}Fe_2O_4$.

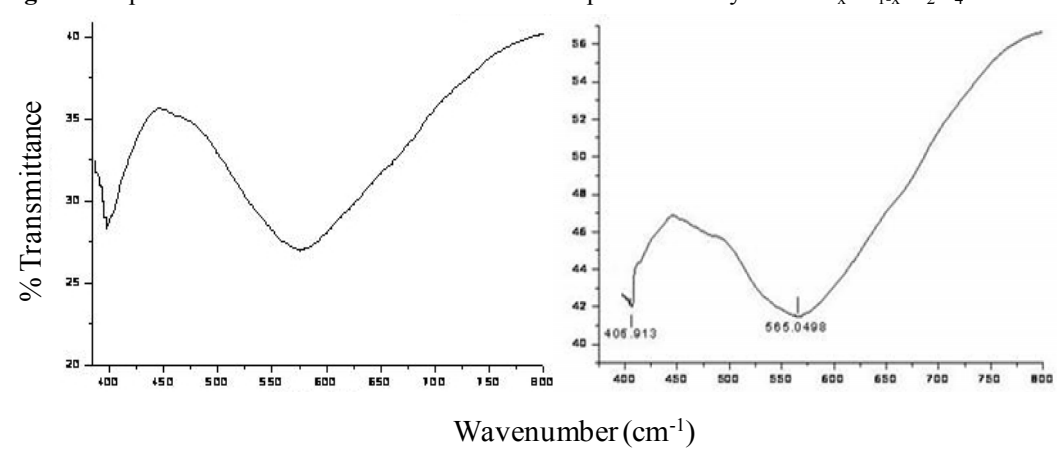


Fig. 3. FTIR spectra of $Mn_xCo_{1-x}Fe_2O_4$ (x=0.0 and 0.2).

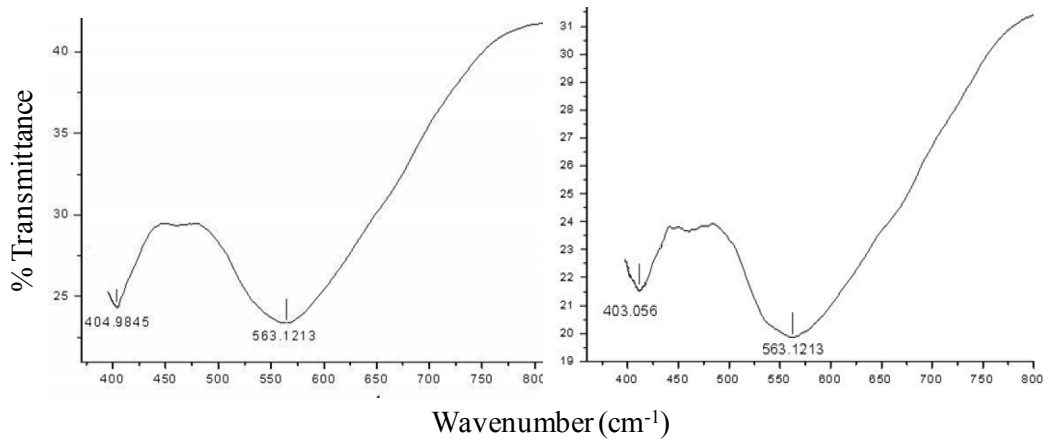


Fig. 4. FTIR spectra of $Mn_xCo_{1-x}Fe_2O_4$ (x=0.4 and 0.6).

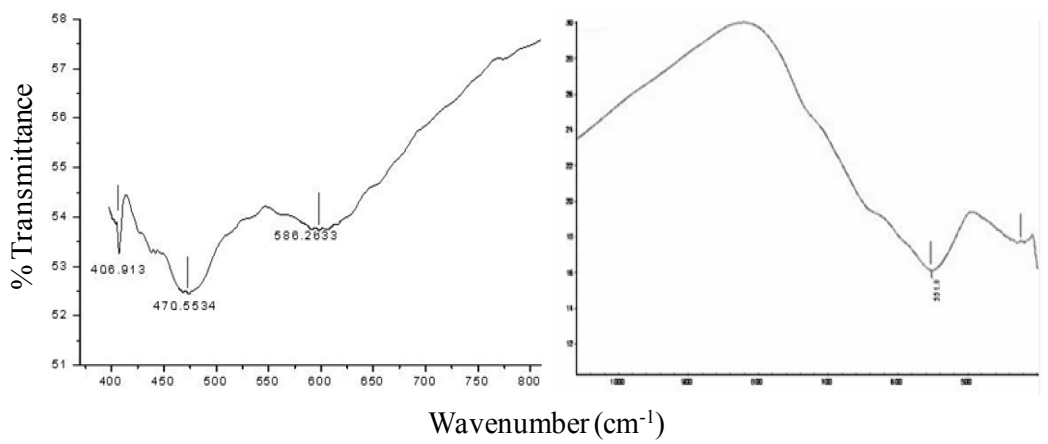


Fig. 5. FTIR spectra of $Mn_xCo_{1-x}Fe_2O_4$ (x=0.8 and 1.0).

TABLES

Table 1. Chemical composition, molecular weight, Lattice constant and X-ray density for $Mn_xCo_{1-x}Fe_2O_4$ spinel ferrite system

x	Composition	Molecular weight	Lattice Constant (a) Å	X-ray density (ρ) gm.cm^{-1}
0.0	$CoFe_2O_4$	234.63	8.379	5.29
0.2	$Mn_{0.2}Co_{0.8}Fe_2O_4$	233.83	8.405	5.23
0.4	$Mn_{0.4}Co_{0.6}Fe_2O_4$	233.03	8.428	5.18
0.6	$Mn_{0.6}Co_{0.4}Fe_2O_4$	232.23	8.455	5.10
0.8	$Mn_{0.8}Co_{0.2}Fe_2O_4$	231.43	8.477	5.06
1.0	$MnFe_2O_4$	230.63	8.503	4.98

Table 2. IR band position (ν), force constants (K), Bulk modulus (B), Rigidity modulus (G), longitudinal wave velocity (V_l), transverse elastic wave velocity (V_t), mean velocity (V_m), Poison's ration (σ), Young's modulus (E) and Debye temperature (θ_D) for $Mn_xCo_{1-x}Fe_2O_4$ spinel ferrite system

Sample → Parameter ↓	0.0	0.2	0.4	0.6	0.8	1.0
$\nu_1(cm^{-1})$	567	565	563	563	580, 471	551
ν_2 (cm^{-1})	401	405	405	403	406	440, 402
K_t(N\m) × 10^2	0.709	0.801	0.758	0.735	0.736	0.749
K_o(N\m) × 10^2	1.995	2.0423	1.901	1.922	1.884	1.852
K_{mean}	1.352	1.422	1.329	1.328	1.310	1.300
B (GPa)	161.2	168.3	157.4	157.4	155.9	153.5
V_l (m/s)	5520	5647	5475	5480	5431	5402
V_s (m/s)	3186	3260	3161	3164	3135	3119
V_m (m/s)	4353	4454	4318	4322	4283	4260
G (GPa)	54.23	56.12	52.47	52.48	51.98	51.18
σ	0.35	0.35	0.35	0.35	0.35	0.35
E	145.1	151.3	141.6	142.3	140.3	145.2
θ_D(K)	591.7	600.8	582.5	583.6	581.0	573.2

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th& Jan. 6th, 2016)

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RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

Extraction and simulation of chitosan from *Squilla sp.* and application of chitosan nanoparticles as a protein delivery carrier

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ABSTRACT

The purpose of the present work was to extract and purify chitosan from shells of *Squilla sp.* and further more nanoparticles of chitosan has been utilized as carrier for protein delivery. The structure and physicochemical properties of extracted chitosan was compared with standard chitosan that showed similar results. With respect to diverse formulation, nanoparticles were prepared depending upon the ionic gelation method of TPP with chitosan. Over and above the examination of nanoparticles with respect to diameter in nano size and appearance by using FTIR and SEM for confirmation of linking of TPP with chitosan ammonium group. The protein delivery with chitosan nanoparticles could be analyzed by deploying standard protein as bovine serum albumin. As initial concentration of BSA and chitosan was increasing, the encapsulation efficiency was decreasing. Low molecular weight extracted chitosan showed higher BSA release as compare to the high molecular weight standard chitosan.

SUMMARY

In the present study, chitosan was extracted and purified from shells of *Squilla sp.* and further it is utilized as protein delivery carrier.

Keywords: Chitosan, Chitosan nanoparticles, bovine serum albumin (BSA), tripolyphosphate (TPP)

INTRODUCTION

Chitosan is consider as polysaccharide made up of N-acetylglucosamine glucosamine.(*1)*. Every year the crustacean processing industries over the world turn out more than 60,000 tonnes of waste. About 10% of chitin on dry weight basis is present in crustacean waste *(2)*.

Chitosan has been found optimum as effective drug carrier for protein. Some studies indicate that to reduce the body impact and applept its bioavilibity, chitosan is used as nanoparticles. Chitosan is also a mucoadhesive polymer. Mucoadhesion is commonly defined as the attractive interaction between a mucosal surface and a pharmaceutical formulation(*4)*. Due to batter stability, simple and mild preparative methods, low toxicity and providing dynamic routes of administration, chitosan nanoparticles are used as drug delivery carrier*(5)*.

To overcome the drawback of chemical cross-linking, physical cross-linking such as ionic gelation method has been applied whichprevents the possibility of damage to drugs, particularly biological agents. Because of the presence of amine groups, CS possess positive charge and efficiently bind with the negatively charged tripolyphosphate (TPP) resulting in nanoparticles of various size which can be efficiently employed in protein and vaccine delivery *(7, 36).*

Few studies have attempted on extraction and characterization of chitosan from *Squillasp*. Therefore, the main focus of present study is to extract chitosan from *Squilla sp.* collected from trash and further preparation of chitosan nanoparticles (CSNPs) with the help of ionic gelation method for protein delivery carrier in order to determine the effect of various parameters. Bovine serum albumin (BSA) was utilized as standared protein and CSNPs prepared from *Squilla sp***.** was compared with the CSNPs prepared from standard chitosan to analyse the encapsulation capability and release rate.

MATERIALS AND METHODS

Materials

Deacytylation degree with 90 % and molecular weight 2743.9±76 of chitosan, 68 kDa molecular weight of BSA and 367.86 kDa molecular weight of TPP were purchased from Hi-media. All other chemicals were of reagent grade.

Sample Collection

Squilla sp. was collected from trash from Royapurum sea coast (13º 6' 26"N 80º 7' 43" E), Tamilnadu, India. Meticulous cleaning of samples was done in D/W and kept in zip lock bag and bring down to laboratory. The collected samples were cleft and the shells of the *Squilla sp.* were detached from other body parts and stored at -20ºC until used.

Proximate Analysis

Proximate analysis was done on wet weight basis of *Squilla sp.* shellswhich includedifferent parameter such asmoisture, ash, lipid and protein. The estimation of moisture content was carried out by tacking accurately 2g of shells into aluminum dish where dish was pre weighted *(9)*. Place the samples in oven up to 105ºC and wait till constant mass was obtained. Content of ash was determined by hammering the sample which is pre dried at 600ºC in a silica crucible up to formation of white ash. Folin and lowry method could be apply to find the total protein in crude sample*(8).*Gravimetric analysis as per Bligh and Dyer method was performed to find lipid content.

Extraction of Chitin

Separation of chitin was achieved in three steps:

(a) Deprotenation

Dry powder of *Squilla sp*. shells was sub divided into three groups and each sub group contains 25 gm of shell powder. Each sub samples were placed in 350 ml of beakers and treated with 3.5% sodium hydroxide (NaOH) for 2hr at 66 °C with solid to solvent ratio of 1:10 (w/v) in order to break up proteins and sugar *(10).*

(b)Deminaralization

After deprotenation, each sub-samples were demineralized with 1 N HCl at ambient temperature with solid to solvent ratio 1:15 (w/v). The samples were allowed to air sock for 12 hrs to remove the minerals mainly calcium carbonate *(10, 11).* The protein and ash content were evaluated after deprotenation and demineralization in order to check how much amount of protein and minerals are removed from shells.

(c)Decolorization

Alkali and acid treatments yields a colored chitin product. For commercial acceptableness, the chitin produced from crustacean sources, inevitably to be decolorized, which is a process to take away astaxanthins and pigments or bleached to produce cream white chitin powder *(10)*. White colored chitin was prepared by treating with acetone and dried for 2 hr at ambient temperature, followed by bleaching with 0.315 % (v/v) sodium hypo chloride solution (containing 5.25% available chlorine) for 5 min with a solid to solvent ratio of 1:10 (w/v), based on dry shell. The remaining chitin was washed with deionized water, which was then drained off *(10).*

Preparation of chitosan from chitin

By removing acetyl group from chitin, be the process of deacetylation, chitosan was obtained from chitin.

Deaceytylation

In this process, the sample was treated with 50% NaOH of 100ml and mixture was boiled up to 70 °C for 20 hrs on hot plate. The samples were then cooled at 30 min at room temperature. Then mixture was cooled at 37 °C and further incubated in oven at 121 °C for 24 hr. After 24 hr of incubation, white cream form of chitosan was obtained *(12).*

Purification of Chitosan

Chitosan obtained by deacetylation of chitin was purified by dissolving it into 0.1 M acetic acid solution and it was filtered to remove insoluble particles. To obtain the purified chitosan, the filtrate was treated with 1M NaOH solution under continuous stirring for 2 h at 70 °C in order to form white precipitate, which was thoroughly washed by using double distilled water and then dried at 40 °C for 48hrs. During the purification process further deacetylation of chitosan was occurred *(13)*.

Characterization of Chitosan by FTIR, XRD, SEM and EDS

Fourier transform infrared (FTIR) spectroscopy is one of the most important and widely used analytical techniques for characterization of chitosan. It is based on the vibrations of the atoms of a molecule. The FTIR spectra of Commercial chitosan and chitosan extracted from *Squilla sp.* were recorded on Perkin-Elmer FTIR spectrometer (1760–X) within KBr pellet. The instrument was operated within the resolution range of 4 cm^{-1} and frequency range of 400-4000 cm^{-1} *(14)*.

X-Ray Diffraction (XRD) measurement was carried out on XPERT-PRO Diffractometer system with CuKα radiation λ = 1.54060 [Å], 45kV, 40mA and scan step time 2[s] *(15)*.

Scanning Electron Microscopy (SEM) with Energy Dispersive X-ray Analysis (EDS) was carried out to know the surface morphology and the elements present in the CS.

Physicochemical properties of Chitosan

Calculation of Degree of Deacetylation (DD)

Degree of deacytylation was determined by Fourier transform infrared spectroscopy [16]. Measurements were carried out on a Perkin-Elmer FT-IR spectrometer (1760–X). The degree of deacetylation was calculated with the help of following Eq. (1).

$$DD\ (\%) = [100- (A_{1655}/A_{3450})] * 115 \text{ -------Eq. (1)}$$

Determination of Molecular weight

Viscometer was exploited to determine molecular weight of chitosan*(17)*.The Mark-Houwink equation $[\eta] = (K) * (Mw)^a$ was utilized to compute the molecular weight. The values of a and K of 0.79 and$1.49*10^{-4}$ cm^3 /g respectively were used *(18)*.

Water and fat binding capacity

By applying Wang and Kinsella (1976) method, Water binding and Fat binding capacity could be found out. In this method, consumption of fat or water was found by weighing 5g of sample in centrifuge tube and add 10 ml of water or soya bean oil and mix the content in vortexer for 1 min to mix the sample well. The mixture was incubated at air temperature for 35 min and apply vigorous shacking for 10 s. Further centrifugation was done for 35 min at 3000 rpm. The pellate was collected and weighted for FBC and WBC. FBC and WBC were computed by Eq. (2) and (3)

WBC (%) = [Water bound (g) / Sample weight (g)] * 100 -----Eq. (2)

FBC (%) = [Fat bound (g) / Sample weight (g)] * 100 ----------Eq. (3)

Preparation of chitosan nanoparticles

The purified chitosan obtained from the *Squilla sp.* Shells as well as the commercial chitosan purchased from the hi-media (Mw- 2743.92±76.43 and DD>90%) were used for the preparation of chitosan nanoparticles according to modified method of Calvo *et al*.(1997) based on ionic gelation of chitosan with TPP anions. A 2% (v/v) of 20 ml acetic acid was used to dissolve chitosan (60 mg) to obtain chitosan solution. Then, Different concentration of TPP ranging from 0.6 to 1.5 mg/ml in D/W was used. Further,

in 5 ml of the chitosan solution, 2 ml of TPP solution was added, opalescent suspension was formed spontaneously under magnetic stirring at room temperature. The opalescent suspension was subjected to freeze drying using freeze dryer. BSA loaded nanoparticles are formed by incorporation of BSA with chitosan solution and flushing with TPP solution. When TPP solution was added to chitosan-BSA mixture, immideatly nanoparticles are forming.

Characterization of chiosan nanoparticles

Fourier-transform Infrared Spectroscopy:

The CSNPs with BSA are prepared from both commercial chitosan and chitosan extracted from *Squilla sp.* were analyzed through FTIR spectrometer. The FTIR instrument was functioned within the resolution limit of 4 cm^{-1}, 400-4000 cm^{-1} for frequency.

Scanning Electron Microscopy (SEM):

SEM was used to examine surface morphology of nanoparticles. The instrument was working at an acceleration voltage 20 kV.

Determination of encapsulation efficiency of nanoparticles

Nanoparticles encapsulation efficiency was determined by, suspension prepared from commercial as well as extracted chitosan from *Squilla sp.* shells were centrifuged at 30,000 rpm at 10°C for 30 min. The free amount of BSA was determined by UV spectrometry at 595 nm using supernatant. Samples in triplicate were examined at regular time interval and encapsulation efficiency of BSA was measured by victimization of Eq. (4) *(13)*.

EE = [(Total amount of BSA – Free amount of BSA in supernatant) / (Total amount of BSA)] * 100 -
----------Eq. (4)

In vitro release study

CSNPs consisting BSA was detached from solution by decanting supernatent, the semi solid CSNP was dissolved in approximate 3.5 ml of PBS under vgorous shaking at room temperature. AT regular time interval, solution of PBS with CSNPs was taken out 150 µl and protein concentration was determined using Bradford method. The suspension in centrifuge tube was replaced with100 µl fresh PBS solution *(13).*

Statistical analysis

Each experimental data was represanted in mean ± standard deviation with triplicates. For computation of onw-way ANOVA PASW statistics 18software was exploiteds for all the experimental data, where data is said to be statisticaly significant by keeping p value<0.05.

RESULTS AND DISCUSSION

Proximate composition

Proximate composition (moisture, ash, crude protein, lipid and carbohydrate) of *Squilla sp*. was tabulated as per their wet weight basis as shown in table 1. Protein and mineral content is decreasing from 22.66±1.52 % to 0.10±0.01 % and18.03±0.62 % to0.057±0.013 %. Hence, protein and minerals are almost removed from shells after deprotenation and demineralization as shown in table 2. Tolaimate *et al.,* (2003) reported that, there is decrease in protein and mineral content after deprotenation and demineralization. The yield of chitin and chitosan obtained in the present work was found to be 10.48±0.68 % and 2.95±0.35 % respectively as those reported in other works *(20, 21).*

Characterization of chitosan

Fourier-transform Infrared Spectroscopy

Chitosan produced from *Squilla sp*. was characterized by FTIR. The IR spectrum of these extracted chitosan was compared with the standard Chitosan (Fig. 1)**.** In spectra of standard chitosan [Fig. 1(A)**]** showed a broad absorption band at 3417 cm^{-1} attributedto the starching vibration of O-H and N-H groups *(22)*. The absorption peaks at 2916.65 cm^{-1}, 1644 cm-1, 1151 cm-1 are due to stretching vibration aliphatic C-H, amide I (-NH deformation of $NaHCO_3$) and C-O-C bonds respectively *(14, 23)*. The purified chitosan extracted from *Squilla sp.* showed the absorption peaks at 3429.98 cm^{-1}, 2921.91 cm^{-1}, 1642.97 cm^{-1} and 1084.25 cm^{-} [Fig. 1(B)] *(24)*, which are similar to the standard chitosan. Mohamed *et al.,* (2012) found similar absorption peaks in their study of nemiticidal activity of chitin and chitosan derived from shrimp shell waste.

X-Ray Diffraction Analysis

In XRD crystallograph, a diffraction pattern of standard chitosan and chitosan *Squilla sp.*is shown in Fig. 2**.** The spectra of standard chitosan shows a characteristic peak at 9.8475° and 20.1672° [Fig. 2(a)]*(23, 25),* which suggest the formation of inter- and intra-molecular hydrogen bonds in the presence of free amino groups in CS. CS from my sp. shows characteristic peak at 9.3023°, 20.9443° [Fig. 2(b)], which are similar to the standard chitosan. Jin *et al.* (2009) and Zhang *et al.*, (2005) observed similar diffraction pattern of chitosan in their study.

Scanning Electron Microscopy

SEM study was performed to analyse the morphological characteristics of nanoparticles. Jun *et al.,* (2011) and Govindan *et al.,* (2012) has observed smooth surface of chitosan as observed in present study. Both standard as well as chitosan extracted from *Squilla sp* showed particle size ranging from 50 to 200 µm as shown in figure(3).

Elemental analysis

The elemental analysis was represented in table 2,which shows percentage of carbon, hydrogen and nitrogen present in standard CS as well as CS extracted from *Squilla sp***.,** which is almost similar [Fig. (4)]. Tuzlakoglu *et al.,* (2004) and Amaral et al., (2005) also observed similar EDS results.

Physicochemical characteristics of chitosan

The physicochemical characteristics of chitosan extracted from *Squilla sp* was determined and compared with standard chitosan shown intable 3.

The intrinsic viscosity of standard CS and CS extracted from *Squilla sp* were found to be 16.4±0.40 cPs and 1.64±0.26 cPs respectively, which corresponds to molecular weight 2743.92±76.43 kDa and 130.18±26.80 kDa respectively, calculated from Mark-Houwink equation *(17)*. The results clearly demonstrate that, as the viscosity reduces molecular weight of chitosan is also reducing (29).

The WBC and FBC differed depending upon the products, ranging from 355 to 611% and 217 to 403% respectively *(30, 31)*. In our study, the WBC and FBC of *Squilla sp*chitosan were found to be 548±11.77 % and 369±19.98 % respectivelythat is lower than the standard CS**.** The degree of deacetylation of chitosan from my species was79%, which is compared with the standard chitosan as shown in table 4. According to No and Meyers (1995), DD of chitosan ranges from 56% to 99%. In our study, the DD of chitosan also falls in this range. Extracted CS as well as standard CS had higher viscosity due to high degree of deacetylation which is very important characteristic of chitosan *(32)*.

Characterization of Chitosan naoparticles (CSNPs)

FTIR was used to confirm the incorporation of TPP into the chitosan matrix and loading of BSA in the prepared nanoparticles *(34)*. The FTIR spectra of CS and CSNPs for standard CS as well as CS from *Squilla sp*. were presented in Fig 1.The two characteristicabsorption bands were observed in standard chitosan (figure 1), at 3417.44 cm^{-1} and 1644.19 cm^{-1}. It is noticeable that this peaks has been shifted to lower wave numbers centered at 3409.95 cm^{-1} and 1631.46 cm^{-1} [Fig. 1(A)], this is an evidence of molecular interaction between chitosan and TPP *(35,36)*. The absorption band shifting also observed in CSNPs prepared from extracted CS from 3429.99 cm^{-1} to 3417.93 cm^{-1} and from 1642.97 cm^{-1} to 1634.29 cm^{-1}, as shown in Fig. 1(B).

The surface morphology of nanoparticles was observed by Scanning Electron Microscopy (SEM). The size of the chitosan reduced from um to nm when it binds to the TPP that indicates the presence of nanoparticles. SEM of CS/TPP nanoparticles revealed a very homogeneous morphology with quite uniform particle size distribution and spherical in shape. The size of particle ranged from 50 nm to 200 nm, represented inFig. 5. Jingou *et al.,* (2012) observed the average size of MTX-loaded FA-CS nanoparticles around 300 nm. Devika *et al,* (2006) also found the similar SEM results in her studies of effect of pH on cross-linking of chitosan with TPP. Qingshen *et al.,* (2012) observed similar SEM micrograph as observed in present study but they used glutaraldehyde as a cross linking agent.

BSA Encapsulation efficiency within nanoparticles

Effect of TPP concentration

Fig. 6 shows that, 0.75 to 1 mg/ml of TPP concentration is optimal for the formation of nanoparticles. As the TPP concentration increases beyond 1mg/ml encapsulation efficiency was decreased from 75.33±1 % to 48.33±2.08% for CSNPs of standard CS and from 51±1.52 % to 21±2.51 % for CSNPs of extracted CS. As the concentration of TPP increase, the pH of the solution increase. With increase in pH, the ionization of amine groups would decrease, that results in decreasing the opportunities for ionic interaction of chitosan with TPP, also prolonged the gelation process *(39, 40)*. Hence, BSA molecules take more time to diffuse out from nanoparticles.

Effect of chitosan concentration

The Encapsulation efficiency of chitosan/TPP-BSA loaded nanoparticles was studied with different concentration of chitosan. The results indicated that by increasing chitosan concentration, the encapsulation efficiency is decreasing. Increasing chitosan concentration made encapsulation extremely

difficult. Calvo *et al.*, (1997) found that fabrication of nanoparticles only affirmable for some particular concentration of chitosan and TPP. This information is also proved in our work in order to deflect the formation of aggregates with large diameter. For the ionic gelation of chitosan and TPP, chitosan and TPP concentration needed to be between 1-3 mg/ml and 1 mg/ml respectively. Particularly only in these concentration we observed opalescent suspension. when concentration of CS is 4 mg/ml and concentration of TPP is 1 mg/ml, foremost opalescent suspension ascertained and dispersed immediately, that displays nanoparticles formation is exceedingly challenging. Fig. 7 shows that, the encapsulation efficiency decreased from 75.33±0.57% to 14.33±2.51 % as the chitosan concentration increased from 1 to 3 mg/ml for CSNP prepared from standard CS. For the extracted chitosan also the encapsulation efficiency is decreasing from 51±1.52 % to 20±2.08 % as the chitosan concentration is increases. Similar effects were also reported by Lim *et al.,* (1997) and Co *et al.,* (2002). Highly viscous nature of gelation medium hinders encapsulation of BSA in the study of chitosan-alginate microspheres.

Effect of BSA concentration

The BSA concentration had significant effect on encapsulation efficiency. As the concentration increase, the BSA encapsulation efficiency was decreasing. Fig. 8 shows that EE of CSNPs of commercial CS and CSNPs prepared from *Squilla sp.* drops quickly from 75.33±0.57% to 31±1 % and 51±1.52 % to 12±2 % respectively with respect to increase in BSA concentration from 1 to 3 mg/ml. Xu and Du (2002) reported the similar effect of BSA concentration on EE but the result fount in the present study was contrary to Berthold *et al.,* (1996) who reported reverse results on BSA encapsulation. The major factor that leads to the association of protein to CS nanoparticles might be the protein-polysaccharide electrostatic interaction, although other mechanism such as reduction of protein solubility near its electrostatic point, hydrogen bonding and hydrophobic interactions might also be involved. Lim *et al.,* (2002)reported that maximum protein association with CS nanoparticles could be achieved only when the protein was negatively charged, in the present study the maximum encapsulation efficiency was found at 1 mg/ml BSA concentration.

Effect of DD and Molecular weight

As the molecular weight of chitosan is increasing, the encapsulation efficiency of BSA is also increasing [Fig. 6, 7, 8]. It is due correlation of molecular weight and spread length of chitosan in solution, which may affect protein interaction and encapsulation. In this study nanoparticles are formed by ionic interaction between positive charges of CS and negative charges of TPP thus the higher viscosity of high Mw chitosan solution containing the longer fragments made its free amino groups harder to protonate and restricted the ionic interaction with TPP. This can explain the low EE of high Mw CSNPs (Yang *et al.,* 2009).Kim *et al.,* (2003) and Sabnis *et al.,* (2000) also reported the similar effect of molecular weight of CS in their studies. The CSNPS prepared from extracted chitosan had low encapsulation efficiency because of its low molecular weight and lower DD as compare with high molecular weight standard CS.

In vitro release study

The in vitro release behavior of CSNPs coated with BSA is shown from Figs. 9-11. At different combination of TPP, CS and BSA concentration, the nanoparticles shows similar results of protein release at 6^{th} hr of incubation time and then slow release at constant but a different rate. The results reveal that, the BSA release rate can be modulated by adjusting various parameters such as chitosan concentration, BSA concentration or Mw and DD of chitosan.

Effect of chitosan concentration

The release profile of BSA with respect to different chitosan concentration ranging from 1 to 3 mg/ml of CSNPs in phosphate buffer was shown in Fig. 9. It was observed that, as the chitosan concentration is increasing, release rate is also increasing. 3 mg/ml chitosan concentration provides the highest release rate 42.38±0.91% for CSNPs prepared from standard CS as well as CS extracted from *Squilla sp.*shows highest burst release 86.61±3.65 % at 6^{th} hr.Yuan *et al.* (2010) stated in study of chitosan–aluminosilicate nanocomposite carrier with controlled and extended drug release behavior that, mucoadhesion and bioavailability of drug that can interact with the gastric and intestinal mucosa can be increased in presence of chitosan in nanocomposite. Hence, the release rate is increased as the chitosan content is increasing. The similar results observed in our study. Lim *et al.* and Kim *et al.* (2002) also found the similar effects.

Effect of BSA concentration

Increase in BSA concentration from 1 to 3 mg/ml increases drug release rate from 21.73±o.56 % to 65.41±0.82 % [Fig. 10(a)]for CSNPs of standard CSand 45.75±0.21 % to 84.6±0.62 %[Fig. 10(b)]for CSNPs ofSquilla sp. Fig. 10shows that high loading capacity of drug provided a fast release rate. Almost linear increase in protein release as the concentration of protein increases. Similar results were also reported by other researchers (Liu *et al.,* 1997). Amidi *et al*. (2010) stated that when protein concentration in CSNP was approximately 30%, it will fastly release in solution of buffer.

Effect of Mw and DD

Protein release studies of CSNPs prepared from high MW standard CS and low MW CS extracted from *Squilla sp.* were shown in figure 11. Figure revealed that BSA release rate is decreasing as the molecular weight is increasing. 130.18±26.80 kDa of CS shows higher release rate 86.61±3.65 %as compare with CS with MW 2743.92±76.43 kDaand release rate 42.38±0.91%.Ko *et al.* (2002) in his study found that as the viscosity of chitosan solution is increasing, molecular weight also increasing that lead to the decreasing drug release rate. Highly viscous chitosan solutions forms high cross-linking density of TPP-chitosan matrix that results in less swelling ability, hence the release of drug decreased. Fernandez *et al.,* (1999) reported that the amount and molecular weight of chitosan did not have significant effect on insulin response.

For the protein release studies DD is the main and most important factor than that of protein loading capacity. Due to the higher DD of chitosan, CS contains high number of ammonium groups that ironically interact with TPP. Hence, slow release rate of drug BSA results due to lower permeability of nanoparticles surface. In the present study, higher DD and Mw of standard CS had low drug release rate as compare to low molecular weight extracted CS*(50).*

CONCLUSION

In the present study, chitosan was extracted and purified from shells of *Squilla sp.* was conformed by FTIR, EDS and XRD and structural morphology by SEM and XRD. The various physicochemical properties of chitosan such as degree of deacetylation, WBC, FBC, viscosity and Mw was found to be 79

%, 548±11.77 % , 369±19.98 % , 1.64±0.26 cPs and 130.18±26.80 kDa respectively and it was compared with commercial chitosan that showed similar results. Further chitosan nanoparticles were prepared by ionic gelation method with the use of TPP as a protein delivery carrier. Various parameters such as TPP concentration, chitosan and BSA concentration, Mw and DD play an important role in protein delivery. At decreased concentration of BSA and chitosan, improved encapsulation efficieny was found but hither Mw and DD. The burst release of protein was observed at 6th of incubation of CSNPs in PBS. Low molecular weight CSNPs prepared from extracted chitosan showed higher release of protein as compare to high molecular weight CSNPs prepared from commercial chitosan. Hence, low molecular weight of chitosan is preferred for preparation of CSNPs as a protein delivery carrier.

FIGURES

Fig. 1 [A] "(a) FTIR spectra of commercial chitosan (b) chitosan nanoparticles"

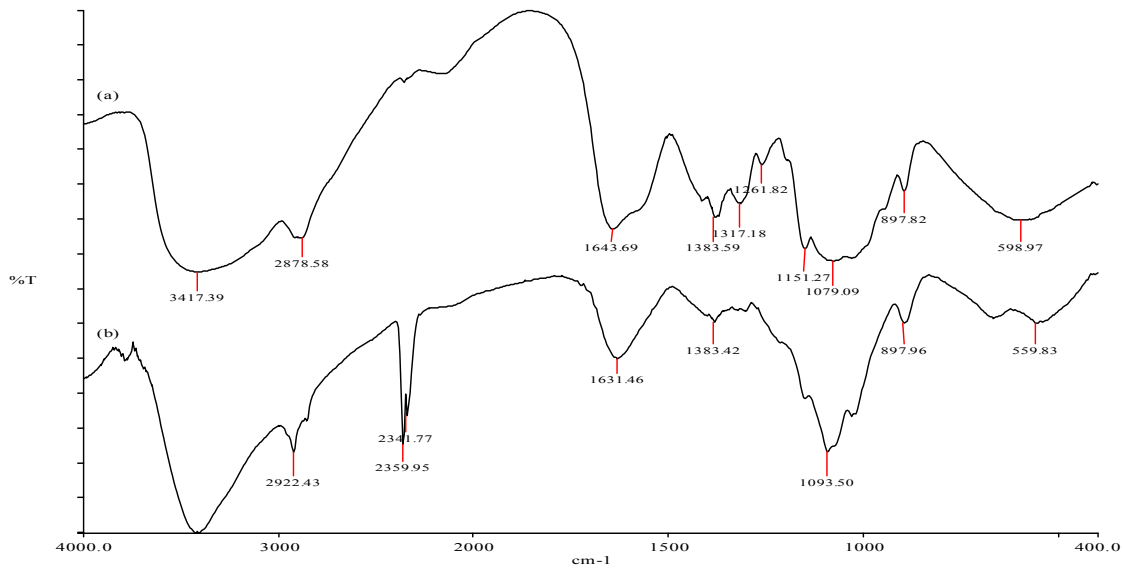


Fig. 1 [B] "(a) FTIR spectra of extracted chitosan (b) chitosan nanoparticles from extracted chiosan"

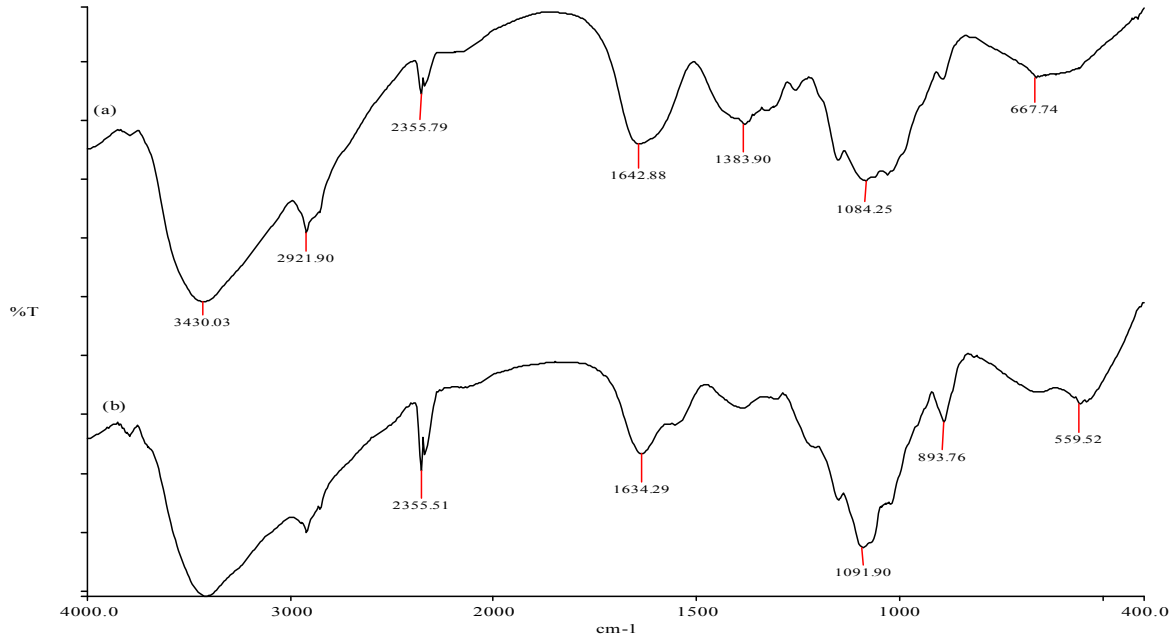


Fig. 2 "XRD crystallograph of commercial CS (a) and CS extracted (b)"

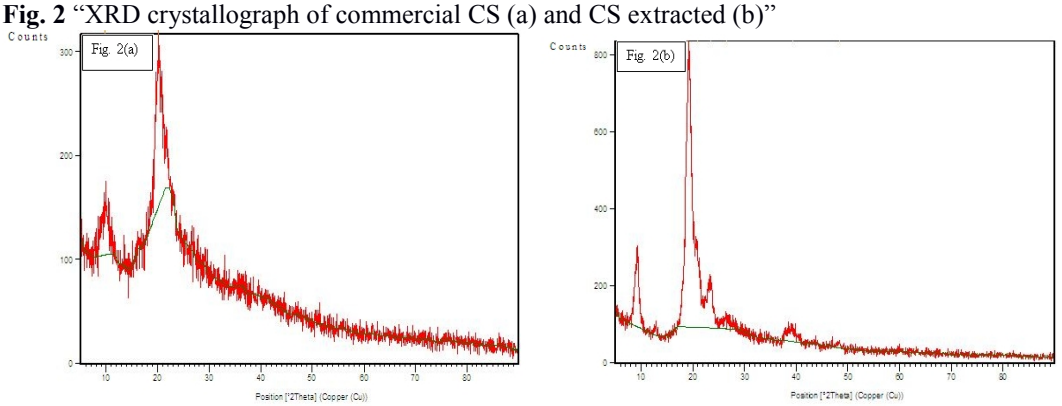


Fig. 3 "SEM images of commercial CS (a) and CS extracted (b)"

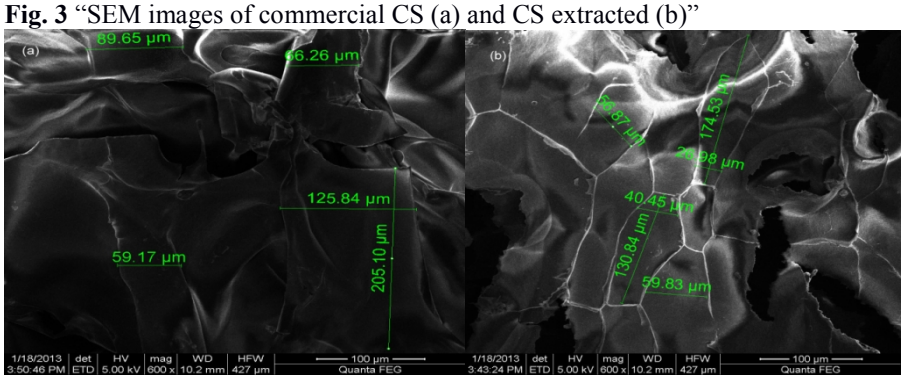


Fig. 4 'Elemental Analysis"

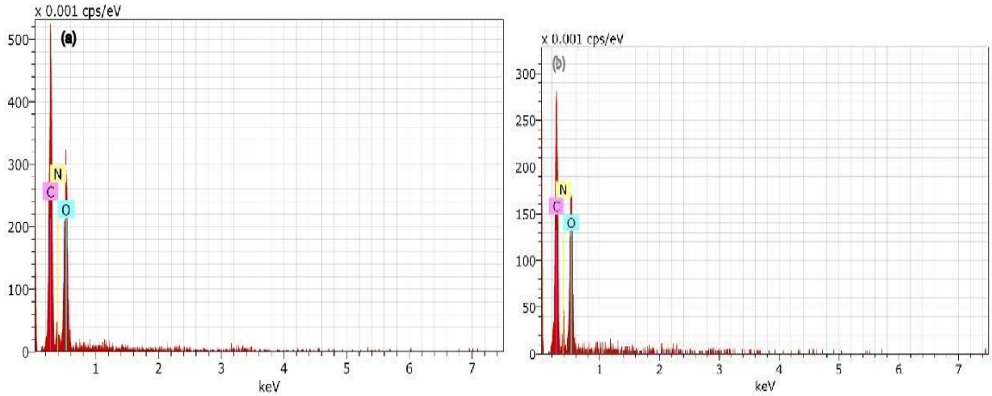


Fig. 5 "SEM images of CSNPs prepared from commercial CS (a) and from extracted CS (b)"

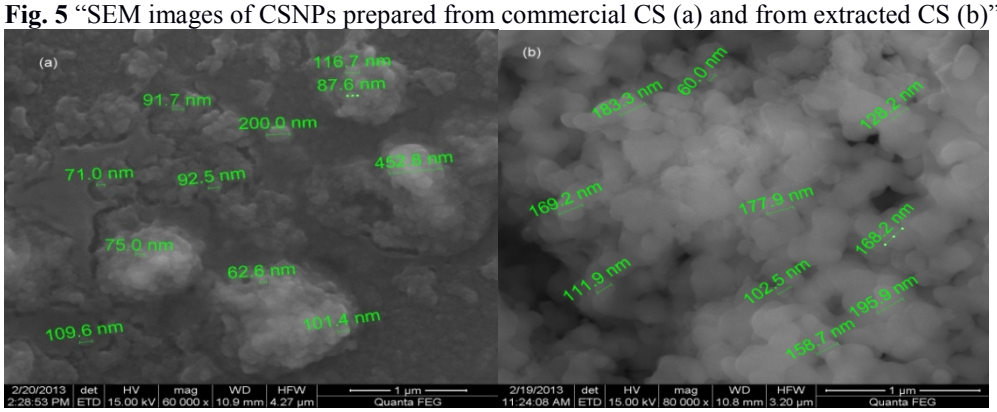


Fig. 6 "Effect of TPP cocentration on encapsulation efficiency (CS-1mg/ml, BSA-1 mg/ml)"

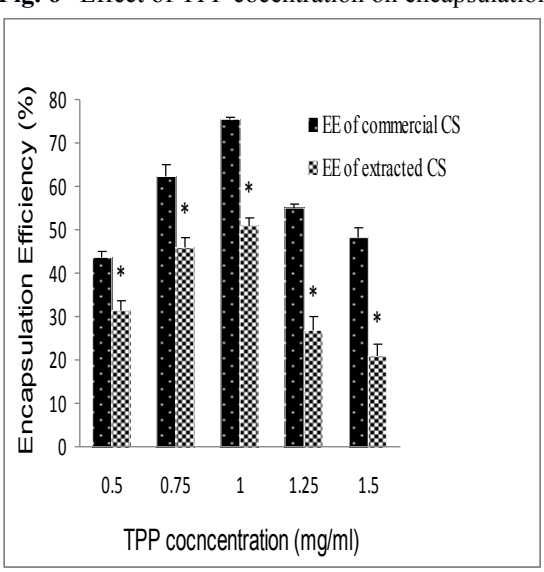


Fig. 7 "Effect of chitosan concentration on encapsulation efficiency (TPP-1 mg/ml, BSA-1 mg/ml)"

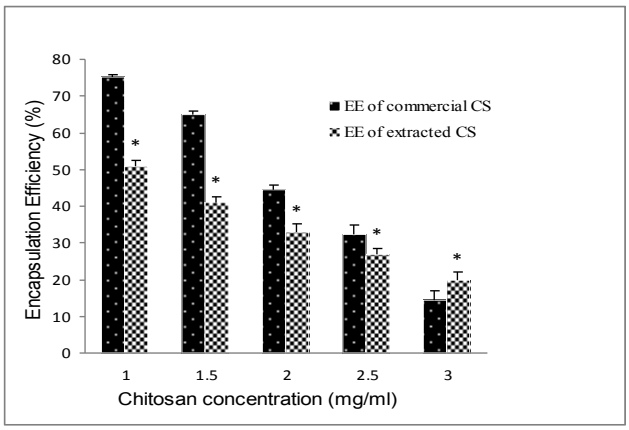


Fig. 8 "Effect of BSA concentration on encapsulation efficiency (TPP-1 mg/ml, CS-1 mg/ml)"

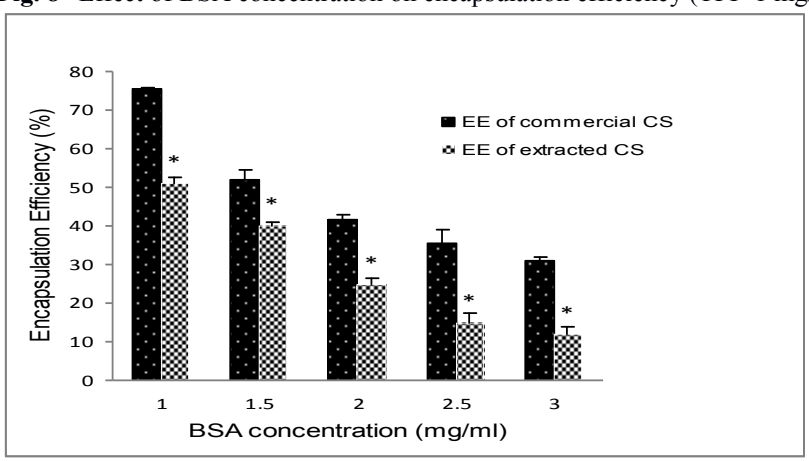


Fig. 9 "Effect of chitosan concentration on BSA release of CSNPs prepared from commercial CS (a) and CS extracted (b) (TPP- 1 mg/ml, BSA-1 mg/ml)"

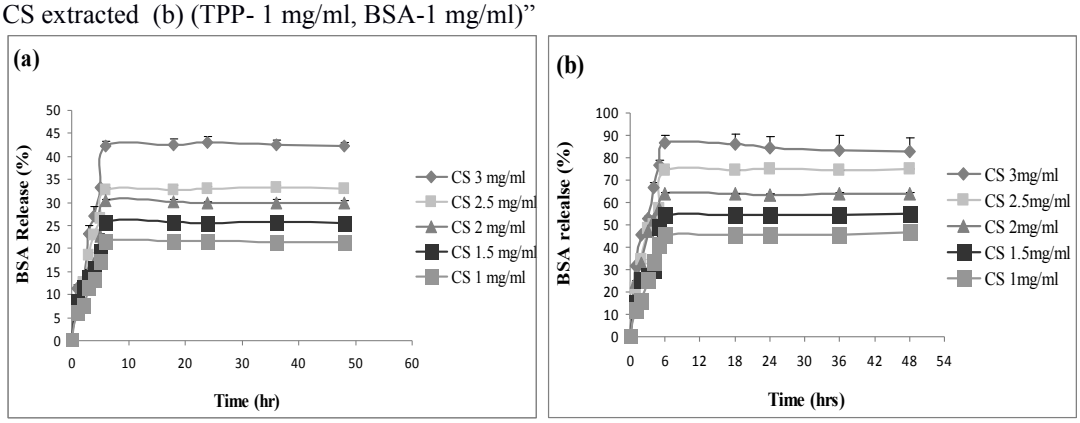


Fig. 10 "Effect of BSA concentration on BSA release of CSNPs prepared from (a) commercial CS and (b) extracted CS (TPP- 1 mg/ml, CS-3 mg/ml)"

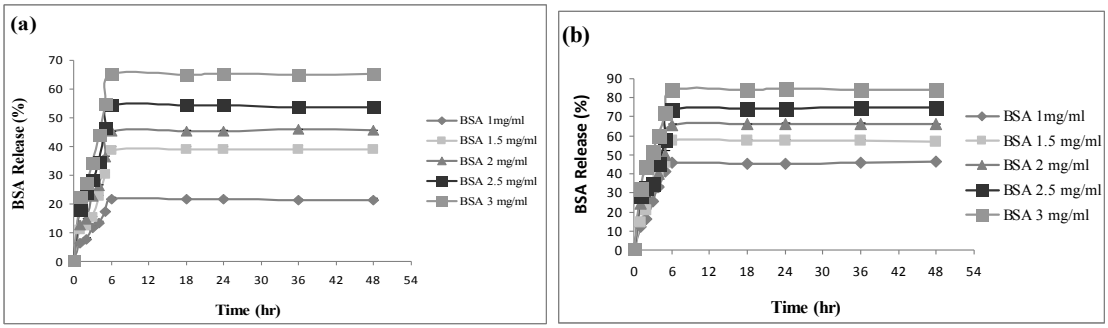
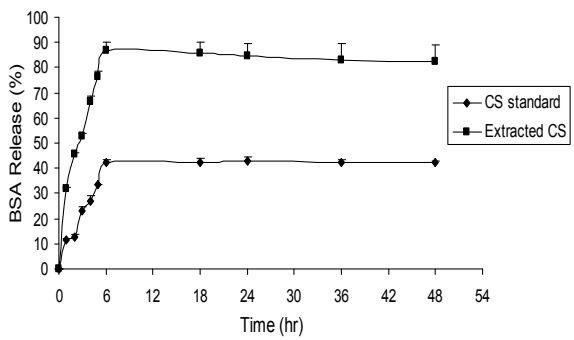


Fig. 11 "Effect of Mw and DD on BSA release (CS-1 mg.ml, TPP- 1 mg/ml, BSA-1 mg/ml)"



TABLES

Table 1. Proximate composition of *Squilla sp*. shells

Moisture - 21.95±0.3 **%**	**Protein -** 22.66±1.52 **%**
Carbohydrate - 1.95±0.46**%**	**Ash -** 18.03±0.62 **%**
Lipid - 7.1±0.46 **%**	

Table 2. Elemental analysis

Elements	Atom % of standard CS	Atom% of extracted CS
C	50.23	49.78
O	38.24	39.20
N	11.54	11.01

Table 3. Physicochemical characteristics of chitosan

	Commercial CS	Extracted CS

Viscosity (cPs)	16.4±0.40	1.64±0.26
Molecular weight (kDa)	2743.92±76.43	130.18±26.80
DD (%)	89.78	79
WBC (%)	573±4.98	548±11.77
FBC (%)	376±14.23	369±19.98

ACKNOWLEDGEMENT

I have taken efforts in this project. However, it would not have been possible without the kind support and help of many individuals and organizations. I would like to extend my sincere thanks to all of them.

I am highly indebted to Dr. R.A Nazeer for their guidance and constant supervision as well as for providing necessary information regarding the project & also for their support in completing the project.
I would like to express my gratitude towards my parents & member of School of bioengineering, SRM University, Chennai for their kind co-operation and encouragement which help me in completion of this project.
I would like to express my special gratitude and thanks to R.K University, Rajkot, Gujarat for giving me such attention and time.

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Comparative analysis of some natural and synthetic edible dye agents

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ABSTRACT

Turmeric and beet root are the natural edible dye which are use in our day today life.Turmeric can be used as powerful healers. Active ingredient which is found in turmeric is curcumini it is mild digestive, being aromatic. Beta vulgaris is is botanically name of beetroot which come under Chenopodiaceae family it consist of number of varieties in number of color rang from yellow to red. Most popular color of beet roots for human consumption is deep red colour, both cooked and raw as salad or juice. In United States amaranth food coloring was replace by Allura Red AC food colour which was originally introduced. It has been proven that tartrazine cause many different side-effects and allergic reactions in people.The natural dyeing can produce a wide range of colors even from the same plant source by applying different dying techniques, extraction methodology, solvent used etc.

SUMMARY

Natural dyes uses is much eco-friendly to human health therefore much use of natural dye should be done in the place of synthetic dye.Include a brief summary of your research work in exactly one sentence.

Keywords: 1, Aromatic 2, Chenopodiaceae 3, Healers 4, Ingredient 5 Tartrazine

INTRODUCTION

Turmeric is one of the natural healing systems since 5,000 year in India. It consist of large no of active ingredient and large no antioxidant properties. Large no of components have been isolated from turmeric. The main component of the turmeric root is a volatile oil, which contains turmerone, and all so have other coloring agents called curcuminoids. Tartrazine is defined as synthetic lemon yellow Azo dye primarily used as a food coloring also known as FD&C Yellow no. 5 and E 102. It is synthetic organic (carbon containing) chemical as well as water soluble. Beta vulgaris is botanically name of beetroot which come under Chenopodiaceae family it consist of number of varieties in number of color rang from yellow to red. Most popular color of beet root for human consumption is deep red color, both cooked and raw as salad or juice. Allura is used for number of products such as puddings, gelatins, custard, dairy products. Food colors are also added to different beverages including milk and milk-based drinks .Most of the natural colors in general belong to the either carotene or antho-cyanin families, which are heat labile.

MATERIALS AND METHODS

EXTRACTION OF NATURAL EDIBLE DYES ITS APPLICATION ON FIBRES AND COMPARATIVE STUDY WITH SYNTHETIC EDIBLE COLOUR:-

Material:-
A dark red variety of beetroot and turmeric are collected.
Substrates For dyeing silk fabrics desized bleached and scoured cotton were used

Bleaching:-

Bleaching is chemical process which is used for removal of natural colouring matter from the substrate. By doing the chemical bleaching chromophore is affected due to which double bond is break which effect source of natural color. The scored fabrics was bleached by treating with2 gms of ascorbic acid and 3 ml of sodium hypochlorite in 200 ml of water and maintained at 90°C for 10 mins. The fabric was dried and washed in sterile water.

Mordanting:-

Mordanting of the fabrics was performed using alum acetate/acetic acid equal to 5% of weight of the fabric (liquor ratio 1:40). The mordant bath was maintained at a temperature of 30°C for about 1hr along with continuous stirring serves as the firm fixative agent.

Chemicals :-

Alum, copper sulphate, stannous chloride and ferrous sulphate (*2*).

Methods:-

The dyeing of cotton with beet root and turmeric was carried out in four stages; Pre-Treatment, Extraction of dyes from beet and termeric, Mordanting (fixing dye with fiber) and dyeing. We carried out the extraction conventional method (*5*).

Coventional Method:-

The basic extraction process we carried out in our experiment was using conventional method that was the sample (beetroot, turmeric) from which dye was to be extracted was taken in round bottom flask, which was a measured quantity and solvent (methanol) was taken in the same round bottom flask and heated in water bath for about 30 min respectively. A condenser was fitted over the round bottom flask in order to condense the vapours coming out while healing. After the extraction time was completed, the residue with solvent was taken out and cooled. Then the filtration was carried out to separate the residue and solvent. The residue was weighed and dried and then once again weighed. This residue can be used as feeding material for plants (bio-fertilizer) (8).

Procedure of Dyeing of cloth: The mordanting samples are immersed in dye bath for 2 hr at a temperature range of 80 ^{O}C and after completion of dyeing; samples are taken out and dried. It is allowed to be aged for a fixed time, and after that soaped with 2 gm/L, non-ionic soap at room temperature for 10 minutes followed by rinsing and line dyeing.

Quality assurance tests of dyed fabric:

Large no of tests has to perform for knowing the all important properties of dyes. As large numbers of dyes are organic compounds so it vulnerable in varying degree to the action of destructive agents. Only selected dyes are used. In dyes numbers of tests are necessary to perform for covering all important properties of dyes. Because of good fastness to one inferior evolution and characterization therefore some test has to be performed in selected fabrics: Rubbing fastness. Washing fastness, and Light fastness (7).

Light Fastness:-

Comparison has been done in the colored portion which is exposed to the light for 24 h with unexposed color material portion (*6*).

Washing Fastness:-

Total 3 sample has been taken in which one white sample of dye is placed between two non dyed pieces and then three sample are held together b stitching the edges. Soap soln pre heated in 60°C at ratio of 1:50 i.e 0.5g/25 mL water. Removal of specimen and cooling is done. So washing fastness recognized by presence of colour in control sample (*6*).

Rubbing fastness:-

The rubbing fastness is mainly done for checking fading of color by rubbing dyed fabrics (*1*).

3.2 Phytochemical Screening

Analysis of phytochemical consists of edible natural and synthetic dyes have been done to evaluate the quality and purity of the extract. The determination is important as it indicates purity of material i.e; presence or absence of any foreign matters.

The following species were selected for present investigation.

1. Curcuma longa
2. Beta vulgaris.
3. Tartrazin (Lemmon yellow).
4. Allura red (red tomato).

Preliminary Phytochemical Screening

The whole plants were used for preliminary phytochemical screening. The plant material was washed thoroughly, dried in shade, ground and stored in paper bags at room temperature. These plant samples were screened to detect different phytochemical. For this, experimental procedure given below was followed.

Experimental procedure:

According to the standard procedures preliminary phytochemical screening of plant was done which was adopted by the various workers (Amarsingham *et al.,* 1964; Das and Bhattacharjee, 1970; Gibbs, 1974; Harborne, 1984,; Chhabra *et al*., 1984; Trease *at el.,* 1985; Danial, 1991).

For 10 days at room temperature the whole plant was dried the dried plant material was milled and ground into fine powder. 10 gm of dried plant material is taken in 100 ml **Petroleum ether** for about 12 - 16 hrs. The extract was filtered by Whatman No.1 filter paper. The extract was collected and residue was dried overnight. The residue was taken in 100 ml **Acetone** for about 12-16 hrs. The extract was filtered and collected and the residue was dried overnight. The residue was taken in 100 ml **Methanol** for about 10-12 hrs. The extract was collected and residue was discard. The petroleum ether, acetone and methanol

extract was tested for the presence of alkaloids, flavonoids, steroids, carbohydrates, saponins, tannins, glycosides, triterpenoids, amino acids and proteins.

Preliminary screening for different phytochemical constituents:

1. Alkaloids:

A few mg of each extract was taken separately in 5ml of 1.5% v/v aqueous hydrochloric acid and filtered. Resulting acidic solution should be divided into four parts. Three parts were tested with **Mayers**, **Wagner** and **Dragendroff's** reagent and the fourth served as blank.

1. **Wagner's Test:** Take 1 ml of extract and add 2 ml **Wagner's reagent**. A brown flocculent precipitate obtains which indicated the presence of alkaloids..
2. **Dragendroff's Test:** Take 1 ml of extract add 2 ml **Dragendroff's reagent** Development of orange precipitate obtain which is the positive test for alkaloids (Dan *et al.,* 1978).

Reagents:

1) **Wagner's reagent:** 1.27gm of iodine and in 5ml of distilled water 2gm of potassium iodide were dissolved and solution was diluted to 100ml.

2) **Mayer's reagent:** 1.36gm of mercuric iodide was dissolved in 60ml of distilled water, solution were mixed and diluted to make up volume 100ml.

3) **Dragendroff's reagent**: Dragendroff's reagent was prepared by mixing **Solution A** and **Solution B** in 1:1 v/v proportion in 7 ml of concentrated hydrochloric acid and 15 ml distilled water.

Solution A: 0.6gm of bismuth sub nitrate was added in 2ml of concentrated hydrochloric acid dissolve in 10 ml of distilled water.

Solution B: 6 gm of potassium iodide in 10 ml of distilled water.

2. Amino acids and Proteins: **1) Ninhydrin test:** violet purple colour indicated the presence of amino acid when 0.1 % ninhydrin n acetone solution was added to extract.

Ninhydrin reagent: 0.1 gm of ninhydrin dissolved in minimum quantity of acetone and make volume up to 100 ml by adding more acetone.

2) Xanthoprotien test: white precipitate formation takes place when 1 ml of extract was added in 1 ml of concentrated nitric acid which indicates presence of protein. Then boiled and remain cooled. On adding 20 5 of sodium hydroxide or ammonia. Then orange colour indicates the presence of amino acid.

3. Carbohydrates and Reducing sugars:

All extracts were tested for the presence of sugars by following tests:

1) **Molish's test:-** 1 ml of extract was placed in a test tube containing 0.5ml of water and mixed with 2 drops of 10% solution of 1-napthol in alcohol. 1 ml of conc. H_2S0_4 was added from the side of test tube. Appearance of red-violet ring at junction of two layers indicated the presence of **carbohydrates**.

2) **Iodine Test:** 1 ml of extract was placed in a test-tube. Add 3 drops of iodine. Appearance of blue colour indicates the presence of starch.

3) **Fehling's test**: few ml of extract taken in test-tube then add few drops of Fehling's solution .heat for 5 minutes. Brick red precipitate forms that presence of carbohydrates and reducing sugar.

4) **Benedict's test:** take 1 ml of extract add 2-3 drops of **Benedict's reagent.** Heat for 2 minutes. Formation of red precipitate indicates the presence of reducing sugar.

4. Flavonoids:

For the presence of flavonoids by **Shinoda's reaction** (Shinoda, 1928) and NaOH test **a**ll extracts were tested

a) **Shinoda's reaction**: A small quantity of plant extract dissolves in 5 ml of ethanol. When Magnesium powder, concentrated hydrochloric acid is added yellow/ red colour generated which indicated the presence of flavonoids.

b) **NaO**H **test:** 0.2 gm of extract dissolves in diluted NaOH. Add few drops of 1% hydrochloric acid. A yellow solution turns colourless indicates the presence of flavonoids.

5. Glycosides:

All extracts were tested for the presence of glycosides by **Keller-Killani test.**

1)Keller-Killani test: 0.5 ml of alcoholic extract is taken in test-tube. Then add 2ml of 3.5% $Fecl_3$ and 2ml of glacial acetic acid then add 2 ml of concentrated H_2SO_4**.** Reddish brown ring obtain at the interphase of the test-tube indicates presence of glycosides.

6. **Steroids**:

All extracts were screened for the presence of steroids. The test carried out by Salkowski reaction and Libermann-Burchard reaction (Maiti, 1968).

1) **Salkowski Reaction:** In 2ml of the chloroform a few mg of residue was taken and by the side of the test tube the 2ml of conc. H_2SO_4 was added. For few minutes test tube was shaken. In chloroform layer the red colour was developed and acid gave greenish yellow fluorescence at lower layer. This colourisation and fluorescence is due to presence of steroids.

2)**Libermann-Burchard reaction:** A few mg of the residue was dissolved in chloroform. To this was added few ml of acetic anhydride and two drops of conc. H_2S0_4 from the side of the tube. The transient greenish colour indicates presence of steroids.

7. Triterpenoids:

For knowing the presence of triterpenoids all extracts were tested by **Libermann-Burchard** reaction. In chloroform a few mg of the residue was dissolved. To this a few ml of acetic anhydride and two drops of conc. H_2S0_4 added from the sides of the tube. The red/ violet colour indicates the presence of triterpenoids.

8. Tannins:

Take few mg extract and concentrated with solvents. Take 0.5 ml of extract and dilute with 1ml of distilled water. Add 2-3 drops of 1 ml of ferric chloride solution. OR.

1 drop of ferric chloride added to 2 ml of extract. Formation of blue-black / greenish black colour indicates presence of tannins.

9. Phenols:

The extract was dissolve in water. 2 ml of ferric chloride solution added to water solution. Formation of green or blue colour indicates phenols.

10. Saponin:

To 5ml of the extract, 5ml of distilled water added and shaken vigorously a warm condition. The formation of honeycomb like foam taken as the indication for presence of saponins.

RESULTS AND DISCUSSION

The qualitative phytochemical analysis of the methanolic plant extracts represented in the Table-1 showed the presence and absence of bioactive components like alkaloids, tannins, terpenoids, phenols, steroids, flavanoids, glycosides, saponins, carbohydrates and proteins, reducing sugar in the selected plant extracts and synthetic edible dyes. It was observed that among flavanoid, saponin, carbohydrate, terpenoids, steroids, tannins, phenols tests. Flavanoids and alkaloid (Wagner's test) present in tartrazine while carbohydrate is present in beetroot and turmeric plant materials, Fehling's test presence partially seen in beet and turmeric, Benedict's test presence only seen in beetroot. Terpenoids are only absent in beet present in remain 3 dyes, Glycosides were present in beetroot, tartrazine and absent in remain 2 dyes, steroids present in beet and partially in tartrazine in salwoski test but in Liebermann test absent in all 4 dyes. Amino acids absent in all 4 dyes, but in xantho-protein test for amino acid present in turmeric and absent in beetroot, allura and tartrazine. The natural dyeing can produce a wide range of colors even from the same plant source by applying different dying techniques, extraction methodology, solvent used etc. In this study seven plant source were chosen for dyeing of cotton fabrics. Each plant source gives different color shades to the fabrics.

The dyeing of the cotton fabrics with these plant extract give different shades of color. These color shading depends on the mordant used during the dying process. Mordants were used for the fixing of the dye to the fabrics. Alum acetate/acetic acid were used as the mordant.

CONCLUSION

The natural dyeing can produce a large range of colors even from plant the plants having same source by applying different dying techniques, extraction methodology, solvent used etc. In this study two plant source and two synthetic colors were chosen for dyeing of cotton fabrics. Each plant source gives different color shades to the fabrics.

The qualitative phytochemical analysis of the methanolic plant extracts represented in the Table-3 showed the presence and absence of bioactive components like alkaloids, tannins, terpenoids, phenols, steroids, flavanoids, glycosides, saponins, carbohydrates and proteins, reducing sugar in the selected plant extracts and synthetic edible dyes. It was observed that among flavanoid, saponin, carbohydrate, terpenoids, steroids, tannins, phenols tests. Flavanoids and alkaloid (Wagner's test) present in tartrazine while carbohydrate is present in beetroot and turmeric plant materials, Fehling's test presence partially seen in beet and turmeric, Benedict's test presence only seen in beetroot. Terpenoids are only absent in beet present in remain 3 dyes, Glycosides were present in beetroot, tartrazine and absent in remain 2 dyes, steroids present in beet and partially in tartrazine in salwoski test but in libermann test absent in all 4 dyes. Amino acids absent in all 4 dyes, but in xantho-protein test for amino acid present in turmeric and absent in beetroot, allura and tartrazine.

The dyeing of the cotton fabrics with these plant extract give different shades of color. These color shading depends on the mordant used during the dying process. Mordants were used for the fixing of the dye to the fabrics. Copper sulphate, ferrous sulphate and natural mordant lemon were used as the mordant. The cotton and synthetic dyed fibers shows different colors.

Quality assurance tests of dyed fabric:

Light Fastness:

Edible dyes which are extracted from beetroot, turmeric, allura and tartrazine shows good light fastness. This happen due to forming of complex with the metal which protects the chromatophore from photolytic degradation. Electron density increase as a particular substituent so as a result either oxidation or reduction takes place.

Wash fastness:-Wash fastness of the dye is influenced due to rate of diffusion which takes place in dye. The dye which is extracted from beetroot and turmeric exhibits good or excellent washing fastness as compare to allura and tartrazine synthetic dye. By using different mordant insolubilizing of dye has been

effected, making it color fast. Electron density increase due to particular substituent of the molecule facilitating oxidation, or in case of reduction electron density with a resultant.

Rubbing fastness:-

Dye which is extracted from the fibres of marigold flower shows good rubbing fastness. Mordant which are used in fibre shows insolubility of dye this is due to mixing mordant with dye. Dye which is obtain from marigold flower shows good fastness properties.

From this current study it was found that the natural dye extracted from the different plant source can be successfully applied to the cotton fabrics to obtain a wide range of color shadings along with the application of the mordant as a fixative agent. These dyes are environmental friendly and harmless when compared to the synthetic dyes. By using vegetative fixing agents color fastness of the dye can be increased.

Natural colours are easier to metabolize than synthetic counert parts. As compared to synthetic dyes Natural dyes produce large number shade. On the other hand, synthetic dyes which widely available at an economical price causes large no of allergy to skin harmfulness to human body produce toxicity chemicals etc (R.Rajendran and B.Thamarai Selvi, 2014).

Natural pigment promotes consumers health protection like pigments or colour obtain from Beta vulgaris for making lipsticks have no side effects on lips (*4*).Without synthetic mix natural pigments allows manufacturing of fully eco-friendly pigment. Occurring organic pigments have been completely displaced by synthetic molecules. In most of the cases of scientific studies it has been proved that both dye properties are comparable with each other. Large much more research and developmental should be done in this area. Traditional practices should be substituted more scientific practices in order to overcome some of the so-called disadvantages of this dye.

TABLE

The preliminary phytochemical investigations of methanolic extract of beetroot, turmeric, allura and tartrazine was performed which shows following results.

Table-1.Phytochemical screening of extract

SR.NO.	TEST FOR	TEST NAME	PLANT MATERIAL

			Beetroot	Allura	Turmeric	Tartrazine
1	**Flavanoid test**	Flavanoid	-	-	-	++
2	**Saponin test**	Saponin	-	-	-	-
3	**Alkaloid test**	Dragandroff's test	-	-	-	-
		Wagner's test	-	-	-	-
		Molish's test (For carbohydrate)	+++	-	+++	-
4	**Carbohydrate and reducing sugar**	Iodine test (For Starch)	-	-	-	-
		Fehling's test (For carbohydrates and reducing sugars)	++	-	++	-
		Benedict's test (reducing sugar)	+++	-	-	-
5	**Glycosides**	Kelle-kilani test (glycoside)	+++	-	-	+++
6	**Tannins**	Tannins	-	-	-	-
7	**Phenols**	Phenols	-	-	-	-
8	**Amino acid**	Ninhydrine (For amino acid)	-	-	-	-
		Xanthoprotien (For protein)	-	-	-	-
		Xanthoprotien (For aminoacid)	-	-	+	-

9	**Steroids**	Salwoski reaction (For steroids)	+++	-	-	++
		Libermann Burchard (for steroids)	-	-	-	-
10	**Terpenoids**	Terpenoids test	-	+++	+++	+++

ACKNOWLEDGEMENT

The work presented in this dissertation was performed at botany department university school of sciences, Gujarat University, Navrangpura, Ahmedabad, December 2014 to April 2015. I would like to thanks Dr. Archana Mankad head, department of botany for granting me permission for dissertation and supporting my scientific development. I also thank Dr. H.A solanki for their valuable impacts in this project. I would like to express my earnest gratitude to Ms. Hiral research scholar in department of botany, Gujarat University School of sciences and my project supervisor for her invaluable guidance, encouragement and support. The lesson I have learned during my lab work will go with me throughout my career.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

(Proceedings available for download at rku.ac.in/icre)

RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

Biosorption of Copper by *Aspergillus sp* from e-waste leachate

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ABSTRACT

The removal Copper E-waste leachate was studied using untreated biomass of *Aspergillus Sp.* Study of various physico-chemical characteristics of leachate was carried out, the metal concentration in leachate is >5000 µg/ml. Different heavy metals present in leachate was Cu, Ni, Cd, Pb, Fe, Cr, Ag etc in different concentration.Leachate was Light green colored, transparent, acidity (200mg/l as per IS), chloride (250mg/l as per IS) and sulphate (200 mg/l as per IS) above the minimum permissible limit as given by standards. So, the leachate cannot be discarded in the stream without any treatment.The effect of different parameters like, variation in p^H, Contact time, Metal concentration, Biomass concentration for biosorption study of copper was carried out. Maximum sorption capacity of Copper is obtained at p^H 5, contact time obtained is 24 hrs, higher % of removal of copper is achieved at 1:5 dilution, at 0.2 g biomass concentration metal uptake is higher.

SUMMARY

Aspergillus sp an efficient biosorbent can use for biosorption of Copper from e-waste leachate.

Keywords: *Aspergillus sp, Biosorption, E-waste leachate*

INTRODUCTION

There is increased use of electronic equipment in day to day life due to modernization and industrialization. That's why electronic waste generated in huge quantity and become serious problem in the world because in the era of the technology increase disposal in to the environment and also result in adverse impact on environment and consequences to human health. E-waste is classified as about 39% heavy metals come from e-waste that is hazardous to human health and environment. And cause various health impact on humans.

Mercury and cadmium containing electronic devices are landfilled both of them leached out into the soil and groundwater. (Jefferies and Firestone, 1984). For the removal of Cu from wastewaters conventional physical and chemical methods, like ion-exchange resins, reverse osmosis, reduction and precipitation and coagulation, these methods are not very effective and also costly and generate toxic compounds . (Bai and Abraham *2001*). To overcome this problem one most effective alternative is biosorption of heavy metals using biological material from waste water. It employs the use of divergent biomass such as seaweed (Yun et al. *2001*), microalgae (Gupta et al. *2001*), fungi (Sag and Kutsal *2002*), bacteria (Nourbakhsh et al. *2002*) and various other plant materials (Sharma and Forster *1993*; Raji and Anirudhan *1998*; Gardea-Torresdey et al. *2000*). The world Health Organization (WHO) recommended a maximum acceptable concentration of Copper in drinking water 1.5 mg/l (Devis, 2010).Copper concentration more than permissible limit cause Wilsons disease and Respiratory difficulty and gastrointestinal bleeding (Danis, 2010) Many authors have reported copper remediation by bacteria, fungi, yeast and algae (Cervantes and Guitierrez, 1994; Qureshi et. Al, 2001; Beolchini et al, 2004; Vijayaraghavan et al 2004; Zaki and Farang, 2010).The isolates of *Aspergillus* sp., *Penicillium* sp. And *Cephalosporium* sp. were tested to evaluate their applicability for heavy metal (Cu, Cd and Pb) removal from industrial wastewaters. The biosorption of dead fungal cells of *Aspergillus* sp., *Penicillium* sp. and *Cephalosprium* sp. Adsorbed Cu (46%), Cd (95%) and Pb (70%) respectively (Hemambika, et al., 2011). Various parameters such as contact time, pH, initial Cu concentration, biomass concentration and temperature influencing Cu biosorption were studied using different biomaterials.

MATERIALS AND METHODS

Sample and culture collection

Aspergillus sp. was procured from Department of Microbiology, Gujarat university, Ahmedabad, India. Collected microorganism was maintained in the medium slant viz. potato dextrose agar medium for *Aspergillus sp.* used for the experiment. Biologically generated electronic waste leachate was procured from Department of microbiology, Gujarat University Ahmedabad, India.

Preparation of biomass

Aspergillus niger was harvested from the medium by physical method filtering and then medium was removed by washing with distilled water. Then both of the biomass was dried in hot air oven at 60 °C for 24 h and then grounded to sieve size 0.7 mm. The sorption experiments was carried out by using prepared biomass.The test solution prepared by diluting stock solution to obtain desired concentration from leachate.

The metal sorption monitored for pH range 2,3,4,5 and 6, pH of leachate was adjusted by 0.1N NaOH or H_2SO_4 (John peter). The sorption experiment was carried out in 250 ml flask with optimum pH and 0.1 g of *Aspergillus sp.* biomass for time interval 1,2,3,4,5 and 24 hrs ;for metal concentration study different dilution in the range 1:2, 1:3, 1:5, 1:10, 1:20 and 1:50 of the original leachate taken; Various concentration of biomass viz. 0.05, 0.1, 0.2, 0.3, 0.4 and 0.5 g at the optimum pH added into 50 ml leachate system.

Biosorption experiment was carried out by 50 ml 1:5 diluted original leachate system and same amount of biomass was added and put it on rotary shaker 150 rpm and reading was taken at suitable time period by SDDC method Optical density was taken at 560 nm and Copper concentration was found from the standard curve and % sorption was calculated using following formula,

$$s = \frac{in - f}{ini} \times 100$$

The amount of metal uptake by the biosorbents was calculated as follows,

$Q = v (C_i - C_f)/m$

Where, Q = Metal Uptake (mg metal per g biosorbent),

v = Liquid Sample volume (ml),

C_i = Initial Concentration of the metal in the solution (mg/l),

C_f = Final concentration of the metal in the supernatant (mg/l),

m = Amount of the added biosorbent on the dry basis (mg). (Yasmin K 2009)

RESULTS AND DISCUSSION

Standard Copper curve :

The standard Copper graph performed using $CuSO_4.5H_2O$ solution, containing 100 ppm concentration. Standard curve used for detection of unknown concentration of Copper in the leachate. Standard curve given in figure 1,

Effect of p^H of solution on Copper sorption

The most important single parameter influencing the sorption capacity is the pH of the adsorption medium. (Goyal et al., 2003). The solution pH plays a major role in biosorption, and seems to affect the solution chemistry of metals and the activity of the functional groups of biomass(vijayaraghvan). For metals, the pH strongly influences the speciation and biosorption availability of the metal ions (Yang and Volesky 1999; Esposito et al 2002). The activity of binding site can also be altered by adjustment of pH. The influence of pH on percentage of sorption and metal uptake is given in figure 2

Figure 2, summarizes the results of the adsorption of Copper ions by *Aspergillus sp* biomass as a function of pH. Maximum metal uptake occurs at pH 5 in the biomass. From this study, we can conclude that at pH 5 maximum sorption of Copper occurs in the biomass. At the lower pH sorption of copper is decreased may be due to protonation of functional group present on cell wall. Furthermore at the more than optimum sorption of copper also decreased may be due to formation of metal hydroxide (chemical precipitation) (Kapoor et al.*1999*).

Effect of contact time on Copper sorption:

The absorption experiments of Copper were carried out for different contact times with a fixed adsorbent dose of 0.1 g / 50 ml system at pH 5 at 30 ± 2 °C. Generally, the biosorption capacity and the % removal efficiency of copper ion by biosorbent become higher with increase contact time. The effect of contact time on the sorption of copper by the both of the biomass *Aspergillus sp* was investigated over the interval from 1, 2,3,4,5, and 24 h.

The figure 3, shows that maximum copper sorption occur in *Aspergillus sp* at 5 h.The % sorption was of *Aspergillus sp,* it gradually increase due to larger surface area of the biomass being available for the adsorption of copper. (Nadaroglu 2015)The copper sorption was rapid due to availability of more number of active site on biomass. As a time passes % sorption slow down due to interference of other metal presence in leachate.

Effect of initial copper ion concentration on Copper sorption:

The adsorption experiment was carried out by making different dilution of leachate. The percentage sorption of copper was decrease with increase in dilution of leachate.

Figure 4 shows that at percentage removal of copper was increase with 1:5 dilution that is 27.5% in *Aspergillus sp* that is maximum due to availability of active site is higher up to this dilution. Table 11 shows that Metal uptake is higher at 1:5 dilution in both biomass. In 1:3 and 1:2 dilution percentage removal and metal uptake of copper decreased due to concentration of copper ion was higher in leachate and saturation of biomass occur.

Effect of biomass concentration on Copper sorption:

The influence of biomass concentration on the percentage sorption of copper was studied using different concentration of both of the biomass. To achieve the maximum biosorption capacity of *Aspergillus sp* for copper, the biomass concentration varied from 0.05 to 0.5 g.

Figure 5 shows that 0.1 g biomass concentration was required for maximum percentage of copper removal that is in case of *Aspergillus sp* 0.2 g biomass concentration was required for maximum percentage of copper removal metal uptake is higher. It is also seen from this result, further increase in biomass does not affect the sorption of Copper greatly. This may be due to blockage of binding site with aggregate formation of biomass.

The given table suggest the metal uptake by *Asprgillus sp* with different parameter study like p^H, contact time, initial metal concentration and biomass concentration.

CONCLUSION

For the removal of heavy metal from environment microorganisms are play very significant role. In this study, biosorbents i.e. with *Aspergillus sp* was carried out with different environmental conditions. *Aspergillus sp* Maximum sorption capacity of Copper is obtained at pH 5 . Further, in 1440 min contact time, at copper concentration 1279 ppm and 4g biomass concentration is optimised from the test organism. From the result obtained, *Aspergillus sp* are efficient for Copper removal from e-waste Leachate.

FIGURES

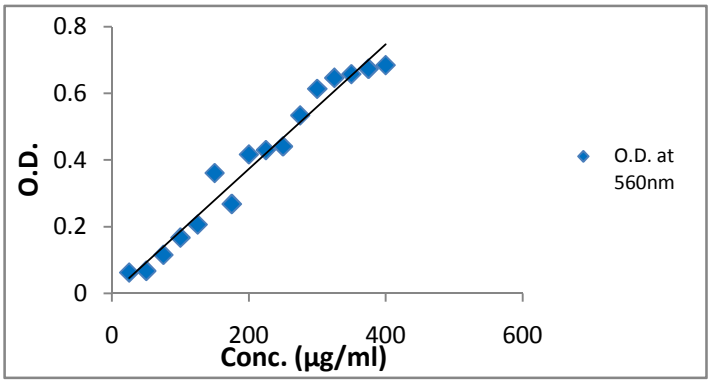


Figure 1: Standard curve of Copper

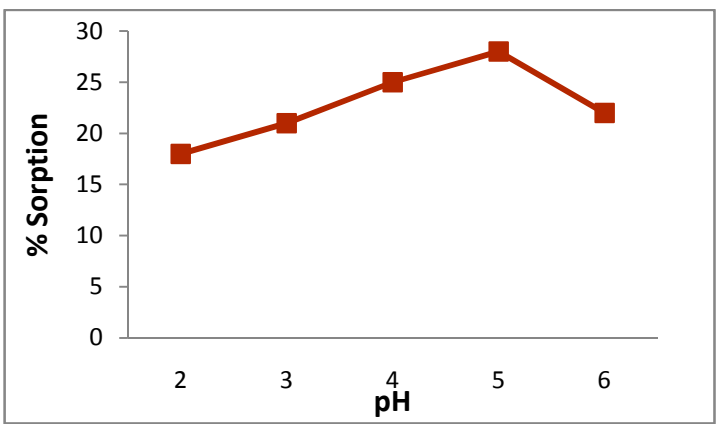


Figure 2: Effect of pH on %sorption

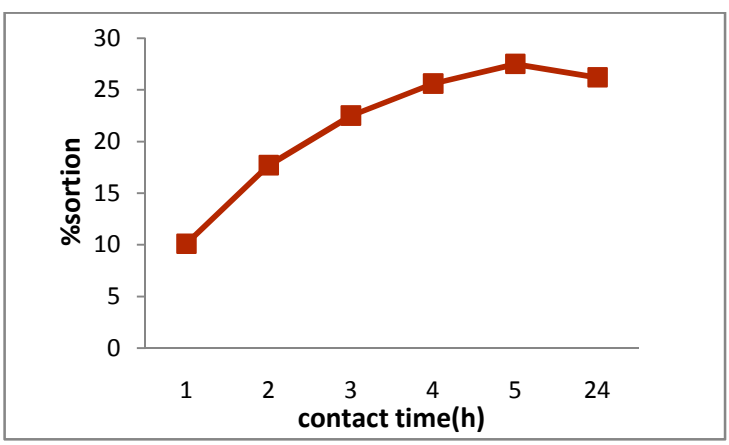


Figure :3 effect of contact time on %sorption

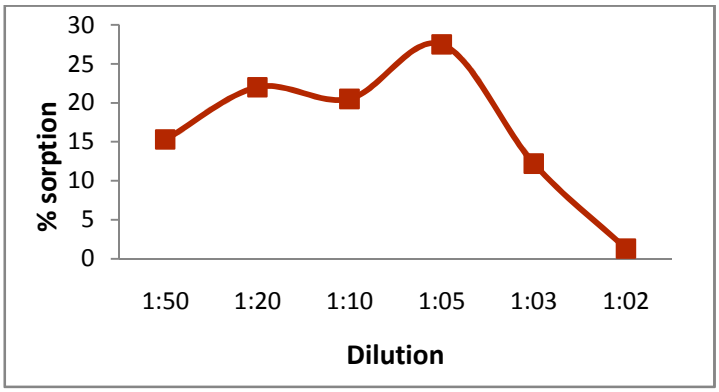


Figure: 4 Effect of initial metal concentration on %sorption

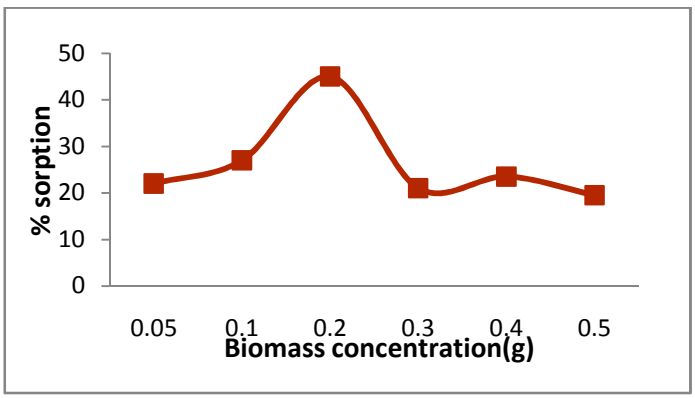


Figure: 5 Effect of biomass concentration on %sorption

TABLES

Table :1 Metal uptake by Aspergillus sp for various parameters

Aspergilus sp	P^H	q_e (mg/g)	Contact Time (hrs)	q_e (mg/g)	Initial Cu^{+2} Concentration Dilution	q_e (mg/g)	Biomass Concentration (g)	q_e (mg/g)
	2	121.3	1	65.15	1:50	1.33	0.05	141.3
	3	134.6	2	113.25	1:20	6.91	0.1	176
	4	164	3	144	1:10	9.63	0.2	288.3
	5	182.7	4	164.5	1:5	35.2	0.3	134.62
	6	145.3	5	176.1	1:3	18	0.4	150.6
			24	184.5	1:2	3.21	0.5	125.26

ACKNOWLEDGEMENT

We gratefully acknowledge Dr. S. R. Dave, Dr. Devyani Tipre, Dr. Baldev Patel, Dr. Meenu Saraf, Ms. Kruti shah and Ms. Monal Shah, Laboratory assistant for their intimate support and Department of Microbiology and Biotechnology, Gujarat Univrsity.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

(Proceedings available for download at rku.ac.in/icre)

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Effect Of Heavy Metals On Biomass And Growth Rate Of Select Pulses

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ABSTRACT

Living cells need metals as micro molecules to do their biochemical functions. From the elements discovered so far few are essential, some are useful whereas few show adverse effects on living organisms. Those which are useful required in very minute amount and their higher concentration cause harm or pose adverse effects on living forms. Copper and manganese are in general counted as essential metals however; chromium is reported as useful in some plants. From the present investigation it was found that higher concentration of copper and manganese showed reduction in biomass and growth rate of all three investigated plants. However, chromium pretence adverse effect on biomass and growth rate of experimental plants even at low concentrations.

SUMMARY

Plants can be effectively used for remediation of copper and manganese heavy metals i.e. control tools for heavy metal pollution.

Keywords: Heavy metals, Biomass, Growth rate, Glycine max, Vigna unguiculata Vigna aconitifolia

INTRODUCTION

Life on this planet has evolved in the presence of metals. Metals have been mined and used since ancient times. Cells learned to make use of the more abundant metals in the Archean oceans at an integral component in their structure and function. Today, we inherit these as the essential metals. The industrial era has seen a sharp increase in both the amounts and variety of metals that have application in industry *(10)*.

All things in nature ultimately succumb to decay. Much of this is a natural consequence of the laws of thermodynamics. Many molecules degrade by the action of oxygen, halogens and radicals naturally found in the environment *(63)*.

Modern industry is to a large degree, responsible for contamination of the environment. Lakes, rivers and oceans are being overwhelmed with bacteria, and wastewater. Among toxic substances reaching hazardous levels are heavy metals *(67)*.

Many uses of heavy metals in several applications lead to their wide distribution in soil, slit, waste and wastewater. Such a pollution of the environment by toxic metals and radio nucleotides arises as a result of many human activities, largely industrial, although sources such as agriculture and sewage disposal also contribute *(13)*.

Heavy metals are among the conservative pollutants that are not subject to bacterial attack or other breakdown or degradation process and are permanent additions to the environment *(16, 34)*.

These metal contaminants pose adverse health effects to those who live near these polluted sites. Breathing, eating, drinking, and skin contact are all possible exposure routes for metal contaminants. Metals such as mercury, lead, and arsenic, potentially can be toxic to the kidneys, decrease mental capabilities, and cause weakness, headaches, abdominal cramps, diarrhoea and anaemia *(66)*. Chronic exposure to these pollutants can cause permanent kidney and brain damage *(66, 1)*.

To solve the water pollution problem by toxic heavy metal contamination resulting from human's technological activities has for long presented a challenge *(67)*.

A key factor to the remediation of metals is that metals are non-biodegradable, but can be transformed through sorption, methylation, and complexation, and changes in valence state. These transformations affect the mobility and bioavailability of metals.

Adsorption, ion exchange, precipitation and complexation with organic matter are mechanisms that limit the amount of metal leaching through surface water or groundwater *(11)*.

There are about 50 metals that are studied with respect to the toxicological importance to plants, animals and man. Such metals accumulate in soil to reach the plant through roots during water absorption and cause serious adverse effect on plants viz., inhibition of seed germination, growth of seedlings and reduction of yield. Though some investigations have been carried out in India throwing light on various aspects of the accumulation and effect of heavy metals in plants, yet such study is not sufficient especially in certain agricultural plants of Gujarat state.

Although a number of techniques have been developed to remove metals from contaminated soils, many sites remain contaminated because economic and environmental costs to clean up those sites with the available technologies are too high *(52)*.

As a rule in nature anything that is present on this earth should be either degraded out or recycled. Heavy metals cannot be degraded out but they can be recycled by changing their ionic stage. Any kind of biomass can be easily degraded out in nature.

Several modes of biotechniques are named as biosorption, phyto-sorption, bioaccumulation, phyto-accumulation, bio-extraction, phyto-extraction, rhizofilteration and rhizodegradation, microorganism stimulation and mobilization, phyto-stabilization and phyto-volatilization etc.

Phytosorption is the technique where plants or plant materials are used to absorb heavy metals. In phyto-accumulation technique plants are used to absorb heavy metals and they are stored in plant parts.

Contamination of heavy metals in biosphere increased drastically since 1900 and expressed severe health and the environmental problems throughout the world *(53, 18)*. The plants that are used for phyto-extraction have tolerance towards the metal(s) targeted and efficient to translocate them from below ground parts to areal parts *(5)*.

The present work was focused towards the toxic effects of chromium and manganese metal on biomass and growth rate on widely cultivated pulses *Glycine max*, *Vigna unguiculata* and *Vigna aconitifolia*.

MATERIALS AND METHODS

The study was carried out in Rajkot city area (22^o 17' Lat. and 70^o 49' Lon.). Experiments on seedling emergence and seedling growth were performed on a coarse loam soil found in the natural habitats where the selected plants cultivated by seed germination. Soil was collected from natural habitats, air dried and passed through a 2 mm sieve. For the study of the effect of copper and ferrous on plant growth rate and biomass development, the soil was mixed with heavy metal salts and prepared for the cultivation of experimental plants. The copper and ferrous metals were added in form of copper sulphate salt ($CuSO_4$) and potassium dichromate salt ($K_2Cr_2O_7$) and manganese sulphate ($MnSO_4\,H_2O$) respectively and mixed in eight different lots of soil (each lot of 10kg). To get 0.2, 0.4, 0.6, 0.8, 1.0, 1.2, 1.4 and 1.6 mM concentrations of metal salt 1.95, 3.0, 6.0, 7.8, 9.74, 11.7, 13.6 and 15.6 grams of copper sulphate 3.3, 6.6, 10.0, 13.3, 16.6, 19.9, 23.2 and 26.6 grams for chromium and 1.04, 2.0, 3.1, 4.2, 5.2, 6.24, 7.3 and 8.3 gram for manganese was used.

The soil mixed with metal salt was placed in polyethylene bags and cultivation of experimental plants was carried out in these bags. The soil without metal salt was control. The initial metal concentration of control soil was negligible and considered as zero. Tap water was added to the soil in polyethylene bags to field capacity and then allowed to dry for 6 days.

The seeds of *Glycine max*; *Vigna unguiculata* and *Vigna aconitifolia* were collected from Sanjiv Agro Center, Rajkot.

Metal salt mixed soils were then raked with fingers and seeds were sown after surface sterilization with H_2O_2. Ten seeds were sown in each bag at the depth of about 8 -10 mm in evening. Immediately after sowing soils were watered and then after watering was carried out at alternate days.

All the seedlings in each bag for each metal concentration were allowed for germination. The study was carried out twice. The results are average of the study of these two sets of germination.

After specific time duration plants were harvested in such a way that the tap root and root hairs were not damaged or damage was minimum. Soil particles were removed from the root by gentle washing.

The plants collected for the study brought in the laboratory, washed with water and carefully blotted on the blotting sheets after washing to remove moisture on their surface. The length of entire plant was measured. The mean of 20 measurements was calculated as final reading. The growth rate of control and treated plant was studied on the basis of length of entire plant five weeks after the germination.

The method of Hunt (1978) was used to study the biomass of experimental plants. The fresh weight of root, stem and leaves was determined separately after blotting in the laboratory. They were cut into small pieces after weighing and placed in brown paper bags separately and kept in oven at 80^o C for a period of 8 days for uniform drying. The dry weight of these organs was recorded.

RESULTS AND DISCUSSION

Effect of copper on fresh weight

In *Glycine max* fresh weight of root was 11.25 gm, of stem was 14.92 gm and of leaf was 3.39 gm in control condition. It was gradually decreased by increasing copper concentration in the treatment (Table 1 - 3). The lowest fresh weight was found at 1.6 mM copper concentration in all three organs.

In *Vigna unguiculata* the root fresh weight was 13.32 gm, stem fresh weight was 18.85 gm and leaf fresh weight was 5.78 gm in control condition. This was gradually decreased due to the increase in copper concentration in treatment except in root at 0.2 mM copper concentration (Table 1 - 3).

In *Vigna aconitifolia* the root fresh weight was 12.29 gm, stem fresh weight was 16.89 gm and leaf fresh weight was 4.59 gm in control. In root of *Vigna aconitifolia* the proportion of fresh weight was higher than control at lower concentration of copper (0.2 mM copper). It was gradually decreased when copper concentration was increased from 0.4 mM to 1.6 mM copper (Table 1). In stem the fresh weight was reduced by increasing copper concentrations. Similar results were obtained for leaves also (Table 2 and 3).

Effect of chromium on fresh weight

The fresh weight in root, stem and leaf of *Glycine max* was 9.97, 13.29 and 4.21 gm respectively which was reduced to 6.19 gm in root, 8.21 mg in stem and 2.86 gm in leaf by 0.2 mM chromium treatment (Table 4 - 6).

The fresh weight of root, stem and leaf of *Vigna unguiculata* was lower than control at 0.2 mM chromium concentration (Table 4 - 6).

In *Vigna aconitifolia* the root, stem and leaf fresh weight was decreased at 0.2 mM concentration of chromium in the treatment. Germination was not recorded at 0.4 - 1.6 mM concentrations of chromium in all three investigated plants (Table 4 - 6).

Effect of manganese on fresh weight

The proportion of fresh weight of *Glycine max* root, stem and leaves was reduced by manganese treatment and was lower than control (Table 7 - 9).

In *Vigna unguiculata* root and stem fresh weight was lower than control in all treatments of manganese. In leaf the fresh weight was gradually decreased by increasing the manganese concentration in treatment. The lowest fresh weight was reported at 1.6 mM manganese concentration in all three organs (Table 7 - 9).

In *Vigna aconitifolia* the root fresh weight was gradually decreased by increasing the concentration of manganese and remained lower than the fresh weight of control (Table 7). Similar results were found for the stem and leaf (Table 8 and 9).

Effect of copper on dry weight

In *Glycine max* dry weight of root was 9.68 gm, of stem was 12.83 gm and of leaf was 2.92 gm in control condition. The root dry weight was higher than control at 0.2 mM copper concentration which was gradually decreased when concentration was raised from 0.4 - 1.6 mM copper (Table 7). Stem and leaf dry weight was gradually decreased by increasing the copper concentration in the treatment (Table 10 and 11). The lowest dry weight of all three organs was found at 1.6 mM copper concentration (Table 10 - 12).

In *Vigna unguiculata* the root dry weight was 11.46 gm, stem dry weight was 16.21 gm and leaf dry weight was 4.97 gm in control condition. This was decreased due to the treatment of different concentrations of copper in all three organs except root where at lower copper concentration (0.2 mM) the dry weight was higher than control (Table 10 - 12). The maximum decrease was at 1.6 mM copper concentration (Table 10 - 12).

In *Vigna aconitifolia* the root dry weight was 10.57 gm, stem dry weight was 14.52 gm and leaf dry weight was 3.94 gm in control. In root of *Vigna aconitifolia* the proportion of dry weight was higher than control in treatment of lower copper concentration (0.2 mM). In all other copper treatments the root dry weight was reduced (Table 10). In stem and leaf the dry weight was reduced due to increase in copper concentration in the treatment (Table 11 and 12).

Effect of chromium on dry weight

The root, stem and leaf dry weight of *Glycine max* was 8.91, 10.36 and 2.92 gm respectively which was reduced to 5.40, 5.86 and 1.81 gm at 0.2 mM chromium treatment (Table 13 - 15).

The root, stem and leaf dry weight of *Vigna unguiculata* and *Vigna aconitifolia* was reduced and found lower than control at 0.2 mM chromium treatment. No germination was observed due to 0.4 - 1.6 mM chromium treatments (Table 13 - 15).

Effect of manganese on dry weight

The root dry weight of *Glycine max* was reduced and was lower than control due to the treatment of different concentrations of manganese (Table 16). The stem dry weight was also lower than control due to manganese treatments (Table 17) except at 0.8 and 1.0 mM manganese concentrations (Table 17). The leaf dry weight was lower than the control (Table 18).

In *Vigna unguiculata* and *Vigna aconitifolia* root, stem and leaf dry weight was decreased by increasing the manganese concentration in treatment (Table 16 - 18). The lowest dry weight of all three organs was reported at 1.6 mM manganese concentration (Table 16 - 18).

Effect of copper on growth rate

The length of *Glycine max* was decreased as the concentration of copper increased in the treatment. The lowest growth rate was found at 1.6 mM copper concentration (Table 19). In *Vigna unguiculata* and *Vigna aconitifolia* also the length of entire plant was reduced due to increase in copper concentration in the treatment. The length of *Glycine max* and *Vigna aconitifolia* was slightly higher than control at 0.2 mM copper concentration (Table 19).

Effect of chromium on growth rate

In *Glycine max*, *Vigna unguiculata* and *Vigna aconitifolia* the length of entire plant was reduced at lower chromium concentration in the treatment. Germination was not observed at 0.4 - 1.6 mM chromium concentrations in these plants (Table 20).

Effect of manganese on growth rate

Manganese showed reduction in growth of *Glycine max*, *Vigna unguiculata* and *Vigna aconitifolia* (Table 21).

Effect of copper on growth rate and biomass

Copper is an essential plant micro nutrient required for the protein components of overall enzymes *(47)*. However, when present in excess quantities, copper is also toxic to plant growth potentially causing damage resulting in complete inhibition of growth *(42)*.

The higher concentration of copper inhibits the growth of root and stem *(42, 43)*. The other workers *(66, 45, 2)* have reported decrease in root and shoot growth due to higher amount of copper in the treatment.

In the present work the decrease in fresh weight and dry weight of root, stem and leaf of *Glycine max*, *Vigna unguiculata* and *Vigna aconitifolia* was observed when concentration of copper was increased

in the treatment. Some workers *(74, 42)* also found decrease in fresh weights due to higher concentration of copper and explained that shoot growth reduction is not due to direct toxicity of copper in the shoots, but rather to nutrient deficiencies resulting from a reduced nutrient uptake by the damaged roots. Reduction in growth of cow pea due to copper treatment has also been reported *(42)*. In the present work also reduction in growth of investigated plants was observed due to higher concentration of copper.

Effect of chromium on growth rate and biomass

Chromium plays an important role in growth and development of plants *(58, 3)*. It is toxic to plants when present in higher concentration and affects the growth *(73, 5, 13, 24)*. Chromium toxicity results in to the decrease in root and shoot length *(33, 23, 5, 15, 20, 61, 9, 36, 63)*, biomass *(70, 69)*, plant weight *(61, 65)* seedling height, killing of seeds *(7)*, root weight *(9, 36)*, fresh weight and dry weight *(63)*.

In the present work only 30 - 40 % seed germination was observed and biomass of all investigated plants was reduced at lower concentration of chromium. Adverse effects of chromium on growth rate have been reported in different plants *(27, 63, 37)*. In the investigated plants also chromium has adverse effect on the growth.

Effect of manganese on growth rate and biomass

Manganese is an essential micronutrient in most organisms *(50, 47)*. In plants, it participates in the structure of photosynthetic proteins and enzymes. Its deficit is dangerous for chloroplasts because it affects the water-splitting system of photo-system II, which provides the necessary electrons for photosynthesis *(8)*. However, its excess seems also to be particularly damaging to the photosynthetic apparatus *(53)*. Thus, manganese has two roles in plant metabolic processes: as an essential micronutrient and as a toxic element when it is in excess *(41, 16)*.

Plant species differ considerably in their normal or adequate manganese concentrations *(9)* and in their susceptibility to manganese deficiency *(59, 47, 49)*.

The studies of several workers have indicated that the excess manganese inhibits the plant growth *(53, 21, 66, 25)* and decreases root, stem and leaf dry weight / biomass. This reduction in fresh / dry weight / biomass of various plant organs was observed in *Lolium perenne (60, 51)*, *Trifolium repens* (60), *Juncus effuses (55*) and *Populas cathayana (46)*. In this study decrease in fresh weight and dry weight of root, stem and leaf of *Glycine max*, *Vigna unguiculata* and *Vigna aconitifolia* was observed by increasing the concentration manganese in the treatment. Reduction in shoot growth and dry weight of various organs of sugar maple seedlings with increasing manganese level in the treatment was noticed *(48)*. The sensitivity of other seedlings of several plant species to excess manganese was reported *(32, 38, 39, 40, 41, 48, 52, 62, 64)*. Excess manganese may induce nutrient deficiencies in plants by interfering with the adsorption, translocation and / or utilization of nutrient elements such as calcium and magnesium *(28, 29, 30, 62)*.

Manganese toxicity may play an important role in poor growth of plants. In several plants reduction in growth rate *(50)*, decrease in plant or shoot height *(55, 46)* and root length *(54)* was found due to effect of excess manganese. The reduction in growth rate was noticed in all three investigated plants by higher manganese concentration in the current work. The reduction in nutrient elements by excess manganese may affect the growth rate *(48)*. The reduced growth during manganese treatment probably is due to the effect of manganese on physiological processes, for example, inhibition of DNA replication *(2)* and protein synthesis *(22)*. The tolerance to an excess of manganese is highly dependent on the plant species and cultivars or genotypes within a species *(22, 30)*.

CONCLUSION

The main points of conclusion which can be derived from the results obtained in present study are as follow:

Reduction in fresh weight and dry weight was observed when the concentration of heavy metals increased. Decrease in fresh and dry weight was observed in following manner, chromium > copper > manganese. The growth rate was reduced by increasing concentration of manganese in the treatment. The growth was decreased even at lower concentration of chromium.

Higher concentration of manganese reduced the growth rate of all three investigated plants. As the concentration of copper increased growth rate and biomass decreased. Chromium has adverse effect on growth and biomass at lower concentration.

TABLES

Table 1: Effect of copper on root fresh weight

Concentration (mM)	*Glycine max* (gm)	*Vigna unguiculata* (gm)	*Vigna aconitifolia* (gm)
Control	11.25	13.32	12.29
0.2	11.44 ± 0.02	13.78 ± 0.01	12.61 ± 0.01
0.4	10.79 ± 0.02	12.59 ± 0.01	11.69 ± 0.01
0.6	10.64 ± 0.02	11.87 ± 0.01	11.26 ± 0.01
0.8	9.81 ± 0.02	10.63 ± 0.01	10.22 ± 0.01
1.0	9.78 ± 0.02	10.14 ± 0.01	9.96 ± 0.01
1.2	8.72 ± 0.02	9.58 ± 0.01	9.15 ± 0.01
1.4	8.34 ± 0.02	9.04 ± 0.01	8.69 ± 0.01
1.6	8.22 ± 0.02	8.84 ± 0.01	8.53 ± 0.01

Table 2: Effect of copper on stem fresh weight

Concentration (mM)	*Glycine max* (gm)	*Vigna unguiculata* (gm)	*Vigna aconitifolia* (gm)
Control	14.92	18.85	16.89
0.2	14.37 ± 0.02	17.64 ± 0.01	16.01 ± 0.01
0.4	13.49 ± 0.02	17.53 ± 0.01	15.51 ± 0.01
0.6	12.79 ± 0.02	17.22 ± 0.01	15.01 ± 0.01
0.8	11.84 ± 0.02	16.96 ± 0.01	14.40 ± 0.01
1.0	10.91 ± 0.02	16.53 ± 0.01	13.72 ± 0.01
1.2	10.89 ± 0.02	15.32 ± 0.01	13.11 ± 0.01
1.4	10.61 ± 0.02	15.18 ± 0.01	12.90 ± 0.01
1.6	10.43 ± 0.02	14.98 ± 0.01	12.71 ± 0.01

N.G. = no germination

Table 3: Effect of copper on leaf fresh weight

Concentration (mM)	*Glycine max* (gm)	*Vigna unguiculata* (gm)	*Vigna aconitifolia* (gm)
Control	3.39	5.78	4.59
0.2	3.27 ± 0.01	5.41 ± 0.01	4.34 ± 0.01
0.4	3.22 ± 0.01	5.26 ± 0.01	4.24 ± 0.01
0.6	2.81 ± 0.01	5.02 ± 0.01	3.92 ± 0.01
0.8	2.63 ± 0.01	4.88 ± 0.01	3.76 ± 0.01
1.0	2.36 ± 0.01	4.59 ± 0.01	3.48 ± 0.01
1.2	2.14 ± 0.01	4.24 ± 0.01	3.19 ± 0.01
1.4	1.92 ± 0.01	3.98 ± 0.01	2.95 ± 0.01
1.6	1.47 ± 0.01	3.56 ± 0.01	2.52 ± 0.01

Table 4: Effect of chromium on root fresh weight

Concentration (mM)	*Glycine max* (gm)	*Vigna unguiculata* (gm)	*Vigna aconitifolia* (gm)
Control	9.97	11.34	10.26
0.2	6.19 ± 0.01	7.58 ± 0.01	7.37 ± 0.01
0.4	N.G.	N.G.	N.G.
0.6	N.G.	N.G.	N.G.
0.8	N.G.	N.G.	N.G.
1.0	N.G.	N.G.	N.G.
1.2	N.G.	N.G.	N.G.
1.4	N.G.	N.G.	N.G.
1.6	N.G.	N.G.	N.G.

Table 5: Effect of chromium on stem fresh weight

Concentration (mM)	*Glycine max* (gm)	*Vigna unguiculata* (gm)	*Vigna aconitifolia* (gm)
Control	13.29	16.45	17.35
0.2	8.21 ± 0.01	10.75 ± 0.01	10.13 ± 0.01
0.4	N.G.	N.G.	N.G.
0.6	N.G.	N.G.	N.G.
0.8	N.G.	N.G.	N.G.
1.0	N.G.	N.G.	N.G.
1.2	N.G.	N.G.	N.G.
1.4	N.G.	N.G.	N.G.
1.6	N.G.	N.G.	N.G.

N.G. = no germination

Table 6: Effect of chromium on leaf fresh weight

Concentration (mM)	*Glycine max* (gm)	*Vigna unguiculata* (gm)	*Vigna aconitifolia* (gm)
Control	4.21	5.12	5.63
0.2	2.86 ± 0.01	3.29 ± 0.01	2.75 ± 0.01
0.4	N.G.	N.G.	N.G.
0.6	N.G.	N.G.	N.G.
0.8	N.G.	N.G.	N.G.
1.0	N.G.	N.G.	N.G.
1.2	N.G.	N.G.	N.G.
1.4	N.G.	N.G.	N.G.
1.6	N.G.	N.G.	N.G.

Table 7: Effect of manganese on root fresh weight

Concentration (mM)	*Glycine max* (gm)	*Vigna unguiculata* (gm)	*Vigna aconitifolia* (gm)
Control	11.25	15.85	13.28
0.2	10.63 ± 0.16	15.32 ± 0.04	13.25 ± 0.01
0.4	10.56 ± 0.16	14.89 ± 0.04	12.18 ± 0.01
0.6	10.87 ± 0.16	14.29 ± 0.04	11.60 ± 0.01
0.8	11.11 ± 0.16	12.99 ± 0.04	10.65 ± 0.01
1.0	11.04 ± 0.16	12.63 ± 0.04	10.20 ± 0.01
1.2	10.70 ± 0.16	12.70 ± 0.04	9.47 ± 0.01
1.4	9.39 ± 0.16	12.54 ± 0.04	8.95 ± 0.01
1.6	8.31 ± 0.16	12.47 ± 0.04	8.59 ± 0.01

Table 8: Effect of manganese on stem fresh weight

Concentration (mM)	*Glycine max* (gm)	*Vigna unguiculata* (gm)	*Vigna aconitifolia* (gm)
Control	14.92	20.67	18.10
0.2	13.64 ± 0.28	19.71 ± 0.04	17.37 ± 0.01
0.4	12.70 ± 0.28	19.47 ± 0.04	16.79 ± 0.01
0.6	13.69 ± 0.28	18.74 ± 0.04	16.27 ± 0.01
0.8	13.72 ± 0.28	17.09 ± 0.04	15.67 ± 0.01
1.0	12.68 ± 0.28	17.07 ± 0.04	14.97 ± 0.01
1.2	12.46 ± 0.28	16.68 ± 0.04	14.36 ± 0.01
1.4	12.39 ± 0.28	17.09 ± 0.04	13.84 ± 0.01
1.6	12.35 ± 0.28	16.63 ± 0.04	13.34 ± 0.01

N.G. = no germination

Table 9: Effect of manganese on leaf fresh weight

Concentration (mM)	*Glycine max* (gm)	*Vigna unguiculata* (gm)	*Vigna aconitifolia* (gm)
Control	3.70	6.30	5.09
0.2	3.58 ± 0.04	6.01 ± 0.01	4.89 ± 0.01
0.4	3.55 ± 0.04	5.85 ± 0.01	4.68 ± 0.01
0.6	3.26 ± 0.04	5.59 ± 0.01	4.41 ± 0.01
0.8	2.86 ± 0.04	5.15 ± 0.01	4.21 ± 0.01
1.0	2.80 ± 0.04	5.02 ± 0.01	3.96 ± 0.01
1.2	2.83 ± 0.04	4.94 ± 0.01	3.71 ± 0.01
1.4	2.86 ± 0.04	4.89 ± 0.01	3.47 ± 0.01
1.6	2.67 ± 0.04	4.66 ± 0.01	3.14 ± 0.01

Table 10: Effect of copper on root dry weight

Concentration (mM)	*Glycine max* (gm)	*Vigna unguiculata* (gm)	*Vigna aconitifolia* (gm)
Control	9.68	11.46	10.57
0.2	9.84 + 0.01	11.85 + 0.01	10.84 + 0.01
0.4	9.28 + 0.01	10.83 + 0.01	10.05 + 0.01
0.6	9.15 + 0.01	10.21 + 0.01	9.68 + 0.01
0.8	8.24 + 0.01	8.93 + 0.01	8.58 + 0.01
1.0	8.12 + 0.01	8.42 + 0.01	8.27 + 0.01
1.2	7.15 + 0.01	7.86 + 0.01	7.50 + 0.01
1.4	6.67 + 0.01	7.23 + 0.01	6.95 + 0.01
1.6	6.41 + 0.01	6.90 + 0.01	6.65 + 0.01

Table 11: Effect of copper on stem dry weight

Concentration (mM)	*Glycine max* (gm)	*Vigna unguiculata* (gm)	*Vigna aconitifolia* (gm)
Control	12.83	16.21	14.52
0.2	12.36 ± 0.01	15.17 ± 0.01	13.76 ± 0.01
0.4	11.60 ± 0.01	15.08 ± 0.01	13.34 ± 0.01
0.6	11.00 ± 0.01	14.81 ± 0.01	12.90 ± 0.01
0.8	9.95 ± 0.01	14.25 ± 0.01	12.10 ± 0.01
1.0	9.06 ± 0.01	13.72 ± 0.01	11.39 ± 0.01
1.2	8.93 ± 0.01	12.56 ± 0.01	10.75 ± 0.01
1.4	8.49 ± 0.01	12.14 ± 0.01	10.32 ± 0.01
1.6	8.14 ± 0.01	11.68 ± 0.01	9.91 ± 0.01

Table 12: Effect of copper on leaf dry weight

Concentration (mM)	*Glycine max* (gm)	*Vigna unguiculata* (gm)	*Vigna aconitifolia* (gm)
Control	2.92	4.97	3.94
0.2	2.81 ± 0.01	4.65 ± 0.01	3.73 ± 0.01
0.4	2.77 ± 0.01	4.52 ± 0.01	3.65 ± 0.01
0.6	2.42 ± 0.01	4.32 ± 0.01	3.37 ± 0.01
0.8	2.21 ± 0.01	4.10 ± 0.01	3.15 ± 0.01
1.0	1.96 ± 0.01	3.81 ± 0.01	2.88 ± 0.01
1.2	1.75 ± 0.01	3.48 ± 0.01	2.62 ± 0.01
1.4	1.54 ± 0.01	3.18 ± 0.01	2.36 ± 0.01
1.6	1.15 ± 0.01	2.78 ± 0.01	1.96 ± 0.01

Table 13: Effect of chromium on root dry weight

Concentration (mM)	*Glycine max* (gm)	*Vigna unguiculata* (gm)	*Vigna aconitifolia* (gm)
Control	8.91	9.02	8.79
0.2	5.40 + 0.01	6.85 + 0.01	6.54 + 0.01
0.4	N.G.	N.G.	N.G.
0.6	N.G.	N.G.	N.G.
0.8	N.G.	N.G.	N.G.
1.0	N.G.	N.G.	N.G.
1.2	N.G.	N.G.	N.G.
1.4	N.G.	N.G.	N.G.
1.6	N.G.	N.G.	N.G.

Table 14: Effect of chromium on stem dry weight

Concentration (mM)	*Glycine max* (gm)	*Vigna unguiculata* (gm)	*Vigna aconitifolia* (gm)
Control	10.36	11.23	11.52
0.2	5.86 ± 0.01	6.17 ± 0.01	6.76 ± 0.01
0.4	N.G.	N.G.	N.G.
0.6	N.G.	N.G.	N.G.
0.8	N.G.	N.G.	N.G.
1.0	N.G.	N.G.	N.G.
1.2	N.G.	N.G.	N.G.
1.4	N.G.	N.G.	N.G.
1.6	N.G.	N.G.	N.G.

Table 15: Effect of chromium on leaf dry weight

Concentration (mM)	*Glycine max* (gm)	*Vigna unguiculata* (gm)	*Vigna aconitifolia* (gm)
Control	2.92	3.94	3.97
0.2	1.81 ± 0.01	1.95 ± 0.01	1.73 ± 0.01
0.4	N.G.	N.G.	N.G.
0.6	N.G.	N.G.	N.G.
0.8	N.G.	N.G.	N.G.
1.0	N.G.	N.G.	N.G.
1.2	N.G.	N.G.	N.G.
1.4	N.G.	N.G.	N.G.
1.6	N.G.	N.G.	N.G.

Table 16: Effect of manganese on root dry weight

Concentration (mM)	*Glycine max* (gm)	*Vigna unguiculata* (gm)	*Vigna aconitifolia* (gm)
Control	9.68	13.63	11.42
0.2	9.14 ± 0.11	13.59 ± 0.02	11.40 ± 0.01
0.4	9.08 ± 0.11	12.80 ± 0.02	10.47 ± 0.01
0.6	9.35 ± 0.11	12.29 ± 0.02	9.98 ± 0.01
0.8	9.33 ± 0.11	10.91 ± 0.02	8.94 ± 0.01
1.0	9.16 ± 0.11	10.48 ± 0.02	8.47 ± 0.01
1.2	8.77 ± 0.11	10.41 ± 0.02	7.76 ± 0.01
1.4	7.51 ± 0.11	9.95 ± 0.02	7.16 ± 0.01
1.6	6.48 ± 0.11	9.80 ± 0.02	6.70 ± 0.01

Table 17: Effect of manganese on stem dry weight

Concentration (mM)	*Glycine max* (gm)	*Vigna unguiculata* (gm)	*Vigna aconitifolia* (gm)
Control	12.83	17.78	15.57
0.2	11.73 ± 0.21	16.95 ± 0.02	14.94 ± 0.01
0.4	10.92 ± 0.21	16.74 ± 0.02	14.44 ± 0.01
0.6	11.77 ± 0.21	16.12 ± 0.02	13.99 ± 0.01
0.8	13.20 ± 0.21	14.35 ± 0.02	13.16 ± 0.01
1.0	13.01 ± 0.21	14.16 ± 0.02	12.42 ± 0.01
1.2	10.22 ± 0.21	13.68 ± 0.02	11.78 ± 0.01
1.4	9.91 ± 0.21	13.67 ± 0.02	11.07 ± 0.01
1.6	9.63 ± 0.21	12.97 ± 0.02	10.40 ± 0.01

N.G. = no germination

Table 18: Effect of manganese on leaf dry weight

Concentration (mM)	*Glycine max* (gm)	*Vigna unguiculata* (gm)	*Vigna aconitifolia* (gm)
Control	3.18	5.41	4.38
0.2	3.08 ± 0.21	5.17 ± 0.02	4.20 ± 0.01
0.4	3.05 ± 0.21	5.03 ± 0.02	4.02 ± 0.01
0.6	2.80 ± 0.21	4.81 ± 0.02	3.80 ± 0.01
0.8	2.40 ± 0.21	4.33 ± 0.02	3.54 ± 0.01
1.0	2.32 ± 0.21	4.17 ± 0.02	3.29 ± 0.01
1.2	2.32 ± 0.21	4.05 ± 0.02	3.04 ± 0.01
1.4	2.29 ± 0.21	3.91 ± 0.02	2.78 ± 0.01
1.6	2.08 ± 0.21	3.63 ± 0.02	2.45 ± 0.01

Table 19: Effect of heavy metals on *Glycine max* growth rate

Concentration (mM)	Length of plant (cm)			
	Control	Copper	Chromium	Manganese
Water	52.00	-	-	-
0.2	-	52.90 ± 0.01	4.90 ± 0.01	49.20 ± 0.17
0.4	-	49.80 ± 0.01	N.G.	48.80 ± 0.17
0.6	-	49.10 ± 0.01	N.G.	50.30 ± 0.17
0.8	-	45.40 ± 0.01	N.G.	51.70 ± 0.17
1.0	-	45.20 ± 0.01	N.G.	51.10 ± 0.17
1.2	-	40.30 ± 0.01	N.G.	49.60 ± 0.17
1.4	-	38.50 ± 0.01	N.G.	43.90 ± 0.17
1.6	-	37.60 ± 0.01	N.G.	38.40 ± 0.17

Table 20: Effect of heavy metals on *Vigna unguiculata* growth rate

Concentration (mM)	Length of plant (cm)			
	Control	Copper	Chromium	Manganese
Water	70.00	-	-	-
0.2	-	67.40 ± 0.02	3.60 ± 0.01	64.00 ± 0.27
0.4	-	63.30 ± 0.02	N.G.	67.50 ± 0.27
0.6	-	60.00 ± 0.02	N.G.	62.70 ± 0.27
0.8	-	55.50 ± 0.02	N.G.	48.80 ± 0.27
1.0	-	51.20 ± 0.02	N.G.	53.70 ± 0.27
1.2	-	51.10 ± 0.02	N.G.	58.60 ± 0.27
1.4	-	49.80 ± 0.02	N.G.	68.10 ± 0.27
1.6	-	48.90 ± 0.02	N.G.	67.90 ± 0.27

N.G. = no germination

Table 21: Effect of heavy metals on *Vigna aconitifolia* growth rate

Concentration (mM)	Length of plant (cm)			
	Control	Copper	Chromium	Manganese
Water	61.20	-	-	-
0.2	-	62.8 ± 0.01	47.5 ± 0.01	61.1 ± 0.01
0.4	-	58.2 ± 0.01	N.G.	54.3 ± 0.01
0.6	-	56.1 ± 0.01	N.G.	51.3 ± 0.01
0.8	-	49.7 ± 0.01	N.G.	47.5 ± 0.01
1.0	-	47.9 ± 0.01	N.G.	44.8 ± 0.01
1.2	-	43.5 ± 0.01	N.G.	41.9 ± 0.01
1.4	-	40.3 ± 0.01	N.G.	39.5 ± 0.01
1.6	-	38.5 ± 0.01	N.G.	37.1 ± 0.01

N.G. = no germination

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

(Proceedings available for download at rku.ac.in/icre)

RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

Avian Biodiversity of Amipur Wetland (Porbandar District, Gujarat) and Threats to it Due to Pesticide Toxicity

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ABSTRACT

Wetlands are some of the ecosystems threatened by the pesticides. Agricultural practices for which the pesticides are applied are carried out on the land adjoining wetlands for the ease of irrigation water availability. As modern, market-oriented agricultural practices make heavy use of the pesticides, the pesticide contaminated run-off or seepage from the cultivation area often drains into the wetland and in turn, harm biodiversity. The Amipur dam wetland in Porbandar is well-known for its huge congregation of migratory cranes, might be facing pesticide toxicity problem. Several cranes and other waterbirds are rescued every year from the agricultural fields within 10 kilometer radius of this wetland and as per circumstantial evidences; pesticide toxicity is the likely reason as pesticides are widely used. The present study brings forth the significance of Amipur dam from the view point of avian biodiversity and draws attention towards threat to wetland and waterbirds from pesticide toxicity.

SUMMARY

The Amipur dam wetland in Porbandar is well-known for its huge congregation of migratory cranes, might be facing pesticide toxicity problem as several cranes and other waterbirds are rescued every year from the agricultural fields within 10 kilometer radius of this wetland and as per circumstantial evidences; pesticide toxicity is the likely reason as pesticides are widely used.

Keywords: Pesticides, Ecotoxicology, Amipur, Wetland, Porbandar, Gujarat, Sustainable

INTRODUCTION

Ecotoxicology is defined as ‘‘the science of contaminants in the biosphere and their effects on constituents of the biosphere, including humans’’[(1)]. A pesticide is usually defined as “a chemical substance, biological agent, antimicrobial or disinfectant used against pests including insects, plant pathogens, weeds, mollusks, birds, mammals, fish, nematodes (roundworms) and microbes that compete with humans for food, destroy property, have a propensity for spreading or area vector for disease or simply a nuisance”[(2)]. Pesticides are supposed to kill only the targeted species, i.e., mainly insects feeding on crops and other weeds competing with crops. Biologist Rachel Carlson had exposed the dark side of pesticides through her book titled *Silent Spring,* which changed the attitude of public towards pesticides[(3, 4)].

In India, more than 400 pesticides belonging to 4 groups are used but, only 234 pesticides are registered[(5)].

1st Generation pesticides include Chlorinated hydrocarbon compounds: DDT, Endosulfon, Heptachlor, Aldrin, Isodrin and Dieldrin etc.

2nd Generation pesticides include Organophosphates: Quinalfos, Monocrotofos, Malathion, Parathion, Trichlorophan, Fenchlorofos, Phoxim, Abate, Dichlorovos, Fenthion and Haloxon etc.

3rd Generation includes Carbamates: Aldicarb, Carbafuron, Carbaryl and Aminocarb etc.

4th Generation includes Synthetic Pyrethroids: Cypermethrin, Deltamethrin, Permethrin and Cismethrinetc[(6)].

As per ‘The Insecticide Act, 1968’ and ‘the Insecticide Rules, 1971 in India’, pesticides have to pass through various registrations and tests before they appear in the market. This process also includes an assessment of the potential risk to wildlife and the environment[(7)]. However, several studies have shown that they also affect non-target species, including birds and other wildlife of wetlands and their environs. Hexachlorocyclohexane (HCH), cyclodienes (aldrin, dieldrin, endrin), chlordanes (heptachlor, heptachlor epoxide, cis-chlordane, trans-chlordane, cisnonachlor, trans-nonachlor, and oxychlordane), HCHs isomers), mirex are the environmentally harmful pesticides[(8)]. Monocrotophos pesticide was responsible for the death of seven Sarus cranes at Keoladeo National Park wetlands, Rajasthan in 2000[(9)]. DDT and its various metabolites are also detected in zooplanktons in wetland based stations extending from Gujarat to Bombay of the Arabian ocean[(10)]. Various isomers of HCH and DDT are also found in flowing water wetlands of Yamuna river and its canals in Haryana and Delhi[(11)]. Isomers of HCH and DDT along with Aldrin and Dialdrin are detected in prevalent amount from west coast of India[(12)]. Ten

commercial fish species of local fish market of Mumbai were found infected with HCH and DDT isomers[13]. Carcass of 16 bird species in multiple specimen were collected from Ahmedabad in 2005-2007 and all of them were detected with either of DDT or HCH isomer[14]. Toxicology division of Sálim Ali Centre for Ornithology and Natural History (SACON) has evaluated and detected organochlorine pesticides in various samples like soils from India, blood samples of vultures and other species of birds, commercial fishes of Mumbai, water samples of Keoladeo National Park wetlands, eggs and tissue samples of various species of birds[15-20].

Many man-made wetlands in India are mainly created for irrigation purpose. They influence agricultural landscape in their command areas and in turn, get influenced by agricultural activities in the environs. One of such influences from agricultural practices is draining or seeping of agricultural residues into the wetlands. Due to heavy use of pesticides in agricultural practices the agricultural run-off easily takes pesticide residues into the wetlands. The pesticides being toxic chemicals have great potential to harm on-target planktons, macro-invertebrates and vertebrates inhabiting the wetlands. Indian wetlands and other important habitats are already contaminated with pesticides but, major work is yet to be done [18-22]. This is despite the fact that pesticide regulations do exist in India (Table 1).

Table 1: Pesticide Regulation in India

No	Banned for manufacture, import and use	Pesticides Refused Registration	Restricted For Use In India
1	Aldrin	Calcium Arsonate	Aluminium Phosphide
2	Benzene Hexachloride	EPM	DDT
3	Calcium Cyanide	Azinphos Methyl	Lindane
4	Chlordane	Lead Arsonate	Methyl Bromide
5	Copper Acetoarsenite	Mevinphos (Phosdrin)	Methyl Parathion
6	CIbromochloropropane	2,4, 5-T	Sodium Cyanide
7	Endrin	Carbophenothion	Methoxy Ethyl Mercuric Chloride (MEMC)
8	Ethyl Mercury Chloride	Vamidothion	Monocrotophos
9	Ethyl Parathion	Mephosfolan	Endosulfan

10	Heptachlor	Azinphos Ethyl	Fenitrothion
11	Menazone	Binapacryl	Diazinon
12	Nitrofen	Dicrotophos	Fenthion
13	Paraquat Dimethyl Sulphate	Thiodemeton / Disulfoton	Dazomet
14	Pentachloro Nitrobenzene	Fentin Acetate	
15	Pentachlorophenol	Fentin Hydroxide	**Pesticide Withdrawn**
16	Phenyl Mercury Acetate	Chinomethionate (Morestan)	1. Dalapon
17	Sodium Methane Arsonate	Ammonium Sulphamate	2. Ferbam
18	Tetradifon	Leptophos (Phosvel)	3. Formothion
19	Toxafen		4. Nickel Chloride
20	Aldicarb	**Banned for import, manufacture and use**	5. Paradichlorobenzene (PDCB)
21	Chlorobenzilate		6. Simazine
22	Dieldrine	1. Methomyl 24% L	7. Warfarin
23	Maleic Hydrazide	2. Methomyl 12.5% L	
24	Ethylene Dibromide	3. Phosphamidon 85% SL	**Banned for use but their manufacture is**
25	TCA (Trichloro acetic acid)	4. Carbofuron 50% SP	**allowed for export**
26	Metoxuron		1. Nicotin Sulfate
27	Chlorofenvinphos		2. Captafol 80% Powder

Source: Web portal of 'Directorate of Plant Protection Quarantine & Storage, Faridabad' as on 15.03.2011

Study Area:

Amipur dam (Fig.1) in Porbandar district constituted the study area.

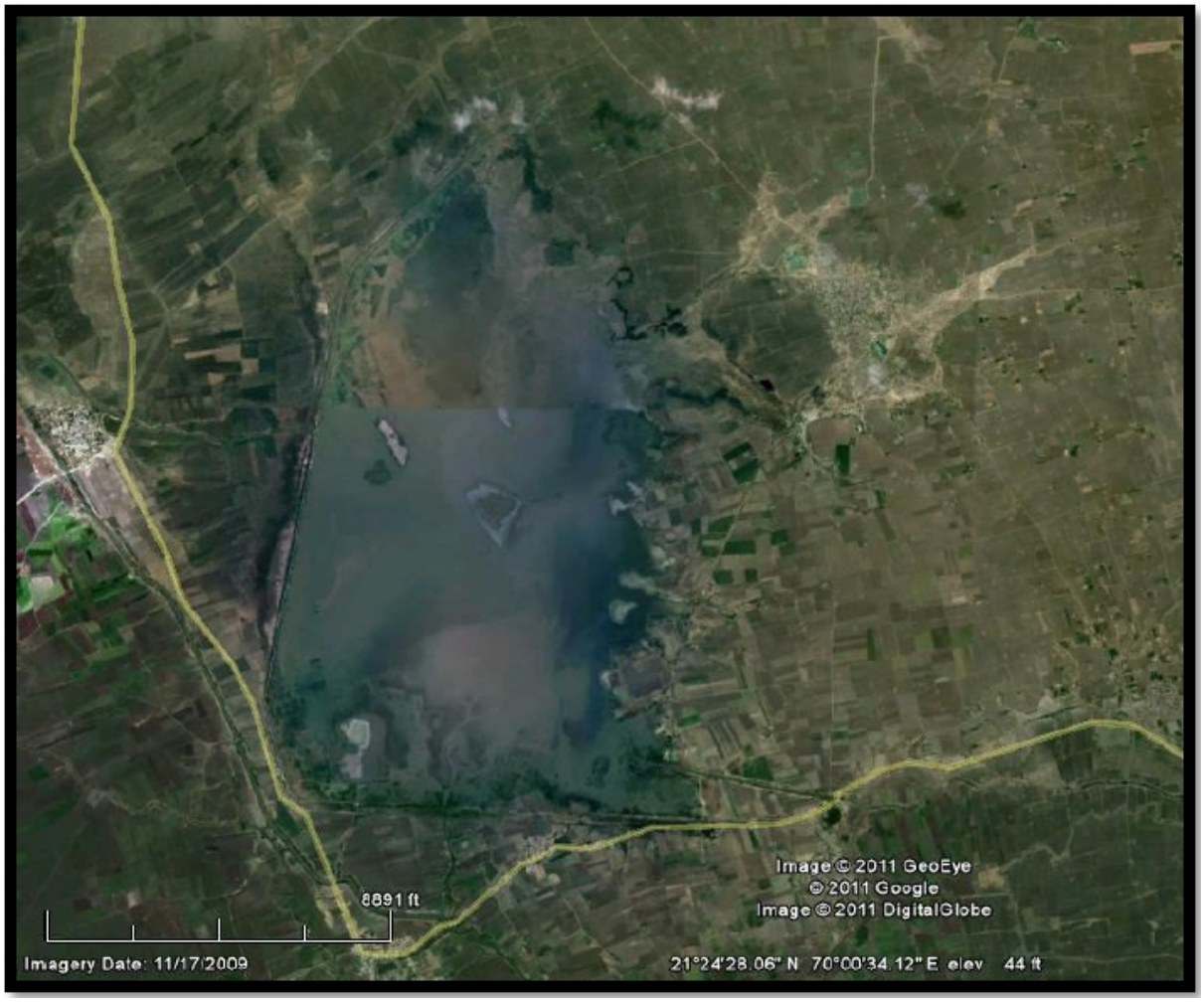


Fig.1 Amipur dam, Kutiyana Taluka, Porbandar District

Amipur dam is located in "*Ghed"* region of Saurashtra. It is located at 21°24'22.57" N latitudes and 69°58'45.34" E longitude near Amipur village (having 251 households and 1229 people) of Kutiyana taluka in Porbandar district, Gujarat of India. It is located at a distance of about 41 km from Porbandar city in its south-east. Apart from Amipur village, there are other villages on the periphery of this wetland, which include Mota Ghed of Kutiyana taluka amd Miti, Hantarpur and Bagasra Ghed of Mangrol taluka. It may be noted that "*Ghed*" is the low lying marshy region adjacent to western Saurashtra coast in north of Porbandar which frequently get flooded during monsoon. "*Sorthi Ghed"* and "*Barda Ghed"* are two distinct sub-regions of Ghed and Amipur Dam is located in "*Sorthi Ghed".* Total 226 wetlands have been

mapped by the Space Applications Center (SAC), ISRO in this area of Porbandar district. Inland wetlands contribute 27.3% of the total wetland area and coastal wetlands contribute 72.7% of the total wetland area[23]. Some significant wetlands of Porbandar district include Mendha creek, Visavada, Kuchhadi, Subhashnagar, Zavar, Karly I, Karly II, Porbandar Bird Sanctuary, Chhaya-I, Chhaya-II and Porbandar Rann, Vanana, Dharampur, Bhadarbara, Bardasagar and Amipur wetlands, out of them Kuchhadi, Subhashnagar, Zavar, Karly I, Karly II, Vanana, Dharampur share physical boundary with Gosabara Mokarsagar Wetland but named accordingly the villages nearby[24]. The entire area is rich in waterbird diversity and abundance. Shree Sahajanand Swami District Community Science Centre (SSDCSC)-Porbandar in the collaboration with Porbandar Forest Division- Porbandar, Indian Coastguard- Porbandar, Green Wildlife Conservation Society and Mokarsagar Wetland Conservation Committee organised Asian Waterbird Census (AWC) on 1st February- 2015 at 21 different wetland sites of Porbandar district and recorded 1,93,358 waterbirds[25]. This is the area, including the area of Amipur dam, is highly favored by a migratory crane species called Demoiselle crane (*Anthropoides virgo*).

MATERIALS AND METHODS

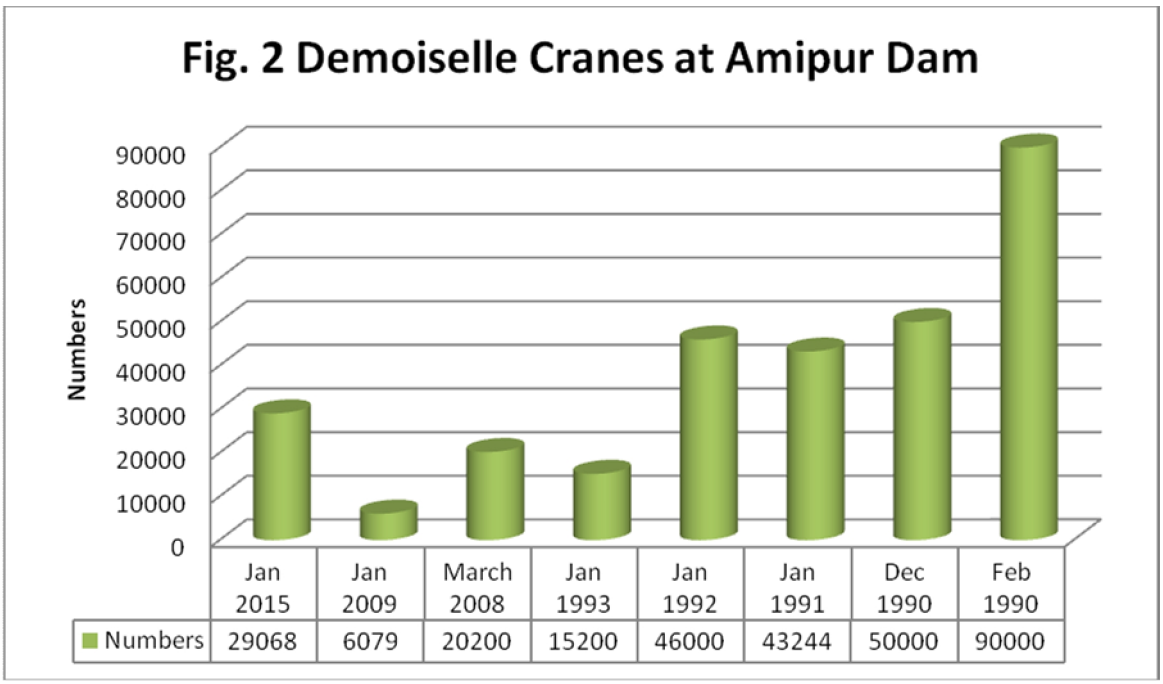
The entire area of Amipur dam was surveyed from October 2015 to the first week of December 2015 for (i) developing waterbird profile; (ii) understanding cropping pattern in the area; (iii) pesticide use by farmers in the area and (iv) understanding waterbird calamities due to likely reason of pesticide poisoning. Visits were paid on fortnightly basis in October and weekly basis in November and December 2015. Birds were observed and counted using pairs of Olympus 10 X 50 binocular and Celestron Ultima 80 Spotting Scope mounted on fluid head tripod. Bird species that did not occur in huge congregation were counted using 'direct head count' method. However, waterbirds like Demoiselle Cranes were present in huge congregations and their population was estimated using 'block count' method. Binoculars were also used to observe any large sized dead birds (cranes, storks, ibises etc.) or to notice any sick waterbirds (with the clinical symptoms like dullness and drooling). About 50 farmers were opportunistically interviewed to understand their preference of crops and to understand the pesticides they use and dosage of pesticides that they normally apply. In addition, secondary data also was used to understand the dosage of various pesticides for various pesticides. Secondary information (literature) was also used to understand Amipur wetland ecosystem and its avian profile.

RESULTS AND DISCUSSION

A total of 75 species of waterbirds were recorded. The significant species and their approximate population (on the scale of "rare" to "abundant" is given below:

Great Crested Grebe (uncommon), Little Grebe (common), Great White Pelican (common), Great Cormorant (uncommon), Little Cormorant (common), Grey Heron common), Purple Heron (uncommon), Pond Heron (common), Large Egret (common), Median Egret (uncommon), Little Egret (uncommon), Painted Stork (Common), Black-headed Ibis (common), Glossy Ibis (common), Red napped Ibis (uncommon), Eurasian Spoonbill (common), Flamingos (two species) (very common), Common/Eurasian Crane (very common), Demoiselle Crane (abundant), Ducks (Common Teal, Northern Shoveler, Northern Pintail, Gadwall, Eurasian Wigeon, Common Pochard, Cotton Teal, Spot-billed Duck, Comb Duck, Lesser whistling Duck) (abundant), Shorebirds (Large Sand Plover, Little Ringed Plover, Kentish Plover, White-tailed Lapwing, Red-wattled Lapwing, Yellow-wattled Laping, Blck-winged Stilt, Avocet, Snipes, Godwits, Eurasian Curlew, Whimbrel, Sandpipers, Stints etc.) (abundant), Gulls and Terns (very common), Rallids (very common), Marsh Harrier (uncommon) ([Note that rare species= less than 10 individuals, uncommon species=11 to 50, common species= more than 50 and upto 500 individuals, very common species= more than 500 individuals but less than 10,000 individuals, abundant species= Above 10,000 individuals]

It may be noted that during Asian Wetland Count (AWC)-2015, a total of 38,658 waterbirds were recorded at Amipur dam that included 29068 Demoiselle Cranes. A waterbird survey in December, 1998 had recorded 36,627 waterbirds[26, 27]. In the winter of 1994, a total of 47,198 waterbirds were recorded at Amipur dam[26, 27]. As far as Demoiselle Crane population in the past is concerned, the observed numbers are shown in Fig. 2[27].



Thus, the waterbird surveys at Amipur clearly indicate that very high waterbird diversity and abundance exists at Amipur dam. Moreover, it supports exceptional congregation of migratory Demoiselle Cranes. Cropping pattern survey indicated that Gram, Ground Nut and Sorghum were the main crops in the area besides Cotton, Wheat and Bajra. Following information was available from the farmers interviewed and published literature for the pesticide dosage used for these crops:

Table 2: Pesticides used for Porbandar's major crops with dosage *(28-34)*

No	Crop	Pesticide		Dosage
		Name	Class	
1	Groundnut	Carbendazim	Fungicide	3 gm/ kg of seeds
		Mancozeb	Fungicide	3 gm/ kg of seeds
		Quinalphos	Organothiophosphate	25 ml/kg
		Chlorpyrifos	Organophosphate	25 ml/kg
		Pendimethalin	Herbicide	3lt/500 lt water/ha

		Fluchloralin	Herbicide	2lt/500 lt water/ha
		Thiram	Fungicide	3 gm/ kg of seeds
		Captan	Fungicide	3 gm/ kg of seeds
		Dimethoate	Organophosphate	20 ml/ 10 lt water
		Demeton-S-methyl	Organothiophosphate	10 ml/ 10 lt water
		Imidacloprid	Neonicotinoids	3 gm/ kg of seeds
		Thiamethoxam	Neonicotinoids	1 gm/ kg of seeds
		Phosphamidon	Organophosphate	3 ml/ 10 lt water
		Carbaryl	Carbamate	40 gm/ 10 lt water
2	Cotton	Imidacloprid	Neonicotinoids	7.5 gm/ kg of seeds
		Fluchloralin	Herbicide	50 ml/ 10 lt water
		Pendimethalin	Herbicide	50 ml/ 10 lt water
		Demeton-S-methyl	Organothiophosphate	10 ml/ 10 lt water
		Phosphamidon	Organophosphate	5 ml/ 10 lt water
		Dimethoate	Organophosphate	10 ml/ 10 lt water
		Imidacloprid	Neonicotinoids	5 ml/ 10 lt water
		Thiamethoxam	Neonicotinoids	3 gm/ 10 lt water
		Acetamiprid	Neonicotinoids	3 gm/ 10 lt water
		Carbaryl 10%	Carbamate	20 kg/ha
		Quinalphos 1.5%	Organothiophosphate	20 kg/ha

		Chlorpyrifos	Organophosphate	20 ml/ 10 lt water
		Bavistin	Fungicide	10 gm/10 lt
3	Wheat	Pendimethalin	Herbicide	10kg/600 lt water/ha
		2,4-D	Herbicide	0.4kg/600 lt water/ha
		Chlorpyrifos 20 EC	Organophosphate	450 ml/ha
		Quinalphos 25EC	Organothiophosphate	0.05%
		Demeton-S-methyl	Organothiophosphate	25 kg/ha
4	Sorghum	Atrazine	Herbicide	0.5 kg/ ha
		Carbosulfan	Carbamate	100 gm/kg
		Thiamethoxam	Neonicotinoids	2 gm/kg
		Carbofuran 3%	Carbamate	7.5 kg/ha
		Quinalphos 5%	Organothiophosphate	7.5 kg/ha and 10 kg/ha
		Dicofol 18.5%	Organochlorine	25 ml in 10 lt water
		Formathion 0.025%	Organophosphate	25 ml in 10 lt water
		Melathion 5	Organophosphate	30 kg/ha
		Ziram	Fungicide	0.2%
		Carbendazim	Fungicide	0.05%
6	Pearl millet(Bajra)	Atrazine	Herbicide	0.5kg/ha
7.	Gram	Acephate 75% SP	Insecticide	150-250 g per acre

Sowing window of major Rabi crops like Wheat and Gram is in between October to November of any one year. This is the time when many of the migratory waterbirds mentioned earlier visit the area. To preserve the seeds from termites and other insects, many times farmers treat the seeds with pesticides prior to sowing day as seed treatment. Cotton is the most pesticide dependent crop of the country. It needs at least 3 kg of pesticides per hectare, using half of the total pesticides in the country and occupying only about 4.5% of the gross cropped area[35].

It is beyond doubt that agricultural runoff from the fields of all these crops might be laden with residues of toxic pesticides mentioned in Table 2 above and ultimately these residues might be finding their way in the open water and marshy areas of Amipur dam. As Amipur dam is an abode for a large variety of waterbirds as shown earlier, the pesticides might be adversely affecting those waterbirds through food-chains. In the past, waterbirds like cranes, storks, ibis and flamingos had to be rescued within 10 km radius of Amipur dam to save them from the sickness developed due to pesticide toxicity as per circumstantial evidences. It may be noted that in December 2012, around 40 Demoiselle Cranes died in Amreli and Surendranagar districts of Saurashtra region of Gujarat state. Large flocks of cranes, with many sick birds were found congregating near Rajula (250 km south-east of Porbandar). Feral dogs and Marsh Harriers were observed preying upon the dying cranes[36].

CONCLUSION

The findings mentioned in the present paper are based on preliminary studies carried out in the initial stage of doctoral research of the first author under the supervision of the second and third authors. They indicate that Amipur dam has high waterbird diversity and abundance on one hand and heavy use of pesticides by the farmers on the other hand. Circumstantial evidences in this area and historical records in other areas of Saurashtra region of Gujarat clearly indicate that pesticide poisoning is very likely in waterbirds occurring at Amipur dam and other wetlands of Porbandar district. At Amipur wetland, agriculture is practiced in post winter to summer period not only in its environs but even in the portion of its basin when water would typically dry up and considering possibility of possibility of pesticide toxicity such practices can pose a major threat to waterbirds and wetland[37]. Therefore, continuous, long-term eco-toxicological monitoring is required. Moreover, education and awareness of farmers about judicious use of the pesticides are highly desirable at Amipur wetland and its environs.

FIGURES

Fig. 1. Amipur dam, Kutiyana Taluka, Porbandar District

Fig. 2. Demoiselle Cranes at Amipur Dam

TABLES

Table 1. Pesticide Regulation in India

Table 2. Pesticides used for Porbandar's major crops with dosage

ACKNOWLEDGEMENT

This paper is based on the first author's recently initiated Doctoral Research at R K University, Rajkot on "Evaluation of Pesticide Toxicity in Some Important Wetlands of Porbandar District, Gujarat State" under the guidance of second and third authors. The first author is thankful to the R. K. University, Rajkot for providing an opportunity to conduct Ph.D. research. He is also thankful to the members of Mokarsagar Wetland Conservation Committee for accompaniment in the field-work and providing important information regarding wetlands of Porbandar whenever required.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

(Proceedings available for download at rku.ac.in/icre)

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Achievement of single phase $BiFeO_3$ through chemical method from mixed minor phases $BiFeO_3$ prepared using Solid State Reaction Technique

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ABSTRACT

Recently multiferroics have attained great attraction due to their ability to show various ferroic properties in a single phase. $BiFeO_3$ (BFO) is the only room temperature single phase multiferroic material which simultaneously possesses anti-ferromagnetic and highly ferroelectric nature. Literature survey shows that BFO is mostly synthesized with Solid State Reaction (SSR) technique. In this method, synthesis of BFO is more critical as temperature stability range is very narrow (800 to 830 ˚C) and generation of minor impurity phases like $Bi_2Fe_4O_9$, $Bi_{25}FeO_{39}$, $Bi_{25}FeO_{40}$, $Bi_{36}Fe_2O_{57}$ and $Bi_{46}Fe_2O_{72}$ diminishes the multiferroic behavior. We have synthesized pure BFO using SSR and for removal of present impure phases, chemical technique was used and finally pure phase BFO was achieved. This method is comparatively low cost method and it can be used to synthesize BFO in the bulk form with use of fewer chemicals then other famous methods, so it can be adaptable for commercial production.

SUMMARY

This paper will give you an idea about synthesizing pure phase $BiFeO_3$ multiferroic in bulk form through conventional Solid State Reaction followed by little chemical processes.

Keywords: Solid State Reaction, Magnetoelectric Multiferroics, Perovskite structure, Leaching

INTRODUCTION

Any single phase material which simultaneously possess two or more primary ferroic properties among the four; which are Ferroelectric, Ferromagnetic, Ferroelastic and Ferrotoroidic, are known as multiferroic material or multiferroic compound (*1*). The first magnetoelectric multiferroic was discovered by E. Ascher and others in 1966 in which simultaneous occurrence of ferroelectricity and weak ferromagnetism was observed in nickel iodine boracite $Ni_3B_7O_{13}I$ (below 61 K, that is -212 ˚C) (*2*), but the field remained inactive until 1994 when Hans Schmid coined the term "*multiferroics"*. In the beginning of this century the novel class of multiferroics was discovered, in which magnetism and ferroelectricity do not just coexist, but in addition to this magnetism causes ferroelectricity, Y. Tokura and others in $TbMnO_3$ (*3*) and N. Hur and others in $TbMn_2O_5$, (*4*) found this effect and J. Wang and others have successfully grew thin films of $BiFeO_3$ or simply BFO (*5*).

The couplings between ferroic orders and their various parameters with each other in the world of multiferroics are described briefly here: Magnetoelectric coupling describes the influence of magnetic (electric) field on the polarization (magnetization) of material, piezoelectric couples a change in strain as a linear function of applied electric field, or change in polarization as a linear function of applied stress, piezomagnetic coupling describes a change in strain as a linear function of applied magnetic field, or a change in magnetization as a linear function of applied stress. Piezoelectric and piezomagnetic nature can be also known as electrostriction and magnetostriction, respectively. Cross coupling allows those ferroic orderings to be tuned by fields other than their conjugates; in magnetoelectric multiferroics an electric field can modify magnetism or vice-versa. Among all multiferroic materials magnetoelectric multiferroics are very famous as these materials coexists ferroelectricity and ferro/antiferro-magnetism together.

Ferroelectric antiferromagnets like $BiFeO_3$, $BaMnF_4$, $REMnO_3$ (RE = rare earth), etc are gained more attraction of scientists. Thin films of epitaxially grown $BiFeO_3$, coupled to a magnetic ordering temperature above RT results promising burst to the interest in magnetoelectric multiferroics (*6*). These new discoveries broke the traditional thinking about multiferroics and opened a new gate for industry: if charge can be controlled by external magnetic fields and spins can be controlled by applied voltage, new devices with varieties of functions may be designed (*7*). The key reason for the interest in multiferroic materials is due to their potential use in commercial applications because of the discovery of "magnetic field control of the electric polarization" or "strong magnetoelectric coupling".

BFO is the only single phase material that shows multiferroic phenomenon at room temperature. It possesses rhombohedrally distorted perovskite crystal structure with R3c space group symmetry. It exhibits both ferroelectric and antiferromagnetic ordering with a curie temperature T_C = 810 ˚C and Neel temperature T_N = 370 ˚C respectively. In BFO the magneto-electric coupling generates a unique magneto electric effect which allows the polarization to be tuned under external magnetic field or magnetization to be tuned under external electric field. Consequently BFO finds use for several applications such as non volatile information storage, spintronic sensors, wireless sensors, digital memories, spin filters etc. This is the main reason for which now-a-days scientists and researchers are focusing on this material's bulk and thin film properties. Now, there are many techniques to synthesize bulk BFO; like, Conventional Solid State Reaction, Sol-gel, hydrothermal, auto combustion and co-precipitation technique. One can prepare BFO using other methods, but the problem is that these methods are either time consuming or costly as high purity chemicals are used. Pure oxides are available from many well-known companies and laboratories like Sigma-Aldrich, Alfa Aesar, Superlab, Koch Chemical Ltd. and Johnson Matthey Chemicals Limited. Oxides are comparatively cheaper and non-toxic materials; by proper heating cycles and leaching technique, one can easily synthesize ultrapure single phase BFO, these are the main reasons why we preferred Solid State Reaction Method to synthesize the sample.

BFO crystallizes in a rhombohedrally distorted perovskte (Fig. 1) structure of R3c symmetry, all metal ions are displaced along the (111) direction relative to the ideal centrosymmetric position and the oxygen octahedra surrounding Fe are rotated alternatively around the axis with $\lambda = 620$ Å, here λ is wavelength of magnetic ordering in BFO. Using Bi_2O_3 and Fe_2O_3 as starting precursors Chandrasekhar and his colleagues have synthesized $BiFeO_3$ which was crystallized in rhombhohedral perovskite phase (*8*). Whereas Lou Yan-Hui and his colleagues have synthesized it using nitrogen atmosphere provided while treating heating cycles (*9*). Sometimes there is presence of common secondary phases like $Bi_2Fe_4O_9$ and $Bi_{25}FeO_{39}$ in BFO. Generally leaching process is used to remove these secondary phases from the sample, many papers have mentioned this process but in many cases it was misleading, sometimes concentrated acids were used and sometimes diluted ones, but no one had mentioned the amount of dilution. As BFO and their impurities both are dissolvable in concentrated acids it cannot be recommended for leaching, while on other hand if one will use highly diluted acids then hardly the impurity phase will be removed and once again the process will be time consuming. Leaching with particular acid in particular diluted amount helps to eliminate the impurity phase easily, here we have used both methods referred in (*8*) and (*9*) and developed an experimental technique through which we achieved single phase BFO. Results are discussed in this paper.

METHOD

We have used high purity Sigma-Aldrich oxides (99.9% pure) of Bi_2O_3 and Fe_2O_3, which were weighted in stoicheometric ratio and then thoroughly mixed and grounded in the alcoholic medium. This media provides easier and homogeneous mixing of oxide powders to lower the amount of impurities, going to be produced while synthesizing the BFO. The sample was divided into two equal parts and subjected to pre-calcinations at 650 ˚C in air and nitrogen atmosphere respectively, for the duration of 60 minutes in muffle furnace and tubular furnace. The calcinated samples once again grounded thoroughly for an hour in dry media and then subjected to heating at 810 ˚C for in air and nitrogen atmosphere for another 60 minutes. Both furnaces were cooled down naturally and the powder samples were finally grounded for XRD characterization. As there were presences of impurities in the samples they were leached in 0.1 M HNO_3 and kept into oven to remove the excess amount of HNO_3 at 200 ˚C. XRD characterization of leached samples shows the partial removal of the impure phases, we again leached those samples with 0.2 M HNO_3, this time XRD confirms the complete removal of impure phases from the sample which was calcinated and sintered in air. The fig. 2 represents the methods used for synthesis of BFO. XRD measurement is much helpful to check the present phases of the material. We have used Philips Xpert MPD; with Cu K_α radiation ($\lambda = 1.5405$ Å), for this characterization which was available at SICART, Vallabh Vidyanagar. The step size was 0.5 degree with total scanning time 30 minute. The XRD patterns of all the samples were analyzed and the result is as follows.

RESULTS AND DISCUSSION

Figure 3(A–E) shows the results of XRD characterizations: Fig. 3(A) is the powder pattern of the sample sintered at 810 ˚C in air and 3(B) is the powder pattern of the sample sintered at 810 ˚C in N_2 atmosphere. After first leaching another XRD measurements were taken for phase purity identification and they were as shown in 3(C) and 3(D) for sample prepared in air and N_2 atmosphere, respectively. The notable decrement in the impurity phase intensities were ranging from 0.37% to 4.80% in the sample heated in air. The sample prepared in N_2 atmosphere was not giving favorable result; hence it was not leached further. Figure 3(E) is the XRD pattern of the sample which is single phase BFO, it was obtained after second leaching of sample sintered in air. The percentage of Impurities obtained from PowderX analysis is listed in Table 1. This XRD pattern was matched with JCPDS No. 86-1518 (*10*) and conforms the pure phase BFO derived from the minor mixed phase BFO synthesized using SSR in air atmosphere**.**

CONCLUSION

We have synthesized 15 gm BFO bulk in one batch using this technique. From the leaching process with 0.1 M HNO_3 followed by 0.2 M HNO_3 one can remove secondary phase, present in the BFO; which was generated while synthesizing BFO through solid state reaction with temperature and duration of heat treatments mentioned in this paper. SSR technique gives mixed phase but leaching is remarkably able to remove the minor mixed phase present in the sample. So as mentioned earlier, this SSR technique can help researchers to synthesize BFO in pure form in bulk and we have successfully achieved single phase BFO though this technique.

FIGURES

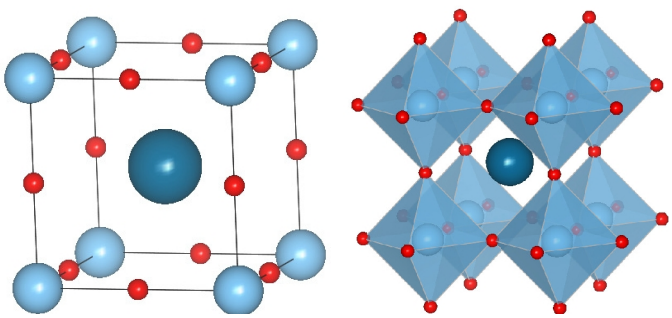


Fig. 1. The schematic diagram of the structure of ABO_3 perovskite, where the darker, the lighter and smaller spheres represent A, B cations and oxygen, respectively.

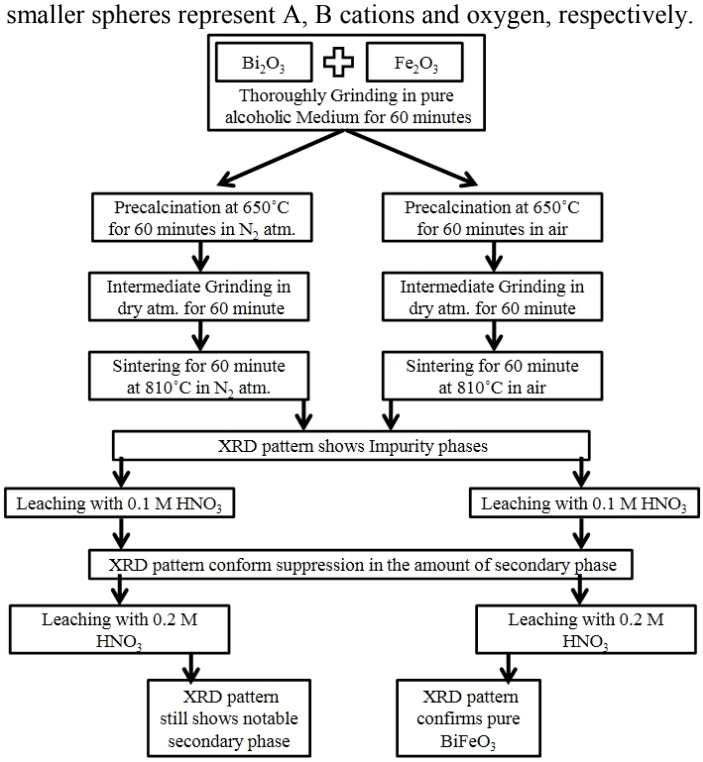


Fig. 2. Synthesis of BFO using Solid State Reaction Method.

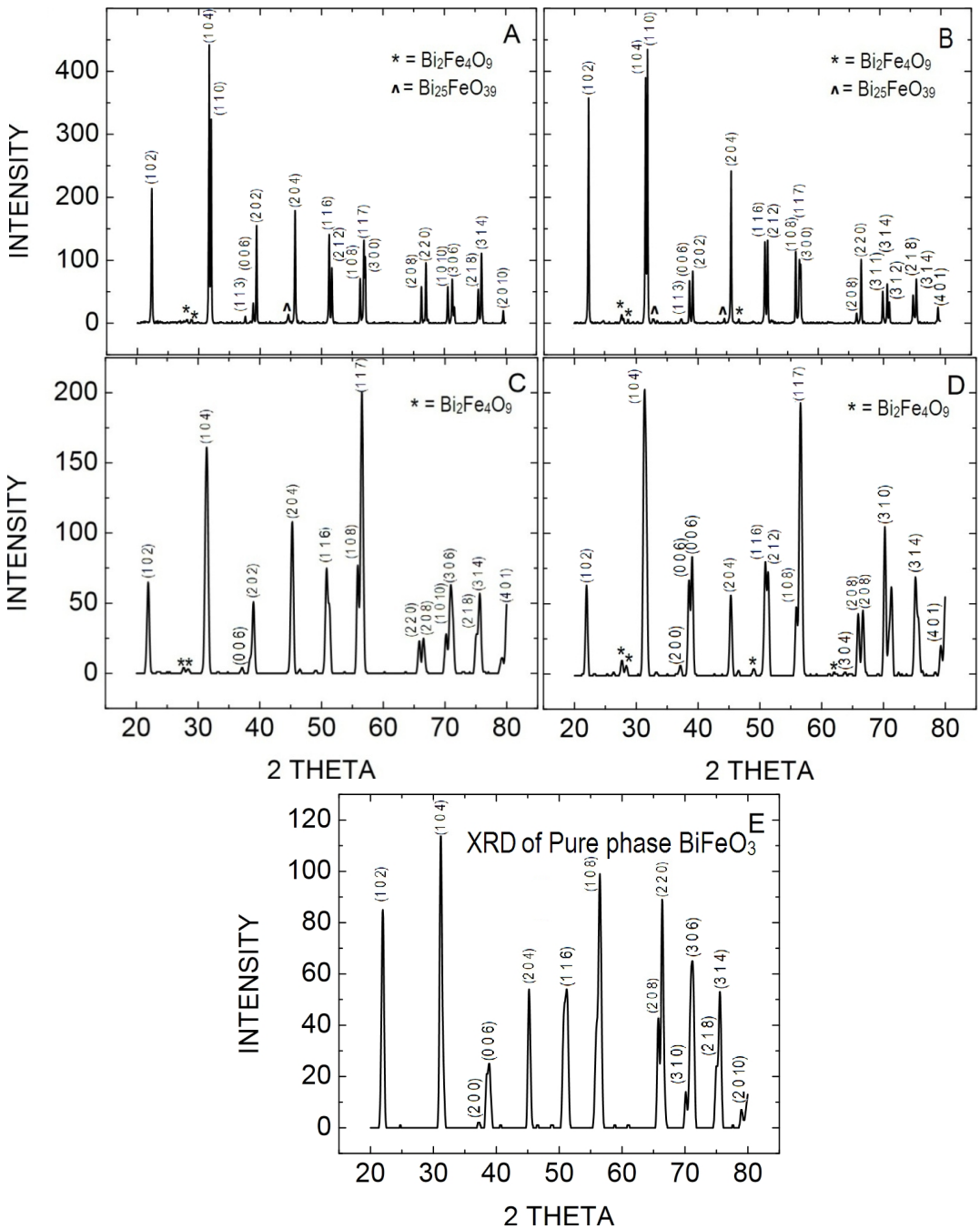


Fig. 3. XRD of BFO sample sintered in air (A) and N_2 gas (B) shows impurity phases before leaching, (C) and (D) corresponds to the patterns of same samples after first leaching. Fig. 3(E) is the pattern of pure BFO, obtained after second leaching of sample treated in air.

TABLE

Table 1. Table of intensity comparison of BFO samples.

Figure	Intensity Maximum		Impurity Peaks			Impurity	Percentage
	I max	2θ	No.	Intensity	2θ		
A	431.34	31.74	3	12.31	28.1	$Bi_2Fe_4O_9$	2.85
				8.11	28.87	$Bi_2Fe_4O_9$	1.88
				20.69	44.63	$Bi_{25}FeO_{39}$	4.80
B	413.95	32.05	5	16.48	27.82	$Bi_2Fe_4O_9$	3.98
				8.88	28.86	$Bi_2Fe_4O_9$	2.14
				8.75	32.94	$Bi_{25}FeO_{39}$	2.11
				8.2	44.64	$Bi_{25}FeO_{39}$	1.98
				9.4	47.01	$Bi_2Fe_4O_9$	2.27
C	210	56.65	2	4.28	27.67	$Bi_2Fe_4O_9$	2.04
				3.17	28.4	$Bi_2Fe_4O_9$	1.51
D	210	31.31	4	12.89	27.72	$Bi_2Fe_4O_9$	6.14
				7.87	28.43	$Bi_2Fe_4O_9$	3.75
				5.92	49.09	$Bi_2Fe_4O_9$	2.82
				3.55	62.01	$Bi_2Fe_4O_9$	1.69
E	153.82	31.2	0	0	0	Pure Phase	0

ACKNOWLEDGEMENT

We are thankful to Department of Physics, Saurashtra University for their financial support and SICART for providing us XRD measurements.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

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RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

ISOLATION OF FUNGI FROM MUNICIPAL SOLID WASTE FOR AMYLASE ACTIVITY

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ABSTRACT

Sixteen fungal isolates from municipal solid waste were isolated . Out of sixteen two fungus were produced high yield of amylase . Which was later identified as *Aspergillus spp*. and *Mucor spp.* when inoculated at different temperature showed maximum enzymatic activity (2.5U/ml) at 35-37^{0}C and *Mucor spp.* Showed maximum enzymatic activity (5.9U/ml) at 30^{0}C.Sodium nitrate was the best nitrogen source in case of *Mucor spp*. (4.4U/ml) whereas Urea was the best nitrogen source in case of *Aspergillus spp.*(6.6U/ml).Sucrose was best carbon source in *Mucor spp*.(5.2U/ml) whereas Glucose in case of *Aspergillus spp*.(6.7U/ml).

SUMMARY

Microbial amylase, break down starches into sugars which has gained profound importance in industries and waste degradation if we provide suitable conditions to them.

Keywords: *Aspergillus niger*, *Mucor spp*. , amylase, Substrate, Solid state fermentation.

INTRODUCTION

Municipal solid wastes soil contains many major groups of microorganisms having bacteria, actinomycetes, fungi, algae and protozoa, and from these bacteria and fungi are the most abundant group and the most important microbe for decomposing waste. Generally they use wastes for their own metabolism and produce some useful compounds which are important for soil health, plant growing. For the hydrolysis of polysaccharides amylases are significant enzymes employed in the starch processing

industries. Several microorganisms can produce amylases enzyme. In industries amylase enzymes plays a major role to decompose starch. Many bacteria and fungi produce amylase such as *Bacillus spp.* and*Aspergillus spp. a*nd many filamentous fungi.
These enzymes are highly demanding in food industries, pharmaceutical industries, starch processing industries, baking industries, alcohol production and brewing industries. Amylase produced by fungi is most preferred than other microbial sources.

MATERIALS AND METHODS

COLLECTION OF SAMPLE: - Partially decomposed part of solid municipal waste was collected from different localities of Solan district of Himachal Pradesh (Chambaghat, Old Bus Stand Solan, Salogra).

ISOLATION OF FUNGI

SERIAL DILUTION METHOD:-
Isolation of mesophilic fungi was done with serial dilution method, serial dilution of soil sample were made in distilled water. One gram of sample was added in 9 ml of distilled water and then serially 1ml was transferred in seven tubes to obtained 10^{-1}, 10^{-2}, 10^{-3}, 10^{-4}, 10^{-5} ,10^{-6} ,10^{-7}and10^{-8} dilution.

AGAR PLATE METHOD:-
0.5ul of sample was taken from 10^{-6}, 10^{-7} and 10^{-8} dilution and dispended over the solidified potato dextrose agar medium plates. The suspension was spread over the medium using the sterilized glass spreader. The plates were incubated at 25^{0}C for 72 hours to isolate fungal colonies.

IDENTIFICATION OF THE ISOLATES:-The selected fungal isolates were identified based on different microscopic and macroscopic characters i.e. (shapes, arrangement of spores, colour, mycelialcolour under light microscope) and pure culture was maintained.

Lactophenol Cotton Blue Staining:- Loops full of fungal cultures were placed on a clean glass slide, and mixed with a one drop of lacto phenol cotton blue stain. A clean cover slip was placed over the culture and the morphology of fungal isolates were observed viewed under the microscope (45 X) and and photographed.

SCREENING OF FUNGI PRODUCING AMYLASE

For amylase production by starch hydrolysis all the isolates were tested. Starch agar medium was inoculated with the test organism and incubated it overnight and after that flooded with iodine solution for 2 minutes after 2 minutes zone of clearance around the microbial growth indicated the production of amylase. Maximum zone of clearance showed by isolates were selected for further studies on amylase production. By using a sterilized dissecting needle amylase-producing fungi were transferred to fresh plates of Potato Dextrose agar containing 1 % starch. Fungal isolates were allowed to grow for 72hours, then store in the refrigerator.

PRODUCTION OF AMYLASE:

Preparation of seed culture
The seed culture (50ml) was prepared by inoculating a loopful of pure culture .The flasks were incubated for 72hrs with continuous shaking (130rpm) at 25°C.

Production of enzyme:
Two ml of seed culture was inoculated in 50ml of production medium Beef extract (0.25%), Peptone (0.15%) and starch (1%) and grown for 72hrs in shaking incubator at temperature 25°C at 130rpm. Flask containing the growing fungus was poured through a funnel fitted withWhattman filter paper No.1. The filtrate remaining contained the crude amylase.

DETERMINATION OF ENZYME ACTIVITY:

AMYLASE ASSAY:
Procedure: 1 ml of crude "enzyme" was pipette into a test tube to it add 1 ml of 1% starch mixed with citrate-phosphate buffer (pH6.5). Incubated the culture in a water bath at 40°C for 30 minutes. A blank was set up consisting of 2ml of the enzyme extract that has been boiled for 20 minutes and added to the starch solution. 2 ml of DNS reagent was added in both test and control to stop the reaction. Boiled for 5 minutes. Cooled and added 20 ml of distilled water. Colour intensity was determined at 540 nm.
One unit (IU/ml) of amylase activity defined as the amount of enzyme that release 1ug of reducing sugar as glucose per gram of dry substrate per minute, under standard assay conditions.

OPTIMIZATION OF CULTURE CONDITION.

DIFFERENT pH EFFECTS: pH in the range of 5,6,7,8 & 9 were examined for their effect on growth of microorganisms by the selected isolate grown in production media. The pH of the medium was adjusted by using 1N HCl or 1N NaOH .The inoculated adjusted at different pH at 25°C for 72hrs. Medium was filtered and mycelium of fungi was discarded. Supernatant was taken for assay of amylase.

DIFFERENT TEMPERATURES EFFECTS:
The effect of temperature were evaluated by incubating the 50 ml of sterile production medium inoculated with 2ml of seed culture at different temperatures (25°C, 30°C, 35°C, 40°C) for 72hr. The medium was filtered. Then amylase assay was done.

CARBON SOURCES EFFECTS ON AMYLASE PRODUCTION:
The effects of various carbon sources on amylase production were studied by replacing sugar in the basal medium with glucose, lactose and sucrose based on their molecular weight. The flasks were inoculated with 2ml seed culture and incubated at 25°C for 72 hrs. Then medium was filtered and amylase assay was done.

NITROGEN SOURCES EFFECTS ON AMYLASE PRODUCTION:
The effects of various nitrogen sources on amylase production were studied by replacing in basal medium with yeast extract, urea, sodium nitrate and ammonium sulphate. The flasks were inoculated with 2ml seed culture and incubated at 25ºC for 72hrs.Then medium was filtered and amylase assay was done.
Fungi were isolated from municipal solid waste (MSW) which was partially decomposed and rich in starchy products. In this study three sample (A, B and C) were collected from different parts of Solan.

RESULTS AND DISCUSSION

The results depicted in table 2 shows from the three samples A, B and C of municipal solid waste sixteen species of different genera were isolated which are designated as A1, A2, B1, B2, B3, C1, C2 andC3. From the sample B and C maximum number of the fungi were isolated.
Identification of the fungal isolates:-

A total of 16 isolates belonging to three different genera's were isolated. The isolated fungi were *Aspergillusniger*,*Mucor spp*. ,*Trichoderma spp.* The isolates were identified by macroscopic and microscopic method shown on Table 3 Seven isolates were identified as *Aspergillusniger,* 6 were identified as *Mucor* spp. and 3 were isolated as *Trichoderma spp.*

Screening of fungi producing amylase:-
All the fungal isolates were grown on starch plates growing isolates were flooded with iodine for 1 min. The data presented in table 4 shows *Aspergillusniger* and *Mucor spp.* showed maximum zone of clearance on the starch plates 8mm and 6mm respectively.

This procedure employed showed a positive result for the *Aspergillusniger* and *Mucor spp.*isolated . The mechanism of clear zone observed was due to the fact that the amylase produced during the growth of the microorganism has hydrolysed the starch around the colony.The un-hydrolysed part of the plate tested positive to the presence of starch hence the blackblue appearance.

Enzyme assay
Enzyme assay was performed for the two fungi *Aspergillusniger* and *Mucorspp*.which showed maximum zone of clearance on starch plates.

Amylase activity was calculated by comparing the O.D value of test sample with glucose standard curve (Graph 1). The perusal of data table 6 shows *Mucor spp.* optical Density (0.910) was when compared with glucose standard curve the concentration of glucose was found to be 5 µl/ml and *Aspergillusniger* optical density (0.199) was when compared with glucose standard curve the concentration of glucose was found 2µl/ml.

The perusal of data table 7 shows that *Aspergillusniger*when inocuated at different temperature 25, 30, 35 and 40^0C showed maximum enzymatic activity of amylase at 35^0C. Amylase activity shown by *Mucor spp.* was maximum at 30 ^{0}C. Optimum temperature for enzyme production by *Mucor spp.* has also been reported as 30^0C.

Table 8 shows *Aspergillus niger* produced high yield of enzyme at 5& 9 pH . According to varalakshi *et al*., 2009 *Aspergillus niger* showed high yield of amylase at pH 9- 9.5. Mohapatra *et al.*, 1998 reported that amylase from *Mucor spp*. showed maximum enzymatic activity at pH 5.0.

Table 9 Shows *Aspergillusniger* showed maximum amylase activity when supplimented with glucose as compared to *Mucor spp*. In *Mucor spp.* Sucrose showed maximum enzymatic activity of amylase. Suganthi*et al*., 2011 has also reported maximum activity of amylase with sucrose.

Table 10 shows Nitrogen sources greatly increased the yield of enzyme produced by *Aspergillusniger* .Panday ,2005 have also reported increased enzymatic activity in solid state fermentation with supplementation of organic and inorganic nitrogen sources.

CONCLUSION

Sixteen fungal isolates were isolated from municipal solid waste.

Three fungal isolates were further purified and identified as *Aspergillus* ,*Trichoderma* and *Mucor* species.

Two fungal isolates show amylase activity that is *Aspergillus* and *Mucor spp.*

Maximum enzymatic activity (4.1U/ml) was recorded with *Mucor* followed by *Aspergillus spp*.(1.8U/ml).

Aspergillus spp. when inoculated at different temperature showed maximum enzymatic activity (2.5U/ml) at 35-37^{0}C and *Mucor spp.* Showed maximum enzymatic activity (5.9U/ml) at 30^{0}C.

Optimum pH for *Aspergillusniger*and *Mucor spp*. was pH 5 and 9.

Sucrose was best carbon source in *Mucor spp*.(5.2U/ml) whereas Glucose in case of *Aspergillus spp*.(6.7U/ml).

Sodium nitrate found best nitrogen source in case of *Mucor spp*. (4.4U/ml) whereas in case of *Aspergillus spp.* urea found to be best nitrogen source(6.6U/ml).

FIGURES

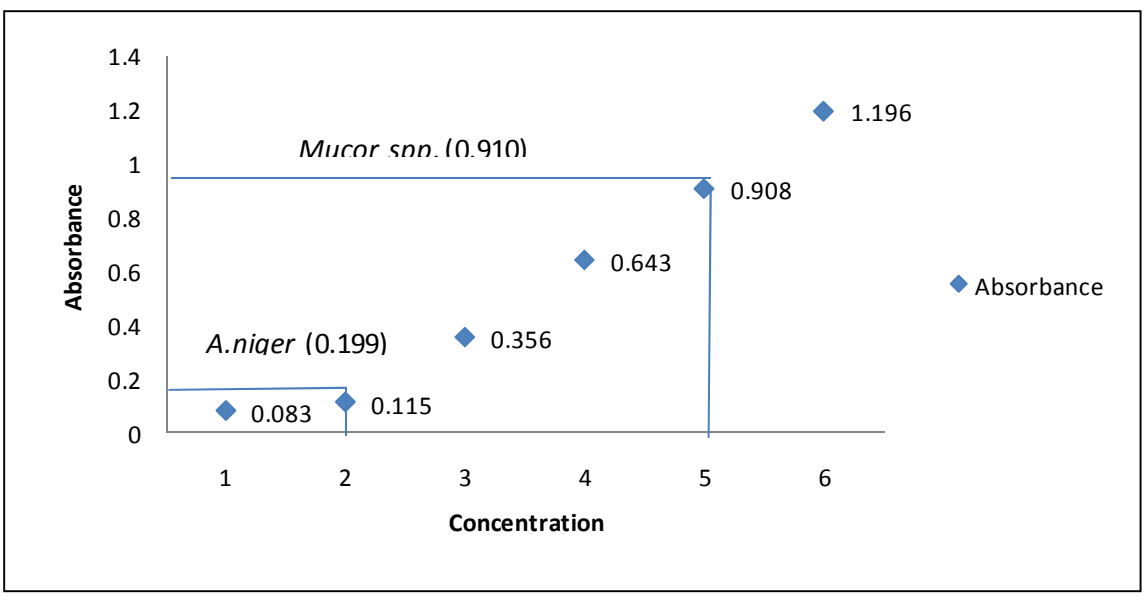


Fig. 1.Glucose Standard Graph

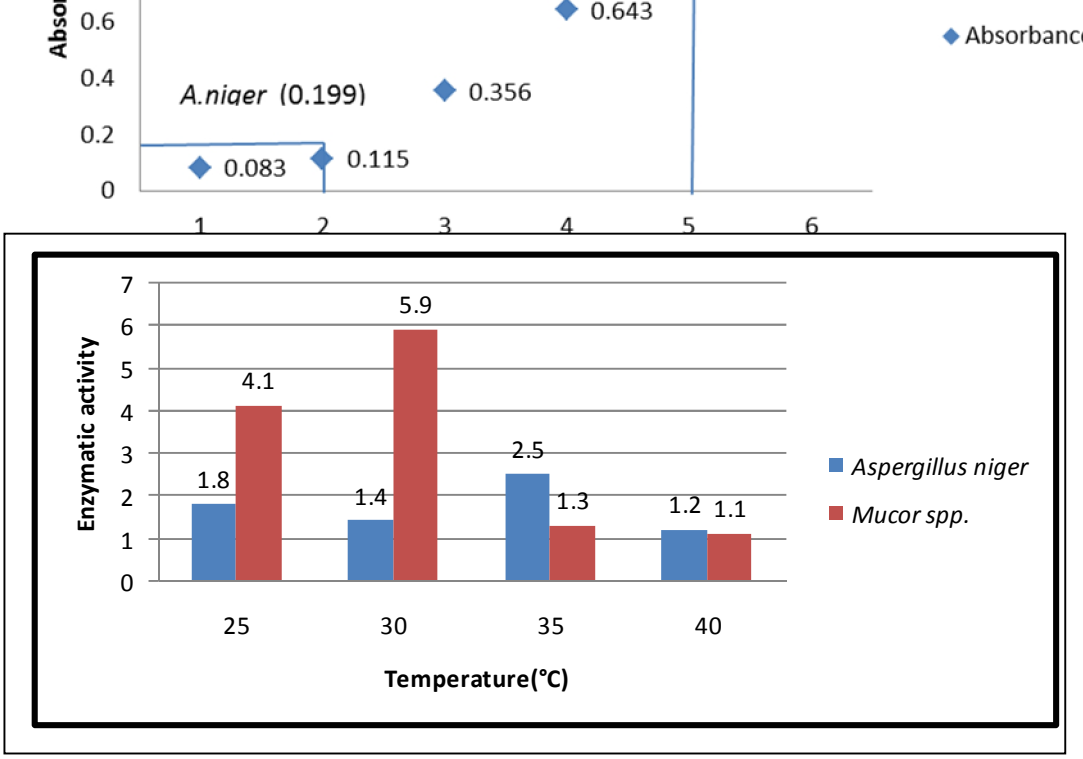


Fig. 2.Temperature effects on amylase production by fungi.

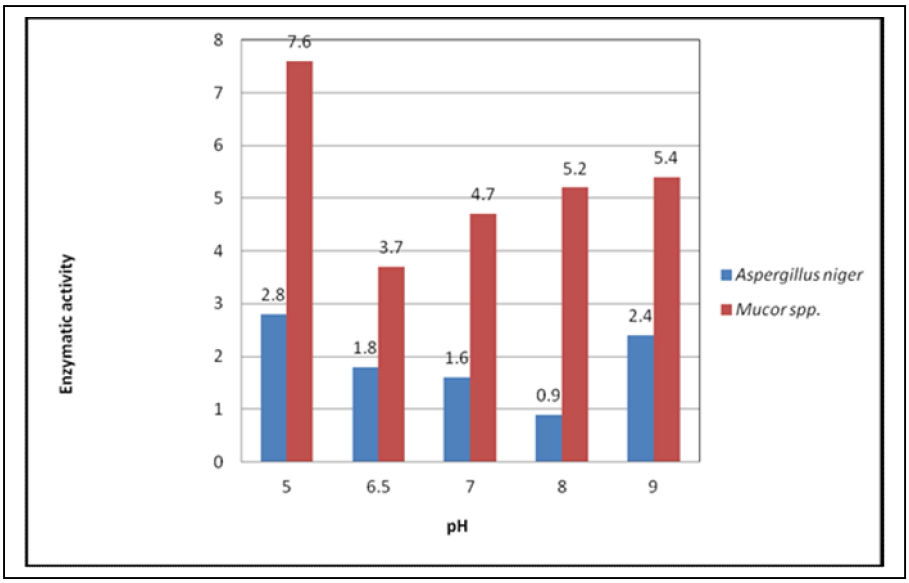


Fig. 3. pH effects on amylase production by fungi.

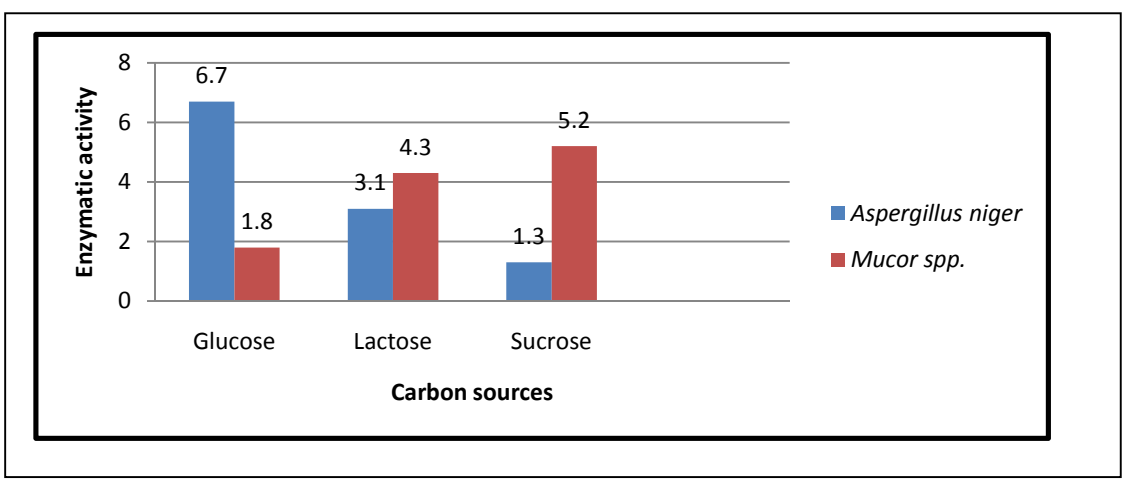


Fig. 4. Carbon sources effects on amylase production by fungi.

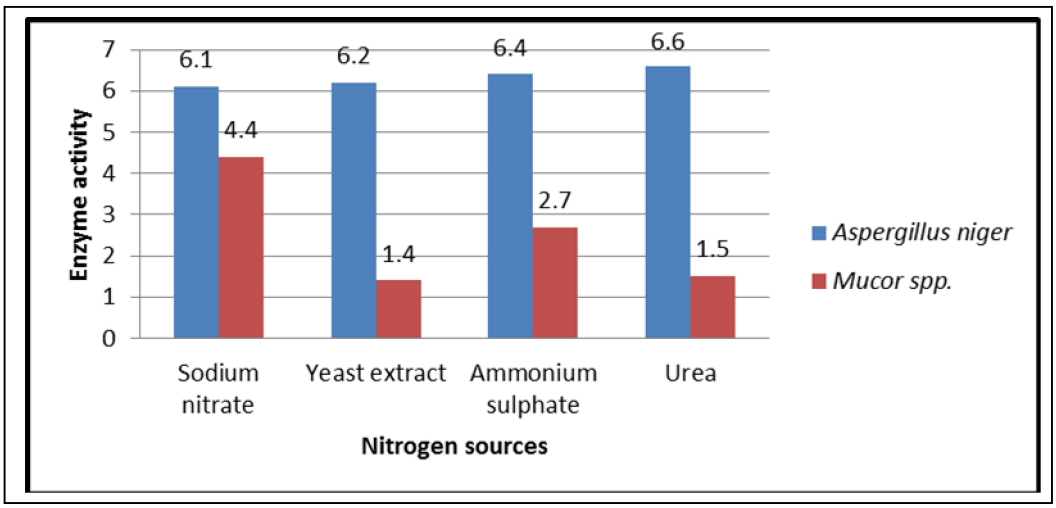


Fig. 5. **Nitrogen sources effects on amylase production by fungi.**

TABLES

Table 1.Collection of samples (Municipal Solid Waste) from different locations of solan.

Serial no.	Sample no.	Place of collection
1	A	Salogra
2	B	Chambaghat
3	C	(Old Bus Stand) Solan

Table 2. Total number of fungi isolated from different municipal solid waste samples.

Serial no.	Sample no.	Fungal isolates	No. of fungal colonies

1	**A**	**A1**	**2**
		A2	**1**
2	**B**	**B1**	**3**
		B2	**1**
		B3	**2**
3	**C**	**C1**	**2**
		C2	**2**
		C3	**3**

Table 3.Macroscopic characters & Microscopic characters of fungus isolates.

Isolates	Fungi	Macroscopic characters	Microscopic Characters
A1, B1, C1=7	***Aspergillusniger***	White colonies became black as culture mature.	Single cell spores in chain developing at the end of the sterigma arising from the terminal bulb of the conidiophores ,the vesicle long conidiophores arise from a septate mycelium
A2, B3, C3=6	***Mucor spp.***	White coloured fungus aerial mycelium cottony and fuzzy	Spores are oval;nonseptate mycelium give rise to single sporangiophores with globular sporangium containing a columella, there are no rhizoids.

B2, C2=3	***Trichoderma spp.***	Green colonies spread all over the petriplate.	Macroconidia and microconidia spread on slide and forming clump with each other.

Table 4.Hydrolysis of starch by the isolated fungus.

Serial no.	**Fungi**	**Diameter(mm)**
1	*Aspergillusniger*	8
2	*Mucor spp.*	6
3	*Trichoderma spp.*	-

Table 5.Glucose Standard Method.

Serial no.	GLUCOSE (µl/ml)	CITRATE PHOSPHATE BUFFER (µl/ml)	di-Nitro Salisilic acid (ml)		O.D at 540nm
1	900	0	3ml		2.084
2	800	100	3ml		1.934
3	700	200	3ml	Incubation Time for	1.563
4	600	300	3ml	15min at 45^0c	1.196
5	500	400	3ml		0.908
6	400	500	3ml		0.643
7	300	600	3ml		0.356
8	200	700	3ml		0.115
9	100	800	3ml		0.083
10	0	900	3ml		0

Table 6 Amylase assay for *Aspergillus*and *Mucor spp.*

Serial no.	Fungus	O.D (540nm) 0f Test sample	Enzymatic activity	Control
1	*Aspergillus spp.*	0.199	1.8	0.016
2	*Mucor spp.*	0.910	4.1	0.148

Table 7. Temperature effects on amylase production by fungi.

Serial no.	Temperature (^{0}C)	O.D (540nm) (*Aspergillus spp.)*	Enzyme activity (IU/ml)	O.D (540nm) (*Mucor spp*.)	Enzyme activity (IU/ml)
1	25	0.205	1.8	0.910	4.1
2	30	0.164	1.4	1.235	5.9
3	37	0.279	2.5	0.386	1.3
4	40	0.148	1.2	0.154	1.1

Table 8.pH effects on amylase production by fungi.

Serial no.	Different pH	O.D (540nm) (*Aspergillus spp.)*	Enzyme activity (IU/ml)	O.D (540nm) (*Mucor spp.)*	Enzyme activity (IU/ml)
1	5	0.305	2.8	1.617	7.6
2	6.5	0.205	1.8	0.910	3.7
3	7	0.184	1.6	1.092	4.7
4	8	0.115	0.9	1.275	5.2
5	9	0.262	2.4	1.215	5.4

Table 9. Carbon sources effects on amylase production by fungi.

Serial no.	Carbon sources	O.D (540nm) (*Aspergillus spp*.)	Enzyme activity (IU/ml)	O.D (540nm) (*Mucor spp.)*	Enzyme activity (IU/ml)

1	GLUCOSE	0.842	6.7	0.481	1.8
2	LACTOSE	0.468	3.1	0.948	4.3
3	SUCROSE	0.282	1.3	1.096	5.2

Table 10. Nitrogen sources effects on amylase production by fungi.

Serial no.	**Nitrogen sources**	**O.D (540nm) (*Aspergillus spp.)***	**Enzyme activity (IU/ml)**	**O.D (540nm) (*Mucor spp.)***	**Enzyme activity (IU/ml)**
1	SODIUM NITRATE	0.887	6.1	0.942	4.4
2	YEAST EXTRACT	0.892	6.2	0.392	1.4
3	AMMONIUM SULPHATE	0.916	6.4	0.633	2.7
4	UREA	0.936	6.6	0.407	1.5

ACKNOWLEDGEMENT

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Analytical Method Development and Validation of Levofloxacin Drug by Fast Reverse Phase-High Performance Liquid Chromatography

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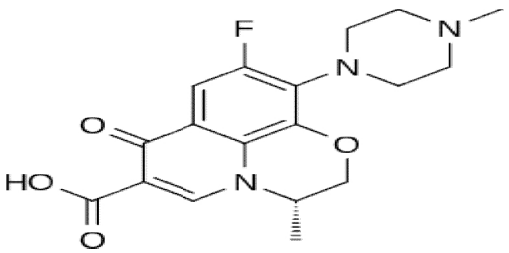
ABSTRACT

A rapid and valuable isocratic chromatographic method has been effectively developed to convincing Levofloxacin drug using fast RP-HPLC. The method is worked in column having specification of RP C-18 (Eclipse Plus (4.6mm I.D. ×100 mm, 3.5 µm), mobile phase consist of a intermixture of ACN: H_2O (68:32 v/v; with phosphoric buffer pH 5.5), the identical rate of flow set at 0.001 L/min with wavelength 295 nm. The linearity curve was found to be linear in the rang 40 to 140 ppm with significantly value of correlation coefficient (r^2> 0.993). LOD as well as LOQ were observed to be 0.1 and 0.4 (µg/ml) accordingly. The % RSD of assay within a day and between two days (intra and inter days) is < 0.70 %. This proposed study was recovered to be precise, reproducible and accurate within the satisfactory range reported to ICH guidelines and can be relevant for the discovery of LF.

Keywords: Fast RP-HPLC, Levofloxacin Tablet, method development and validation.

INTRODUCTION

In 1987 Levofloxacine (LF) was prototypal patented by Daiichi Pharmaceutical Co., Ltd. (European patent) and it was authorized by the U.S., F.D.A since 20 December 1996 for the treatment of bacterial sinus infection, bacterial bronchitis, pneumonia, simple infections of skin , complex infections of urinary tract, and acute pyelonephritis.*(1).* This development challenged by Lupin Pharmaceuticals who is a generic drug manufacturer ; presented that the levofloxacine is an individual isomer of previously authorized antibacterial levofloxacine, and is not a "novel active component" and hence inappropriate for patent term delay.



Chemical structure of levofloxacin

According to IUPAC the name of drug LF is (S)-9-fluoro-2,3-dihydro-3-methy(4methyl piperazin-1-yl)7oxo-7*H*-pyrido[1,2,3]-1,4-benzoxazine-6-carboxylic acid. M.F of LF is $C_{18}H_{20}FN_3O_4$ and mass of LF is 361.368 g/mol. Class of drug LF is a antibiotic class. *(2).* and the LF is the (L) isomer of its ofloxacinand the spectrum of LF is actexcludes, the majority of bacterial pathogen strains including gram(- ve) and gram (+ ve) responsible for infections of respiratory, urinary tract, gastrointestinal, and abdomen, and different bacterial pathogens*(3).*LF is utilized selected in or bind with few other antibacterial drugs to treat definite infections caused by bacteria apart from pneumonia,*(4)* urinary tract health problem, *(5,6).* and skeletal strength infections.*(7).*

MATERIALS AND METHODS

Instrumentation

HPLC (Model: Agilent Intinity 1220 LC with 1260 Fraction collector) , detector DAD (190nm to 948nm), electronic balance (Shimadzu) was used for the purpose of weighing samples were injected manually on to HPLC system .

Materials and reagents

Levofloxacine (LF) kindly provided as a sample by cipla, ACN (HPLC grade), Me-OH (HPLC grade), H_2O (HPLC grade). All the chemical agent used of analytical grade and used without promote cleanup.

Chromatographic conditions

The mobile phase for the proposed method was acetonitrile: water (68:32) (pH adjested to 5.5 with phosphoric buffer) and it was strained by 0.2 µm (N66) filter paper (membrane). It was degassed with tank to the column (Eclipse Plus RP C-18 (4.6mm I.D. ×100 mm, 3.5 µm) .The rate of flow is 1.0 ml/min and ran time is 1 min. the column temperature was kept up before injecting the LF drug solution ; according to system rules column was equilibrated minimum 1 hour aline the mobile phase . The eluent was show at 295nm wavelength.

Stock Solution Preparation

Accurate weight of LF i.e 10 mg was weighted individually and shift into two a choice of 10 ml calibrated volumetric flask. LF was wet minor quantity of Me-OH and fill the flask the up to the mark with Me-OH, which was give to concluding solution containing 5.0µg/ml of LF. The mixture was introduced to ultra sonification for 10 min will separated out with 0.2 µm (N_{66}) (membrane) filter paper.

Choice for Mobile Phase

LF was inserted into the fast RP-HPLC instrument and run in various solvent systems. Various mobile phase like ACN, Me-OH : H_2O, ACN : Me-OH, THF, Me-OH: HCOOH, ACN : Me-OH : 0.1% TEA etc. at were tested with variable mobile phase quantitative relation was arrange to find the high-grade situation for the modification of LF. The different chromatograms was found with different mobile phase , and the ACN : Water (68:32) (pH adjusted to 5.5 with phosphoric buffer) is chosen as mobile phase.

Mobile Phase preparation

ACN:Water (HPLC grade) was filter by 0.45 µm, 47 mm membrane filter paper , then it was ultra sonicated for 10 minutes on ultrasonicator and finally the Mobile phase was prepared by mixture of ACN: Water(68:32).

RESULTS AND DISCUSSION

Method Development and Optimization

the drug LF was finished by propose method i.e analytical method development and validtion , which is shows the high-grade elution of the peak. The particularity experiment report shows the analytic chromatogram is not qualified to more than one constituent. (Fig.1)

Validation of the Method *(8)*

Linearity of Levofloxacin (Calibration curve)

Six points standardization calibration curve was got by plotting Levofloxacin concentration v/s peak area, there is the concentration range was 5 µg/mL (correlate to 40 to 140 ppm, severally of the experimental mixture concentration) (Fig.2). The linear equation of regression for proposed method was y = 27.271x + 1038.49 where x = concentration in (µg/mL) and y = peak area in (absorbance units); the correlation of correlation was obtain 0.993. (Table-1, 2).

Accuracy Study

one of the most important parameter for measurement of the recovery i.e accuracy.In this method the three different concentrations (related to 100, 120 and 140% of experimental solution) .The amount of Levofloxacin (i.e, known amount 5 µg/ml) was added to test solution. This amount of LF was recovered, in the presence of test intervention. For to each one concentration, three sets was make and inserted in reproduction. The % Recovery of LF was calculated at each level and recorded which as shown in below (Table-3). The mean recovery of was between 95.58- 99.75 which is satisfactory.

LOD and LOQ study

The LOD as well as LOQ which were observed by the calculating the signal to noise (S/N) ratio of the LOD preparation and LOQ preparation. The concentration for LOQ and LOD which is found to be 0.4 µg/ml and 0.1 µg/ml separately. (Table-4).

PRECISION

Repeatability Data for Levofloxacin

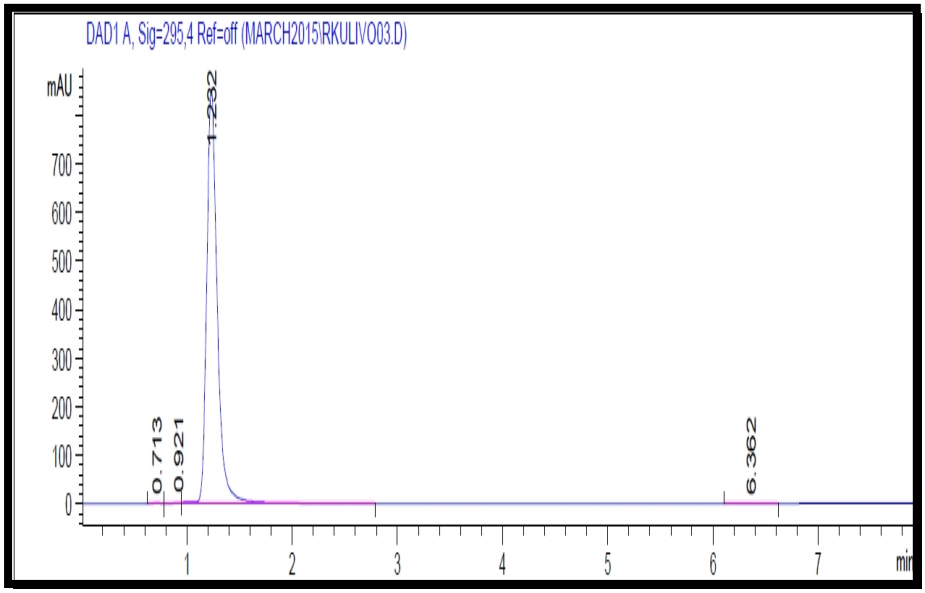
Calculation of standard error (S.E), Standard deviation (S.D.)and relative standard deviation (% R.S.D.) for LF, indicating high degree of precision of the method show in table. The % relative standard deviation (% R.S.D.) which is Found less than 1% as essential by ICH and USP guidelines. (Table-5, 6, 7)

From above found data of the calibration curve shows linear response above the range of concentration used and precision data shows the reputability of the assay procedure which was satisfactory. The accuracy of the method was observed by recovery studies of LF. The recovery studies was carried out by the calculation of % recovery.

CONCLUSION

The present paper describes the estimation of Levofloxacin by fast RP-HPLC which is less time required then simple HPLC method representative UPLC like performance at HPLC backpressures therefore, the analysis time requirement as well as solvent consumption will be less. Hence, the method is cost-effective. The method was found to be specific, precise, reproducible and accurate, as depicted through the statistical analysis of data. The Results of validation parameters confirmed that the analytical study is suitable for its proposed purpose as routine quality control method for analysis of Levofloxacine as such and in different pharmaceuticals products.

FIGURES



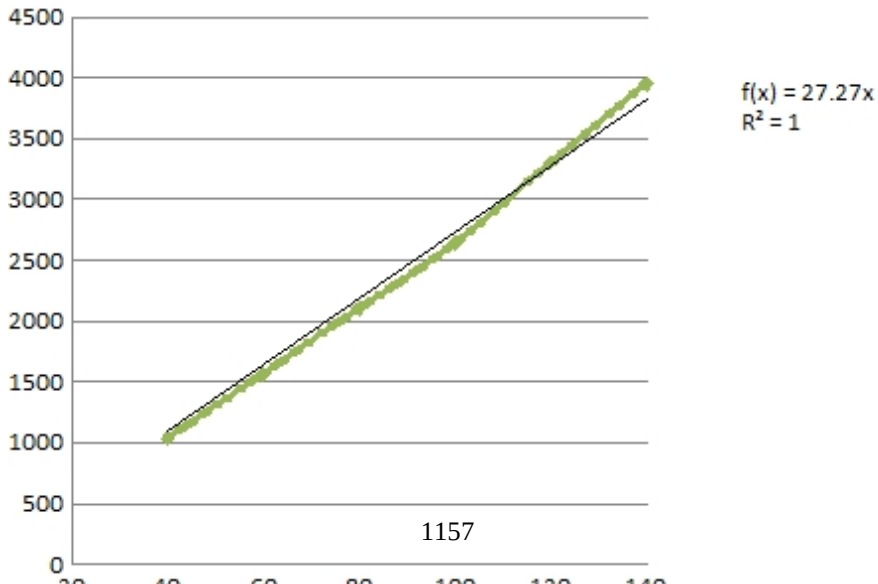


Fig. 2: Linearity curve for Levofloxacin

TABLES

Parameter	**Mean**
Slope	27.271
Intercept	1038.49
RegressionCoefficient(r2)	0.9933

Table-1:Statistical validation of Liner Regression of Levofloxacin standard (n=3)

Sr. No	**Concentration ppm**	**Area**
1	40	1038.49
2	60	1566.55
3	80	2101.03
4	100	2637.8
5	120	3302.63
6	140	3946.97

Table -2 : Linearity table of Levofloxacin (n=6)

Concentration Level (%)	**Mean Recovery (%)**	**Standard Deviation**	**Relative Standard Deviation (%)**	**Standard Error**
100	99.75	0.03536	0.035	0.025

120	87.5	1.0607	1.212	0.750
140	95.58	0.676	0.707	0.465

Table-3: Statistical Validation of Recovery Studies Levofloxacin (n=3)

Drug	LOD (µg/ml)	LOQ (µg/ml)
Levofloxacin	0.1	0.4

Table 4: LOD and LOQ Data for Levofloxacin (n=1)

Drug Levofloxacin	Mean (%)	S.D.	%R.S.D	S.E
Standard	99.96	0.02887	0.2888	0.01667
Sample	99.96	0.02887	0.2888	0.01667

Table-5: Repeatability Data for Statistical Validation (n=2)

Drug Levofloxacin	Mean (%)	S.D.	% R.S.D	S.E
Standard	**99.97**	**0.03536**	**0.0356**	**0.025**
Sample	**95.85**	**0.6576**	**0.6860**	**0.4650**

Table-6: Intra-day precision data for Statistical Validation of Levofloxacine (n=2)

Drug Levofloxacine	Mean (%)	Standard Deviation	Relative Standard Deviation (%)	Standard Error
Standard	99.97	0.03536	0.0356	0.025

Sample	95.85	0.6576	0.6560	0.4650

Table -7: Inter-day precision data for Statistical Validation of Levofloxacine (n=2)

n is the number of replicates.

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Functional modification of κ-carrageenan: A facile synthesis of a fluorescent κ-carrageenan-guanine derivative

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ABSTRACT

A new fluorescent polymeric material was synthesized by grafting the nucleobase guanine on to the backbone of κ-carrageenan. The synthesis involved a rapid water based method under microwave irradiation using potassium persulphate (KPS) as an initiator. The emission spectrum of the modified κ-carrageenan recorded in 0.1 M aqueous NaOH ($5x10^{-5}$ M) solution exhibited emission maxima ($\lambda_{em,max}$) at 340 nm by excitation at 274 nm. The emission intensity was enhanced by ca.50% compared to that of pure guanine solution of the same concentration. When the concentration of the pure guanine solution is made equivalent to the concentration of the guanine molar component ($3.18x10^{-5}$) present in $5x10^{-5}$M solution of modified κ-carrageenan, then ca.73% enhancement in emission intensity was observed. The remarkable fluorescent activity of the κ-carrageenan-guanine derivative may have potential uses as sensor in various applications.

SUMMARY

κ-carrageenan of *Kappaphycus alvarezii* was grafted with nucleobases guanine.

Keywords: Carrageenan, Guanine, Fluorescence

INTRODUCTION

Fluorescence phenomenon was harnessed to study agarose gelling system.[1] Polysaccharide conjugates were prepared with fluorescein to distinguish underivatized polysaccharides as well as for localizing and quantifying cell surface proteins in cell biology research.[2] Other fluorescent polysaccharides and their conjugates were prepared with an eye to identifying biomolecules, sensing pH as well as preparing cellulose based organic light emitting diode.[3-7] Modification of cellulose with amino compounds and their fluorescence properties have reported.[8] A facile synthesis of a fluorescent agarose-guanine derivative has been reported.[9] Synthesis and fluorescent properties of pyrene-lableled guanine base was reported for studying the secondary structures of G-rich DNA.[10] There exist numerous reports in the literature on the modification of polysaccharides employing various strategies e.g. grafting, cross linking etc.

In an ongoing program of the author's laboratory on the modification of seaweed polysaccharides for preparing new materials with improved functional properties.[11-20], we report herein functional modification of κ-carrageenan (Fig. 1) by grafting guanine (Fig. 1) on to κ-carrageenan by a water based method. This guanine modified κ-carrageenan exhibited exceptionally strong fluorescent properties. Guanine is 2-amino-6-hydroxypurine, which is one of the four nitrogenous bases found in nucleic acids.[21] Carrageenans represent yet another prominent class of gelling polysaccharide obtainable from red seaweeds known for its versatility envisaging wide areas of applications which include food, feed, pharmaceuticals and agri-horticulture. Major carrageenans are termed ι-, κ-, λ-carrageenans. Structurally, these carrageenans are consisted of sequences of: D-galactose-4-sulphate and 3,6-anhydro-D-galactose-2-sulphate (ι-carrageenan), D-galactose-4-sulphate and 3,6-anhydro-D-galactose (κ-carrageenan) (Fig.1). D-galactose-2-sulphate and D-galactose-2,6-disulphate (λ-carrageenan).[22] To our knowledge, this carrageenan derivative and its effects are being reported for the first time.

MATERIALS AND METHODS

2.1 Materials

κ- carrageenan used in this study was extracted from the seaweed *Kappaphycus alvarezii (Craigie and L).* Other chemicals used in this study (e.g. sodium hydroxide, potassium persulphate (KPS) and guanine, LR grade) were purchased from S.D. Fine Chemicals Ltd. Mumbai (India).

2.2 Synthesis of carrageenan-graft-guanine

A known weight of κ-carrageenan (100 mg) was dissolved in 20 ml of hot water, to which 10.0 mg (0.738 mM) of KPS was added and mixed well. In a beaker, a known weight (50 mg) of guanine was dissolved in 20 ml of 0.5 M aqueous sodium hydroxide (pH ca. 11.6), and then mixed with the hot carrageenan sol and KPS mixture under stirring condition followed by microwave irradiation for 5 min. The colorless reaction mixture got converted slowly into a dark yellow mixture. The yellow colored product was isolated from the reaction mixture by precipitation with isopropanol (IPA) (reaction mixture:IPA=1:2 v/v), and was air dried. The dry product was redissolved in 0.1 M NaOH solution (ca. pH 9.5), when unreacted guanine (superimposable IR spectra) got precipitated out, leaving behind the yellow product in the solution. Unreacted guanine was filtered off. To the filtrate was added slowly dilute HCl (0.1 M) to bring the pH ca. 7.7, when onset of clouding of the solution was observed. The product was the isolated by precipitation with isopropanol (IPA) (reaction mixture:IPA=1:2 v/v) at pH 7.7. The weights of unreacted guanine and carrageenan-*graft*-guanine were determined.

2.3 FT-IR spectra

The non-modified and modified κ-carrageenan was characterized by FT-IR analysis using a Perkin-Elmer FT-IR machine (Perkin-Elmer Spectrum GX FT-IR System, USA), by taking 10.0 mg of

sample in 600 mg of KBr. All spectra were average of two counts with 10 scans each and resolution of 4 cm^{-1}. IR spectra were recorded as KBr pellets.

2.4 UV-Vis and Fluorescence spectroscopy

The UV-vis absorption spectra of the modified and non-modified κ-carrageenan were obtained on a Varian CARY 500 UV-VIS-NIR spectrophotometer, Pittsburgh, USA. The fluorescence spectra were recorded at room temperature on a Perkin-Elmer Spectrofluorimeter LS-50B, USA. The fluorescence emission spectra of κ-carrageenan, guanine, and κ-carrageenan -*graft*-guanine were measured at a concentration 5 $x10^{-5}$ M in 0.1 M NaOH as well as additionally at a concentration of $3.18x10^{-5}$M of guanine using excitation and emission slits 5.0/5.0 nm. Guanine and modified κ-carrageenan were excited at 274 nm with an emission at 340 nm.

2.5 Optical rotation and Circular dichroism

Optical rotations were measured on a Digipol 781 automatic polarimeter (Rudolph Instruments Inc., NJ, USA) (c 0.5%, 0.1 M NaOH) at 35^{o}C. Circular dichroism (CD) spectra were recorded on JASCO model J-815 CD Spectrometer, Tokyo, Japan, in the range 190-250 nm using sample concentration of ca. 0.8 mg/ml (800 ppm). Molar ellipticity values, [θ] are reported in units of deg cm^2 $dmol^{-1}$. All measurements were performed at room temperature using 1.0 cm quartz cells.

2.6 ^{13}C NMR spectroscopy

The NMR spectra of κ-carrageenan and κ-carrageenan-*graft*-guanine were recorded on a Bruker AVANCE II 500 MHz spectrometer, Switzerland, at 70^{o}C. κ-carrageenan was dissolved in D_2O (50 mg/ml), κ-carrageenan-*graft*-guanine was dissolved in D_2O and NaOH (50 mg/ml) and the spectra were recorded at 70^{o}C with 7000-7500 accumulations, pulse duration 11.25 μs, acquisition time 1.048 s and

relaxation delay 6 μs using DMSO as internal standard (ca. d 39.5). Guanine was dissolved in D_2O/NaOH (20mg/ml) and spectra was recorded at room temperature with 2000 accumulations, pulse duration 9.40 μs acquisition time 1.048 s and relaxation delay 6 μs using DMSO as internal standard.

2.7 Other characterizations of carrageenan-graft-guanine

Thermogravimetric analysis (TGA) was done on a Mettler Toledo Thermal Analyzer, model TGA/SDTA 851e, Switzerland. Total nitrogen was estimated by Kjeldahl method on a KEL PLUS- KES 201 Digestion unit attached to a KEL PLUS-CLASSIC DX Distillation unit (M/s PELICAN equipments, Chennai, India). Crude protein content was calculated multiplying the nitrogen content by the factor 6.25; the results were calculated as means ± SD of four replicates.

RESULTS AND DISCUSSION

3.1 Yield and grafting pattern

Yield of the product was 87% which was calculated on the basis of the nitrogen content of the product (Kjeldahl's estimation) with respect to the total quantities of κ-carrageenan and guanine that were used in the synthesis. Grafting percent (G%) in the product was 135%, whereas its total conversion (C%) value was 65%.[14]

3.2 FTIR spectroscopy

Strong bands at 1643 for carrageenan (bonded H-O-H) [24-25] and 1673 and 1697 cm^{-1} for guanine for amide carbonyl were observed in the FT-IR spectra (Fig. 2).[26-27] The spectrum of carrageenan-*graft*-guanine (Fig. 2) exhibited strong bands at 1673 and 1696 cm^{-1} as well as a shoulder at 1642 cm^{-1}, indicating the presence of polysaccharides and guanine moieties in the product. In the IR spectrum of guanine, the sharp band for C-2″-NH_2 at 3335 cm^{-1} (νNH_2;)[27] was not observed clearly in the spectrum of

carrageenan-*graft*-guanine (Fig. 2), which indicated the involvement of C-2″-NH_2 in the chemical transformation to form a new compound. In the products, several bands e. g. 1414, 1117, 687, 605 cm^{-1} were observed, whereas in guanine a relatively stronger band appeared at 1418 cm^{-1} (Fig. 1 and 2), indicating a discernible difference in the structure of the product in comparison with those of pure carrageenan and guanine. The bands near 1421 cm^{-1} in the product and at 1418 cm^{-1} in guanine are presumably due to the "ring pinching mode of guanine",[28] while the bands at 1117 cm^{-1} and 1120 cm^{-1} may be attributed respectively to C-NH- / C-NH_2 bending vibrations.[26] The remaining bands at 691, 605 cm^{-1} in the product and 693 and 602 cm^{-1} in guanine may have arisen due to -CH deformations.[26] A broad shoulder is observed around 3110 cm^{-1} in carrageenan-*graft*-guanine whereas guanine exhibited a sharp band at 3113 cm^{-1} for -NH- stretching.[27] Furthermore, characteristic band at 927 (3,6-anhydro moiety of carrageenan), 778 and 741 cm^{-1} for β-skeletal bending of basic carbohydrate moieties in the IR spectrum of the copolymer indicated that during grafting reaction the carrageenan polymers did not get decomposed.[25] The important IR bands of guanine and carrageenan (Fig. 1 & 2) are given below.

Guanine [26, 27]: (cm^{-1}) 3335, 3113 (νNH_2, νNH); 1697, 1673 (amide C=O); 1559 (ring double bonds); 1473, 1418 (ring vibration); 1120-949 (C-NH_2 bending); 879-780 (skeletal vibration); 693, 602 (-CH deformations).

κ-carrageenan [25]: (cm^{-1}) 3392 (-OH stretching); 1643 (bonded H-O-H); 1376 (-CH_2-OSO_3^{-2} linkage at C-6, which is not shown in the idealized repeating units of κ-carrageenan in Fig. 1; 1072 (C-O-C glycosidic linkage); 929 (3,6-anhydrogalactose linkage). 848 and 737 cm^{-1} (β-skeletal bending of basic carbohydrate moieties).

3.3 Thermal analysis (TGA)

The TGA patterns of the guanine grafted κ-carrageenan were comparable, in fact superimposable to that of guanine in repeated experiments. The initial TGA traces of κ-carrageenan and the grafted

product up to 250°C present a reversed pattern having a cross over point at ca. 250°C whereat κ-carrageenan started decomposing very fast till 500°C. The grafted product, however, started decomposing at ca. 250°C very slowly till ca. 460°C. Then the TGA traces exhibited even slower decline. The thermal stability of the grafted product with respect to κ-carrageenan indicated the formation of a new material with an aromatic compound, the latter being highly thermally stable.

The thermogravimetric (TGA) analysis curves of κ-carrageenan, guanine and κ-carrageenan-*graft*-guanine are shown in Fig. 3. The mass losses in κ-carrageenan, guanine and κ-carrageenan-*graft*-guanine were observed in three stages (i) 12%, 5% and 1% up to 175°C; (ii) 74%, 36% and 45% up to 400°C, and (iii) 65% and 67% up to 750°C respectively, while carrageenan showing decomposition in the range 250-500°C (Fig. 3). The first step indicated loss of bound water in the polysaccharide, which is much lower in the grafted product presumably due to the enhanced hydrophobicity as a result of grafting of κ-carrageenan reflecting the nature of guanine.

The TGA pattern of the guanine grafted carrageenan was comparable, in fact superimposable to that of guanine in repeated experiments (Fig. 3). One obvious conclusion to this was the confirmation of formation of new materials. The initial TGA traces of polysaccharide (carrageenan) and the grafted products up to 250°C present a reversed pattern having a cross over point at ca. 250°C whereat carrageenan started decomposing very fast till 500°C. The grafted products, however, started decomposing at ca. 250°C very slowly till ca. 460°C. Then the TGA traces exhibited even slower decline. The thermal stability of the grafted products with respect to carrageenan indicated the formation of a new material with an aromatic compound, the latter being highly thermally stable.

3.4 ^{13}C NMR

^{13}C NMR spectra of carrageenan, guanine, carrageenan-*graft*-guanine are given in Fig. 4a-c, having indicated the chemical shift values and the probable assignments. Five carbons of guanine

appeared at 120.3, 150.5, 160.6, 162.8 and 169.3 ppm, which were assigned to C-5, C-8, C-2, C-4 and C-6 (Fig. 1) respectively. The assignments were done by comparison with the corresponding data obtained from ChemDraw v10.0. The carbon chemical shifts of carrageenan was assigned by comparison with the data reported by Prasad et al. (2006a).[26] To our knowledge there exists no report of experimental ^{13}C NMR data of guanine or its derivative in the literature. The 14 peaks could be assigned to any of the three possible structures of the grafted products based on carrageenan C-6/C-2/C-2' and guanine C-2 NH_2 linkages (Fig.1). Appearance of a ^{13}C NMR peak at δ 49.9 indicated a new C-N bond formation (vide mechanism in *Section 3.11* below). The assignments were done by comparison with the values obtained for guanine and carrageenan in this study.

While assigning the ^{13}C NMR resonances, initially four possible structures were conceived e.g. carrageenan C-6, C-2, C-2' - substituted or C-6, 2'-disubstituted products on the basis of linkages with guanine C-2 NH_2. The di-substituted product was ruled out on the basis of the nitrogen content of the product which indicated insertion of one guanine moiety on to the carrageenan polymer.

Furthermore, there are possibilities that the bonding may happen between C-6 of carrageenan and one appropriate ring N of guanine e.g. N-9″ (Fig. 1). However, the ^{13}C δ-values of the guanine residue of the product were in excellent agreement with those of the experimental values of guanine. Therefore, there was least perturbation on the electronic environ of the guanine residue indicating thereby the involvement of the guanine C-2″–NH_2 group in the new N-C bond formation.

3.5 Optical rotation

The optical rotation values of parent guanine $[\alpha]_{589}^{30}$ (c 0.25, 0.1 M NaOH solution) and carrageenan $[\alpha]_{589}^{30}$ (c 0.25, H_2O) were +43.78° and +67.64°, respectively, while of κ-carrageenan-*graft*-guanine $[\alpha]_{589}^{30}$ (c 0.25, 0.1 M NaOH solution) was +128.967°. The modified $[\alpha]_D$ value of κ-

carrageenan after grafting indicates changes in the molecular symmetry profiles as a result of functionalization with guanine.

3.6 Nitrogen content

The total nitrogen contents in κ-carrageenan, guanine and κ-carrageenan-*graft*-guanine were 0.21±0.001%,, 46.0±0.50% and 7.72±0.1% (Kjeldahl's estimation data), respectively, indicating addition of guanine to κ-carrageenan.

3.7 UV-Vis analysis

The absorption maxima in the UV-Vis spectrum appeared at ca. 274 nm in guanine and κ-carrageenan-*graft*-guanine (in 0.1 M NaOH solution), while κ-carrageenan did not have any absorption bands in the UV-visible region. The product ($5x10^{-5}$ M) exhibited absorption maxima with ε274 15720 M^{-1} cm^{-1}, whereas that of guanine was 2460 $M^{-1}cm^{-1}$ at the same concentration. At a concentration $3.18x10^{-5}$ M guanine exhibited molar extinction coefficient value, ε_{274} 7190 $M^{-1}cm^{-1}$. This concentration was equivalent to the actual molar content of guanine in the $5x10^{-5}$ M solution of κ-carrageenan-*graft*-guanine. Hence, there was a 2.2 fold enhancement in the ε_{274} value in the latter, indicating that κ-carrageenan polymer became substantially more UV sensitive in presence of guanine moiety compared to the parent κ-carrageenan, which was UV transparent. This phenomenon points to the fact that there happened a transformation in the molecular make up of the polymeric architecture of the parent polysaccharide on substitution with guanine (cf. Section 3.6). The same trend of significant enhancement in the fluorescence intensity in the product compared to that of guanine was also observed in the fluorescence emission spectra (*vide infra section 3.9*).

3.8 Fluorescence measurements

The fluorescence emissions (λ_{max} 340 nm) of pure guanine, κ-carrageenan and the carrageenan-*graft*-guanine derivative were measured at 5 x10^{-5} M concentration (Fig. 5). κ-carrageenan at this concentration exhibited negligibly low emissions. The emission spectrum of the modified κ-carrageenan recorded in 0.1 M aqueous NaOH (5x10^{-5} M) solution exhibited emission maxima ($\lambda_{em,max}$) at 340 nm by excitation at 274 nm. The emission intensity was enhanced by ca.50% compared to that of pure guanine solution of the same concentration. When the concentration of the pure guanine solution is made equivalent to the concentration of the guanine molar component (3.18x10^{-5}) present in 5x10^{-5}M solution of modified κ-carrageenan, then ca. 73% enhancement in emission intensity was observed. Fluorescence yield of guanine is poor probably because of intermolecular interaction leading to quenching of emission intensity (Fig. 5).[29] In the product the guanine residue is placed well apart for inter-guanine molecular interaction to take place (Fig. 1). Therefore, possibly this has contributed to the enhancement of fluorescence intensity.

3.9 Circular dichroism (CD)

The CD curve of κ-carrageenan showed a negative trend having the peak value of [θ] -22.00 at 180 nm and trough [θ] -155.00 at 190 nm. Carrageenan-*graft*-guanine carrageenan-*graft*-guanine showed peak at [θ] 1.55 at 208 nm (Fig.6), suggesting significant chiroptical changes in the nucleo base grafted polymers.[30-33] Furthermore; it can also be explained on the basis of peak/trough ratios of the parent and modified polymeric materials. The peak-to-trough ratios of guanine 1.48 (>1) and κ-carrageenan was 0.86(<1) while that of grafted product κ-carrageenan-*graft*-guanine 1.82 (>1), suggesting significant chiroptical modification of the carrageenan polymer.[30-33] This is yet another demonstration of the fact that the presence of certain molecules or substance in the parent polymer induces conformational changes in

the polymeric chain under appropriate physical conditions, resulting in pronounced changes in the factor Mol. ellipticity with wavelength.[33]

3.10 Mechanism of formation of the grafted product

The plausible mechanism of formation of the grafted product is shown in Fig. 1. The reaction is proposed to take place via free radical mechanism where sulphate anion radical is formed first from KPS under microwave irradiation conditions. Then the radical ion generated the carrageenan free radical on C-6 carbon (the predominant possibility) with the elimination of OH radical, as well as the guanine radical on the C-2"-NH (the most likely possibility). The two radical species thus produced subsequently got coupled to form a new C-N bond resulting in the carrageenan-*graft*-guanine product as evident from the ^{13}C NMR data (new peak at 49.9 ppm). The simple nature of the ^{13}C NMR spectrum as well as the absence of complementary carbon resonances other than those of the product shown in Fig.1 indicated that the thermodynamically more stable product was predominantly formed in the radical coupling reaction. Chemical formation of the grafted product was thus confirmed by the FT IR, ^{13}C NMR spectral data which were supplemented by the XRD pattern, CD spectrum and thermal behavior as well as UV and fluorescence spectra.

CONCLUSION

A facile water based synthesis and characterization of κ-carrageenan-guanine derivative has been described. This derivative exhibited substantially enhanced fluorescence emission, e.g. 73% greater than guanine at $5x10^{-5}$ M concentration. The remarkable fluorescent activity of the carrageenan-guanine derivative predisposes it for its potential uses as sensors in various applications including biomedical ones.[34]

FIGURES

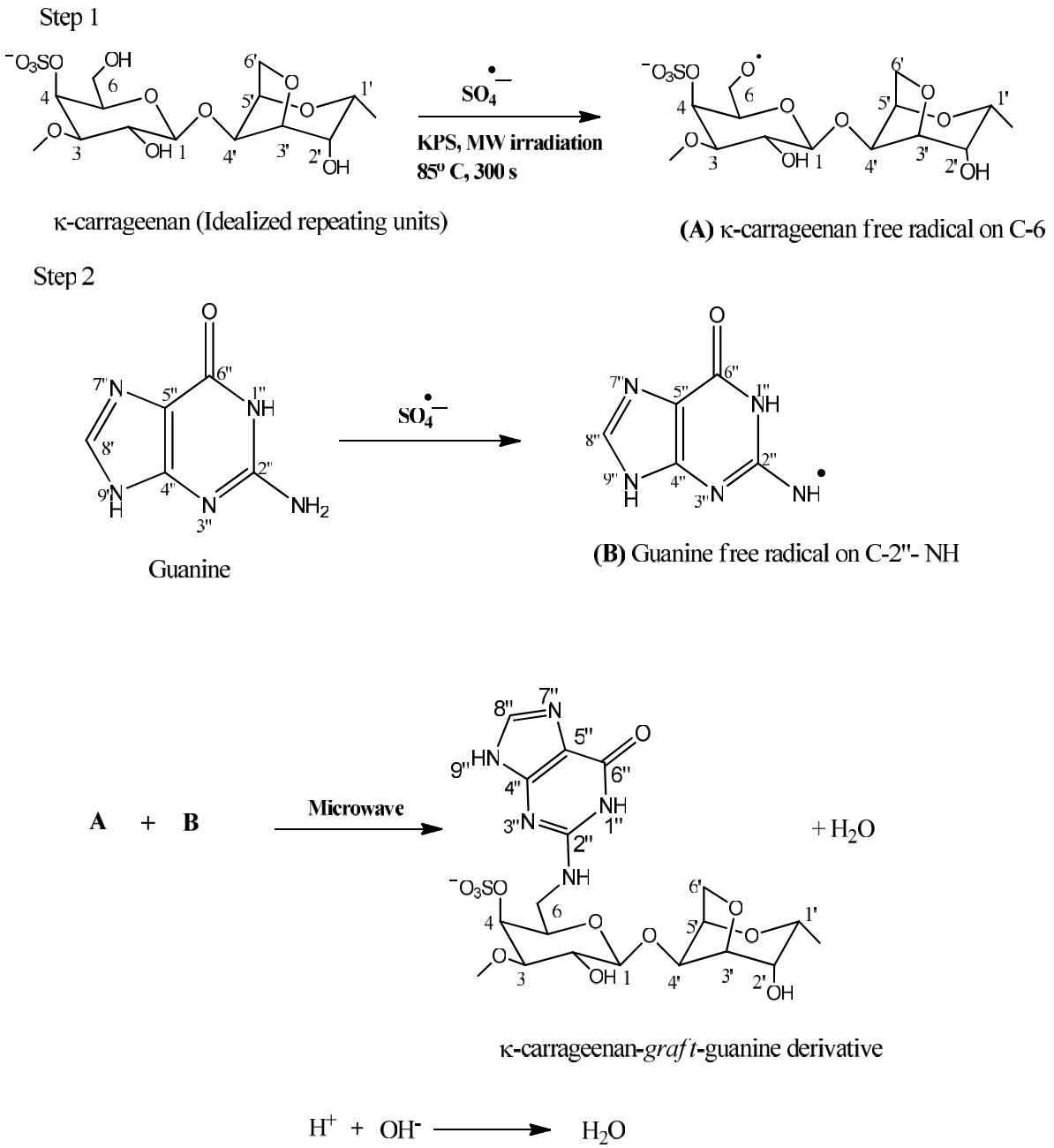


Fig. 1 Plausible mechanism of formation of carrageenan-*graft*-guanine

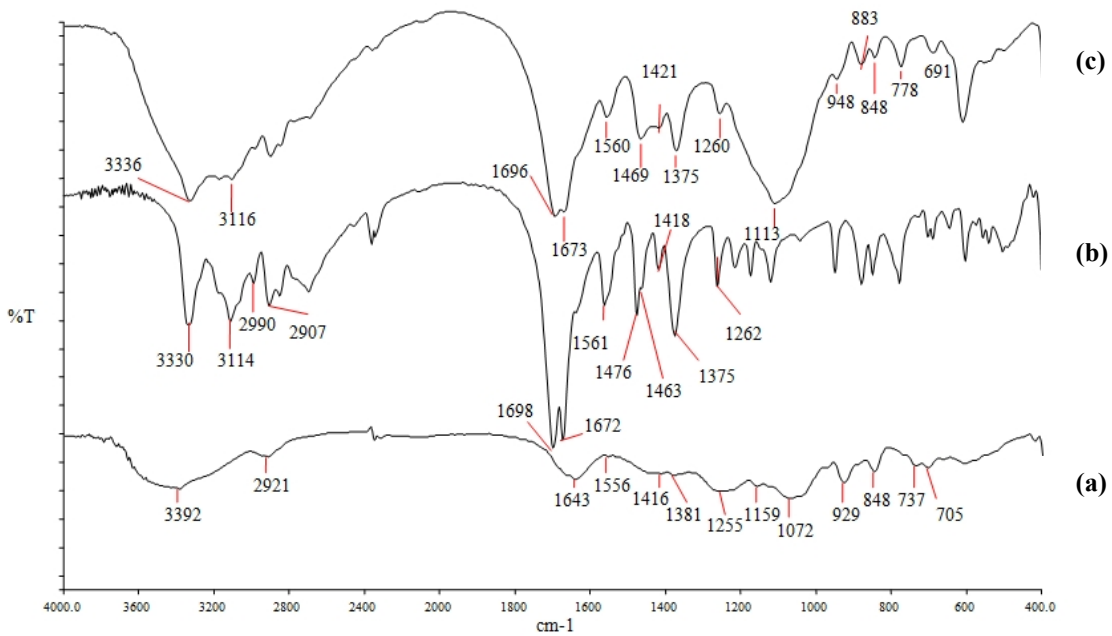


Fig. 2 FTIR of (a) carrageenan (b) guanine and (c) carrageenan-*graft*-guanine

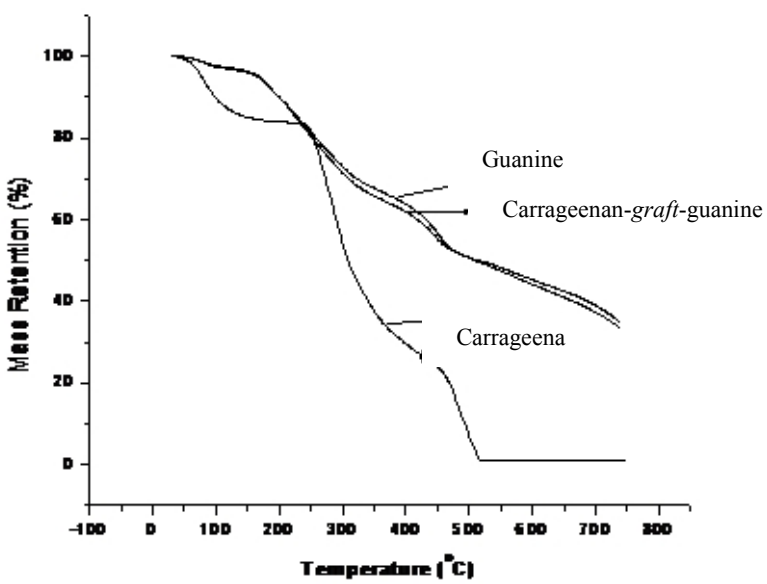


Fig. 3 TGA of (a) carrageenan (b) guanine (c) carrageenan-*graft*-guanine

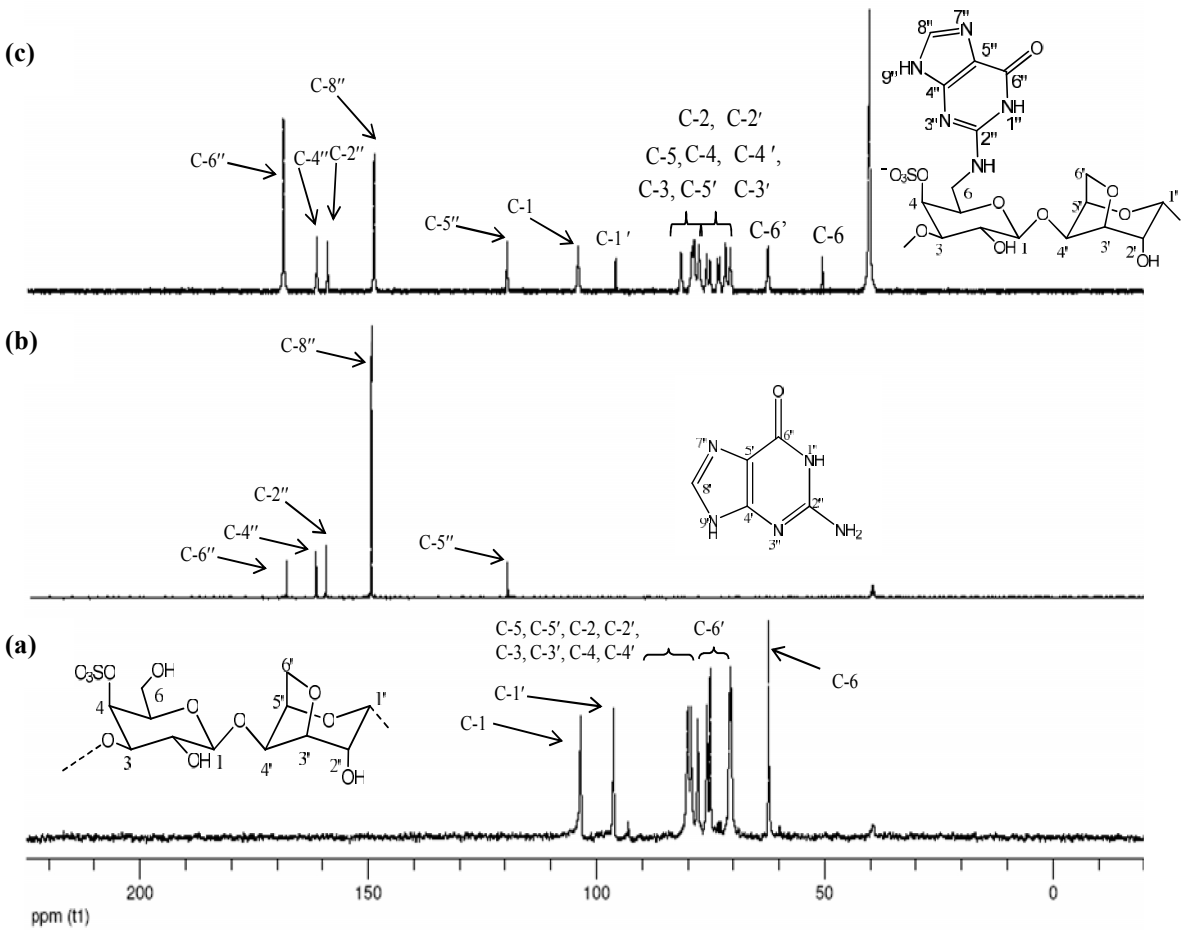


Fig. 4[13]C NMR of (a) carrageenan (b) guanine (c) carrageenan-*graft*-guanine

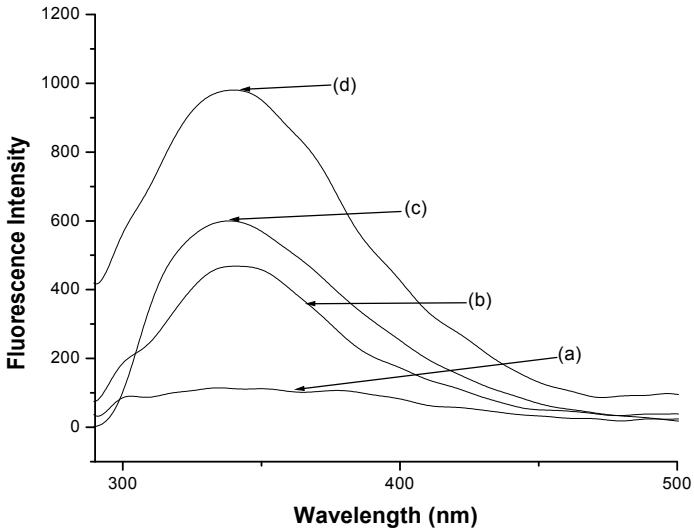


Fig. 5 Fluorescence emissions of (a) carrageenan, $5x10^{-5}$ M (b) guanine, $3.18x10^{-5}$ M, containing 0.032 mM guanine; (c) guanine, $5x10^{-5}$ M, containing 0.051 mM guanine; (d) carrageenan-*graft*-guanine, $5 x10^{-5}$ M, containing 0.037 mM guanine.

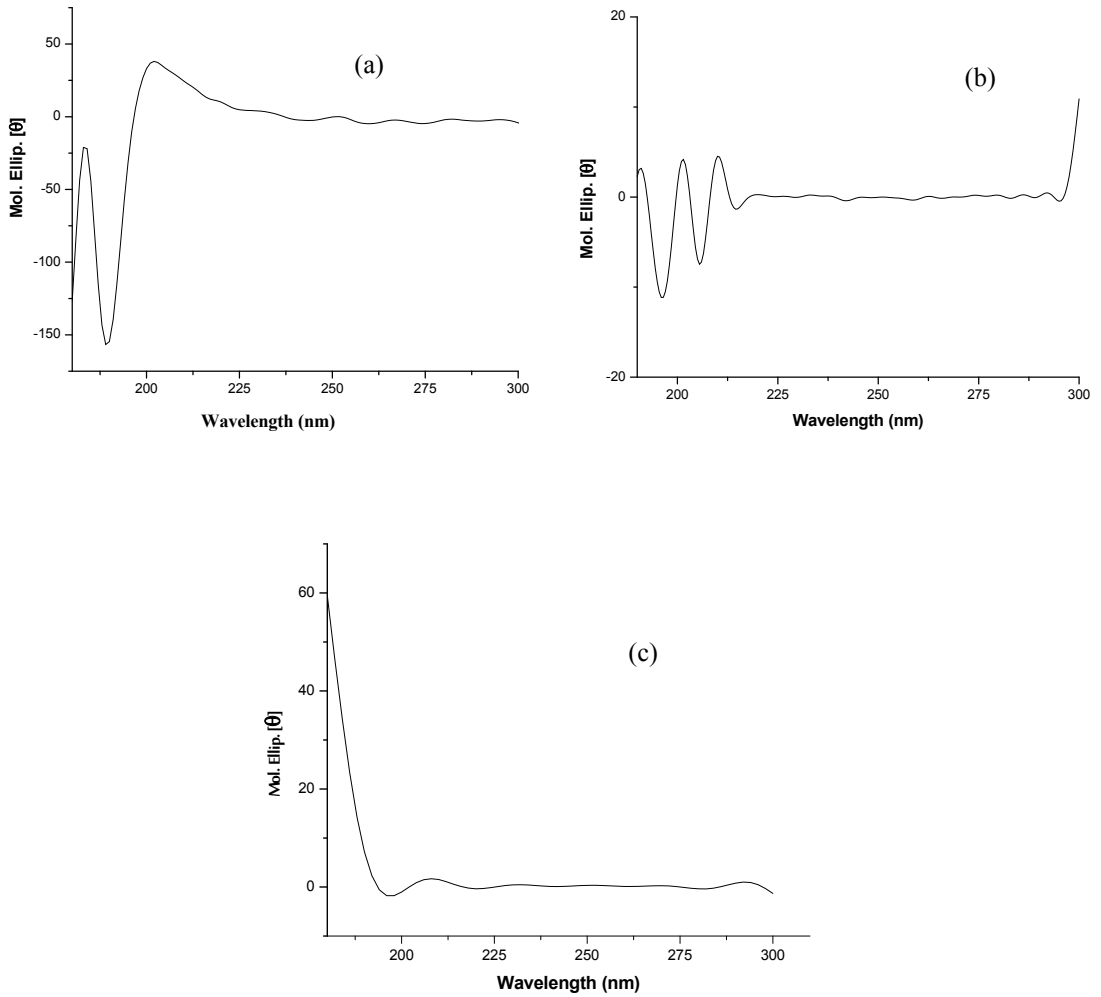


Fig. 7 Circular dichroism (CD) spectra of (a) carrageenan (b) guanine (c) carrageenan-*graft*-guanine

TABLES

Table 1 ^{13}C NMR data of carrageenan, guanine and carrageenan-*graft*-guanine

	^{13}C Chemical shifts (ppm)			
[a]Carrageenan		**Guanine[b]**	**Carrageenan-*graft*-guanine[c]**	
Units/Carbons			**Carrageenan C-6 substituted**	**Carrageenan C-2' substituted**
		169.3 (168.8) C-6"	169.5	169.5
		162.8 (162.2) C-4"	162.1	162.1
		160.6 (153.9) C-2"	159.7	159.7
		150.5 (144.8) C-8"	149.4	149.4
		120.3 (119.6) C-5"	119.9	119.9
G: C-1	103.5		104.1	104.1
C-3	80.1		81.3	81.3
C-5	75, 73.1		78.4	78.4
C-4	75.7		75.6	75.6
C-2	70.6		71.4/72.7	71.4/72.7
C-6	62.3		49.9	62.0
A: C-1'	96.2, 93		95.8	95.8
C-3'	79.9		78.8	78.8
C-4'	79.2		74.9/73.2	74.9/73.2
C-5'	77.7		77.3	77.3
C-2'	72.7		70.4	49.9
C-6'	70.9		62.0	70.4

Assignement were done by comparison with the δ values, cf. Figure 1; [b]obtained from ChemDraw v10.0 (given in brackets); [c]of carrageenan and guanine given in this Table (Figure 1).

ACKNOWLEDGEMENT

The Ministry of Earth Sciences, New Delhi (MoES/9-DS/6/2007-PC-IV) and CSIR, New Delhi, are gratefully acknowledged for the award of fellowship to MDO. AKS thanks CSIR, New Delhi, for the award of Emeritus Scientist Scheme. Sincere thanks are accorded to the Division of Analytical Sciences for the spectral and SEM analyses.

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Synthesis and characterization of Schiff's bases

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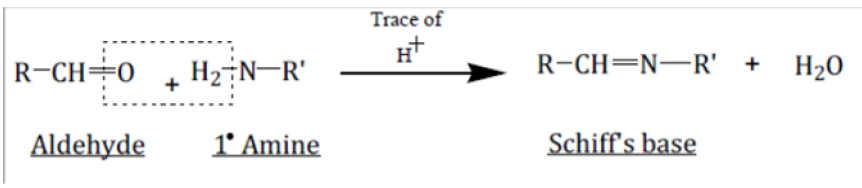
ABSTRACT

In this research paper we have synthesized some Schiff bases from salicyladehyde and 4-fluro benzaldehyde. Schiff's bases (azomethine) are the chemical derivatives having a carbon attach with nitrogen double bond in which nitrogen atom linked with aryl as well as alkyl group without hydrogen. All the propose derivatives were characterized by mass spectrometric, ^{1}H NMR and IR spectroscopy. They consist general formula $R_1R_2C = NR_3$.

Keywords: Schiff bases, azomethane, aryl group, alkyl group.

INTRODUCTION

Schiff bases were initially proposed by Hugo Schiff in 1864 (*1, 2*). It is obtain by the condensation of 1˚ amine with aryl carbonyl group (*3*).They are also known as 'anils' 'imines' or 'azomethines'. When these compounds were prepared by aldehydes, they are known as the 'aldimines' and when prepared by ketones they are known as 'kitimines'. General formula of Schiff bases is RCH=NR' where >C=N is the azomethinegroup(*4*).



Due to the presence of this azomethine (>C=N) linkage Schiff base shows color and can differ by introducing other auxochromic groups. It shows the phenomenon of tautomeris(*5*), and geometrical isomerism(*6*).Schiff bases possess both types of hydrogen bonding i.e. intermolecular as well as intramolecular(7).

MATERIALS AND METHODS

Schiff bases are obtained by condensation method. First take aromatic aldehyde (0.04 mole) dissolved it in absolute methanol (50 ml) then add aniline and it's derivatives (0.05 mole) and mixture was stirred at room temperature (*8*).After that reflux the reaction mixture for 6 hours at 85˚-95˚C.on cooling mixture the color precipitate formed, collect it by filtration, dried it and recrystallized from ethanol.

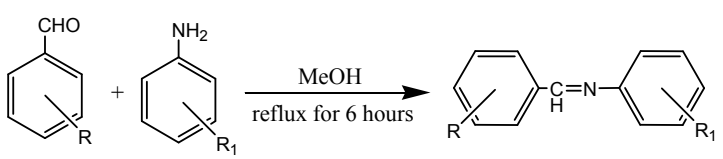
RESULTS AND DISCUSSION

We are carried out synthesis of Schiff's bases by taking one specific aldehydes and 6 different amine derivatives in methanol as a solvent and catalyst (H_2SO_4) resulted in new series of Schiff's bases with the general formula RHC =N-R_1.(*9*)

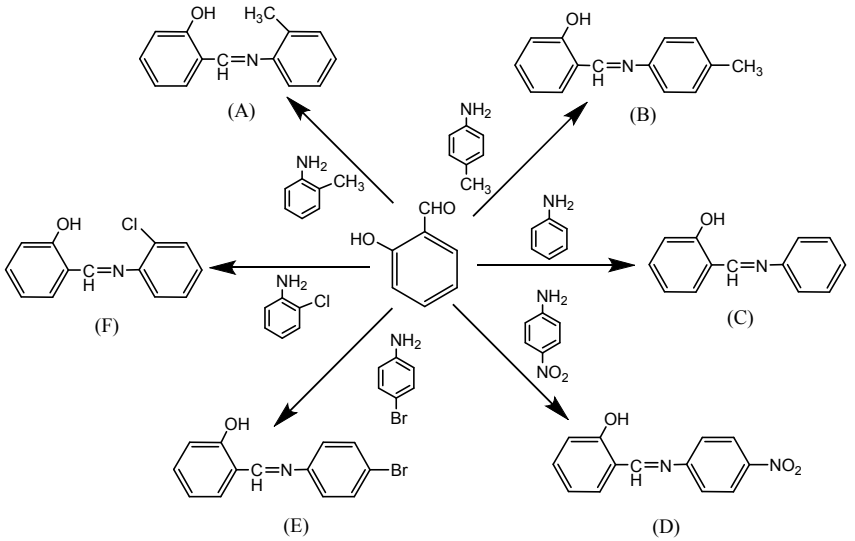
Here, for scheme 1; R is salicyladehyde and R_1 are o-toludine, p-toludine, aniline, p- nitro aniline, p- bromo aniline, and o-chloro aniline.

In scheme 2; R is 4- fluorobenzaldehyde and R_1 are o-toludine, p-toludine, aniline, m-nitro aniline, p-fluoro aniline, and o-chloro aniline.

- General scheme for preparation of Schiff base



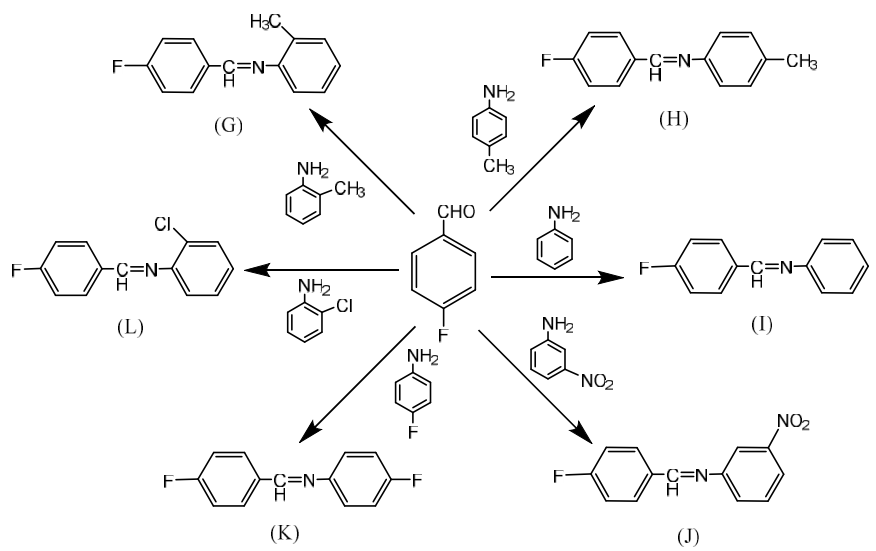
- For scheme 1:-
  Here we take salicyladehyde and six different substituted amine for synthesis of Schiff bases.



(A): 2-((2-aminophenylimino) methyl)phenol, (B):2-((4-aminophenylimino)methyl)phenol
(C):2-((phenylimino) methyl) phenol, (D): 2-((4-nitrophenylimino) methyl) phenol.
(E): 2-((4-bromophenylimino) methyl) phenol, (F): 2-((2-chlorophenylimino) methyl) phenol

- For scheme 2:-

Here, we take 4-floro benzaldehyde and six different substituted amines for synthesis of Schiff bases.



(G):N-(4-fluorobenzylidene)-2-methylbenzenamine, (H): *N*-(4-fluorobenzylidene)-4-methylbenzenamine, (I): N-(4-fluorobenzylidene)benzenamine (J): N-(4-fluorobenzylidene)-3-nitrobenzenamine, (K): N-(4-fluorobenzylidene)-4-fluorobenzenamine, (L): *N*-(4-fluorobenzylidene)-2-chlorobenzenamine

Physical properties of the prepared Schiff's bases

The structure of the recently synthesize Schiff's bases was identify by IR spectroscopy, 1H NMR. Spectra of prepared compounds are given bellow.

- Spectral data for scheme 1 compound E. [2-((4-bromophenylimino)methyl)phenol.]
  1. IR: 3018.70 (C-H str); 1569.18 (C=C str); 1569.18 azomethine (C=N str); 1417.73 (-OH ben), 600-500 (C-Br str).
  2. NMR: δ : 13.48 (s,1H), 8.64(s,1H), 7.40(m, 2H), 7.25(m, 4H), 7.09 (m,1H).
  3. Mass (m/z) = 275$[M+1]^+$
- Spectral data for scheme 2 compound K. N-(4-fluorobenzylidene)-4-fluorobenzenamine
  1. IR: 3051.49 (C-H str), 2991.67 (C-H ben), 1597.11(C=C str), 1597.11 (C=N str) 1354.07-1232.55 (C-F str)
  2. NMR: δ : 7.08 (m, 2H), 7.77 (m, 3H), 8.38 (s, 1H).
  3. Mass (m/z) = 216$[M+1]^-$

CONCLUSION

Six novel schiff bases of salicyladehyde and six schiff bases with 4-floro benzaldehyde with aniline derivatives like F, Cl, Br, CH_3, NO_2, etc have been successfully synthesized. However, it was noted that the product increased efficiently when the reaction was conducted under mild acidic conditions. The reason behind this fact was protonation of carbonyl site increases the attack of nucleophile of the 1° amine (10).

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A Novel Synthetic Route For Blattellaquinone: A strong German cockroach Pheromone

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ABSTRACT

The alternative path to synthesize Blattellaquinone with aim to prepare eco-friendly pesticide agent. German cockroach the synthetic course consists of three steps to obtain. Final product which found more suitable as compare to method. Noted ever before each organic product were recognize for their purity by TLC and verified characterization made by mass spectrometry and ^{1}H NMR.

SUMMARY

Synthesis of new quinone having nature of bio effectiveness on German cockroach.

Keywords: Pheromones 1, Blattellaquinone 2, German cockroach 3

INTRODUCTION

Pheromones are a naturally occurring chemical compound found in all insects, animals, and humans. The term pheromone was introduced by Peter Karlson and Martin Lusche in, based on the Greek word pherein means to transport and hormone means to stimulate. They are also sometimes classified as ectohormones. [*2, 3*] Pheromones are molecules that are evolved signals, in defined ratios in the case of multiple component pheromones, which are emitted by an individual and received by a second individual of the same species, in which they cause effect on hormone levels or behavioral changer specific reaction.[*4, 5*] For example, stereotyped behavior or developmental process. Pheromones are found in living things and are the most ancient form of animal communication. Pheromones are natural scents

which play an important role in sexual communication. These Pheromones aromas convey signals relating to mood, status, drive and health to the subconscious awareness of the opposite sex. This philosophy holds well in the animal world, pheromones are consciously detected over considerable distances and serve at times in place of real communication. They help animals to mark territory, recognize mates, and signal sexual interest. For example, female dogs in heat leave their pheromones and can attract male dogs over a mile away. Pheromones are produced by ectodermal glandson the abdomen and associated with mandibles of hymenopterans and wings of lepidopteran insects. Butenandt et al. first discovered sex pheromone in the silk worm (Bombyxmori). [*1*]

A sex pheromone that eluded natural product chemists for several decades has been characterized for the German cockroach, Blattellagermanica, one of the most important residential and food-associated pests worldwide. Movement of these cockroaches between human and animal waste and food materials allows them to acquire, carry, and mechanically transfer pathogens. [*6, 7*]

Types of insect's pheromones

Pheromones are subdivided into several types based on the nature of the interactions between emitters and receivers. Furthermore, releaser pheromones (e.g. alarm pheromone) bring about immediate changes in the behavior of receivers whereas primer pheromones (e.g. 9-keto-2-decenoic acid or queen honeybee substance) cause relatively slow and longer-term physiological changes (Ginzel, Law and Regnier). [*8, 9*]

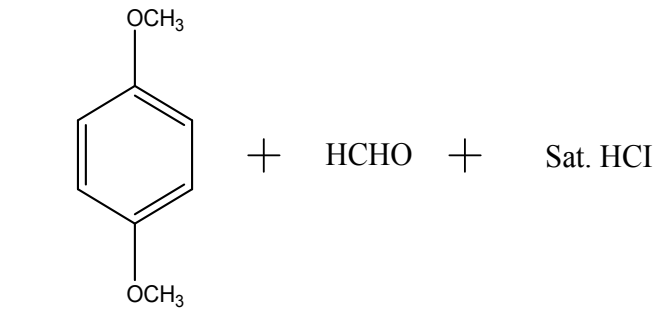
1. Sex Pheromones
2. Alarm Pheromones
3. Aggregation Pheromones
4. Anti-Aggregation Pheromones
5. Oviposition-Deterring or Epideictic Pheromones

MATERIALS AND METHODS

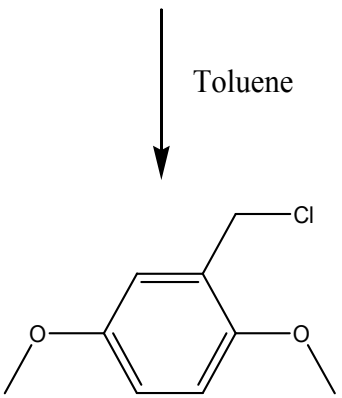
The synthesis of (3,6-dioxocyclohexa-1,4-dienyl)methyl 3-methylbutanoate requires three steps. The first step is the synthesis of 2-(chloromethyl)-1,4-dimethoxybenzene which further react with isovaleric acid. This reaction is a nucleophilic acyl substitution reaction and yields the ester 2,5-dimethoxybenzyl 2,3-dimethylbutanoate. 2,5-dimethoxybenzyl 2,3-dimethylbutanoate reacts with ceric ammonium nitrate salt to form (3,6-dioxocyclohexa-1,4-dienyl)methyl 3-methylbutanoate.

Scheme 1 : The Synthetic scheme for the preparation of compounds (3,6-dioxocyclohexa-1,4-dienyl)methyl 3-methylbutanoate.

Raw material
1,4-Dimethoxy benzene, Hydrochloric Acid, Formalin solution,
Product
2-(chloromethyl)-1,4-dimethoxybenzene.



1,4-dimethoxybenzene Formaline

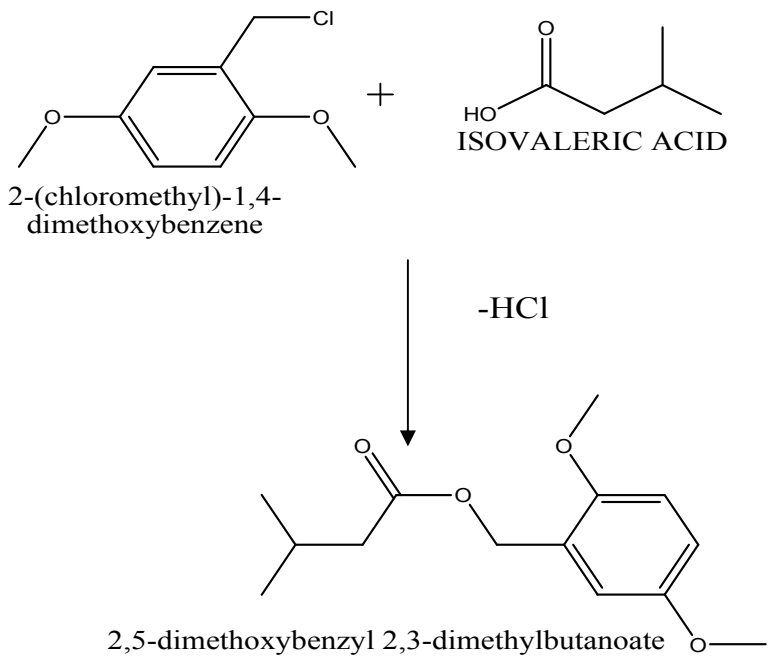


2-(chloromethyl)-1,4-dimethoxybenzene

A stirred solution of 55g of 1, 4-dimethoxybenzene in 100 ml of benzene and 200 ml. of concentrated HCl was saturated with hydrogen chloride. With a slow stream of HCl, 30 g. of formalin solution (37%) was added dropwise during 1 h with intermittent cooling in an ice bath. The HCl stream was then increased and the mixture was stirred for 1 h; the temperature rose to 55°C. The mixture was then extracted with benzene and the benzene layer was washed with water and NaHCO3, dried, and evaporated. Recrystallization of the solid residue from heptane gave 45g (61%) of colorless crystals, (m.p. 70-72°C). The purity of the compounds was checked by thin layer chromatography (TLC). Silica gel plates kiesel gel 0.25 mm, 60G F254, precoated sheets obtained from Merck, Darmstadt (Germany) were used for TLC. Developing solvent system of n-hexane: methanol (7:3) was used and the spots were visualized by ultraviolet light (254nm) as visualizing agent. ^{1H}-NMR (DMSO, δ ppm): 4.64 (2H, -CH_2), 6.66 (H, -CH), 6.66 (H, -CH), 6.66 (H, -CH), 3.73 (3H, -CH_3), 3.73 (3H, -CH_3); Anal. Calcd. $C_9H_{11}ClO_2$; C: 57.92%, H: 5.94%, Cl: 19.00%; O: 17.14%. Found: C: 57.82%, H: 6.04%, Cl: 19.07%; O: 17.07%.

Scheme 2 : The Synthetic scheme for the preparation of compounds (3,6-dioxocyclohexa-1,4-dienyl)methyl 3-methylbutanoate.

Raw material
2-(chloromethyl)-1,4-dimethoxybenzene, Isovelric Acid
Product
2,5-dimethoxybenzyl 2,3-dimethylbutanoate



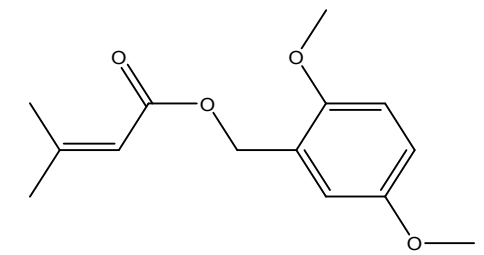
The second step is the reaction of 2-(chloromethyl)-1,4-dimethoxybenzene with isovaleric acid. This reaction is a nucleophilic acyl substitution reaction and yields the ester 2,5-dimethoxybenzyl 2,3-dimethylbutanoate.

The above reaction has to be carried out in the presence of a weak base (triethylamine) in order to trap the HCl which is formed during the reaction because the acid can promote the hydrolysis of the ester, undoing the esterification reaction. After the esterification reaction is complete, the work-up of the mixture includes a wash with aqueous ammonium chloride to remove the amine (m.p. 93 - 94°C). The purity of the compounds was checked by thin layer chromatography (TLC). Silica gel plates kiesel gel 0.25 mm, 60G F254, precoated sheets obtained from Merck, Darmstadt (Germany) were used for TLC. Developing solvent system of toluene:acetonitrile:methanol (5:3:2) was used and the spots were visualized by ultraviolet light (254nm) as visualizing agent. ^{1}H-NMR (DMSO, δ ppm): 6.59 (1H, -CH), 6.59 (1H, -CH), 6.59 (1H, -CH), 3.73 (3H, -CH_3), 3.73 (3H, -CH_3), 5.34 (2H, -CH_2), 2.48 (H, -CH), 1.24 (3H, CH_3), 2.46 (H, -CH), 1.01 (3H, -CH_3), 1.01 (3H, -CH_3); Anal. Calcd. $C_{15}H_{22}O_4$; C: 67.64%, H: 8.33%, O: 24.03%. Found: C: 67.52%, H: 8.41%, O: 24.07%

Scheme 3 : The Synthetic scheme for the preparation of compounds (3,6-dioxocyclohexa-1,4-dienyl)methyl 3-methylbutanoate (blattellaquinone).

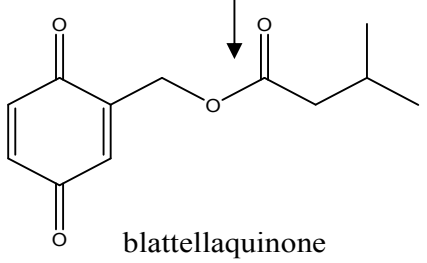
Raw material
2,5-dimethoxybenzyl 2,3-dimethylbutanoate, Ceric ammonium nitrate (CAN)

Product
(3,6-dioxocyclohexa-1,4-dienyl)methyl 3-methylbutanoate



2,5-dimethoxybenzyl 2,3-dimethylbutanoate

$(NH_4)_2Ce(NO_3)_6$(CAN)
acetonitril / water



blattellaquinone
(3,6-dioxocyclohexa-1,4-dienyl)methyl 3-methylbutanoate

The third step in the synthesis of (3,6-dioxocyclohexa-1,4-dienyl)methyl 3-methylbutanoate is the oxidation of 2,5-dimethoxybenzyl 2,3-dimethylbutanoate with ceric ammonium nitrate (CAN) to form the pheromone (3,6-dioxocyclohexa-1,4-dienyl)methyl 3-methylbutanoate. The product obtained was collected, dried and Re-crystallized from methanol. m.p found 141^{o}C. The purity of the compounds was checked by thin layer chromatography (TLC). Silica gel plates kiesel gel 0.25 mm, 60G F254, precoated sheets obtained from Merck, Darmstadt (Germany) were used for TLC. Developing solvent system of CCl_4:n-hexane:methanol (1:1:8) was used and the spots were visualized by ultraviolet light (254nm) as visualizing agent. ^{1}H-NMR (DMSO, δ ppm): 6.78 (1H, -CH,), 6.78 (1H, -CH,), 4.75 (2H, -CH_2,), 2.21 (2H, -CH_2), 2.47(2H, -CH_2), 1.01(3H, -CH_3), 1.01 (3H, -CH_3), 6.77 (1H, -H); Anal. Calcd. $C_{12}H_{14}O_4$: C: 64.85%, H: 6.35%, O: 28.80%. Found: C: 64.97%, H: 6.43%, O: 28.60%.

RESULTS AND DISCUSSION

Laboratory chemicals were supplied by Oxford India Ltd., and Angel Scientific Ltd. Melting points of the synthesized compounds were determined in open-glass capillaries on Stuart-SMP10 melting point apparatus and were uncorrected. The purity of the compounds was checked by thin layer chromatography (TLC). Silica gel plates kiesel gel 0.25 mm, 60G F254, precoated sheets obtained from Merck, Darmstadt (Germany) were used for TLC. Developing solvent system of n-hexane: methanol (7:3) was used and the spots were visualized by ultraviolet light (254nm) as visualizing agent.

CONCLUSION

In the present study it is crystal clear that the propose molecule was synthesized with completely new method and recongnized by different spectral analysis. Due to anavailibility of animal testing we are not able to present the application but literature it self is evidence of usefulness of 3,6-dioxocyclohexa-1,4-dienyl)methyl 3-methylbutanoate as strong German cockroach pheromones lead as ecofriendly pestisides.

ACKNOWLEDGEMENT

We are thankful to the management of RK University, Rajkot to provide advance laboratory condition and lab wear.

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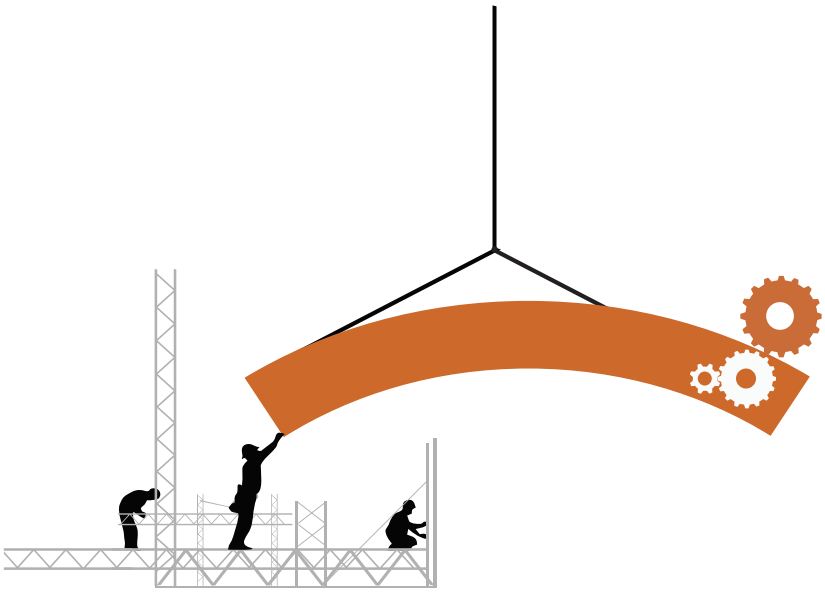
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SECTION NO. 6: TECHNOLOGY RESEARCH

In line with the main theme of the conference (research and entrepreneurship, with special focus on the role of India in shaping the 21st century), engineering and technology researchers who have published their work in the technology section have focused on research outcomes based on the use/incorporation of innovative materials, experiments, studies, analysis, and simulation experiments in the field of civil engineering, mechanical engineering, electrical engineering, agricultural engineering, electronics engineering, computer engineering, and information technology. The specific methods and mechanisms used to establish these research outcomes have been highlighted in the submitted manuscripts.





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ISBN: 978-93-5254-061-7 | www.rkuconference.org | JAN. 5 & JAN. 6, 2016

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Theoretical Analysis of Fluid Parameters influence on flow rates and Power in Magneto caloric Effect using Response Surface Methodology

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ABSTRACT

Magneto caloric effect is one of the methods of getting motive power without using any types of conventional energy and without any environmental effect. In the present study, an attempt has been made to investigate the effect of fluid parameters on flow rates and power in magneto caloric effect. The analysis was carried out on response surface methodology (RSM). The magneto caloric effect produced using this system mainly depends on the flow rate, velocity and power produced, which in turn depends on the process parameters such as diameter of tube, induced magnetic field and temperature differences. Analysis of Variance (ANOVA) test has been conducted to determine the statistical adequacies of the developed models. Analytical results show that power output is directly proportional to applied magnetic field. Also temperature places an important role in the flow rate. However, transportation media i e. Tube diameter has no effect.

SUMMARY

The theoretical analysis of fluid parameter indicates that flow rate and power increases as temperature of fluid increases.

Keywords: Magneto caloric effect, Magnetic field, Response Surface Methodology, Analysis of Variences.

INTRODUCTION

Property of some material which exerts an attractive or repulsive force on some other material is known as magnetism. The attractive/repulsive force is generated by two origins. The first is due to orbital motion of electrons within the atoms and another is due to spinning motion of electron on its axis. The magnetic theory postulates the electrons of atoms which are orbiting the nucleus create this force. The

electron is nothing but a small current loop which results in producing very little magnetic field which in fact creates magnetic moment along its axis of rotation. The basic concept is the magnetic field of the magnetism. This field exists in the volumetric space whenever there is change in volume. This is vector quantity and it is represented by H. In nature basically there are some other types of material like ferromagnetic, ferromagnetic, paramagnetic etc. which inherently magnetic.

In magnetic material two poles exist i.e. North Pole and South Pole. Both the poles creates magnetic field. The property of the magnetism is today provides many applications of our present technology. The most important is generation of electricity which at present provides the fundamental source of energy devices of the life time of technology which include electrical power generators and transformers, electric motors, radio, television, telephone, computer and other component of sound and videos (*1*). A new automatic cooling device using temperature sensitive magnetic fluid consists of an electromagnet, a heat source, a heat sink and a tube containing a temperature sensitive magnetic fluid. The fluid is suspension of Mn-Zn ferrite particles in water. The heat source is placed in the field region where the applied field strength has negative gradient. The experimental result indicates that the thermal energy given to the magnetic fluid is converted in to the mechanical energy of the fluid (*2*).

A magneto caloric pump for micro fluidic applications used fluids with $Mn_{0.5}Zn_{0.5}Fe_2O_4$ nanoparticles suspended in water and in oil, with a curie temperature of approximately 150°C. The experiment was carried out in such a way that the heat input, the magnetic field and the temperature gradient inside the pump are at constant. The only variable was the type of ferrofluid. The result showed that a maximum velocity of 2.1 mm/s obtained for a temperature gradient of 13° C for the water based ferro fluid and 1.59 mm/s for the oil based ferro fluid. Thus it shows that the MnZn ferrite ferrofluid is a good candidate for magneto caloric energy conversion (*3*). A new engine system consists of a permanent magnet, kerosene based magnetic fluid, and a rotor is the basic components of the thermo magnetic effect of a temperature sensitive magnetic fluid. A miniature motor has been developed based on the thermo magnetic effect of a temperature sensitive magnetic fluid in a loop shaped channel where heat is converted into kinetic energy of the fluid and power output is measured with a rotor. The rotor was driven by the thermal convection of the magnetic fluid in the presence of an external magnetic field. The result shows that the rotation speed of the rotor increases as the input heat load increases, or as the heat sink temperature decreases (*4*). The flow characteristics and heat transfer of a thermo sensitive magnetic fluid were investigated experimentally. In this experiment, there are three different conditions with respect to the magnetic pole like no magnets, a pair of magnets and a magnet on one side. When there is no magnet the magnetic fluid is in the steady flow condition, the velocity at the centre of the channel increases and the velocity distribution shows a Newtonian fluid. When magnets of the same strength are placed on both sides of the channel, the temperature difference was 15°C, and the velocity was 4.5 mm/sec. when a magnet is placed on only one side of the channel, the temperature difference was 14°C and the velocity was 4.7 mm/sec (*5*).

MATERIALS AND METHODS

The analytical data is obtained from the experiments conducted on magnetic fluid based prototype designed at our laboratory. And thereby to study the influence of flow rates and power in magneto caloric effect using response surface methodology.

1. Experimental setup

The paper deals with data obtained from the experimental set-up developed in Department of Physics, M K Bhavnagar University, Bhavnagar. Fig. 1 shows the prototype of equipment design. When heat is applied to one end of the heating section; it starts to heat the fluid. As the temperature of the magnetic particles is increased their magnetization reduces and reaches a very low level. The magnet is put before the heating section. When the magnetic fluid is placed in a magnetic field, it would be held there by magnetic force. On increasing the temperature of fluid (70°C) the hot and less magnetic fluid will be replaced by the cold and more magnetic fluid. Thus magneto caloric effect develops and displacement of fluid takes place by just the application of heat and induced magnetic field.

2. Process parameters

The three fundamental elements of magnetic fluid based engine are magnetic fluid, magnetic field and temperature.

2.1 Magnetic Fluid

Magnetic fluids are stable colloidal suspension of magnetic nanoparticles dispersed in a liquid carrier as shown in Fig.2. The size of magnetic nanoparticles has specific size range so that they remain suspended in the liquid. The specific size range is between 3 to 15 nanometre and their volumetric loading between 5 to 10 %. The reason of this particle size is to provide thermal agitation to disperse them uniformly in liquid carrier providing magnetic response. All liquids Brownian motion takes place this motion retards the settling of magnetic particles. Magnetic particles have a property of aggregation, and aggregate particles sediment quicker than unaggregated particles. Therefore the particles are coated with special stabilizing dispersing agent. To prevent agglomeration and subsequent settling, use of surfactant and other type of coating is used. The surfactant or coating substance should be such that it should appropriate to the carrier fluid such that it overcomes the mutual attractive force. So that nanoparticles do not agglomerate even when there is strong magnetic field.

2.2 Magnetic Field

The basic principle which is used in magnetic fluid based engine is that when temperature of fluid increases, the magnetic field of fluid starts losing its magnetism till it reaches to the Curie temperature. The temperature at which ferromagnetic material convert in paramagnetic material is known as Curie temperature. Therefore when a portion of magnetic fluid is surrounded by magnetic field and subjected to temperature gradient creates pressure gradient due to the cooler fluid. The ultimate result is fluid flow without any mechanical force. But problem is that when the operational temperature is far below the Curie temperature, in this case the change of magnetization would be almost very little. Thus it is very important to use the magnetic fluid which has a Curie temperature near to working temperature. The schematic operational design is shown in Fig. 3.

2.3 Temperature

In a ferromagnetic material when temperature increases due to inherent tendency of the magnetic dipoles to position or orient themselves in a specific way. This continues till the temperature increases beyond a certain point. The material loses its intrinsic domain formation and becomes paramagnetic at that particular temperature is known as Curie temperature. This temperature varies material to material including different types of magnetic fluid.

The Curie-Weiss law follows the magnetic susceptibility of the paramagnetic material beyond the Curie temperature,

$$X = C/T - T_C \quad \text{---------- (1)}$$

Here C = Constant and
T_C = Curie temperature

The value of T_C, i e. Curie temperature also provides the quantity of energy required to break up the domain range needed for the material. The domain magnetization is a function of temperature which is shown in Fig 4.

It is easy to assume that the magnetism and temperature follows linear function under Curie temperature (*6*). Thus the magnetization can be found from equation 1, as:

$$M = K(T_c - T) \quad \text{----------} \quad (2)$$

In the above equation K is the pyromagnetic coefficient and it can represent as:

$$K = -\frac{\partial M}{\partial T} \quad \text{----------} \quad (3)$$

The curve representing domain magnetism and temperature can be used to find Curie temperature simply by extrapolating the linear portion till it intercepts the temperature line. However the value so obtained is not an exact one. An alternate method may be used for this purpose. The intersection point of two tangents i.e. of ferromagnetic and paramagnetic behavior. Yet also Curie temperature can be found out by using domain magnetization vs. temperature curve in which maximum curvature point of the curve is taken. In this process measured values by two times differentiate (d^2M/dT^2). The maximum value

from the second derivative with respect to the maximum curvature point gives a good estimate of the Curie temperature (*7, 8*).

Properties of magnetic fluid, diameter of tube, induced magnetic field and range of temperature has been considered as independently controllable process parameters with significant contribution on magneto caloric effect. Table 1 shows the ranges of the input process parameters used for conducting the experiments. The volumetric flow rate, velocity and power output are considered as responses for the effect of magnetic fluid based engine are shown in design matrix by Table 2.

RESULTS AND DISCUSSION

Last three columns of Table 2 shows the values of three parameter of the experiment i.e. volumetric flow rate, velocity and power output. The three variable parameter provides 3^3=27 observations. The ranges of each parameter are shown in Table 3.

The Design Expert software is used for analysis. The Analysis if Variance (ANOVA) results in the form of partial sum of square is shown. The summary table by ANOVA is usually used to give summary. The test gives the regression model, significance factors and their interaction and lack of fit test. The test is significant if the value of 'Prob>F' is less than 0.005. Lastly % contribution column is provided in the Table 4 (*9*).

Table 4 shows that model; magnetic field (B) and temperature (C) are three significant factors. Table 4 also shows the various R^2 statistics of the volumetric flow rate. The R^2 = 0.911066 value (F-value) for flow rate indicates that the 91.10% variation is explained by the model. The statistic adjustment of R^2 is for the model size. Again the value $R^2_{Adj.}$ = 0.876978 indicates that 87.69% of the total variability is explained by the model after considering the significant factors and R^2_{Pred} = 0.647909 shows that the model would be expected to explain 64.79% of the variability in new data. The improved precision and reliability of the analysis is indicated by the lower value of the coefficient of variation (C.V. = 6.17%). The similar procedure was repeated for power and the ANOVA results are shown in Table 5.

After removing the insignificant factors, the response surface equations for flow rate and power are obtained in the actual values are as follows:

R1-Flow rate = − 0.276132418 – 0.0006415*A + 0.007464102*C

R2- Power = 21.13230516 – 0.072703367*A – 48.55925811*B – 0.246910347*C + 0.96*B*C

Where A = Tube Diameter, B = Magnetic Field, and C = Temperature

The normal probability plot of the residuals for flow rate and power is shown in Fig. 5-6, respectively. It indicate that the residuals lie reasonably close to the straight line which support that terms mentioned in the model are significant (*9*).

Fig. 7-8 shows the flow rate at 60°C and 70°C for various diameter of tube Vs. magnetic field. The tube diameters are 4, 6 and 8 mm and magnetic fields are 0.5, 1.0 and 1.5 T respectively. It indicates that flow rate remains constant at given temperature irrespective of tube diameter and applied magnetic field. Thus it can prove that flow rate is independent of tube diameter and magnetic field for any given temperature of fluid. However as temperature increases flow rate increases. Fig. 9-10 shows the effect of tube diameter and temperature on flow rate at different magnetic fields. In each case it indicates that flow rate is almost constant for any magnetic field but as temperature increases flow rate increases. Thus it is clear that the flow rate is independent of tube diameter and magnetic field but it is directly proportional to temperature of fluid.

Fig. 11-12 shows the output power in different diameter of tube. Power is same and it is independent of tube diameter for the same magnetic field. Also power output increases as applied magnetic field increases and it is directly proportional to magnetic field. Fig. 13-14 indicates the effect of tube diameter and temperature on power output at different magnetic fields. In each case power output is linear and proportional to fluid temperature.

CONCLUSION

This paper presents the theoretical analysis of magnetic fluid parameters like tube diameter, magnetic field and temperature influences on flow rates and power in magneto caloric effect using response surface method and following conclusions are drawn.

The velocity and power output were obtained in the range of 3.2 mm/sec to 21.3 mm/sec. and 7.1 milliwatt to 35.8 milliwatt respectively. The increase in flow rate is due to increase in temperature of fluid. Also the power output increases as applied magnetic field increases. The value of $R^2 = 0.911066$ for flow rate indicates that the 91.10% of the total variations are explained by the model. The value of the $R^2_{Adj.} = 0.876978$ for flow rates indicates that 87.69% of the total variability is explained by the model after considering the significant factors. $R^2_{Pred} = 0.647909$ shows that the model would be expected to explain 64.79% of the variability in new data. The value of $R^2 = 0.912674$ for power indicates that the 91.26% of the total variations are explained by the model. The value of the $R^2_{Adj.} = 0.881853$ for power indicates that 88.18% of the total variability is explained by the model after considering the significant factors. $R^2_{Pred} = 0.636457$ shows that the model would be expected to explain 63.64% of the variability in new data. Flow rate is independent of tube diameter and magnetic field for any given temperature of fluid. However as temperature increases flow rate increases. Power output increases as applied magnetic field increases. However tube diameter has no effect.

FIGURES



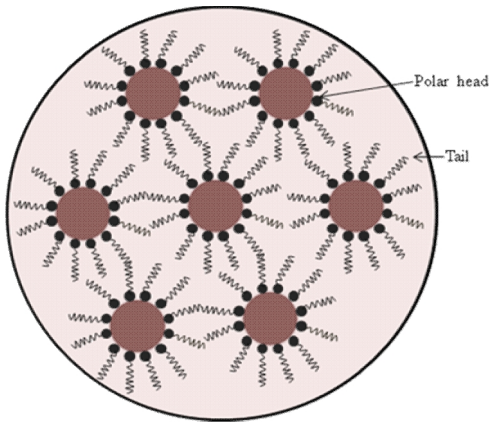


Fig.1. Prototy

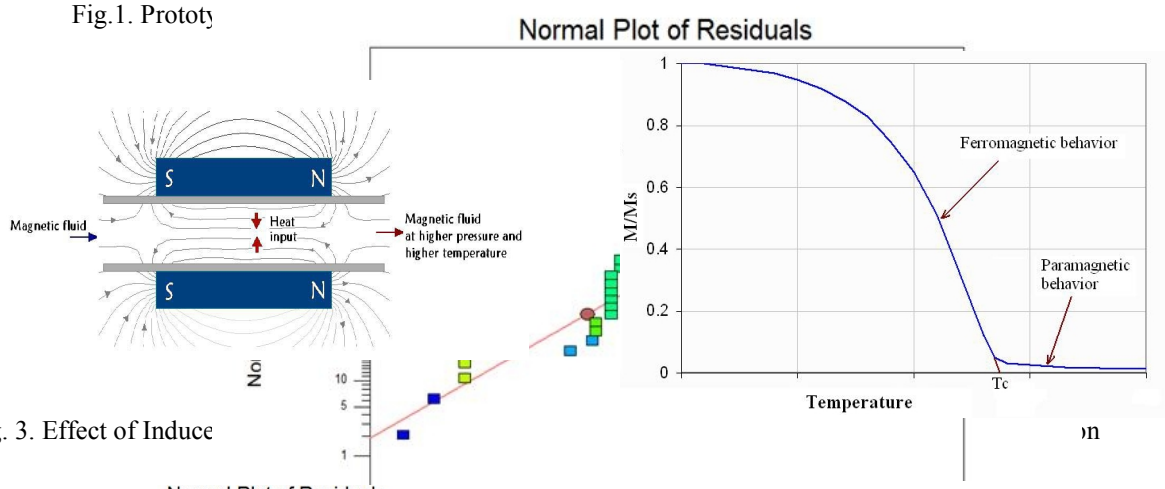


Fig. 3. Effect of Induce

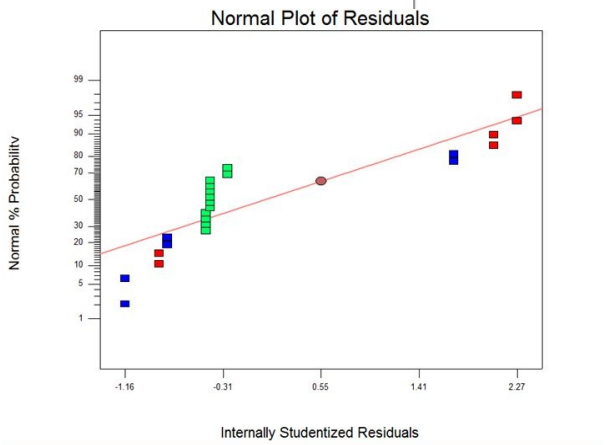


Fig. 5. Normal probability plot of residuals for flow rate

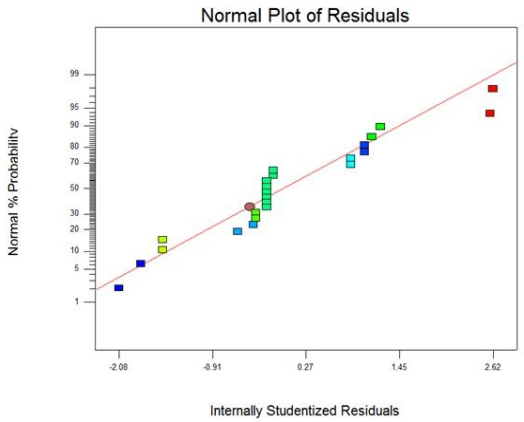
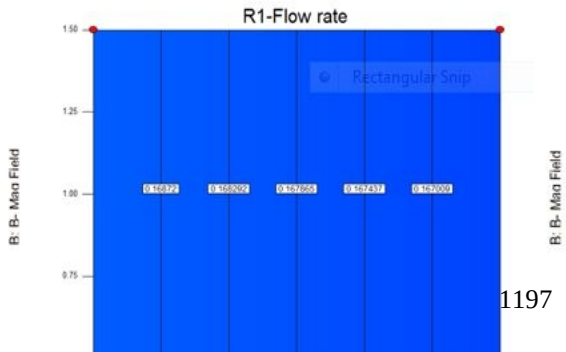


Fig. 6 Normal probability plot of residuals for power



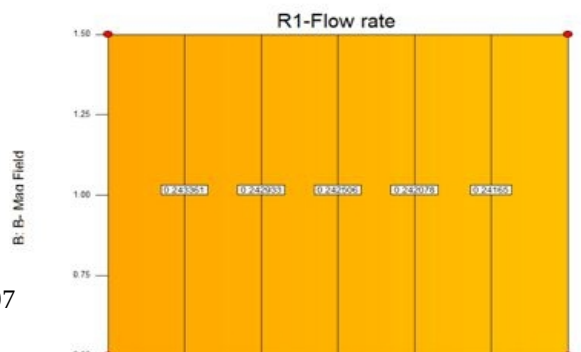


Fig. 7-8. Effect of temperature on flow rates at 60°C and 70°C.

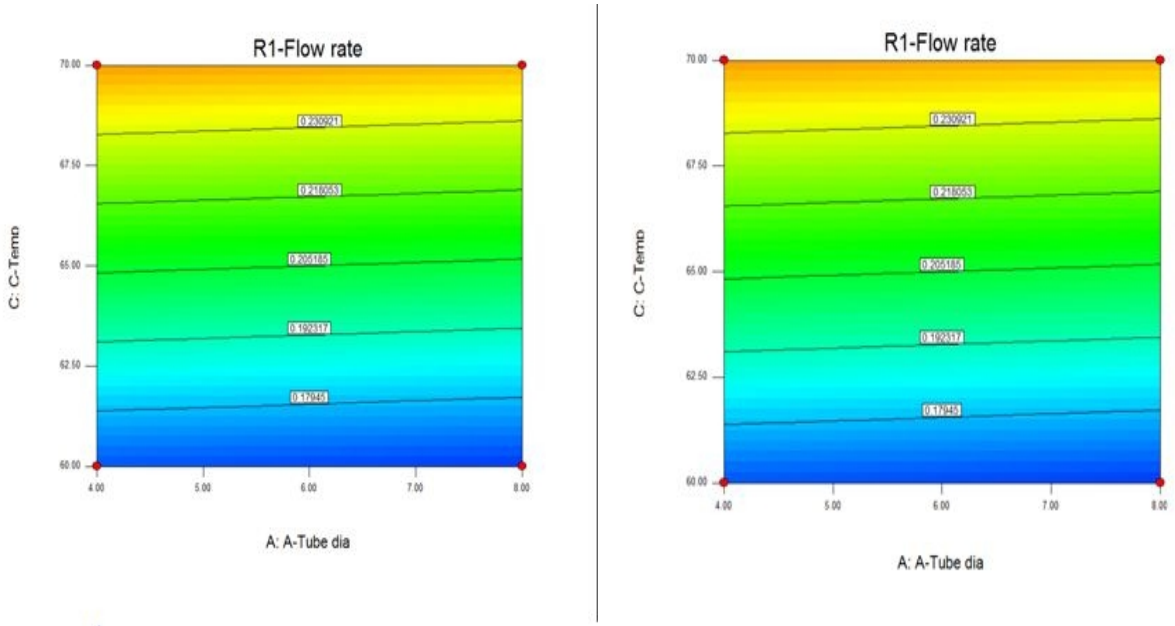


Fig. 9-10. Effect of magnetic fields on flow rates at 0.5T and 1.5T.

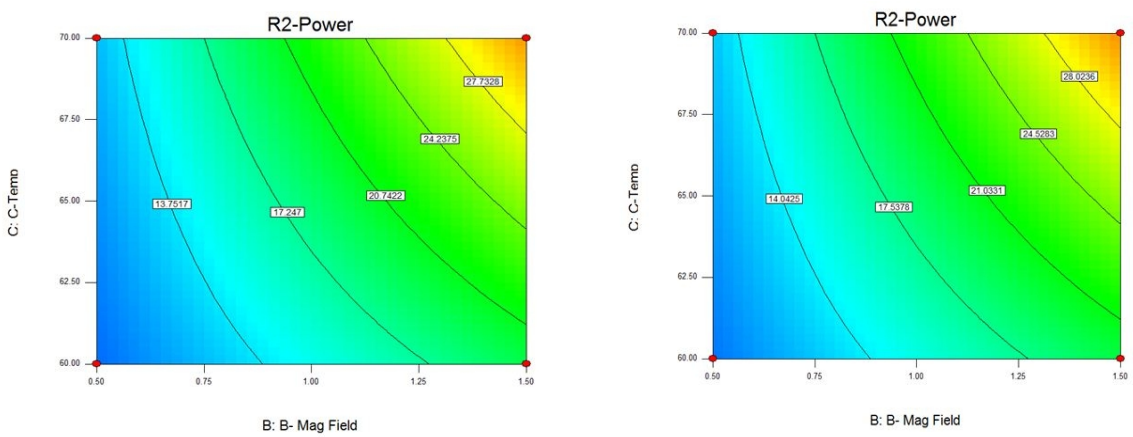


Fig. 11-12. Effect of Tube diameter on power at 4mm and 8mm**.**

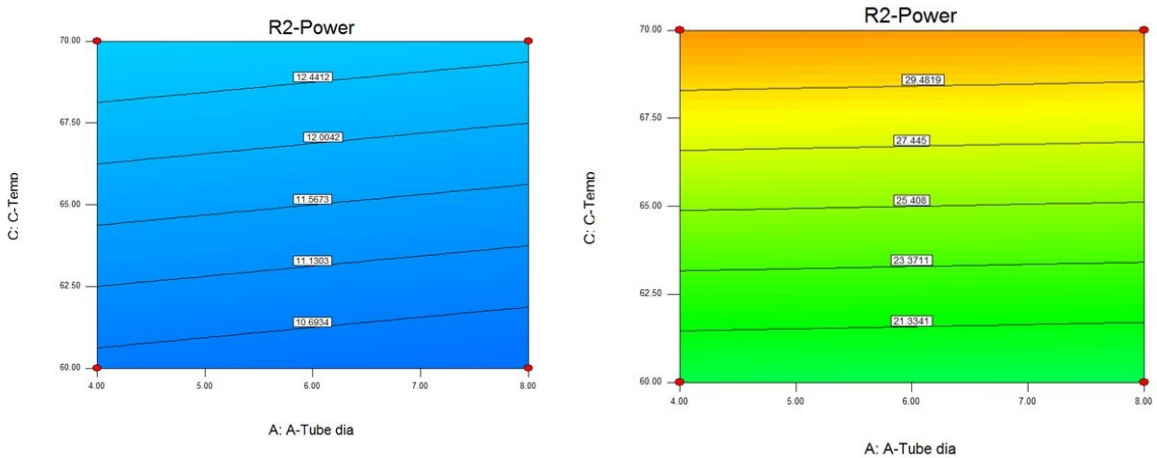


Fig. 13-14. Effect of magnetic field on power at 0.5T and 1.5T**.**

TABLES

Table 1. The process parameter levels

Parameter	Levels		
	Low (-1)	Medium (0)	High (+1)
Diameter of Tube (mm)	4	6	8
Magnetic Field (Tesla)	0.5	1.0	1.5
Temperature (°C)	60	65	70

Table 2. Deign matrix with response

Runs	Tube Dia.(m)	Mag. Field B (Tesla)	Temp. (°C)	Flow Rate Q (ml/sec)	Velocity V (mm/sec)	Power P (milliwatt)
1	8	0.5	70	0.26	5.34	11.9
2	8	1.5	65	0.2	4	26.8
3	4	1.5	70	0.26	21.3	35.8
4	4	1.5	65	0.2	16.0	26.8
5	8	0.5	65	0.2	4	8.9
6	4	1	70	0.26	21.3	23.8
7	6	1	60	0.16	5.69	14.3
8	6	1	65	0.2	7.12	17.9
9	6	0.5	60	0.16	5.69	7.1
10	4	1	65	0.2	16.0	17.9
11	6	1	70	0.26	9.49	23.8
12	8	1	70	0.26	5.34	23.8
13	8	0.5	60	0.16	3.2	7.1
14	4	0.5	65	0.2	16.0	8.9

15	6	0.5	65	0.2	7.12	8.9
16	8	1	60	0.16	3.2	14.3
17	6	0.5	70	0.26	9.49	11.9
18	6	1.5	60	0.16	5.69	21.4
19	6	1.5	70	0.26	9.49	35.8
20	4	1.5	60	0.16	12.8	21.4
21	4	1	60	0.16	12.8	14.3
22	4	0.5	70	0.26	21.3	11.9
23	8	1	65	0.2	4	17.9
24	6	1.5	65	0.2	7.12	26.8
25	8	1.5	70	0.26	5.34	35.8
26	8	1.5	60	0.16	3.2	21.4
27	4	0.5	60	0.16	12.8	7.1

Table 3. Range of parameter

Parameters	Range	
	Minimum	Maximum
Flow rate (ml/sec)	0.16	0.26
Velocity (mm/sec)	3.2	21.3
Power (milliwatt)	7.1	35.8

Table 4. Analysis of Variance (partial sum of square Type III) for flow rate

Source	Sum of Square	df	Mean Square	F-value	Prob>F	% Contribution	Remark
Model	0.027878629	6	0.004646	29.0256	<0.0001	0.199636	Significant
A-A Tube dia	2.22222E-05	1	2.22E-05	0.138819	0.7141	-0.00855	
B-B Mag Field	0	1	0	0	1.0000	-0.00597	Significant
C-C Temp	0.027856406	1	0.027856	174.0148	<0.0001	0.031352	Significant
AB	0	1	0	0	1.0000	-0.00944	
AC	0	1	0	0	1.0000	-0.00944	
BC	0	1	0	0	1.0000	-0.00944	
Residual	0.002721371	17	0.00016				
Lack of Fit	0.002721371	7	0.000389				
Pure Error	0	10	0				
Cor Total	0.0306	23					
Std. Dev.	0.012652299		R-Squared	0.911066			
Mean	0.205		Adj R-Squared	0.876978			
C.V.%	6.17853088		Pred R-Squared	0.647909			

Table 5. Analysis of Variance (partial sum of square Type III) power

Source	Sum of Square	df	Mean Square	F-value	Prob>F	% Contribution	Remark
Model	1258.444539	6	209.7407566	29.61239581	<0.0001	17.32046676	Significant
A-A Tube dia	0.285432099	1	0.285432099	0.040298931	0.8433	1.673613973	

B-B Mag Field	957.8306805	1	957.8306805	135.2319963	<0.0001	5.66482058	
C-C Temp	254.2484269	1	254.2484269	35.89624246	<0.0001	2.309897901	
AB	0	1	0	0	1.0000	1.985199437	
AC	0	1	0	0	1.0000	1.985199437	
BC	46.08	1	46.08	6.505837116	0.0207	0.414800563	
Residual	120.4087938	17	7.082870226				
Lack of Fit	120.4087938	7	17.20125626				
Pure Error	0	10	0				
Cor Total	1378.853333	23					
Std. Dev.	2.661366233		R-Squared	0.912674691			
Mean	18.46666667		Adj R-Squared	0.881853993			
C.V.%	14.4117305		Pred R-Squared	0.636457247			

NOMENCLATURE

Symbol	Physical Quantity
X	Magnetic susceptibility
T	Temperature (°C)
T_C	Curie temperature (°C)
M	Magnetization vector (A m^{-1})
K	Pyromagnetic coefficient
B	Magnetic Field (Tesla)

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

ISBN: 978-93-5254-061-7 *(Proceedings available for download at rku.ac.in/icre)*

RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

Shunt Active Power Filter Using Three Level T-Type Inverter with Self Balancing Capacitors Voltage

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ABSTRACT

In this paper novel T type three level inverter is used for shunt active power filter of low voltage applications. T type three level inverter can reduce size of ripple filter for shunt active power filter. Efficiency of T type three level inverter is more than two level voltage source inverter. Size of T type three level inverter is less than neutral point clamped inverter. This paper presents design, simulation and analysis of T type three level inverter based shunt active power filter using PSIM software. In this work, Fast Fourier Transform algorithm is used for reference current prediction of shunt active power filter. Also PI controller has been used to stabilize DC link voltage of inverter. The novel self balancing capacitor voltage control has been used in this work for shunt active power filter application.

SUMMARY

Novelty of this work is combination of control algorithms for shunt active power filter using T type three level inverter for low voltage applications.

Keywords: Shunt Active Power Filter, T type Three Level Inverter, Fast Fourier Transform, PI Based Current Control Method, Self Balancing Capacitors Voltage.

NOMENCLATURE

Vdc*: Reference DC Link Voltage
Vdc : DC Link Voltage
Vdc1 & Vdc2: Upper & Lower DC Link Capacitors Voltages
L: Filter Inductance
PCC: Point of Common Coupling
Vsa, Vsb and Vsc: Supply Phase Voltages
I_tri1 and I_tri2 : Triangular Carrier Waves
I_ref : Reference Current Wave (Output of PI Controller)
Ia_load: Load Current
Ia_compensating : Compensating Current
Ia_source: Source Current
Vab_inverter : Three Level Output Voltage of Voltage Source Inverter

INTRODUCTION

Harmonics are generated in power systems are due to the non-linear load. Day by day use of non linear load increases in domestic and industrial use for various applications. So harmonics issues are serious today because sensitive equipments may be damaged or cannot work satisfactorily. Active power filters have been used to solve voltage and current harmonics problems.
The T type three level inverter are used for active power filters for better performance at low voltage level. The efficiency of three-level T-type inverter is good than two level voltage source converter. The panel size of the T-type converter is small than three-level other power topologies of converters.
Pinkal J. Patel *et al. (1)* described about the review of shunt active power filters using three level voltage source inverter. The capacitor voltage unbalance operation has to be compensated by a natural balancing due to PWM scheme like is shown in *(2)*.
Diode clamped power topology has disadvantage of capacitors voltage unbalancing. Sun Hui *et al. (3)* have discussed for The NPC three-level converter with self- voltage balancing through clamping switching and clamping diodes used for active power filter.
Space Vector Pulse Width Modulation (SVPWM) method can be used for three level diode clamped voltage source inverter and using redundant vector capacitors voltage to be balanced *(4)*.
Space vector hysteresis current control with two coordinate system has been used for two level and three level VSI with simulation and FPGA based hardware results *(5)*.

The comparisons of Carrier PWM strategies for cascaded and neutral point clamped multilevel inverters have been discussed *(6)*.
Fast Fourier Transform algorithm is modification of Discrete Fourier Transform (DFT) algorithm. FFT algorithm speed is fast than DFT algorithm. S.D. Round and D.M.E. Ingram *(7)* have described about various techniques (Instantaneous Reactive Power Theory, d-q reference frame, Sine Subtraction and FFT) used for reference current generation and their comparisons. Fast Fourier Transform algorithm performance is good for Steady-state condition, unbalanced load and unbalanced supply voltage. Lucian Asiminoaei et al. *(8)* also discussed the reference current generation methods evaluations for active harmonics filter.

Simulation, Analysis and Modelling of a Shunt APF using cascaded multilevel inverter is discussed in *(9)*. PSIM simulation package can be used for the FFT and pq theory based shunt active power filter for current harmonics compensation *(10)*. Peng Xiao *et al. (11)* have discussed about Seven-Level Shunt Active Power Filter for High-Power Drive Systems. Todays, multilevel inverters have shown some significant advantages over traditional two-level inverters *(12-14),* especially for high-power and high-

voltage applications. The Pulse Based Dead Time Compensation (PBDTC) method with self voltage balancing space vector pulse width modulator has been used for diode clamped three level inverter fed induction motor drive on 45 kW (60 HP) induction motor with experimental results *(15)*. PBDTC method should be used for shunt active harmonics filter.

Hirofumi Akagi and Takaaki Hatada *(16)* have discussed about the capacitors Voltage balancing control for diode clamped power topology characterized by superimposing a sixth harmonic zero-sequence voltage on the active harmonics filter voltage reference in each phase is introduced to the three-level converter with triangle carrier modulation. For Low Voltage Applications and High Efficiency Three-Level T-Type Converter has been discussed *(17)*. S. Lee and S. Sul *(18)*, has described about harmonic reference frame based current controller for active filter applications.

In this paper simulation of T type three level inverter based shunt active harmonics filter is discussed with self balancing capacitors voltages.

THREE LEVEL T TYPE VSI BASED SHUNT ACTIVE POWER FILTER

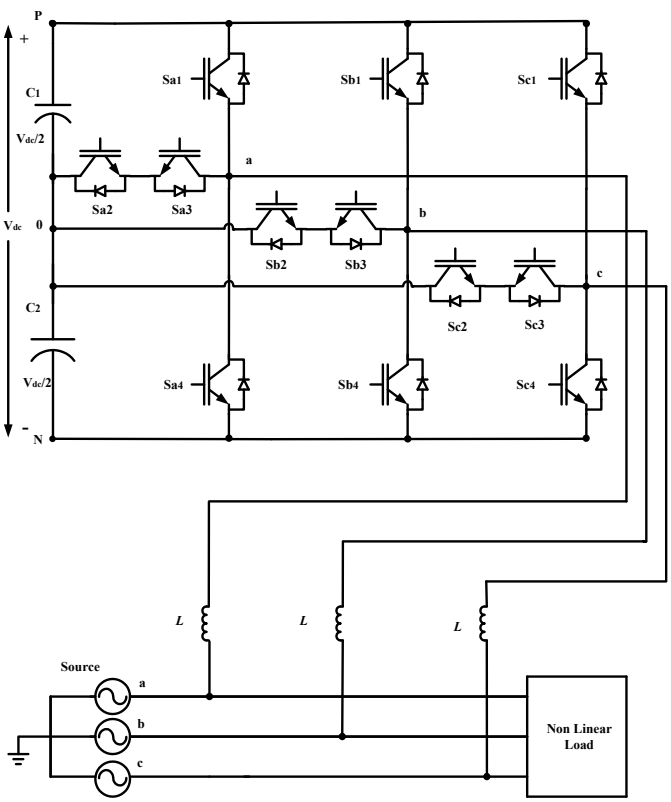


Fig.1. Three Level T Type VSI for the SAPF

Fig.1 shows the configuration of the standard T type three-level voltage source inverter for the Shunt Active Power Filter application. T type power topology is used for low voltage applications with high efficiency. No need of clamping diodes for T type power topology like Neutral Point Clamped three level

inverter, so overall size of active harmonics filter panel can be reduced. SPWM and SVPWM modulation strategies are same as NPC power topology. Size of ripple filter can be decreased using three level inverter based shunt active power filter. The model of T type power topology is same as NPC topology. The six IGBTs voltage rating is use double than DC link voltage and six middle IGBTs connected with split capacitors have been used half of DC link voltage.

Table 1 shows switching states of four switches of phase a. It is same as NPC three level voltage source inverter. Switch Sa1 and Sa3 are inverted switches and it is required to put dead time between these two switches to avoid DC link to be short circuited. Same as Sa2 and Sa4 are inverted switches in phase a. Similarly switching states are applicable for b and c phases as shown in Fig.1.

Table 1. Switching States

State	Output Voltage	Sa1	Sa2	Sa3	Sa4
P	+ Vdc/2	ON	ON	OFF	OFF
0	0	OFF	ON	ON	OFF
N	- Vdc/2	OFF	OFF	ON	ON

OVERALL SYSTEM CONTROL

Fig.2 shows overall control schemes block diagram for shunt active power filter. Fast Fourier Transform (FFT) method is used for harmonics current reference prediction from the non linear load. FFT method can work satisfactory for unbalanced load or unbalanced supply voltage.

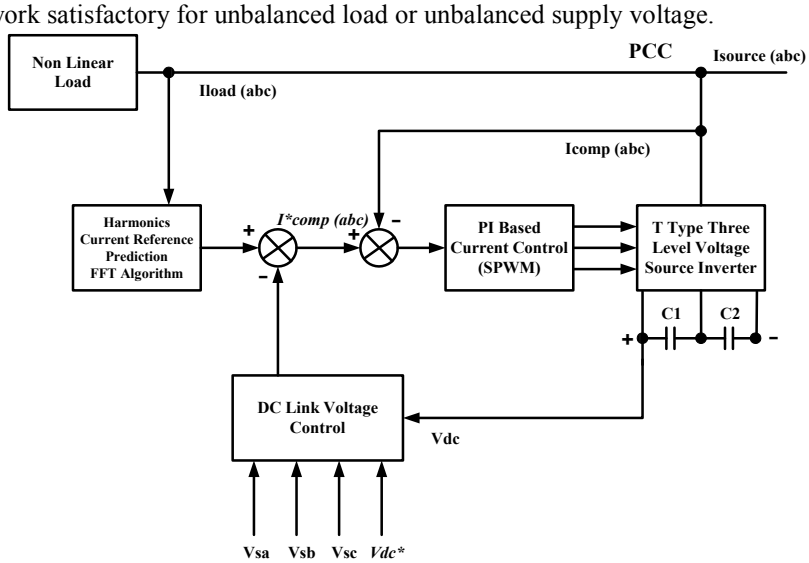


Fig.2. Control Block Diagram for the SAPF

Following equations of FFT algorithms have been used for C - programming of reference current predictions.

$$\overline{X}_h = \sum_{n=0}^{N-1} x(n) \bullet \cos\left(\frac{2\pi hn}{N}\right) - j\sum_{n=0}^{N-1} x(n) \bullet \sin\left(\frac{2\pi hn}{N}\right) \quad (1)$$

$$\overline{X}_h = X_{hr} + jX_{hi} \quad (2)$$

$$|\overline{X}_h| = \sqrt{X^2_{hr} + jX^2_{hi}} \quad (3)$$

$$\phi_h = \arctan\left[\frac{X_{hi}}{X_{hr}}\right] \quad (4)$$

$$I_{refj}(k) = -Ki \bullet I_{Lj}(k-2) \quad (5)$$

Where,
N : number of samples per fundamental period,
$x(n)$: input current signal at point n,
$\overline{X}_h$: complex Fourier vector of *h* harmonic of input signal
X_{hr} : real part of X_h
X_{hi} : imaginary part of X_h
$|\overline{X}_h|$: magnitude of the vector
k : Computation Index
$I_{refj}(k)$: Vector of Reference current harmonics
$I_{Lj}(k)$: Vector of Load current harmonics

PI based current control scheme is used for generation of PWM. This method is used level shifted carrier (Triangular) waves for Sine Pulse Width Modulation (SPWM). The main advantage of PI based current control is to limit switching frequency of IGBTs. Output of PI Controller is compared with triangular carrier wave for 12 PWM generations for 12 IGBTs. In each phase two middle switches ON and OFF time must be equal for balancing of capacitors voltages. To get equal ON and OFF time for middle two switches, PI controller must be properly tuned. This is called self balancing of capacitors voltage. For DC link voltage control, three phase voltage is superimposed with dc link voltage PI controller output and accordingly subtracted with three references of current. Trial and Error method has been used for tuning of PI controllers in this work.

RESULTS & DISCUSSIONS

Fig.3 shows PSIM Simulation steady state Model of Shunt Active Power Filter with dead time. In this work, Three phase uncontrolled rectifier is used as a non linear load to generate current harmonics in source current. Three level T type voltage source inverter is used for current harmonics compensation. In control systems, DLL (Dynamic Link Library) Block is used for FFT algorithm. FFT algorithm is used for reference harmonics current prediction. PI controllers have been used for current control. Outputs of PI controllers have been compared with triangular carrier wave as shown in Fig.4. Outputs of comparators have been given input to the dead time circuits. Dead time is set 5 µs between two inverted switches to avoid dc sources to be short circuited. Fig.5 shows Load current, compensating current and source current waveforms with THD. Load current THD is 31.33 %. Source current THD is 4.3 % after compensation. IEEE 519-1992 standard has been complying in this work. FFT plot for non-linear load current, compensated current & source current are shown in Fig.6. As shown in Fig.7, balanced upper and lower capacitors voltages and dc link voltage. Self balancing control algorithm has been work satisfactory for balancing capacitors voltages. DC link PI controller is also working satisfactory for dc link voltage control of inverter. Fig.8 shows line to line three level output voltage wave of T type inverter. In this work L filter is used for current ripple minimization. So the overall size of shunt active power filter panel size will be reduced for hardware implementation. The Switching frequency is 7 kHz used for this simulation. System parameters of simulation are mentioned in Table 2.

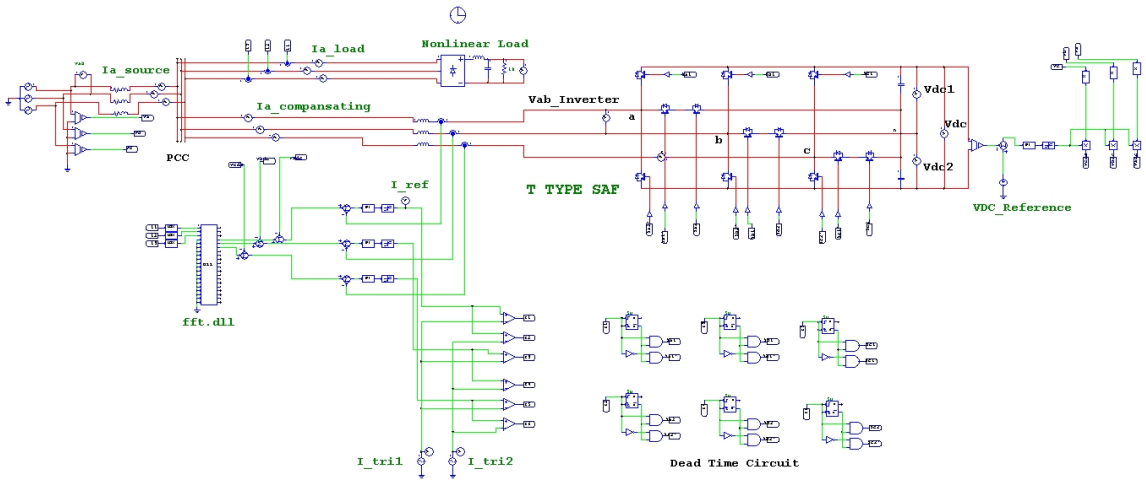


Fig.3. PSIM Simulation Model of T type Shunt Active Power Filter

Table 2. System Parameters

Line to Line Voltage rms voltage	415 V
Line Frequency	50 Hz
Filter Inductor	0.3 mH
DC capacitance of active filter	4700 µF
DC Voltage of Active Filter	715 V
Switching Frequency	7 kHz
RMS value of Load Current	45 A

This steady state simulation and analysis shows that novel shunt active power filter using three level T type voltage source inverter with self voltage balancing can work satisfactory for low voltage application with high efficiency (17).

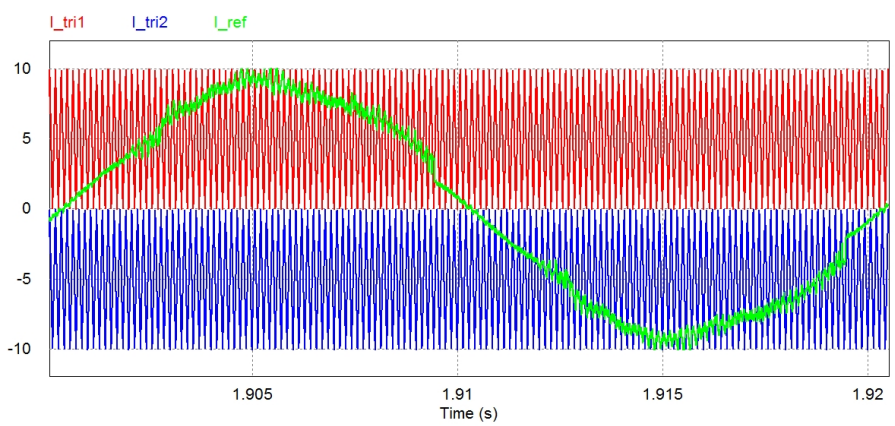


Fig.4. Output of PI controller compared with level shifted triangular carrier wave

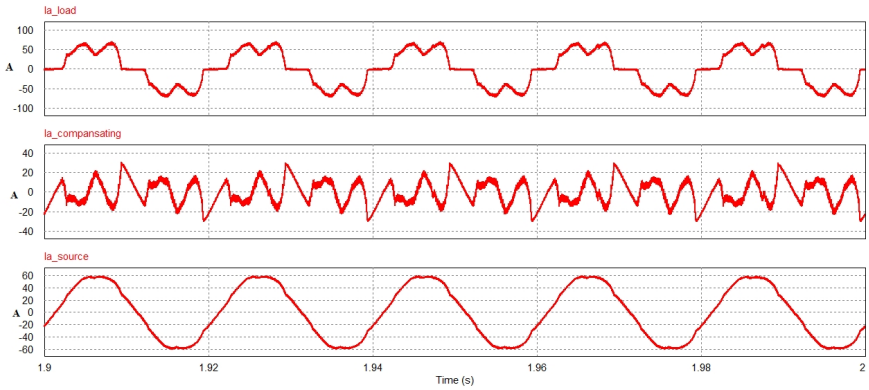


Fig.5. Load current, compensating current and source current waveforms

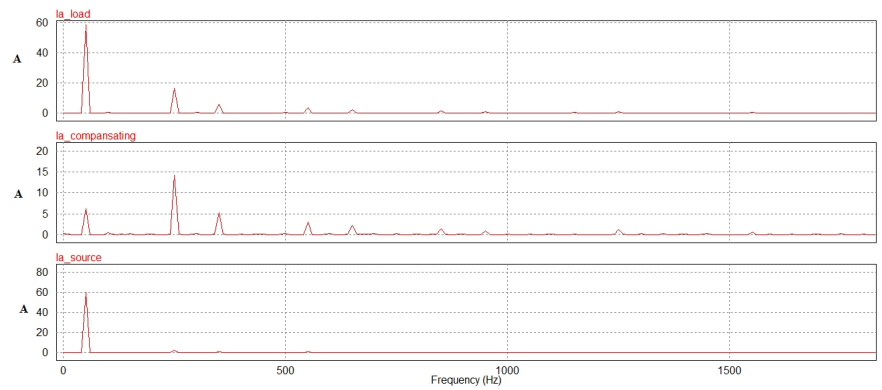


Fig.6. FFT plot for non-linear load current, compensating current & source current

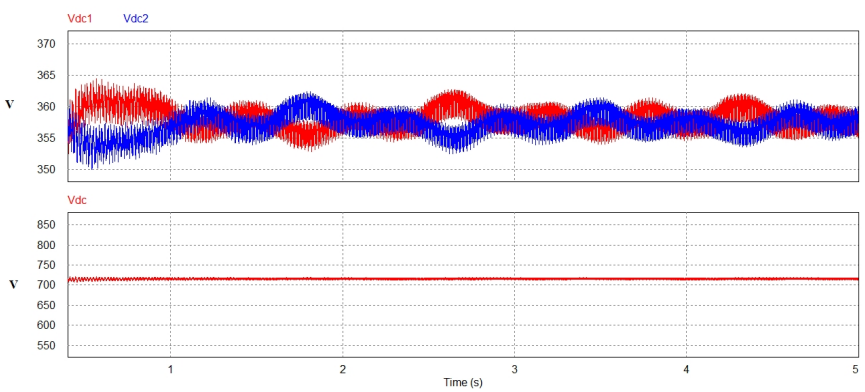


Fig.7. Upper and lower capacitors voltage and dc link voltage

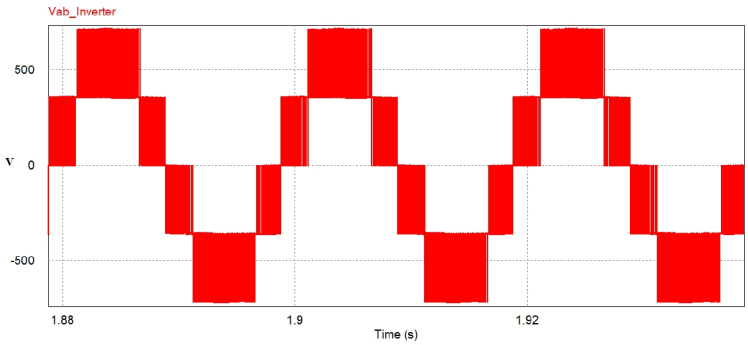


Fig.8. Line to line output voltage wave of three level T type inverter

CONCLUSION

Three Level T type voltage source inverter can be used for low voltage applications with high efficiency. It does not require to use clamping diodes like NPC inverter. It can reduce size of ripple filter. So overall size of active harmonics filter panel will be reduced. Novel self balancing control algorithm can work satisfactory to balance dc link upper and lower capacitors voltages. Source current THD has been reduced less than requirement of IEEE 519-1992 standards.

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A New Approach for Human Face Recognition Invariant to Illumination

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ABSTRACT

In this paper, we have design and implemented new preprocessing technique to eliminate illumination effect from human face images. The different stages include adaptive histogram equalization (AHE), Gaussian filtering, Log transform, difference of AHE + Gaussian filtering+ Log image, and AHE + Log image, and normalization. These steps are performed to get the image without illumination effect. We have used principle component analysis (PCA) and speed up robust features (SURF) for features extraction and for classification we have used euclidean distance, SURF matching and support vector machine (SVM). We have used different six combinations of techniques with and without using proposed preprocessing technique, feature extractions and classifications. We have calculated performance evaluation parameters like recognition rare (RR) and error rate (ER) of all six combinations of techniques on standard face databases like YaleB and Extended YaleB.

SUMMARY

Our proposed preprocessing technique and the combination of PCA and SVM give better recognition rate on standard YaleB and Extended YaleB face databases.

Keywords: Adaptive Histogram Equalization (AHE), Gaussian filtering, Log transforms, Principle Component Analysis (PCA), Speed Up Robust Features (SURF), Support Vector Machine (SVM), Recognition Rare (RR), Error Rate (ER).

1. INTRODUCTION

In the current era face recognition with uneven illumination effect is one of the most challenging tasks in the biometric pattern recognition techniques. Computerized systems can recognition the human face but whenever there is varying illumination in the face images it will degrade the performance of the system.

Human face images are captured under the uncontrolled environment due to that illumination is also varying in the face images.

Face recognition is one of the popular research areas in biometrics. It has been considered by researchers from diverse areas of sciences and those from diverse areas of computer sciences (*2*). Researchers doing research on machine recognition of human faces mostly deal with the computational aspects of face recognition. Figure 1 shows the sketch of any pattern recognition system.

1.1 Face recognition challenges

There are many challenges to human face recognition system. Some of possible challenges for human face recognition system are primarily (*1, 3*).

1) Changes in facial expression 2) Illumination change 3) Aging 4) Rotation 5) Image size 6) Profile vs. Frontal 7) Occlusion

MATERIALS AND METHODS

2. Proposed preprocessing technique

The preprocessing approaches are generally useful to reduce the illumination effects in the human face images in face recognition problem. Some samples images from YaleB face database with different illumination effects are given in fig. 2.

In our proposed preprocessing we have tried to eliminate illumination effect from the human face images. The block diagram of proposed preprocessing technique is given in fig. 3.

The input image is given as input to the adaptive histogram equalization (AHE). It divide the image into 8 x 8 sub regions and computes the histogram of each sub regions centered at a specified pixel to find out the mapped value for that pixel, It will accomplish a local dissimilarity enrichment. The output image of AHE is given as an input to the gaussian filter (*3*). Gaussian is a high pass filter so it will remove low frequencies and retain the high frequencies components in the human face image. Mainly the illumination effects are there in the low frequency component of the image, thus gaussian filter remove illumination effect and noise from image but it will blur the image. It uses various kernels those represent the nature of the gaussian hump. Equation 1 is used for the gaussian filtering.

$$G(x,y)=\frac{1}{2\pi\sigma^2}e^{-\frac{x^2+y^2}{2\sigma^2}} \quad 1$$

Where σ indicates standard deviation of gaussian distribution and G(x, y) is the output image. In our experiment we have used σ=0.5. The AHE+Gaussian output image is very high brightness and blurred. To remove these effects AHE+Gaussian output image is given as an input to the Log transformation. Log transform enhance the low frequency parts and compress the higher frequency parts. It is also helpful for varying lighting circulation and shadowed images. The next step of the preprocessing technique we take the difference of AHE+Gaussian+Log output image with AHE+Log output image and then we perform the normalization so it will gives the illumination free image. The output of each steps of our new proposed preprocessing approach is given in fig. 4.

3. Feature extraction techniques

We have used principle component analysis (PCA) and speed up robust feature (SURF) feature extraction techniques in our experimental study to calculate performance parameters.

3.1 Introduction to principle component analysis (PCA)

PCA was invested by Turk and Penland (*4*), for dimensionality reduction in the images. PCA is used to convert the 2-D image into 1-D feature vector. It is known as the eigenfaces. It represents the facial features of the images. The eigenfaces created as outcome of PCA translation makes use of facial features

to illustrate dissimilar training images or eigenfaces from the face dataset. The features vector obtained is projected on to a face database, which consequences in a key reduction in a size of image (*5*). PCA also finds the features of the test image and it is projected on the face space and compared with the training images in the face dataset. This will helps in reducing the largely examination time. The sample eigenfaces images are shown in fig. 5.

3.2 Introduction to speed up robust feature (SURF)

In image processing sometimes there is an interest to detect points or regions in an image (*6*). For this purpose there are algorithms to identify and illustrate limited features in images. From the human face image we are finding the interest points to provide feature description of the face image. This feature description is used to match the feature description of the test image and according to the feature matches we identify the face image from the dataset.

Another essential attributes of these features are those, the comparative positions among them in the original image don't alter from one face image to another face image. For example, if only the face template is used as features, It will work despite of the face position, but if the face components like, eyes, lips, nose etc. features are used then the recognition would be fail if the eyes are closed or mouth is opened. However for performance measurement SURF finds and uses a large number of features from both test and train images. It will eliminate the possibilities of the errors due to local variations in the all similar matching features errors (*7*). SURF can vigorously recognize objects under disorder and under biased occlusion; because of its SURF features descriptor is invariant to lighting conditions, affine distortion, size, and direction. In our experiment we had find strongest 100 features from the face image as shown in fig. 6.

4. Classification techniques

There are so many classifications techniques are available but we have used euclidean distance, SURF matching and support vector machine (SVM) in our experiments.

4.1 Euclidean distance and SURF matching

The test image is given as an input to the PCA method and finding the euclidean distance that distance is compared with the euclidean distance of the training images. The minimum euclidean distance image is classified as the output image. In SURF matching (*8*) we are finding the features of the test image and features of all images in the training set then we are matching each features of the test image with the features of every image in the training set images. The image which gives highest matching pairs between test and training image is classified as the output image.

4.4Support Vector Machine (SVM)

Support Vector Machines were designed for binary classification. It has been proposed by Vapnik and his co-workers (*9*) for biometric pattern identification. In classification problem we have given set of points belong to different classes. SVM determines the hyperplane which separates the set of points in the same class and maximize the distance from other class to the hyperplane. This hyperplane will reduce the threat of misclassification of images in the training set as well as unknown images in the test set. We have used multiclass classification SVM. It is the combination of binary class classification. We have used linear kernel function in our experiments.

5. Introduction to face databases

To demonstrate the performance of our technique, we have used the standard human face databases, YaleB and Extended YaleB (10). YaleB face database contains 10 subjects' images captured with varying 64 illumination surroundings, which are separated into 5 subsets according to lighting angle as shown in table 5.1. The total images in database are 10x64=640 images. The image dimension is 640 x 480 pixels. We had resized the image into 170 x 170 pixels. The Extended YaleB face database has total 16128 images. It contains 28 human persons under 9 poses and 64 lighting variations. The image naming

convention and dimension of images are similar to YaleB face database. We had resized the image into 170 x 170 pixels.

RESULTS AND DISCUSSION

In our experiment we had used different six combinations of techniques, in which three technique without using proposed preprocessing and three with using proposed preprocessing techniques. We had used varying number of test set and training set images of YaleB and Extended YaleB face databases as shown in table 6.1 respectively. The columns of the tables shows the subset number, number of training images, number test images and database used for the experiments. We have applied six different combinations of techniques on YaleB and Extended YaleB face databases. We have used varying number of test set and training set images as shown in table 6.1 and calculated recognition rate and error rate of each technique on YaleB face database. The evaluation parameters are shown in table 6.2. The first column indicates the techniques names, second column onwards indicates the RR and ER of each subsets in percentage. In the first column techniques abbreviations are PP+SF+SM (Proposed Preprocessing + SURF Features + SURF matching), SF+SM (SURF Features + SURF matching), PP+PCA+SVM (Proposed Preprocessing, Principle Component Analysis and Support Vector Machine), PCA+SVM (Principle Component Analysis and Support Vector Machine), PP+PCA+Euc. (Proposed Preprocessing, Principle Component Analysis and Euclidean distance), PCA+Euc. (Principle Component Analysis and Euclidean distance). By observing the parameters in the table 6.2, we conclude that PP+PCA+SVM combinations give better results compare to other techniques. It is highlighted in table 6.2. We had applied same combinations of techniques on the Extended YaleB face database and calculated RR and ER of each technique as shown in table 6.3. Number of test images and training images in each subset is shown in table 6.1. By observing table 6.3 we conclude that PP+PCA+SVM give better results compare to other techniques.

From both table 6.2 and 6.3 we observed that when we apply proposed preprocessing technique it will increase the recognition rate and decrease the error rate.

Comparison with existing methods

Many researchers had proposed so many techniques to eliminate illumination effects from the face images and recognize it. We had compared our result with the existing techniques on the YaleB face database (*2, 11-14*). The comparisons are shown in fig 7. The graph shows that PP+PCA+SVM give better results compared to other techniques.

CONCLUSION

In this paper we had anticipated new preprocessing technique to eliminate illumination effects from the face images. Also we had applied different combinations of techniques to eliminate the illumination effect and increase the face recognition rate and decrease the error rate. To measure the performance of techniques we had used two standard face databases, YaleB and Extended YaleB with different number of training and test images. We proved that PP+PCA+SVM combinations give better recognition rate and less error rate in all subsets in both the databases. The recognition rate and error rate depends on the selection of training and test images selection. In last section we had compared our techniques with existing techniques and proved that our PP+PCA+SVM give better recognition rate.

FIGURES

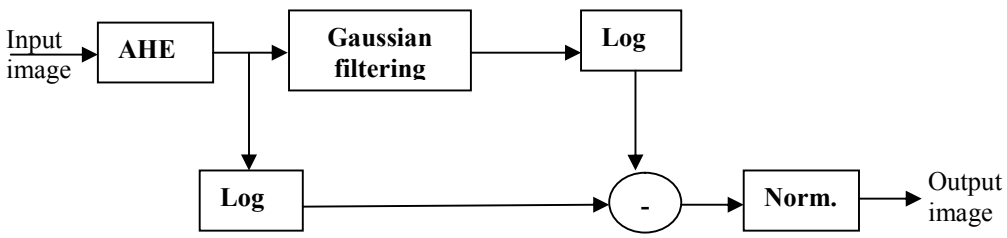
"**Fig1.** Sketch of pattern recognition system."



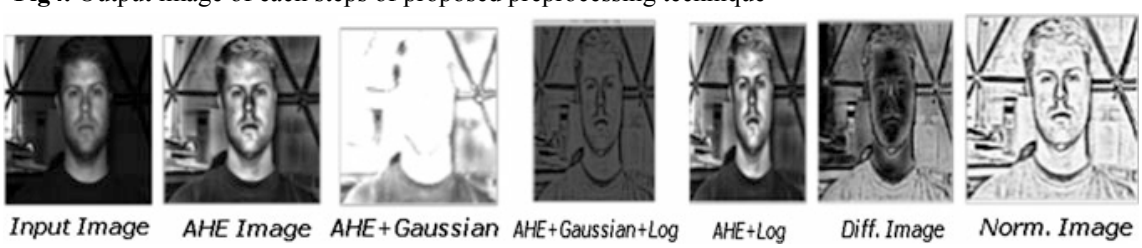
"**Fig2.** Sample images of varying illumination."



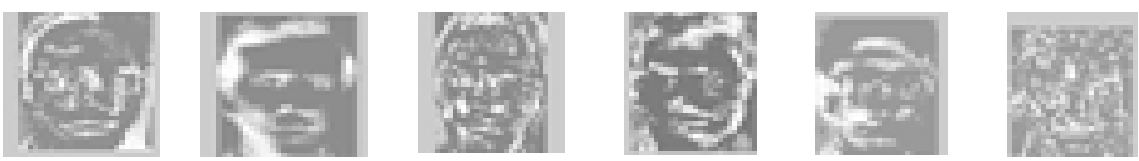
"**Fig3.** Block diagram of proposed preprocessing technique."



"**Fig4.** Output image of each steps of proposed preprocessing technique"



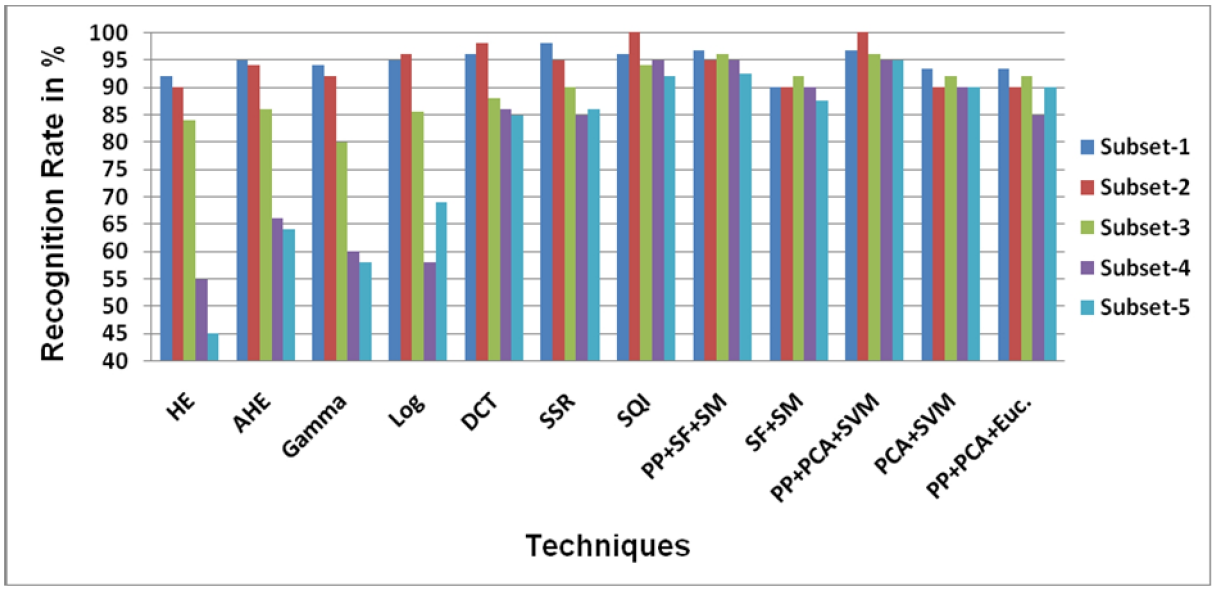
"**Fig5.** Sample Eigenfaces from ORL face dataset"



"**Fig6.** SURF features"



"**Fig7.** Comparison of proposed and existing techniques"



TABLES

"**Table 5.1** Subset division of YaleB Database."

Subset No	Lighting angle	No. of images
1	0 – 12	140
2	13 - 25	100
3	26 – 50	100
4	51 – 77	120
5	>77	180

"**Table 6.1** Number of training and test images from YaleB and Extended YaleB face database."

Subset No	Train Images	Test Images	DB	Train Images	Test Images	DB
1	110	30	YaleB	392	84	Extended YaleB
2	80	20		270	60	
3	95	25		336	84	
4	80	20		280	70	
5	140	40		504	100	
Total	505	135		1782	398	

"**Table 6.2** Recognition rate and Error rate of different techniques on subsets of YaleB face database."

Techniques	YaleB Subset-1		YaleB Subset-2		YaleB Subset-3		YaleB Subset-4		YaleB Subset-5	
	RR (%)	ER (%)	RR (%)	ER (%)	RR (%)	ER (%)	RR (%)	ER (%)	RR (%)	ER (%)
(PP+SF+SM)	96.67	3.33	95.00	5.00	96.00	4.00	95.00	5.00	92.50	7.50
(SF+SM)	90.00	10.00	90.00	10.00	92.00	8.00	90.00	10.00	87.50	12.50
(PP+PCA+SVM)	**96.67**	**3.33**	**100.00**	**0.00**	**96.00**	**4.00**	**95.00**	**5.00**	**95.00**	**5.00**
(PCA+SVM)	93.33	6.67	90.00	10.00	92.00	8.00	90.00	10.00	90.00	10.00
(PP+PCA+Euc.)	93.33	6.67	90.00	10.00	92.00	8.00	85.00	15.00	90.00	10.00
(PCA+Euc.)	90.00	10.00	85.00	15.00	88.00	12.00	80.00	20.00	85.00	15.00

"**Table 6.3** Recognition rate and Error rate of different techniques on subsets of Extended YaleB face database."

Techniques	YaleB Subset-1		YaleB Subset-2		YaleB Subset-3		YaleB Subset-4		YaleB Subset-5	
	RR (%)	ER (%)	RR (%)	ER (%)	RR (%)	ER (%)	RR (%)	ER (%)	RR (%)	ER (%)
(PP+SF+SM)	96.43	3.57	95.00	5.00	95.24	4.76	97.14	2.86	94.00	6.00
(SF+SM)	95.24	4.76	93.33	6.67	94.05	5.95	94.29	5.71	91.00	9.00
(PP+PCA+SVM)	**96.43**	**3.57**	**96.67**	**3.33**	**97.62**	**2.38**	**95.71**	**4.29**	**96.00**	**4.00**
(PCA+SVM)	90.48	9.52	95.00	5.00	96.43	3.57	94.29	5.71	94.00	6.00
(PP+PCA+Euc.)	92.86	7.14	91.67	8.33	88.10	11.90	91.43	8.57	94.00	6.00
(PCA+Euc.)	89.29	10.71	90.00	10.00	86.90	13.10	88.57	11.43	90.00	10.00

ACKNOWLEDGEMENT

We are very thankful to our principal Dr. F.S. Umrigar and Dr. D.G. Thakore, Head Computer Engineering Department and TEQIP-II Coordinator, BVM Engineering College and RK University, Rajkot for encourages us to write this research paper. Our special thanks to Ralph Gross for providing the database.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th& Jan. 6th, 2016)

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Entrepreneurial opportunity of using adsorption cooling system

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ABSTRACT

Increasing global warming and its ill effect on the environment has triggered the development in research of adsorption refrigeration system. Thiscommunication lucidly presents the principle of operation of adsorption cooling system. A comparison is made between the VCRS & SARS based on thermodynamic platform with performance parameters like COP & SCP comprising ofsame capacity to bring in the advantage of zero Ozonosphere depletion potential (ODP), zero global warming potential (GWP), size, scale and practical applicability as entrepreneurial opportunity. Finally cost analysis is done for both the system based on initial cost, running cost and replacement cost. It is found that though the adsorption cooling system is more costly as compared to conventional VCRS but due to long life duration and environmentally friendly operation of the former system makes it a preferred candidate.The size of evaporator for the SARS (A_{eva}= 1.48m^2) is about 65% more than conventional VCRS (A_{eva}= 0.8m^2).

SUMMARY

For the same capacity of 3.5 kW cost of for SARS is far more than VCRS. The size of evaporator for the SARS is about 65% more than conventional VCR system.Activated carbon-methanol is more suitable for ice making application, as it can produce 14 kg of ice per day with COP of 0.34 and later system is suitable for producing chilled water with COP of 0.42.

Keywords: Adsorption, Refrigeration, Silica-gel, solar, heat & mass transfer

INTRODUCTION

In today's modern world the field of refrigeration and air-conditioning is one of the most important aspects of our life whether it may be field of food preservation or comfort living. A majority of the refrigeration and air conditioning devices used today are running on electricity. Majority of the electricity is produced by burning of fuel like coal and natural gas which are non-renewable in nature, thus constitutes to release of the greenhouse gasses in the atmosphere which leads to the global warming. The overall temperature on the earth surface has raised during the last century due to global warming this has resulted in the imbalance of the climate around the world this has further increased the demand of the air conditioning and refrigeration. This is like never ending loop and therefore the time has come that to reduce the consumption of fossil fuels to produce electricity and shift towards the clean and renewable energy at least in the field of refrigeration and air-conditioning.

One such option of using the clean or green energy is the solar energy. It is the main source of energy and available in plenty. The fact that peak cooling requirement coincide with the peak availability of the solar energy will only help. In recent years many projects have been developed justifying the use of solar cooling for cooling of residential and commercial buildings.

The solar cooling is broadly classified in two main categories one is solar thermal application and solar electric application. The solar thermal application uses the heat energy gained from solar radiation for pressurisation of the refrigerant and the solar electric application uses the solar photo voltaic module to run the compressor of the conventional Vapour compression refrigeration system. In solar thermal application there are further sub division namely solar adsorption cooling, solar absorption cooling and solar decent cooling.

As shown in figure 1, Li et al. (1) performed experiments with a solar powered ice maker that had activated carbon–methanol as working pair. The icemaker, which is shown schematically in Fig. 8, had a COP ranging from 0.12 to 0.14, and produced between 5 and 6 kg of ice per m^2 of collector area. Analysing the temperature gradient within the adsorbent bed, the authors concluded that in order to improve the performance of system, the heat transfer properties of the adsorber bed must be enhanced. This could be achieved by increasing the number of fins or using composite adsorbent.

As shown in figure 2, In 2010 HuilongLuo at el *(2)*, had developed a solar-powered adsorption chiller with heat and mass recovery cycle. Their system consists of a solar water heating unit, a silica gel-water adsorption chiller, a cooling tower and a fan coil unit. The adsorption chiller included two identical adsorption units and a second stage evaporator with methanol working fluid. The effects of operation parameters on system performance were tested successfully. Test results indicated that the COP (coefficient of performance) and cooling power of the solar-powered adsorption chiller could be improved greatly by optimizing the key operation parameters, such as solar hot water temperature, heating/cooling time, mass recovery time, and chilled water temperature. Under the climatic conditions of daily solar radiation being about 16–21 MJ/m^2, this solar-powered adsorption chiller can produce a cooling capacity about 66–90W per m^2 collector area, its daily solar cooling COP was about 0.1–0.13.

Our focus in this communication will be on the comparison solar adsorption cooling and vapour compression cooling system. Both the systems are compared on the thermodynamic platform and their detail working is explained. Pressure-enthalpy (p-h) & Temperature-Entropy (t-s) characteristics are considered for VCR System and Clausius-clapeyron and dhurring diagram are considered to gauge the performance of the solar adsorption cooling. Performance both the systems are compared on various performance parameters like Coefficient of Performance (COP), specific cooling power and thermodynamic analysis. Finally cost analysis is done for the life cycle of systems considering initial, running and replacement cost. Comparison is made between the adsorption refrigeration systems with Activated-carbon- methanol and silica gel-water as working pairs.

MATERIALS AND METHODS

Working principle of Vapour compression Refrigeration system:

The vapour compression refrigeration system works on the principle of second law of thermodynamics. According to clausius's statement the heat cannot flow from the colder body to hotter body on its own there is a need to supply some external work, for the process to complete. That external work is supplied by the compressor.

As shown in figure 6, the compressor of the conventional vapour compression refrigeration system is replaced by adsorber for adsorption refrigeration system. The driving energy in case of VCRS is the electric energy where here in case of adsorption system heat energy is the driving energy and compresses the refrigerant by way of constant pressure heating. The heat energy can be of any form either waste heat energy or solar energy or it may be a special heat source in form of furnace or a boiler.

Working principle of solar Adsorption refrigeration system:

Figure7shows adsorption refrigeration cycle which works on three temperature levels namely the desorber, ambient and evaporator temperature having two sources and two sinks. The energy required to drive the thermal compressor is obtained from the source at desorber temperature, this is similar to the compressor work in the Vapour compressor refrigeration system.

Where as the working principle of the Vapour compression refrigeration system is explained in figure 3,figure 4 shows basic setup of vapour compression refrigeration system & figure 5 shows p-h & t-s

diagram. the electrical energy is used to drive the compressor where the compressor compresses the vapour refrigerant from the evaporator thus increasing its pressure and temperature.

Figure 8 shows the duhring diagram for adsorption cooling system which shows the relation of pressure with the temperature for whole operation of the system. Here the pressure is in kPa and Temperature is in °C.

1-2 Preheating mode:

During preheating mode the pressure increases and adsorption bed is made ready to start desorption process. Heat is supplied to the bed in form of hot water flowing through the adsorption bed.

2-3 Desorption mode:

Refrigerant water vapours starts leaving from adsorption bed to condenser resulting in the decreasing the concentration ratio of bed. Pressure and temperature reaches to highest value in whole cycle during this process. Further heat is rejected in the condenser and refrigerant vapours are converted to liquid.

3-4 Pre cooling mode:

Now the stored heat energy in form of sensible heat of the adsorbent bed is released to the cooling water during this precooling mode, thus bed is prepared for the adsorption of vapours from the evaporator.

4-1 Adsorption mode:

During this mode the adsorption bed is connected to evaporator and low pressure triggers the evaporation of the liquid refrigerants and once again concentration ratio of bed increases. Cooling is produced in this mode.

As shown in figure 9, the detail working of the adsorption refrigeration can be understood by the clapeyron diagram. The system works between condenser pressure and evaporator pressure both the pressure are being in vacuum.

Isobaric cooling (Adsorption-Evaporation) Process 4-1:

Evaporation of the refrigerant is triggered by adsorption in the bed at evaporator pressure due to the adsorption the concentration of the refrigerant in the adsorption bed is increasing from low concentration line to the high concentration line at a constant pressure resulting in cooling effect in evaporator shown by process 4-1.

Isosteric heating (constant concentration) Process 1-2:

Once the adsorption bed is saturated with the vapour refrigerant the heat energy is required to remove the vapour refrigerant from the bed this energy is supplied by flowing the hot water through the bed this increases the pressure of the bed to the saturation pressure of the refrigerant and pressure is increases to the condenser pressure shown by process 1-2.

Isobaric heating (Desorption-condensation) Process 2-3:

After the isosteric heating the high pressure vapour refrigerant enters the condenser from the bed where the further heat rejection takes place in the condenser at constant pressure. As more and more vapour refrigerant is taken out from the bed its concentration reduced to low concentration shown by process 2-3.

Isosteric cooling (Constant concentration) process 3-4:

Now the liquid refrigerant from the condenser enters the evaporator via expansion device thus pressure reduces to the evaporator along the constant concentration line shown by process 3-4 in the figure.

Design procedure for solar adsorption cooling system:

The adsorption cooling system works under vacuum. In our case water is used as refrigerant. It is found from the table of saturated water and steam temperature table the vapor pressure of the water is 2.5 kPa at a temperature of 20.5°C *(3)*. The complete working of the adsorption cooling system is divided in to two process that is Adsorption and desorption both having equal duration of 900 s each. Whole design is carried out considering 3.5kW of cooling capacity. The adsorbate-adsorbent pair is water-silica gel.

Step 1: To compute amount of refrigerant required to produce 3.5kW of cooling effect.

The cooling effect produced due to vaporization of 1 kg of water is calculated from equation:

$$Q_c = \frac{q_{eva,ads}}{T_{ads}}$$

Where $q_{eva,ads}$ isheat of vaporization of 1 kg of water at evaporator pressure and temperature. T_{ads} is the duration of adsorption process in sec

Q_c= 2733.56 w/kg of refrigerant.

For calculating the amount of refrigerant required to produce 3.5kW of cooling.

$$V_{ref} = \frac{Q_{cap}}{Q_c}$$

$= 1.280*10^{-3}$ m^3.

Step 2: To compute the amount of silica gel.

It is found from literatures that the silica gel can absorb 30 % of water of its dry weight in 1 hour when the height of bed is 1m and velocity of air is 0.2 m/s with relative humidity of 60% *(4, 5)*.

Based on the above data it is possible to find the relation between the absorptivity of the silica gel and bed physical parameters.

$$A_{900} = A_{60} * 0.9 * \frac{T_{ads}}{T_{60}}$$ (g of water/100 g of silica gel)

= 6.3 g of water/100 g of silica gel

Now to absorb 1280 gm of water refrigerant in 900 s the amount of silica gel required is calculated and come to 20.31 kg.

Therefore the amount of silica gel required is selected as 21 kg.

Step 3: To compute the heating power to regenerate the silica gel for adsorption/ desorption process:

$$Q_{reg} = q_{bed,des} * \frac{V_{ref}}{T_{des}}$$ kW

= 5870*1280/900

=8348 W or 8.3 kW.

Step 4: To compute area of solar collector to provide heating power for regeneration of the adsorbent bed

$A_{sc} = Q_{reg} / \eta_{sc} * A_{rad}$ m^2

Where A_{rad} average solar radiation for ahmedabad region= 5.76 Kwh/m^2/day *(6)* = 0.48 kW/m^2

η_{sc} = efficiency of solar collector 50%

A_{sc}= 35.58 m^2

Step 5: To compute the storage tank capacity for heating and cooling water.

$V_h = [Q_{reg} / C_{ref} * \Delta T_{ads}]$*3600*10 $^{-3}$$m^3$

= 1.02 m^3

$V_{ch} = [Q_{eva} / C_{ref} * \Delta T_{eva}]$*3600*10 $^{-3}$$m^3$

=0.602 m^3

Step 6: To compute surface area of evaporator, condenser and adsorber bed

Evaporator surface area:

$$A_{eva} = \left\{-Ln\left[1 - \frac{Q_{eva}}{\Delta T_{eva} * K}\right] * K\right\} / U_{eva} \text{ m}^2$$

Consider copper as the material for the construction of evaporator, taking thermal conductivity and heat transfer coefficient of copper:

K =400 W/m K ;U_{eva} = 1000 W/m^2 K

= 1.48 m^2

Condenser surface area:

$$A_{cond} = \left\{-Ln\left[1 - \frac{Q_{cond}}{\Delta T_{cond} * K}\right] * K\right\} / U_{cond} \text{ m}^2$$

Consider copper as the material for the construction of condenser, taking thermal conductivity and heat transfer coefficient of copper:

K =400 W/m K ;U_{eva} = 1000 W/m^2 K

= 1.68 m^2

Adsorber bed surface area:

$$A_{bed} = \left\{-Ln\left[1 - \frac{Q_{bed}}{\Delta T_{bed} * K}\right] * K\right\} / U_{bed} \quad m^2$$

Consider aluminum as the material for the construction of adsorber bed, taking thermal conductivity and heat transfer coefficient of copper:

K =205 W/m K ;U_{eva} = 500 W/m^2 K

= 2.34 m^2

Step 7: To compute the mass flow rates of chilled water, cooling water and hot water

Mass flow rate of chilled water for evaporator:

$M_{ch} = 3.6 * Q_{eva} / C_{ch} * \Delta T_{ch}$ m^3/hr

= 0.32 m^3/hr

Mass flow rate of hot water for adsorber bed:

$$M_{hw} = 3.6 * \frac{Q_{bed}}{C_{hw} * \Delta T_{hw}} \text{m}^3/\text{hr}$$

= 0.41 m^3/hr

Mass flow rate of cooling water for adsorber bed:

$$M_{cw} = 3.6 * \frac{Q_{cond}}{C_{cw} * \Delta T_{cw}} \text{m}^3/\text{hr}$$

= 0.38 m^3/hr

Step 8: To Compute specific cooling power of adsorption cooling system:

$$Specific\ cooling\ power = \frac{Q_{eva}}{W_s}$$

= 166 W / kg of silica gel

Step 9: To Compute Coefficient of Performance.

$$COP = \frac{Q_{eva}}{Q_{reg}}$$

= 0.42

As our main focus is on the adsorption cooling system therefore detail design is not studied for the vapor compression refrigeration system and so all data system parameters and operating conditions and performance equations are extracted from *(7-9).*

Refrigerant: R134a
Evaporator pressure: 200 kPa
Condenser Pressure: 1400 kPa
Refrigerant mass flow rate: 0.09m^3/hr
Degree of super heat: 10°C
Degree of sub cooling: 5°C
Mass of refrigerant: 1.15 kg

Various detail of Vapour compression refrigeration system (Refrigerant thermo physical properties, material of construction, specifications) at evaporator pressure and condenser pressure are shown table 1:

Evaporator detailsare given in Table 2.

Thermophysical properties of refrigerant R134a are given in table 3.
Based on the above mentioned parameters for the vapour compression refrigeration system, a simple ideal design steps are discussed in the following section:

Design procedure for vapour compression refrigeration system
Assumptions:

1. Changes in kinetic and potential energy are neglected while considering the different components
2. Compression process is considered isentropic process
3. Heat loss from evaporator is negligible
4. No heat transfer and pressure drop in connecting pipe lines
5. Steady flow process in all components

Step 1: calculate refrigeration capacity

$$Q_{eva} = m_r * (h_{eva,out} - h_{eva,in})$$ kW

=3.317 kW

Step 2: work input to compressor:

$$W_c = m_r * (h_{com,out} - h_{com,in})$$ kW

η_{comp} = 88%

$h_{com,out}$= 301.74 KJ/Kg

$$W_c = m_r * (h_{com,out} - h_{com,in})$$ kW

= 1.21 kW

Step 3: Heat rejected by condenser

$$Q_{con} = m_r * (h_{con,out} - h_{con,in})$$ kW

= 4.53 kW

Step 4: Pressure drop at expansion device

Considering throttling process ie. Constant enthalpy process $h_{expin} = h_{expout}$

The exit quality of the refrigerant is two phase , so using dryness fraction to find the quality of the refrigerant at exit of expansion device.

$$h_{out} = h_f + (Xh_{fg})$$ kj/kg

whereh_f enthalpy of saturated liquid refrigerant, h_{fg} = latent heat of vaporization at evaporator pressure.

$h_{expin} = h_{expout}$=120.39KJ/kg

Step 5: Calculating the Coefficient of performance (COP)

$$COP = \frac{Q_{eva}}{W_c} = \{m_r * (h_{eva,out} - h_{eva,in}) / m_r * (h_{com,out} - h_{com,in})\}$$

COP = 2.72

Comparison of Adsorption refrigeration system using Activated carbon-methanol as working pairs with silica gel-water pair for same capacity air conditioning application are shown in Table 3.

Table shows the comparison between the adsorption cooling systems comprising of different working pairs ie Activated carbon –methanol and silica gel-water for same cooling capacity of 3.5 kW.

As seen from the data in table for same capacity the size of activated carbon-methanol system is more than silica gel-water system mainly due to higher operating temperatures and low latent of vaporization of methanol as compared to water.

A system with activated–carbon and methanol is suitable for ice making application due to lower freezing point of the methanol. The same system is able to produce 14kg of ice per day with COP of 0.23 for ice making application. Long cycle time facilitates better cooling capacity but low COP.

Cost analysis for Vapour compressin system and solar adsorption cooling system are shown in table 4.

It is found that though the adsorption cooling system is more costly as compared to conventional Vapour compression refrigeration system (VCRS) but due to long life duration and environmentally friendly operation of the former system makes it a preferred candidate.

Further the cost of solar adsorption system will reduce over time and cost of electricity will increase day by day so adsorption refrigeration have bright future ahead.

RESULTS AND DISCUSSION

Table 3 shows the comparison for two types of solar adsorption refrigeration systems for activated carbon-methanol and silica gel-water as working pairs. It is found that for same capacity AC-Methanol pair can produce 14 kg of ice per day with COP of 0.34 and silica gel-water pair is suitable for producing chilled water with COP of 0.42.The AC-Methanol adsorption system is more in size due to lower heat of vaporization of methanol as compared to water.

Table 4 shows cost analysis of solar adsorption refrigeration system and vapour compression refrigeration. The initial cost of VCRS is very less as compared to SARS; the running cost of SARS is negligible as compared to VCRS. Solar adsorption refrigeration systems are more bulky in size and less efficient in operation due to lower thermal inertia and interminent nature of the adsorption and desorption process.

Table 5 for the same capacity of 3.5 kW cost of for SARS is far more than VCRS. The size of evaporator for the SARS (1.48m^2) is about 65% more than conventional VCRS (0.8m^2).

CONCLUSION

Solar Adsorption systems are heavy and bulky in size due to lower thermal inertia and coefficient of performance is much lower as compared to the conventional vapour compression refrigeration system. The use of solar collector to supply the heat of adsorption ie, driving energy further increases the cost and space requirement. As the adsorption systems work under the vaccum pressure there is a great requirement to maintain the pressure below the atmosphere. As seen from the table for the same cooling capacity the evaporator size for the adsorption system is more than 65% as compared to the vapour compression system. As the availability of solar energy is internment in nature, the necessity to supply cooling for continuously for 24 hours leads to the additional components like chilled water and hot water storage tanks, which increases the cost. Considering the future prospects of environmental problems these non-conventional refrigeration systems provides definitively a bright solution.

Considering the present state of research in the field of adsorption refrigeration system there are high chances that these system will dominate the conventional compression based refrigeration systems over a time. Once their COP increases and mass production will lead to reduction of cost so here lie the entrepreneurial opportunities.

FIGURES

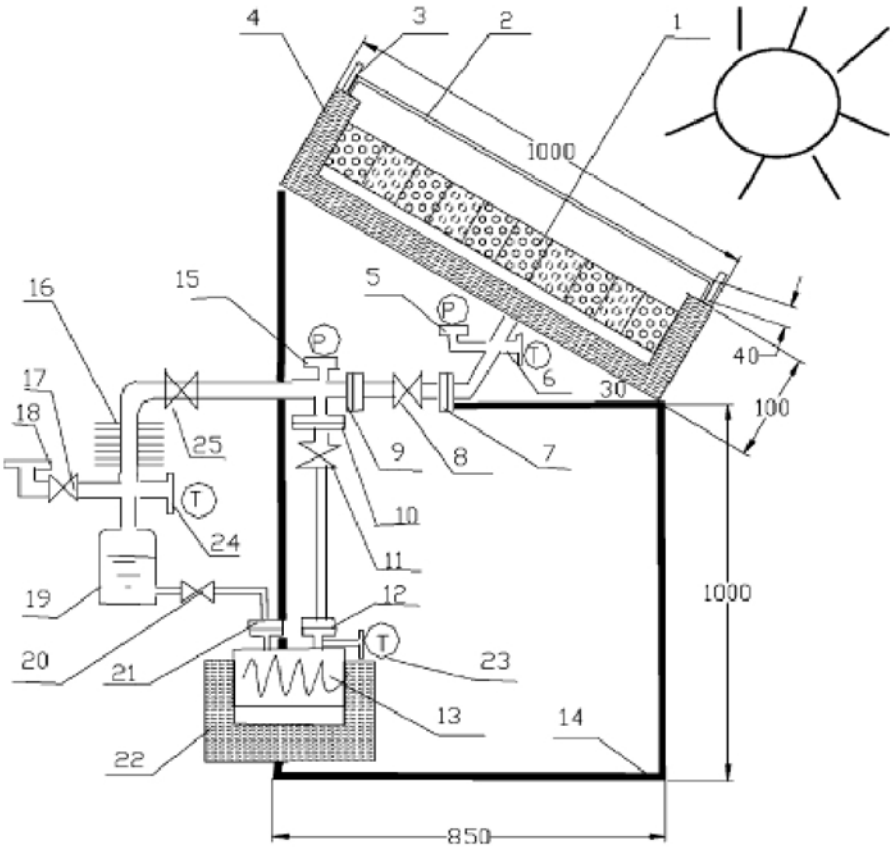


Fig.1: Schematic of the solar solid-adsorption ice maker: (1) adsorbent bed; (2) glass cover; (3) damper; (4) insulated material; (5,15) pressure gauges; (6,23,24) temperature gauges; (7,9,10,12) connecting flanges; (8,11,17,20,25) valves; (13) evaporator; (14) bracket; (16) condenser; (18) refrigerant input pipe;(19) Reservoir; (22) ice box. *(1)*

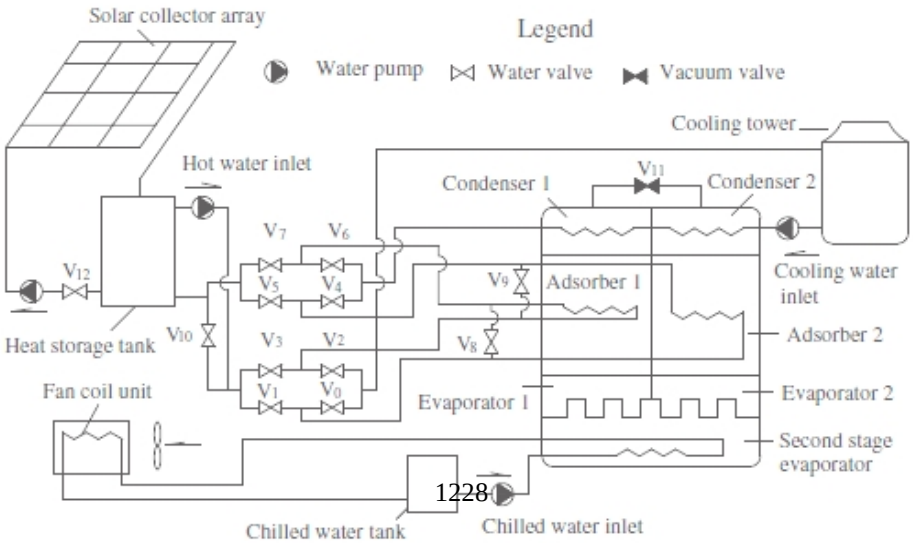


Fig.2: Schematic Diagram of Solar powered Adsorption Chiller. *(2)*

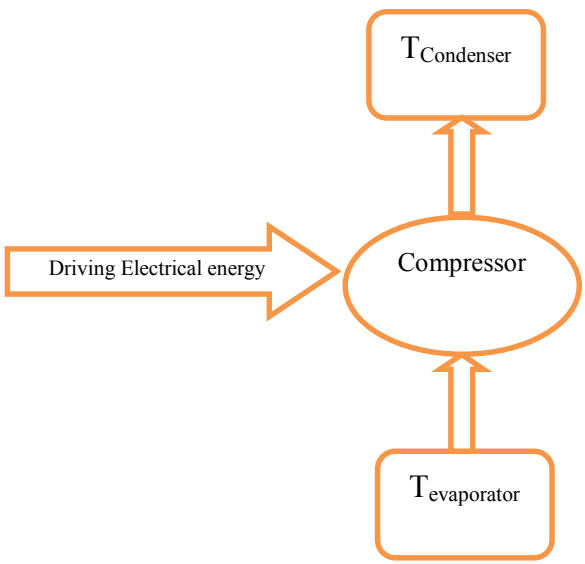


Fig.3: Working Principle of Vapour compression refrigeration system

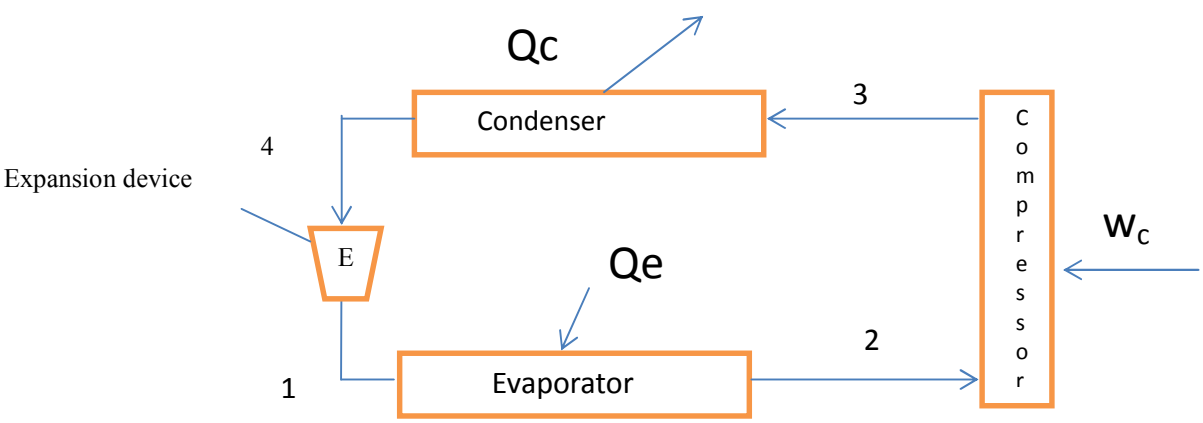


Fig.4: the basic diagram of Vapour compression refrigeration system

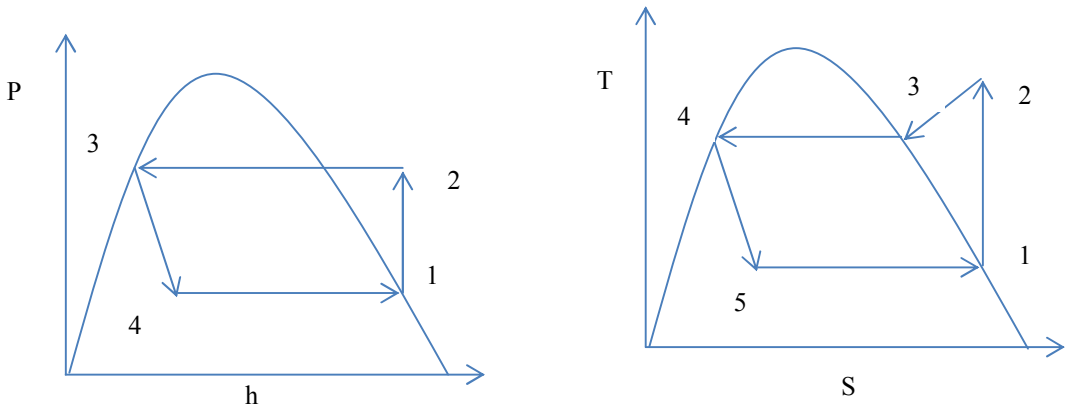


Fig.5: p-h & t-s Diagram for Vapour compression refrigeration system

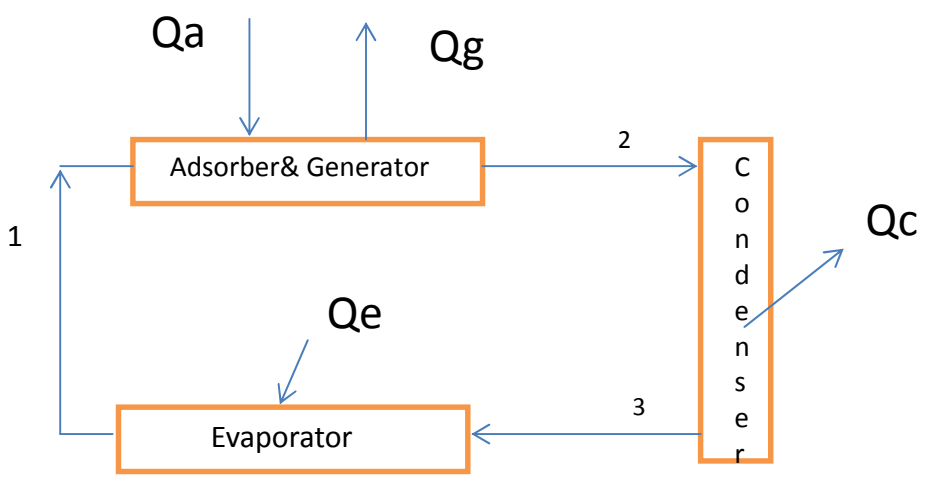


Fig.6: Adsorption refrigeration system

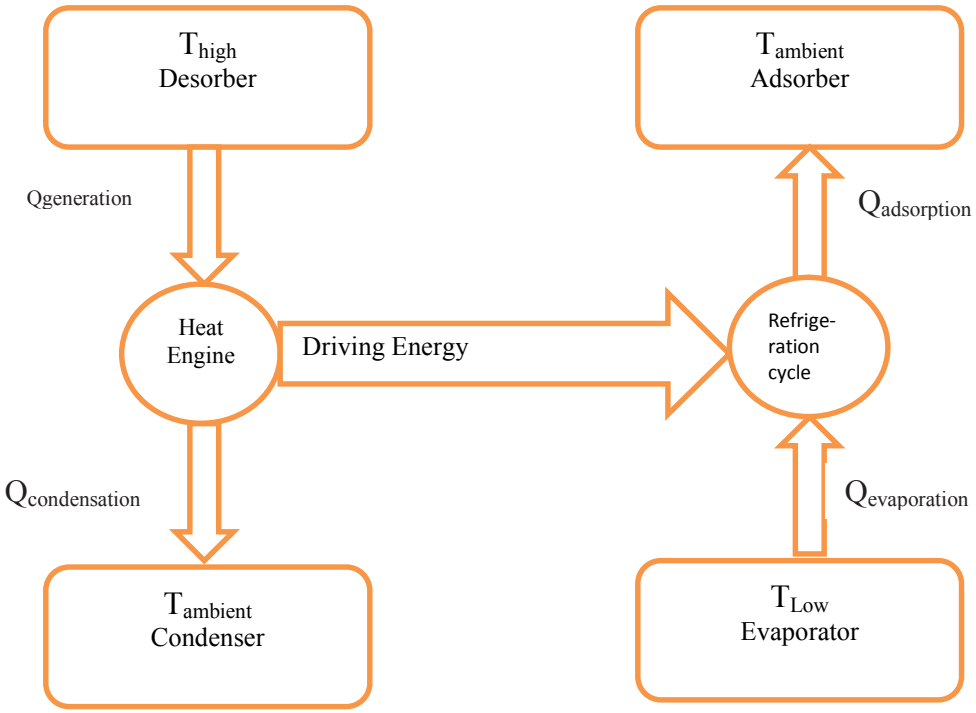


Fig.7: Working Principle of Adsorption Refrigeration cycle

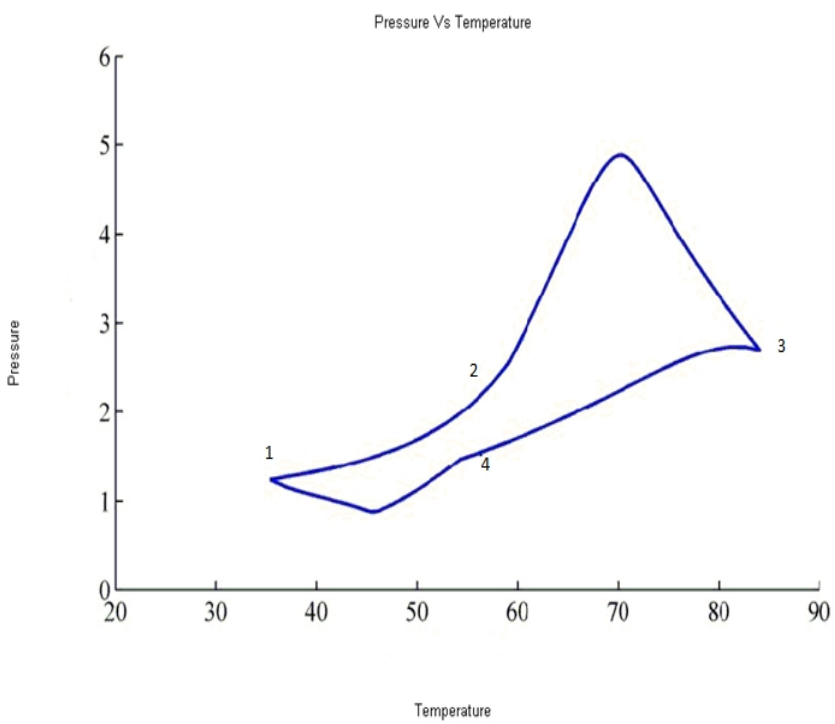


Fig.8: Duhring Diagram

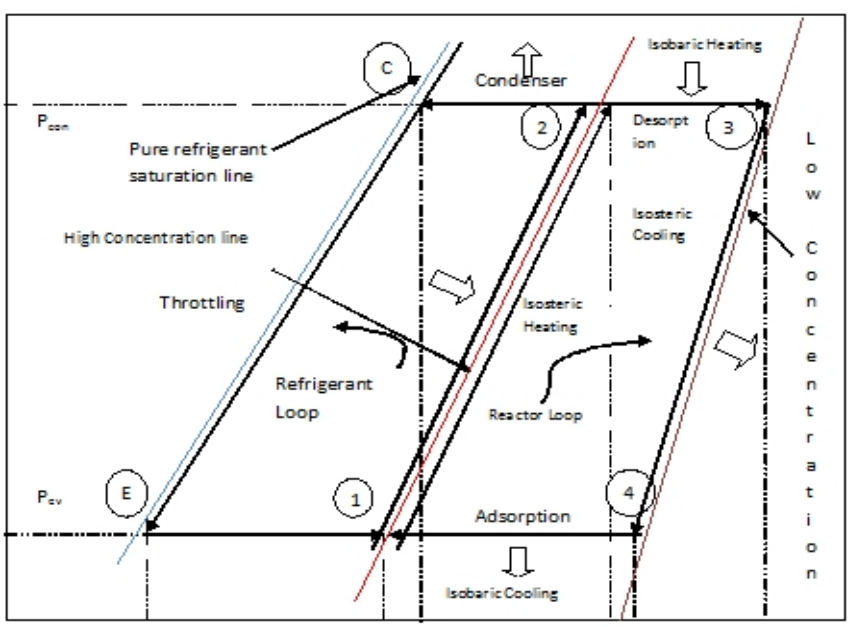


Fig.9: Adsorption Clapeyron Diagram

TABLES

Condenser Pressure: 1400 kPa	
T_{sat}	52.4°C
T_{cond}	48°C
$h_{expin} = h_{expout}$	120.39KJ/kg
Degree of sub cooling	
Material of construction:	
Copper tube with aluminum fins	K= 400 w/m.K ; U= 1000 w/m^2.K
Electric motor efficiency	100%
Outer Diameter	9.5 mm
Inner diameter	8 mm
Length	13 m
Surface area	2.4m^2 (air cooled)

Table 1.Condenser Specification

Evaporator Pressure: 200 kPa	
Refrigerant	R134a
$T_{eva,in}$	-10 °C

h_{compin}	253.05 KJ/kg
Entropy S_{compin}	0.9698 KJ/kg.K
Material of construction:	
coil type with internal fins of copper	K= 400 w/m.K ; U= 1000 w/m^2.K
Outer Diameter	17 mm
Inner diameter	15.5 mm
Length	0.9m or 900 mm
Surface area	0.8 m^2

Table 2. Evaporator Specification

Comparison for Various parameters for solar adsorption refrigeration system using Silica gel- water pair and Acvitaved Carbon- methanol(AC-Methanol)		
System parameters	VCRS(AC-methanol)*(11)*	SARS(silica-water)
Refrigeration capacity	3.46kW	3.5kW
Refrigerant mass	0.9 kg	1.280kg
Adsorbent mass	26 kg	21 kg
Heating power/Compressor work	7.2 kW	8.3kW
Solar collector area	Not required	35.58m^2
Evaporator surface area	2.5 m^2	1.48 m^2
Storage tank Volume(Hot water)	Not required	1.02m^3
Storage tank Volume(Chilled water)	Not required	0.60 m^3
Condenser surface area	3.2 m^2	1.68 m^2
Adsorber bed surface area	5 m^2	2.34 m^2
Mass flow rate of refrigerant/Chilled water	1.157m^3/hr	0.32 m^3/hr
Coefficient of Performance(COP)	0.34	0.42
Evaporator Pressure	15kPa	2.5 kPa
Condenser Pressure	101kPa	101 kPa
Cooling/heating cycle time	2400 s	900 s

Table 3. Comparison of two types of adsorption systems for air conditioning application *(11).*

Cost analysis for Vapour compressin system and solar adsorption cooling system for capacity of 3.5 kW		
Type of system	VCRS	SARS
Initial cost	30,000 Rs	5 lacs(approx.)
Running cost per year	18000 Rs	Negligible as compared to vcrs
Replacement cost(3 years)	30,000	Nil
Environmental pollution	High	No pollution
Break even	------	10 years

Table 4. Cost analysis of VCRS & SARS

Comparison for Various parameters for Vapour compression refrigeration system and solar adsorption refrigeration system using Silica gel- water pair.		
System parameters	VCRS	SARS
Refrigeration capacity	3.5kW	3.5kW
Refrigerant mass	0.6 kg	1.280kg
Adsorbent mass(Silica gel)	----------	21kg
Heating power/Compressor work	1.21kW	8.3kW
Solar collector area	----------	35.58m^2
Evaporator surface area	0.82m^2	1.48 m^2

Storage tank Volume(Hot water)	----------	1.02m^3
Storage tank Volume(Chilled water)	----------	0.60 m^3
Condenser surface area	2.4 m^2(air cooled)	1.68 m^2
Adsorber bed surface area	----------	2.34 m^2
Mass flow rate of refrigerant/Chilled water	0.09m^3/hr	0.32 m^3/hr
Coefficient of Performance(COP)	2.72	0.42
Evaporator Pressure	200kPa	2.5 kPa
Condenser Pressure	1400 kPa	101 kPa
Cooling/heating cycle time	Temperature cutoff	900 s

Table 5. Comparison of VCRS & SARS.

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NOMENCLATURE

p	pressure	kPa
h	Enthalpy	kJ/kg
t	temperature	°C
S	entropy	kJ/kg.K
W	watt	J/s
s	second	time
w	work	J
Q	heat	J
V	volume	m^3
k	thermal conductivity	W/m.K

SUBSCRIPT

cond	condenser
eva	evaporator
g	generator
a	adsorber
ads	adsorption
des	desorption
ref	refrigerant
reg	regeneration
sc	solar collector
h	hot
ch	chilled
exp	expansion
A900	adsorption quantity in 900 s

ABBREVIATIONS

VCRS	vapour compression refrigeration system
ODP	ozonosphere depletion potential
GWP	global warming potential
COP	coefficient of performance
SARS	solar adsorption refrigeration system

APPENDIX

Molar mass	102 kg/Kmol	
Normal boiling point	-26°C	
Critical temperature	101°C	
Critical pressure	4.06 MPa	
properties at saturation(0°C)		
Pressure	Liquid	Vapour
	0.29MPa	0.29MPa
Volume	Liquid	Vapour
	0.77 m^3/Kg	69.31 m^3/Kg

Specific heat capacity	Liquid			Vapour	
	C_p=1.34 KJ/Kg.K		C_V=0.88KJ/Kg.K	C_p=0.90KJ/Kg.K	C_V= 0.76 KJ/Kg.K
Viscosity	Liquid		Vapour		
	$271.08*10^{-6}$Pa.S		$10.73*10^{-6}$Pa.S		
Thermal Conductivity	Liquid	Vapour			
	0.092 W/m.K	0.012W/m.K			
Surface tension	0.012 N/m				
Heat of vaporization	198.6 KJ/Kg.K				
Ozone Depletion Potential(ODP)	0				
Global Warming potential (GWP)	1300				

Appendix 1. Thermophysical Properties of refrigerant R134a. *(10)*

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Biometric Image Protection Using Compressive Sensing and DCT based Watermarking Technique

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ABSTRACT

In this paper, a new watermarking technique proposed for security of biometric image in multibiometric system. In this proposed technique, sparse watermark biometric data embed into discrete cosine transform (DCT) coefficients of the host biometric data. In this proposed technique, the compressive sensing theory is used to provide more computational security to watermark biometric data. For our experimentation, face and fingerprint image is used where measurements of the watermark fingerprint image which is generated using compressive sensing theory are watermarked into face image. The results show that this proposed watermarking technique is fragile compared with existing watermarking techniques available in the literature when watermarked face image is subject to watermarking attacks.

SUMMARY

A proposed watermarking technique based on Compressive Sensing Theory and DCT for providing security to biometric image in multibiometric system.

Keywords: Biometrics, Compressive Sensing, Discrete Cosine Transform, PSNR, Watermarking

1. INTRODUCTION

With the current technology used for person recognition, the security of biometric data against modification or spoofing attacks in biometric system is major concerned *(1)*. The applications such as E-banking, ATM, smart cards and high security of biometric data, either while biometric data is modified in the database. This makes implementation of the biometric data protection technique a hot research topic. The biometric system is providing automatic authentication to individual using their biometric characteristics. The popularity of biometric systems is increasing nowadays because it is differentiated between authorized and unauthorized user *(1)*. Schneier described that biometric system work properly only if the biometric data came from the sensor is genuinely individual and provide security for enrolled biometric data of genuine individual *(2)*. Ratha and its research team are pointed out various vulnerable points in biometric systems *(3)*. The biometric system is vulnerable against eight different types of attacks such as fake biometric, a replay of stored biometric data, change result of feature extraction, modified biometric feature at feature extraction modules, modified matcher modules according to attacker requirement, modification attack at system database, modification of template over non-secure communication channel between system database and matcher, changing decision result by an attacker *(3)*. Also, when designing any multibiometric system, two problems with system such as design biometric image protection technique against modification attack at system database and design of the fusion technique for biometric image *(4)*.

One of the approaches to address the problem of modifying attack at system database is to embed a biometric image as invisible data to the other biometric image. This approach is known as watermarking technique. The various watermarking techniques are proposed for biometric data protection in the last decade. The watermarking techniques combined with biometric data *(4 –11)* are used to enhance security of biometric image. Paunwala and Patnaik *(4)* described watermarking techniques embed fusion fingerprint and iris data into low frequency AC DCT coefficients of the host image to improved security of biometric data in multibiometric system. Behera and Govindan *(5)* described a watermarking technique for security of face features when fingerprint feature is hidden in it. In this technique, phase congruency model applied on DCT coefficients of face image to get low frequency DCT coefficients of face image. These low frequency DCT coefficients of face image are modified according to the watermark fingerprint bit.

Bedi et al. *(6)* described PSO based watermarking technique for biometric data protection. The authors used PSO algorithm to find suitable DCT coefficients of host face image for watermark embedding. Edward and Sumanthi *(7)* described a multibiometric watermarking technique for Digital Rights Management (DRM) application. The authors used ridge let transform on a watermark fingerprint image before embedding into a host face image. Naik and Holambe *(8)* presented biometric watermarking technique where the watermark fingerprint image is embedded into low frequency DCT coefficients of host face image. Vatsa et al. *(9)* described spatial domain biometric watermarking techniques using correlation properties of PN sequence and the DCT. These techniques provide two levels of privacy using iris and face image where iris image used for cross verification of individuals.

Ratha et al. *(10)* described a robust blind watermarking technique for fingerprint image protection. In this technique, the randomly selected quantization integer values are used for watermark embedding. The LSB of wavelet coefficients of the host image replaces by watermark bit to generate a watermarked image. At the extraction, the random collected LSB of wavelet coefficients of host image is used to generate watermark bit. Jain et al. *(11)* presented a watermarking technique for protection of fingerprint images. In

this technique, facial features which are generated using eigenvalue are inserted into fingerprint minutiae points.

The problems of security of biometric image against modification attacks concern with the biometric system used in high security applications. So keeping security issue of biometric image in concerns, a biometric watermarking technique for security of biometric image against modification attacks on multibiometric system is proposed in this paper. This proposed technique is based on Compressive sensing theory and DCT. The compressive sensing theory is providing more computation security and fragility to proposed technique compared to traditional watermarking techniques available in the literature. In this technique, measurements of the watermark biometric data are generated by using compressive sensing acquisition framework at embedder side. These measurements are embedded into DCT coefficients of a host biometric data. At detector side, extraction measurements of the watermark biometric data from watermarked biometric data using the reverse procedure of watermark embedding. The watermark biometric data are reconstructed from extracted measurements using the compressive sensing recovery procedure. Section 2 describes the proposed watermarking technique. The results obtained are illustrated in Section 3. Finally, the conclusion of the paper is given.

2. PROPOSED WATERMARKING TECHNIQUE

In this section, the proposed watermarking technique based on compressive sensing *(12, 13)* and Discrete Cosine Transform *(14)* is described. This proposed watermarking technique is divided into two procedures such as watermark preparation & embedding and watermark extraction & reconstruction. The compressive sensing acquisition procedure is used for converting watermark biometric image into its measurements at embedder side. At detector side, the compressive sensing recovery procedure is used for reconstruction of watermark biometric image from its extracted measurements. These two steps provide more computational security in proposed technique compared to the traditional watermarking techniques.

2.1 Watermark Preparation & Embedding

The steps of watermark preparation and embedding are given below:

- Take a watermark biometric image with size of N×N and compute the size of the image.
- Generate Discrete Wavelet Transform (DWT) basis matrix with size of N×N using Daubechies (db1) wavelet filter.
- Then a watermark biometric image is converted into sparse coefficients by multiplying DWT basis matrix with a watermark biometric image.
  $$x = \Psi \times W \quad (1)$$
  Where x = Sparse Coefficients of a Watermark Biometric Image, Ψ = DWT Basis Matrix, W = Watermark Biometric Image.
- Generate measurement matrix A with size of N×N using the normal distribution. This matrix is same for embedder and detector side.
- Generate measurements of a watermark biometric image by multiplication of the measurement matrix with sparse coefficients of a watermark biometric image.
  $$y = A \times x \quad (2)$$
  Where y = Measurements of a Watermark Biometric Image, A = Measurement Matrix, x = Sparse Coefficients of a Watermark Biometric Image.

- Then multiply sampling factor with measurements of a watermark biometric image which is denoted as W_S. This is same for embedder and detector side.

$$W_S = \beta \times y \quad (3)$$

Where W_S = Sparse Watermark Information, y = Measurements ofWatermark biometric Image, β = Sampling Factor.
- Take host biometric image with size of N×N and compute the size of the image.
- Apply 2D Discrete Cosine Transform (DCT) on the host biometric image, to converting into its DCT coefficients. Then choosing all DCT coefficients for sparse watermark information hiding. The reason behind choosing all DCT coefficients is that the advantages of watermark information in hiding low frequency DCT coefficients and high frequency DCT coefficients are explored in this proposed watermarking technique.
- Then sparse information of the watermark biometric image is embedded into DCT coefficients of a host biometric image using multiplicative watermarking equation *(15, 16)*.

$$I_{W_DCT_Coefficients} = I_{DCT_Coefficients} * (1 + k \times W_S) \quad (4)$$

Where $IW_{DCT_Coefficients}$ = Modified DCT Coefficients of a Host Biometric Image, $I_{DCT_Coefficients}$ = Original DCT Coefficients of a Host Biometric Image, W_S = Sparse Watermark Information, k = Gain Factor.
- Then apply inverse 2D Discrete Cosine Transform (DCT) on modified DCT coefficients of a host biometric image to get a watermarked biometric image.

2.2 Watermark Extraction & Reconstruction

The steps of watermark extraction and reconstruction are given below:
- Take a watermarked biometric image and applied 2D Discrete Cosine Transform (DCT) applied to the watermarked biometric image convert into its DCT coefficients.
- Take an original host biometric image and applied 2D Discrete Cosine Transform on it to convert into its DCT coefficients.
- Extracted sparse information of a watermark biometric image using the reverse procedure of watermark embedding.

$$W_E = \frac{\left(\frac{I_{W_DCT_Coefficients}}{I_{DCT_Coefficients}} - 1\right)}{k} \quad (5)$$

Where $IW_{DCT_Coefficients}$ = DCT Coefficients of a Watermarked Biometric Image, $I_{DCT_Coefficients}$ = DCT Coefficients of a Host Biometric Image, $W_{Extracted}$ = Extracted Sparse Watermark Information, k = Gain Factor.
- Then extracted measurements of the watermark biometric image are divided sampling factor which is used at embedder side to get actual measurements of the watermark biometric image. After getting measurements of the watermark biometric image, then reconstructed actual watermark biometric image from its measurements using CS theory recovery procedure.

$$Recover_y = \frac{W_E}{\beta} \quad (6)$$

Where $W_{Extracted}$ = Extracted Sparse Watermark Information, Recover_y = Extracted Measurements of Watermark Biometric Image, β = Sampling Factor.
- Then apply the orthogonal matching pursuit algorithm *(17)* on extracted sparse measurements of the watermark biometric image along with measurement matrix. The output of OMP algorithm is sparse coefficients of a watermark biometric image.

$x_{E?} = O \quad (R \quad _y, A, M)$ (7)

Where $x_{Extracted}$ = Extracted Sparse Coefficients of a Watermark Biometric Image, Recover_y = Extracted Measurements of Watermark Biometric Image, OMP = Orthogonal Matching Pursuit, A = Measurement Matrix, M = Row Size of a Watermark Biometric Image.

- Finally, the inverse DWT basis matrix is multiplied with extracted sparse coefficients of a watermark biometric image to get reconstructed watermark biometric image at detector side.

3. EXPERIMENTAL RESULTS

For testing of proposed watermarking technique, 8 bit grayscale face images from Indian face database *(18)* as the host biometric image and 8 bit gray scale fingerprint image from FVC 2004 DB4 set B *(19)* as the watermark biometric image is taken. The size of host image and watermark image is 128×128 pixels selected. Figure 1 shows an original host face image and watermark fingerprint image.



(a) Host Face Image



(b) Watermark Fingerprint Image

Fig. 1. Test Biometric Images

For a generation and embedding of watermark fingerprint image below procedure is adopted. First generate Discrete Wavelet Transform (DWT) basis matrix with size of 128×128 using Daubechies (db1) wavelet. Then multiply DWT basis matrix with the watermark fingerprint image to get sparse coefficients of a watermark fingerprint image with size of 128×128. Then generate a measurement matrix with size of 128×128 using a Gaussian distribution with mean = 0 and variance =1. The measurements of watermark fingerprint image with size of 128×128 are generated using $y_{128\times128} = A_{128\times128} \times x_{128\times128}$. These measurements of the watermark biometric image which are used as watermark information and it is denoted as W_S.

Watermarked face image is generated using equation 4 at embedder side where DCT coefficients of host face image are modified according to gain factor and sparse information of the watermark fingerprint image. At detector side, extract sparse information of a watermark fingerprint image using equation 5 and then reconstructed watermark biometric image form extracted sparse measurements using below procedure. The gain factor k value set is 0.2 and sampling factor β value set is 0.0001.

For reconstruction of watermark fingerprint image, applied OMP algorithm *(17)* on extracted measurements of a watermark fingerprint image. The input of OMP algorithm is measurements, measurements and level of sparsity which is 128 in this case. The output of OMP algorithm is sparse coefficients with a size of 128×128 for watermark fingerprint image. Then an inverse DWT basis matrix is multiplied with extracted sparse coefficients of a watermark fingerprint image to get reconstructed watermark fingerprint image at detector side. Figure 2 shows watermarked face image, reconstructed watermark fingerprint image, original sparse & extracted measurements of the watermark fingerprint image.



(a) Watermarked Face Image



(b) Reconstructed Watermark Fingerprint Image



(c) Measurements of Watermark Fingerprint Image



(d) Extracted Measurements of Watermark Fingerprint Image

Fig. 2. Experimental Results of Proposed Watermarking Technique

The quality measures such as peak signal to noise ratio (PSNR), normal cross correlation (NCC) *(18)* is used for quality check between original face and watermarked face image at embedder side while the structural similarity index measure (SSIM) *(19)* is used for quality check between original fingerprint and reconstructed fingerprint image at detector side. This proposed watermarking technique is tested by applying various watermarking attacks such as JPEG compression, addition of noise, applied different image filter, histogram equalization and cropping on it. The quality measures of proposed watermarking technique are summarized in Table 1.

Table 1. NCC, PSNR and SSIM Values Proposed Watermarking Technique at Gain Factor = 0.2

Attack	**NCC**	**PSNR (dB)**	**SSIM**	**Decision about Authentication**
No Attack	1.00	43.62	0.985	Authentic
JPEG Compression (Q = 70)	0.99	36.54	0.676	Unauthentic
Gaussian Noise (μ =0, σ=0.0001)	1.00	36.27	0.641	Unauthentic
Salt & Pepper Noise (Noise Density = 0.0005)	0.99	35.37	0.670	Unauthentic
Speckle Noise (Variance = 0.0004)	1.00	29.09	0.676	Unauthentic
Median Filter (Size = 3 × 3)	0.99	35.60	0.671	Unauthentic
Mean Filter (Size = 3 × 3)	0.98	25.49	0.664	Unauthentic
Gaussian Low Pass Filter (Size = 3 × 3)	0.98	31.20	0.665	Unauthentic
Histogram Equalization	0.97	19.32	0.675	Unauthentic
Cropping	0.97	15.87	0.663	Unauthentic

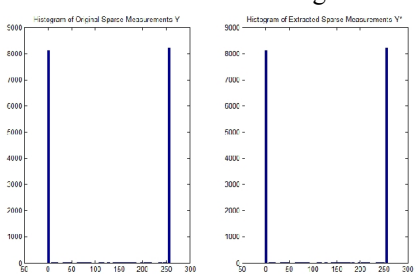
In the watermark embedding process, the gain factor is multiplied with sparse information of watermark biometric data, and then embedded in the frequency coefficients of host biometric data. Therefore, the gain factor has an effect on the watermarked biometric image and the watermark biometric image. Table 2 shows the effect of the gain factor changes on both the PSNR value of the watermarked biometric image and SSIM value of the watermark biometric image.

Table 2. The Gain Factor Effect on Proposed Watermarking Technique

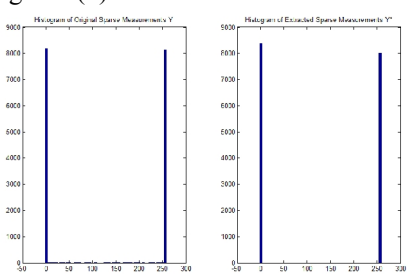
Gain Factor	PSNR (dB)	SSIM
0.1	50.35	0.985
0.2	43.62	0.985
0.3	38.25	0.985
0.4	34.78	0.985
0.5	36.35	0.985
0.6	28.10	0.985
0.7	28.82	0.985
0.8	26.42	0.985
0.9	24.83	0.985
1.0	22.61	0.985

For individual authentication, SSIM value between the original watermark biometric data and reconstructed watermark biometric data must be greater than 0.90. SSIM values in Table 1 are indicated that when watermarking attacks is applied on watermarked face image, then the watermark fingerprint image can't reconstruct successfully at detector side. This situation indicated that the proposed watermarking technique is fragile against various watermarking attacks.

The reason behind achieving fragility for this proposed watermarking technique is that the measurements of the watermark fingerprint image are destroyed when watermarking attacks is applied on watermarked face image. Figure 3 shows the histogram of measurements and extracted measurements of watermark image without watermarking attacks and under watermarking attacks. Figure 3(a) indicated the histogram of measurements and extracted measurements of the watermark fingerprint image is same when no watermarking attacks is applied on watermarked face image, but it is different when watermarking attacks in applied on watermarked face image which is indicated in Figure 3(b).



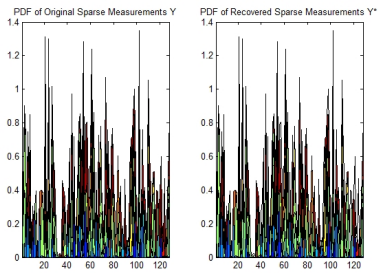
(a) A Histogram of Measurements of Watermark Fingerprint Image Without Attacks



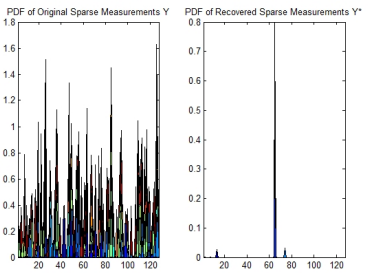
(b) A Histogram of Measurements of Watermark Fingerprint Image With Attacks

Fig. 3. Histogram of Measurements in Proposed Watermarking Technique

Also normalized probability density function (PDF) of measurements of the watermark fingerprint image is calculated for checking fragility of this proposed watermarking technique. Figure 4 shows normalized PDF of measurements and extracted measurements of watermark image without watermarking attacks and under watermarking attacks. Figure 4 (a) indicated the normalized PDF of measurements and extracted measurements of the watermark fingerprint image is same when no watermarking attacks is applied on watermarked face image, but it is different when watermarking attacks in applied on watermarked face image which is indicated in Figure 4 (b).



(a) A Normalized PDF of Measurements of Watermark Fingerprint Image Without Attacks



(b) A Normalized PDF of Measurements of Watermark Fingerprint Image With Attacks

Fig. 4. Normalized PDF of Measurements in Proposed Watermarking Technique

The proposed watermarking technique is compared with existing watermarking techniques available in literature with various features and parameters are summarized in Table 3. The existing watermarking techniques available in the literature are robust against attack while this proposed watermarking technique is fragile against attacks.

Table 3. Comparison of Proposed Watermarking Technique with Existing Watermarking Techniques available in the Literature

Features & Parameters	Type of Watermarking Technique	Computational Security Achieved	PSNR (dB)	SSIM
Paunwala Technique (2014) et al. *(4)*	Robust	No Such Scope	38.23	Not mention
Behera Technique (2013) et al. *(5)*	Robust	No Such Scope	45.64	0.981
Bedi Technique (2012) et al. *(6)*	Robust	Particle Swarm Optimization Algorithm	40.89	0.954
Edward Technique (2011) et al. *(7)*	Robust	No Such Scope	45.00	0.942
Naik Technique (2010) et al. *(8)*	Robust	No Such Scope	36.66	0.991
Proposed Watermarking Technique	Fragile	CS Theory Procedure	43.62	0.985

This proposed watermarking technique is achieved computational security using the compressive sensing theory procedure while in existing watermarking techniques in the literature, computational security achieved by using gain factor, PN sequences and particle swarm optimization (PSO) algorithm. This

proposed watermarking technique provides additional computational security using compressive sensing theory with more quality measure values compared to some existing DCT based watermarking techniques available in the literature.

4. CONCLUSION

A non-blind multibiometric watermarking technique based on the Discrete Cosine Transform and Sparsity property of Discrete Wavelet Transform is proposed in this paper. This technique is fragile against various watermarking attacks. This technique provides more payload capacity compared to existing watermarking techniques available in the literature. This technique provides security to biometric image against modification attack because it is difficult to imposter to generate two biometric characteristics where one biometric characteristic is encoded by a compressive sensing theory which is embedded into another biometric characteristic.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

(Proceedings available for download at rku.ac.in/icre)

RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

Simulations on Standalone Wind Solar Hybrid Generation System

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ABSTRACT

The present work focuses on the hybridization of PV and Wind power for standalone application. The said hybrid system in which problem is variation in solar intensity and wind speed. This problem can be sorted of using complementation between one source to another. Considering this aspect of proposed hybrid system is simulated with 28kW of mechanical power from wind and 3.6kW DC power from solar PV system. This operation of proposed hybrid system is tested for various permutations and combinations of type and capacity of loads. The system is then optimally operated by application of MPPT algorithm. Both the systems are operated at constant voltage. The hybrid system is simulated in PSIM ® 9.3.4 software and results are obtained at constant wind speed and different radiation levels.

SUMMARY

This paper focuses on wind solar hybrid generation system for standalone application supplying power to DC loads applying MPPT algorithm.

Keywords: Hybrid Generation System, Maximum power point tracking, Solar, Standalone, Wind.

INTRODUCTION

Renewable energy has its own importance nowadays because of the fossil fuel exhaustion and environmental effects. Compared with other renewable energy, such as solar energy and wind power is more suitable for some applications with relatively low cost compared to other renewable energy sources.

The curves of IV and PV are usually plotted at standard test conditions (STC) that is 1000W/m^2 radiation and 25°C temperature. For wind energy conversion system, the wind turbines are broadly classified as horizontal axis wind turbine and vertical axis wind turbine. Rated power can be drawn at rated wind speed and 0° pitch angle and its overall theoretical efficiency is 0.59.*(14)*

Present study focuses on the wind solar hybrid generation for standalone system. The load is fed with combined wind and solar power generation. In this study we are assuming that all the considered loads are remain ON during simulation time and resistance has been represented by equivalent DC resistance. The parameters of load wattage rating and system will mention in upcoming sections. During entire study batteries have not been used to make the system cost effective, however some useful outcomes using batteries and dump loading system used nowadays have discussed in literature.

REVIEW OF LITERATURE

Present study and simulation focuses on the wind solar hybrid with MPPT based charge controller, in this section some latest MPPT techniques have been discussed briefly and among that two of the P&O (Perturb and Observe) techniques have been adopted for wind and solar energy conversions system.

The main concept of maximum power point tracking (MPPT) algorithms to achieve fast as well as accurate tracking performance and minimize the oscillations due to varying weather conditions, in case of solar it could insolation with module temperatre and in case of wind it could be a wind speed. There are many algorithms to extract the maximum energy from the renewable energy source is available, among that present work focuses on the combined power generation with wind energy conversions system, solar PV and battery in PSIM ® 9.3.4 environment. There are different MPPT techniques have been reported in literature for wind and solar energy systems.

A comparative study of different power point tracking methods is reported in *(1)*. Authors of the same compared almost 24 different MPPT techniques that are used for solar PV systems, among that

incremental conductance (INC) and perturb and observe (P&O) widely used in the area of PV power generation. The efficiency of such algorithm depends upon how fast it will track maximum power under sudden change in irradiance. Satishkumar K et. al.*(2)* have developed variable perturbation size adaptive P&O MPPT algorithm for sudden change in irradiance. They have proposed two stage variable size algorithm. They have also proposed a novel algorithm in *(3)* by adding a new loop in existing algorithm. They have explained why adaptive P&O is better and where other methods particularly INC fails.

Dezso Sera et. al in *(4)* discussed selection process of P&O and INC for different applications. They have analyze both the method mathematically and practically after thoroughly reviewed the same. Their mathematical analysis reveals that there is no difference between two. They have also explained the boundary conditions between two algorithms.

L. Piegari and R.Rizzo in *(5)* proposed adaptive P&O algorithm for PV MPPT that works with faster dynamics and improved stability compared to traditional P&O. Proposed P&O method has faster dynamics and improved stability compared to traditional P&O.

Zakariya M. Dalala et. al. in *(6)* proposed a new scheme for small scale wind energy conversion system, that is implemented in this paper. They have used P&O technique for change in wind speed within few seconds and variable change in wind speed (means change must be observed after some finite time). They have also proved the same with hardware results in that they have used Wind turbine as an Induction Motor. In this paper same technique is used for hybrid generation for extracting maximum power from wind generator. They have taken DC current as a perturbing variable and through DC link voltage slope they have detected the change in wind speed. In *(7)* they have implemented stall control method for small scale wind energy conversion system.

Yuanye Xia et. al. in *(8)* proposed a new MPPT technique for PMSG based wind energy conversion system using two combined methods. One is adaptive P&O and other is optimum relationship based (ORB) control. They have also surveyed and citied other MPPT methods for small scale wind energy conversion system. In this paper they have proposed a new method combining P&O and ORB, adaptive P&O to evaluate wind condition and ORB for training mode. In this method they do not require an anemometer reading of wind turbine or in other words pre-knowledge of the system. In *(9)* they have developed wind power coefficient C_P analysis& implementation of the same in MPPT technique. To implement the same they have developed the linear relation between DC voltage and DC current in terms of V_{dc}^2 and I_{dc}.

Nishad Mendis et. al. in *(10)* proposed PMSG and DFIG based hybrid wind energy conversion system with battery. This can be explained & done for remote area power supply (RAPS) application with improved voltage and frequency control together. They followed a particular sequence for control co-ordination of wind speed based hybrid RAPS system.

Fernando and Paul in *(11)* proposed supervisory control for standalone generation system using wind and photovoltaic energy. The main consideration in this paper to manage the flow of power from wind, solar PV and battery plus state of charge (SOC) of battery bank ensure constant power supply.

M. Hashem Nehrir et. al. in *(12)* proposed an approach to evaluate the general performance of standalone wind and PV generation system. They combined 10kW of wind power with 3kW of PV and with backup generator as well as battery. They have also used electric water heater as a dump load.

Toshiro Hirose et. al. in *(13)* developed standalone hybrid wind solar power generation system applying a dump load power control without dump load. They system is useful for isolated island and rural location. They have proposed a unique standalone hybrid wind solar power generation system which is characterized by PLL control and dump power control.

Photovoltaic systems normally use a maximum power point tracking (MPPT) technique to continuously deliver the highest possible power to the load when variations in the isolation and temperature occur as well as there is change in load power. The purpose of MPPT is to adjust the power from PV at its maximum value so generated power is utilized. In order to continuously gather the maximum power from the PV array, they have to operate at their MPPT despite of the not uniform change in environmental conditions. This maximum point can achieved with change in load.

SYSTEM DESCRIPTION AND BLOCK DIAGRAM

Hybrid system which can be considered in this study is combination of PV and Wind energy conversion system used in residential applications. This can also be known as DC microgrid up to some aspects. This concept of hybridization is used in that manner is that solar can handle mandatory loads up to 2.5kW at lower radiation levels (when there in not suitable amount of wind is available). The readings during simulation study in PSIM ® 9.3.4 software at rated wind speed 11.5m/s for wind energy conversion system and for solar PV at 1000W/m^2 to 800W/m^2. Fig. 1 represents entire system block diagram. Wind turbine of 28kW is coupled with 30kW of permanent magnet synchronous generator type (PMSG). The difficulty of selecting wind turbine rating is has no globally accepted standard. In this study for 28kW wind turbine at 15m/s speed or say rated wind speed of 11m/s is connected with 30kW of generator than SRC (specific rated capacity)*(14)* is defined as per equation (1),

$$\text{SRC}=\text{Generator Electrical Capacity}/\text{Rotor Swept area} \quad (1)$$

According to Betz's disc theory, the maximum theoretical power coefficient is limited to 59% with one rotor, so from data used in wind turbine of 28kW at least generate power at the value of power co-efficient of 0.25. Now at this value of C_P mechanical power is generated around 15kW and it coupled with PMSG of 30kW, considering all the losses PMSG will generate 14kW DC power when above load is connected. In this entire study we have taken $V_{pk}/k_{rpm} = 2400$. The above value can change with change in climatic conditions.

The said is hybridized with solar PV energy conversion system, here after surveying some review paper of wind solar hybridization we have decided at least solar PV can handle mandatory loads (say fan, lights, laptops and other light loads) when wind speed is not up to the mark or prior to cut in speed as per fig. 1. In this configuration this proposed hybrid system 28kW (gross mechanical power) is hybridized with 30 panels in series each of 60W make one string and other same string of 30 panels is connected in parallel. Generated DC power by PV panels is regulated through MPPT controller using boost converter and MPPT technique (Adaptive P&O *(2)*).

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This system is connected with 28kW of wind energy conversion system with MPPT controller (7) of the wind and both are operated at same DC voltage level.

SIMULATION RESULTS AND ANALYSIS FOR WIND SOLAR HYBRID STANDALONE SYSTEM

STANDALONE WIND ENERGY CONVERSION SYSTEM AND ITS RESULTS

- **A test on Wind turbine at constant mechanical load.**

Fig. 2 shows simulation of wind turbine connected with constant mechanical load of 198Nm via speed and torque sensors. Readings have taken from cut in speed to rated wind speed at 0° pitch angle and mechanical power developed using generalized equation,

$$P = \frac{2\pi NT}{60} \quad (2)$$

Where, in fig. 2, K represents a constant of $2\pi/60$. To obtain these power param sweep block is used in PSIM and reading have taken from cut in to rated wind speed at 0° pitch angle. Fig. 3 shows the variation in power coefficient at constant mechanical load at different wind speed. Fig. 4 shows output power at constant rated wind speed. C_P (Power coefficient) is obtained for different wind speeds at fixed mechanical load. In characteristics of wind turbine generally C_P is considered as one of the most important parameter. Actually C_P is increasing with increase in wind speed from cut in speed to rated speed. After that there is a decrement in C_P maintaining output power constant.

- A test on Standalone Wind energy conversion system connected with DC loads.

  In this segment wind turbine is connected with 30kW of permanent magnet synchronous generator. The output of generator is connected with uncontrolled rectifier and output of rectifier is connected with boost converter with MPPT extraction technique proposed by algorithm by Zakariya M. Dalala et al. in *(7)*. This later on connected with wattmeter for measurement the total consumption of DC power. This entire test is performed at constant wind speed of 11.5m/s and 0° pitch angle. Certain more assumption has been made as under in table 2. The details of different loads have given in table 1. Here the entire test is performed with equivalent resistance of 27Ω and 54Ω connected in parallel. Considering the resultant of all the components DC resistances at 600V and its power consumptions at that voltage have been shown in table 1. The MPPT algorithm is coded in DLL block of PSIM the code has been done in Microsoft visual studio C++ and from that reference current is generated. Later on after tuning on the PI block it can generate gate pulse and so MPPT algorithm. Results are shown in fig. 5 in fig. 6 output power consumed by DC load is shown.

- **A test on single module PV system.**

Fig. 7 gives the basic idea of solar module used as a power generation and track it's MPPT; here single panel of 240W is simulated in PSIM ® 9.3.4.

In fig. 7 single module of 250W is tested in PSIM software. Using utility block all the parameters of modules are inserted in single PV module and then it has been tested with boost converter with constant load resistance of 15Ω using MPPT technique coded in DLL library of PSIM, the algorithm is proposed in *(2)*.

Results of fig.8 are compared with fig.9 and fig.10 where perturbation is applied in case of solar radiation and following results are obtained. Applying perturbation on solar PV module measuring MPPT efficiency by superimposing two curves of available maximum power (P_{max}) and tracked power (Power) are at different radiations. Similarly results may be obtained with changing in loads as well as module temperature.

Fig. 10 shows actual power consumed by wattmeter at different radiation and fixed DC load. As seen from the graph the power consumed is decreased with change in radiation. Similarly in fig. 9 maximum power and tracked power are compared with different radiations. Among that five reading two of them are shown in fig. 9. For example Pmax_05.01 is the total power available from the panel and Power_05.01 is the tracked power with MPPT algorithm. Here also changing in the radiation can be done using param sweep block in PSIM software. Similar the things can be done in hybridization concept.

- **A test on Hybrid system with constant load of 20Ω (equivalent to all the loads).**

In this section hybrid system is designed for constant load of 20Ω at 350V DC and equivalent load of 20Ω represents the constant load of 15kW considering all the loads in table 1. Now this system is simulating at constant wind speeds of 11.5m/s constant wind speed and 0° pitch angle for wind energy conversion system. Moreover solar PV is operating with 1000W/m^2 and 800W/m^2 radiations. 60 modules of 60W each is connected in series combination of 30 modules and that of 30 modules in parallel to that so it can feed the sufficient amount of current.

Fig. 11(1) shows the output of solar energy conversion system and its share with combined output. Here little worst results are obtained in wind energy conversion system as compared to wind energy conversion system. In solar PV 1000W/m^2 and 800W/m^2 radiation levels have taken in consideration. Fig. 11(2) shows combined power and share of wind power at rated wind speed has been shown. Definitely share of

wind turbine is more, but at low wind regime or there is no sufficient amount of wind solar will take care of all mandatory loads. In fig. (12) 1 & 2 the constant voltage across load and the current have shown. The load voltage is maintained constant with change in radiation of PV modules. In fig. 13 (1) boosted voltage of PV module is shown and compared with total voltage from PV and in the same case boosted voltage from the wind fig. 13(2) is same as the boosted voltage from solar PV, so both the system are working with same voltage level make hybridization worth. Moreover wind energy system has taken care of 80% load and 20% load can be shared by solar PV systems.

CONCLUSION

The concept of hybridization is successfully demonstrated meeting variable load demand. The solar PV generating systems and wind generation systems showed their suitability for complementing each other in power generation with respect to changes in daily & seasonal solar radiation and wind speed. This becomes a viable way to produce electrical energy where grid connections are not available especially in rural areas. The performance of hybrid system increases the system reliability and continuity of supply. So this system can find its place as eco-friendly, low maintenance alternative power solution..

FIGURES

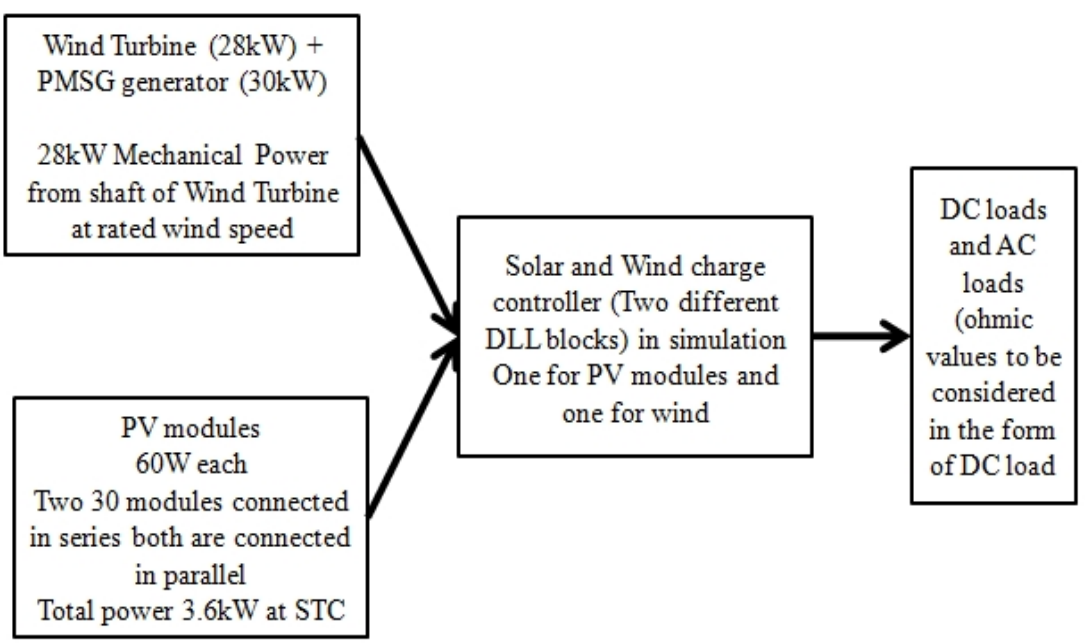


Fig.1. System Block diagram

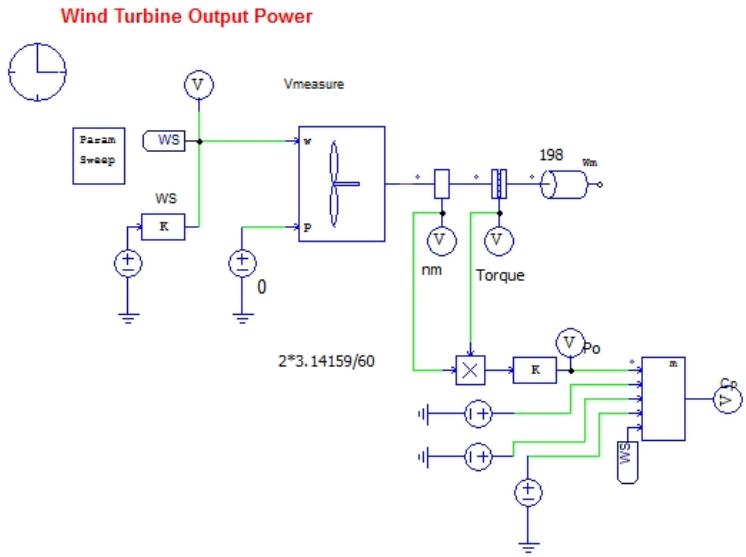


Fig.2. A test on Wind Turbine at constant speed mechanical load

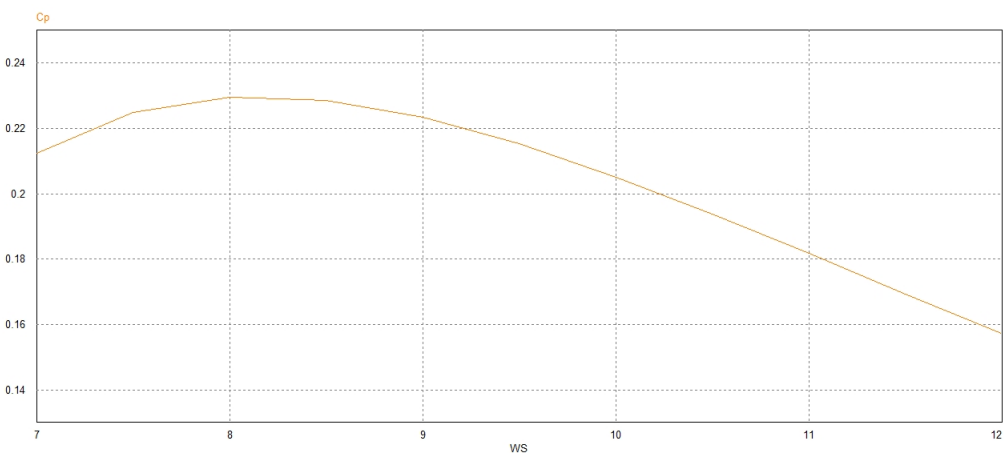


Fig.3. Output graph of C_P v/s Wind speed at constant mechanical load of 198Nm

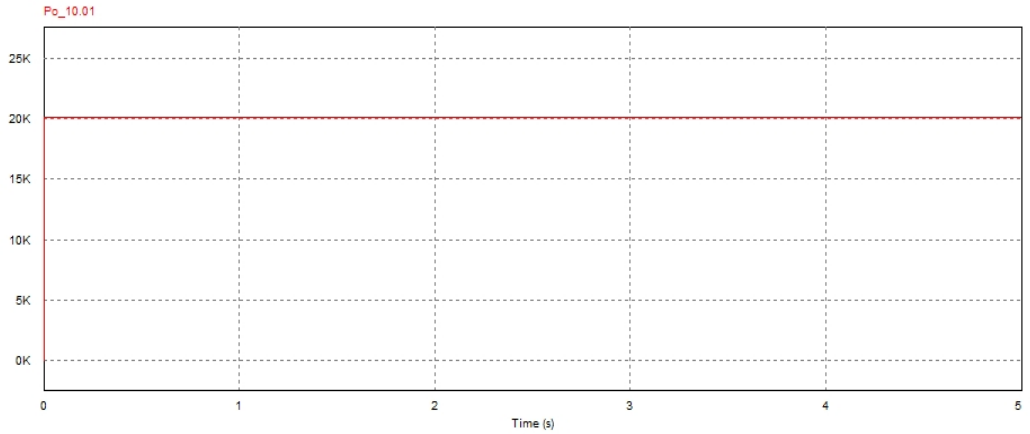


Fig.4. Output graph Power v/s Wind speed (11.5m/s rated) at constant mechanical load of 198Nm

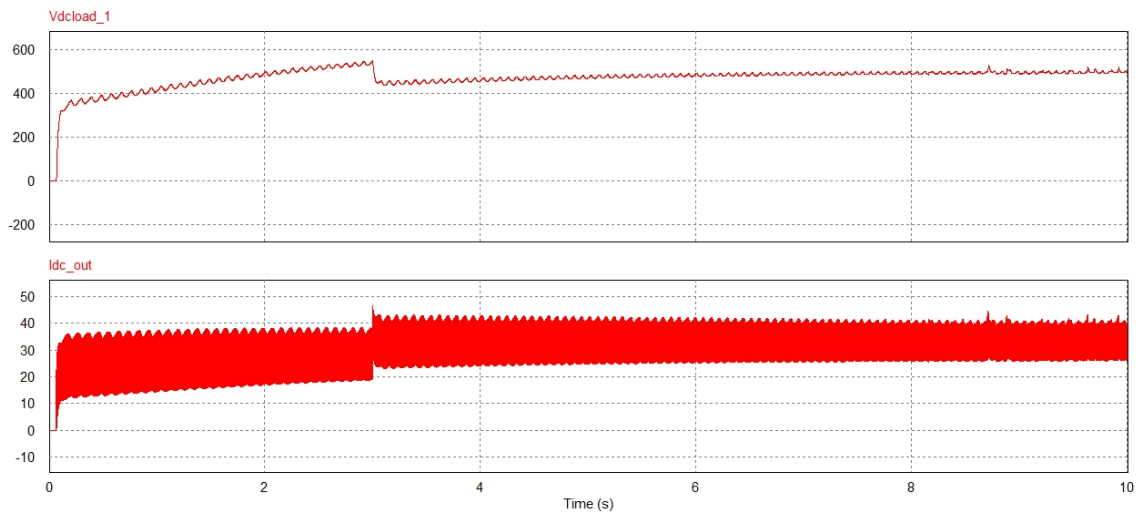


Fig.5. Output voltage and current at rated wind speed

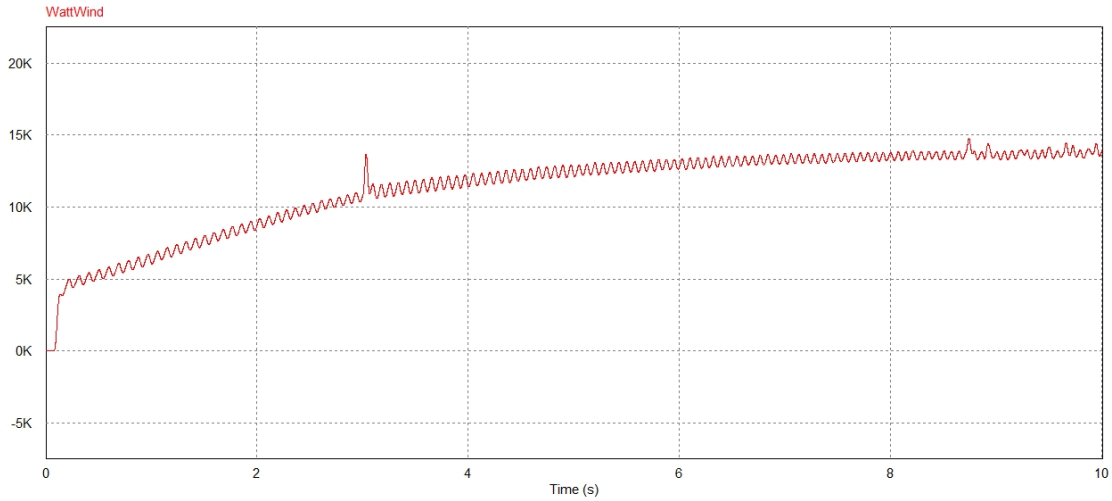


Fig.6. Output power consumed by load

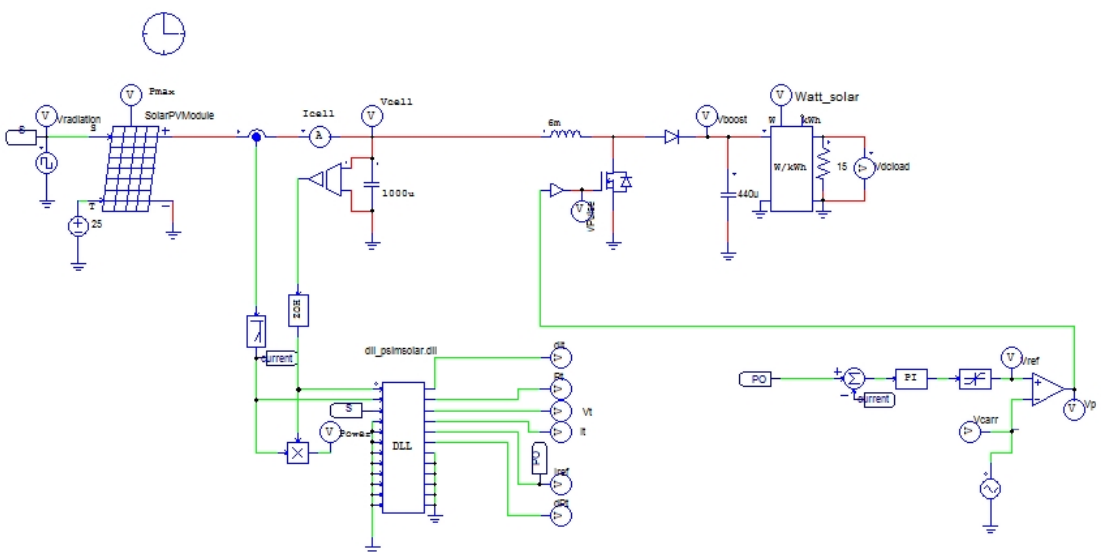


Fig.7 PSIM based simulation circuit with P&O MPPT algorithm - Single PV module – PSIM based current perturbation model.

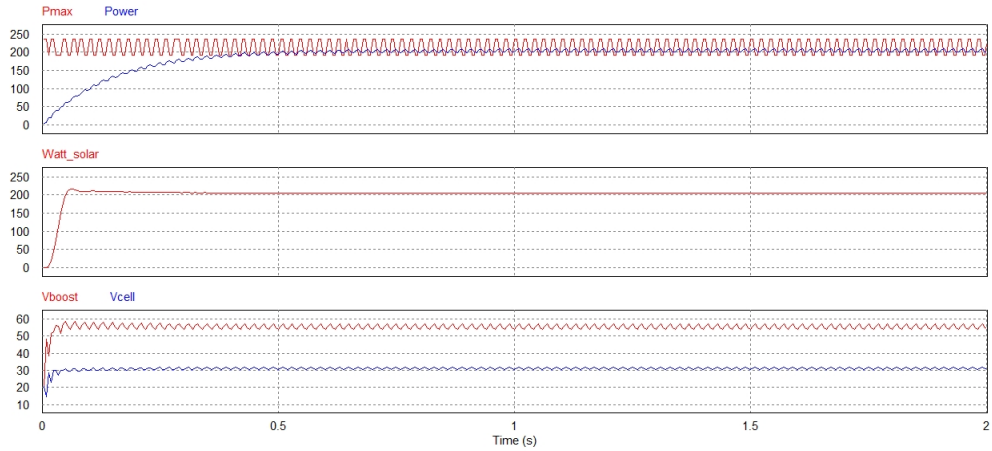


Fig.8 Result of power consumed, tracked power and boost converter at constant radiation. (MPPT efficiency 86.91%)

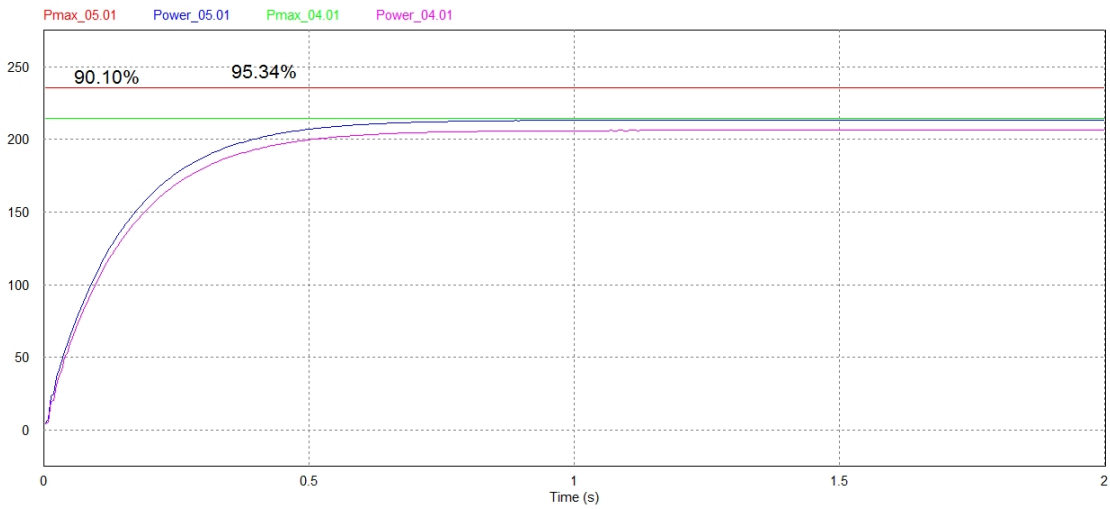


Fig.9 Maximum Power and tracked power at different radiation with its efficiency.

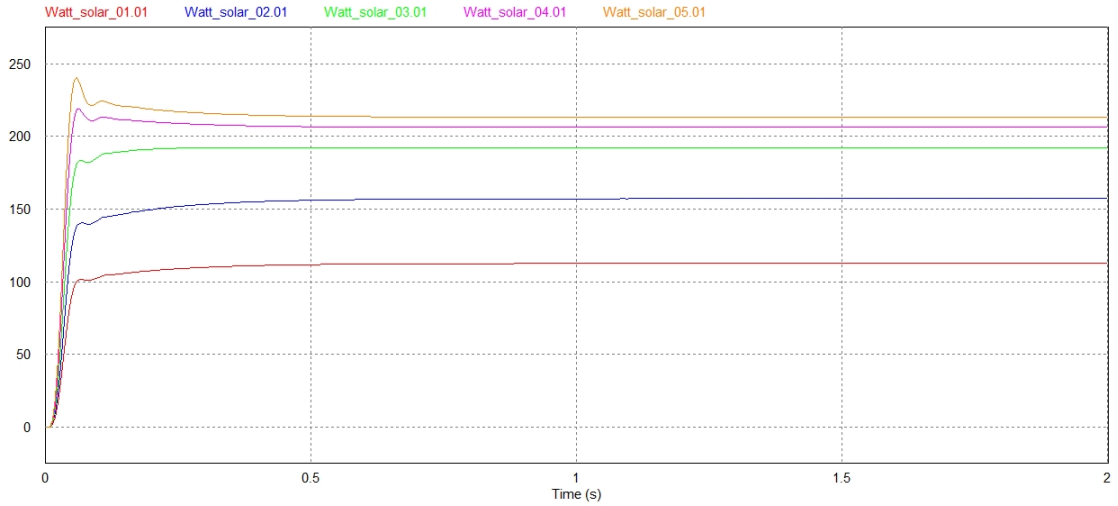


Fig.10 Actual power consumed at different radiations by wattmeter.

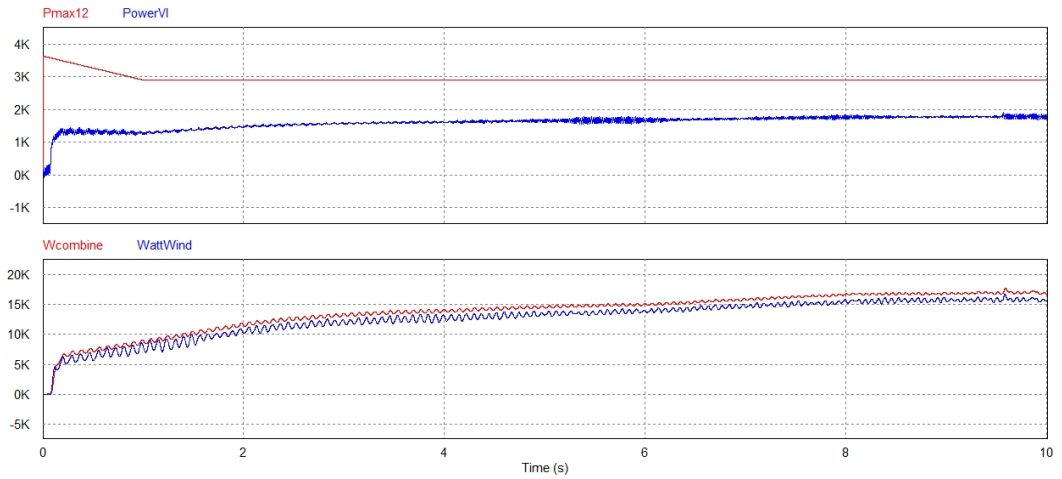


Fig.11 (1) Total available power (Pmax12) and tracked power (PowerVI) (2) Wcombine = Combined wind and solar power & Wattwind = Power generated/shared by WECS

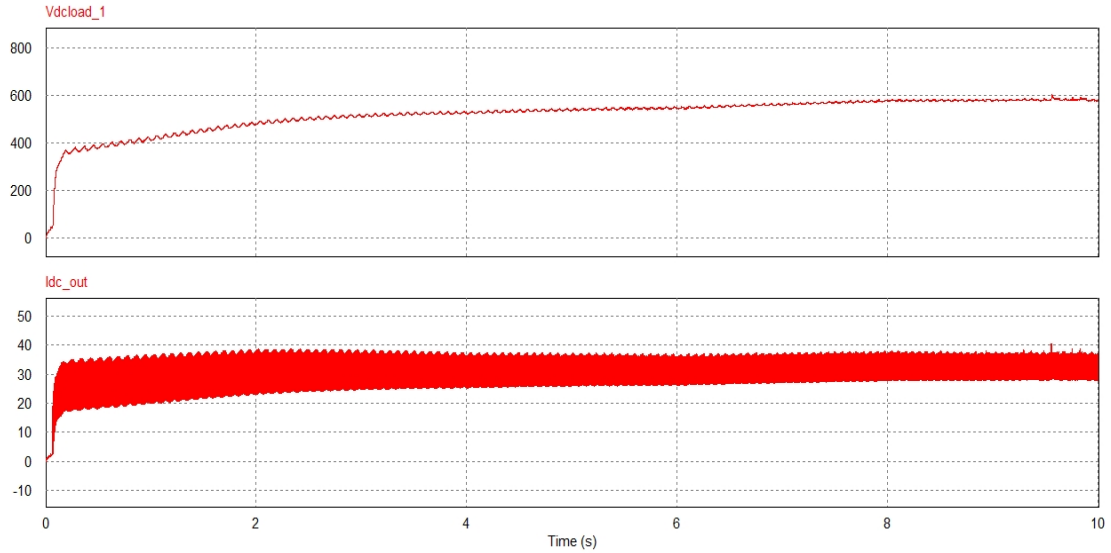


Fig.12 (1) Vdc load1=Load voltage of combined system (2) Idcout = Total current drawn from the system

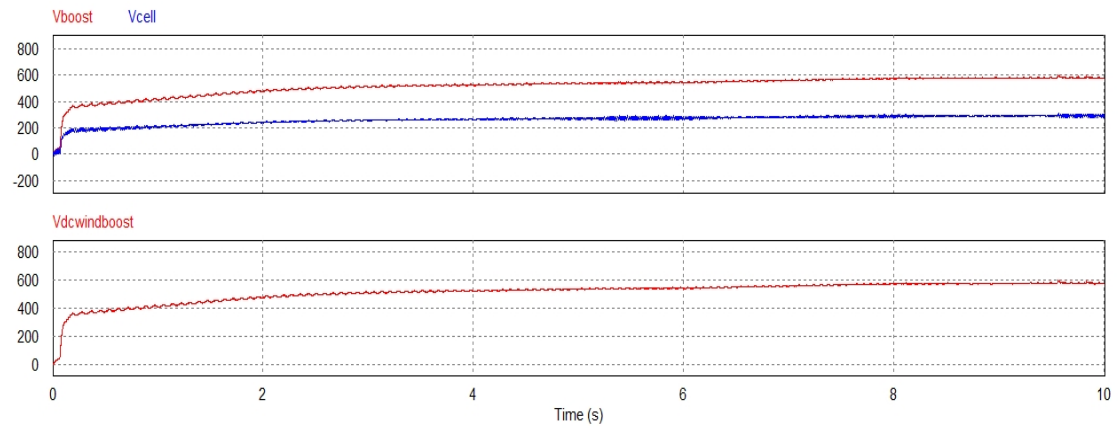


Fig.13 (1) Vboost = Boosted voltage from Solar PV and Vcell is the actual voltage from PV modules at a particular radiation. (2) Vdcwindboost = Boosted voltage from WECS

TABLES

Table.1 Description of load in Watt and value of resistance used *(15)*

Sr. No.	Load	Total No. of Units	Power Consumption in Watt at 600V DC
1	Refrigerator	1	300W
2	Dishwasher	1	1450W
3	Large Burner	1	2100W
4	Ceiling Fans	4	100*4=400W
5	Cloth Iron	1	1000W

6	Tube lights	4	60*4=240W
7	Window Air Conditioner	2	1200*2=2400W
8	3 Star Air Conditioner	2	3000*2=6000W
9	Vacuum Cleaner	1	1000W
10	Television	2	150*2=300W
11	Water Pump	1	450W
12	Toaster	1	1400W
13	Laptop/Computer	4	500W

Table. 2 Parameters taken while simulating the Wind energy conversion system

Sr. No.	Name of the parameters	Value
1	Constants K_0, K_1, K_2	 1,0.1,0.09
2	Simulation time	10 seconds
3	PMSG's Peak line-to-line back emf constant, in	2400
4	Wind speed	15m/s
5	Value of DC resistance	27Ω (1 to 10 seconds) 54Ω (3 to 10 seconds)

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Study and Performance Analysis of Different Modulation for Space Time Block Coding and VBLAST MIMO for Wireless Communication

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ABSTRACT

Across space and time domain, structured codes need to apply for combining diversity gain in wireless communication. This Space-Frequency Block Code is useful to achieve diversity gain specially for currently demanded wireless system like MIMO-OFDM. Alamouti code is most frequently used Space Time Block Code for narrowband system. Also this paper includes study and performance analysis of another MIMO technique called VBlast which reduces interference significantly in received signal. To increase data rate, different modulation techniques are used for Alamouti Space Time Block Code as well as VBlast MIMO and Bit Error Rate performance with Signal to Noise Ratio is analyzed.

SUMMARY

BER performance of STBC as well as VBlast MIMO which are one of the important MIMO coding techniques have been analyzed over different modulation techniques.

Keywords: MIMO, MMSE, STBC, VBlast, ZF

INTRODUCTION

The main advantage of multiple input and multiple output(MIMO) is higher data rate without additional bandwidth or power. MIMO system mainly consists three parts-transmitter(TX), receiver(RX) and channel(H).Figure 1 shows block diagram of MIMO system.

The channel is demonstrated by N_r x N_t matrix (N_r is number of receivers and N_t is number of transmitters):

$$H = \begin{pmatrix} h_{1,1} h_{1,2} \ldots\ldots h_{1,N_t} \\ h_{2,1} h_{2,2} \ldots\ldots h_{2,N_t} \\ \vdots \quad \vdots \quad \vdots \\ h_{N_r,1} h_{N_r,2} \ldots\ldots h_{N_r,N_t} \end{pmatrix} \quad (1)$$

where each term $h_{a,b}$ shows phase shift and attenuation between b^{th} transmitter and a^{th} receiver. With assumption of spatially independent Rayleigh channel and independent and identically distributed (i.i.d.) AWGN noise symbols with mean zero and variance N_0 , received signal is represented by

$$y = Hx + n \quad (2)$$

Here y is received signal, x is transmitted signal, n is noise and H is channel matrix(6-10). Using multiple antennas, the reliability of data transmission in wireless communication can be achieved using Space Time Code where transmitting multiple, redundant copies of data to achieve efficient decoding at receiver with the fact that few of detected copies of data will be better. So it results in higher probability to decode received data correctly. Using space time coding, maximum information can be extracted as possible in optimum way. The Space Time Code may be classified in two types ,one is Space Time Block Code(STBC) and second one is Space Time Trellis Code(STTC). To achieve diversity gain in STTC, trellis code is used over multiple antennas. It sends multiple, extrinsic copied of code distributed over time and space. On the other hand, STBC transmits block of data at a time to achieve diversity gain with much more easy in implementation than STTC. That is why STBC is used here for simulation to check performance of different modulation techniques. Figure 2 shows block diagram of STBC. Orthogonal Space Time Block Code is attractive one of Space Time Block Code because of easily detected at receiver with simple linear operations. It is very popular in wireless standards. By S Alamouti(*1*), simple block code was given and later on developed space time bock code by others(*2,3)*. Alamouti code is not a just code but technique to achieve reliability of data transmission in wireless communication. According to Alamouti code, two transmit and receive antennas are used for Space Time Block Code. For first time slot, antenna 1 and 2 simultaneously transmit symbol s_1 and s_2 respectively. For consecutive second time slot, antenna 1 and 2 transmit $-s_2^*$ and s_1^* respectively. For two consecutive time slots, received symbols can be represented as

$$[y_1 \;\; y_2] = [h_1 \;\; h_2] \begin{bmatrix} s_1 & -s_2^* \\ s_2 & s_1^* \end{bmatrix} + [n_1 \;\; n_2] \quad (3)$$

So each symbol has been transmitted twice and that is how data redundancy is achieved for efficient decoding at receiver. Hence, code rate is 1.Also it is assumed that channel is constant for a symbol block and changing from one block to other(*6*). Encoder consists calculation and transmitting data symbols from antennas for different time slots. For digital modulation like BPSK,QPSK, QAM, there are 2^d constellations, in which d is bits per symbol. At any time slot, 2d bits come at encoder and pick up two constellation symbols. By conjugating matrix given in (3),it has been observed that two columns (first one for s_1 and second one for s_2) become orthogonal. So detection rule at receiver converts in two separate, orthogonal issues. then

$$d_1 = [h_1^* \quad h_2] \begin{bmatrix} r_1 \\ r_2^* \end{bmatrix} \quad (4)$$

$$d_2 = [h_2^* \quad -h_1] \begin{bmatrix} r_1 \\ r_2^* \end{bmatrix} \quad (5)$$

Now using Maximum Likelihood rule, if de = Re{ d_1 } ≥ 0 then detected data symbol is 1 otherwise it is considered as 0(*7,8*).

Another MIMO technique called Vertical Bell Lab layered Space Time (VBLAST) proposed in (11,12) is an efficient wireless communication technique. VBLAST is spatially multiplexing technique which divides main data

stream into sub streams and transmit by multiple antennas. At receiver, the first step of VBLAST is interference suppression by projecting the received vector onto null subspace and second step is interference cancellation by using different detectors like Zero Forcing(ZF) ,Minimum Mean Square Error(MMSE).Using interference suppression, detection variables g can be demonstrated by(10-13)

$$g = My \tag{6}$$

where nulling matrix M of order N_t x N_r for ZF and MMSE detector can be shown by

$$W = [H^* H]^{-1} H^* \quad \text{For ZF detector} \tag{7}$$

$$W = [H^* H + N_o I]^{-1} H^* \quad \text{For MMSE detector} \tag{8}$$

RESULT

Simulation has been done on MATLAB software to see the effects of different modulation techniques like BPSK ,QPSK ,QAM on Alamouti Space Time Block Coding techniques. Performance analysis is done over Rayleigh channel to understand response on real practical channel scenario. AWGN channel is used as a reference channel to see the effects of channel noise on received data symbols. In simulation, coded Alamouti Space Time Block Coding has been observed with Uncoded wireless communication system where it has only one transmitter and receiver. And for Alamouti Space Time Block Code, two transmitter and two receiver have been taken. In this way, Bit Error Rate performance with respect to Signal to Noise Ratio is analysed of coded Multiple Input and Multiple Output(MIMO) system with Uncoded Single Input and Single Output(SISO) system. Uncoded wireless system is not preferred because of fading effects in channel but here it is taken as reference for comparison with 2x2 Alamouti Space Time Block Coding(*4,5,9*).

Here Bit Error Rate (BER) vs Signal to Noise Ratio(SNR) is analyzed over 0 to 35 dB SNR range. To increase data rate, some digital modulation techniques are used which are BPSK, QPSK, 8PSK, 16PSK, 4QAM, 8QAM, 16QAM and these all modulation techniques have been applied on 2x2 Alamouti STBC as well as Uncoded SISO wireless communication system and analyze BER vs SNR performance. In first simulation result which is shown in Figure 3 BER vs SNR performance is observed of Alamouti Space Time Block Code to Uncoded wireless system with digital modulation techniques-BPSK, QPSK, 8PSK, 16PSK. Observed BER at 10, 20, 30 and 35 dB has been noted in Table.1.In addition, simulation result of BER vs SNR for digital modulation techniques-QPSK, 4QAM, 8QAM, 16QAM for Alamouti Space Time Block Code with Uncoded wireless system is shown in Figure 4 and observed BER data is given in Table.2 at 10, 20, 30, 35 dB SNR respectively.

From Matlab simulation, it is clear that BPSK and 4QAM gives better BER performance compare to all other modulation techniques for Alamouti STBC. But BER response for BPSK is achieved for very less SNR range almost stick to 5 dB which does not fit current high SNR demand for wireless communication. 4 QAM gives better response which fits low SNR required application stick to almost 15 db.

With going higher modulation order, BER response become worst with increasing data rate. So this factor also needs to be considered for design engineer of wireless communication and it is needed to choose modulation order according to application performance required. Uncoded system gives linear response but because of real time wireless communication system suffers from interference, noise and fading components ,it is not preferred over coded communication system.

Figure 5 shows simulation of BER performance for VBlast MIMO system and Table 3 shows simulation result at some particular SNR values. This result is very important to study performance analysis between two popular MIMO techniques ,Diversity gain(STBC) and Spatially multiplexing(VBlast-ZF, VBlast-MMSE).From observation ,it is very clear that Vblast gives steady and better response especially at high SNR. So for high data rate as well as high power requirement, VBlast would be healthy compare to STBC and it fulfills current application requirement. Among VBlast-ZF and VBlast-MMSE, VBlast gives comparatively efficient response throughout taken SNR range.

CONCLUSION

In Alamouti STBC, Channel state information is not required at transmitter .Because of no feedback, channel estimation is not required and overall system become simple. Also there is less complexity at decoder due to orthogonality in code. But some disadvantages are also there in Alamouti STBC. One of major is, if channel is

varying during two consecutive time instants ,this code is not useful. Also there is high complexity at receiver because of using Maximum likelihood detection technique. Therefore, efficient detection techniques are required and VBlast is good option to mitigate these all problems. By analyzing Vblast result, It will definitely give very good response in high order MIMO-OFDM applications.

FIGURES

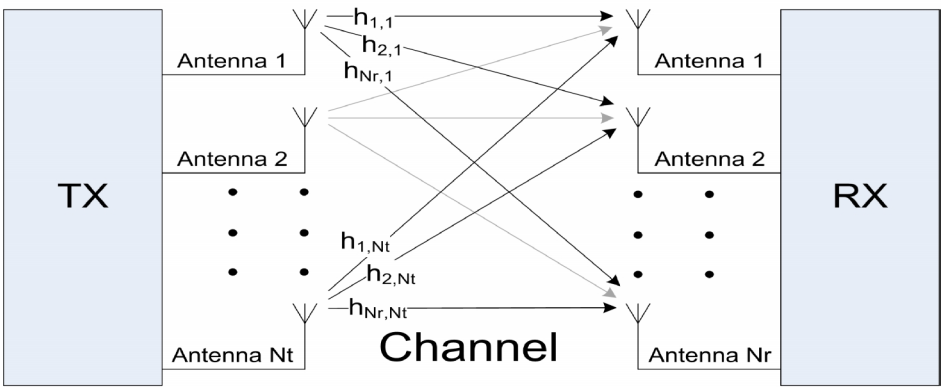


Fig. 1. Block Diagram of MIMO system(10).

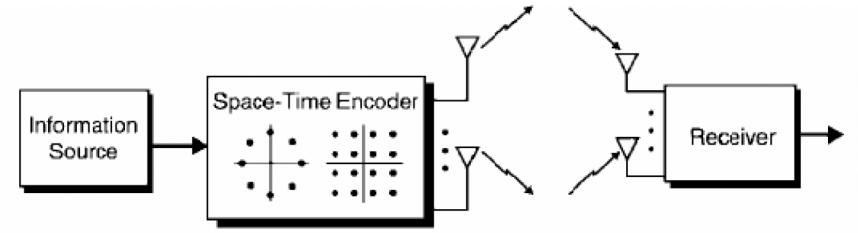


Fig. 2. System Bock Diagram of STBC(*2*).

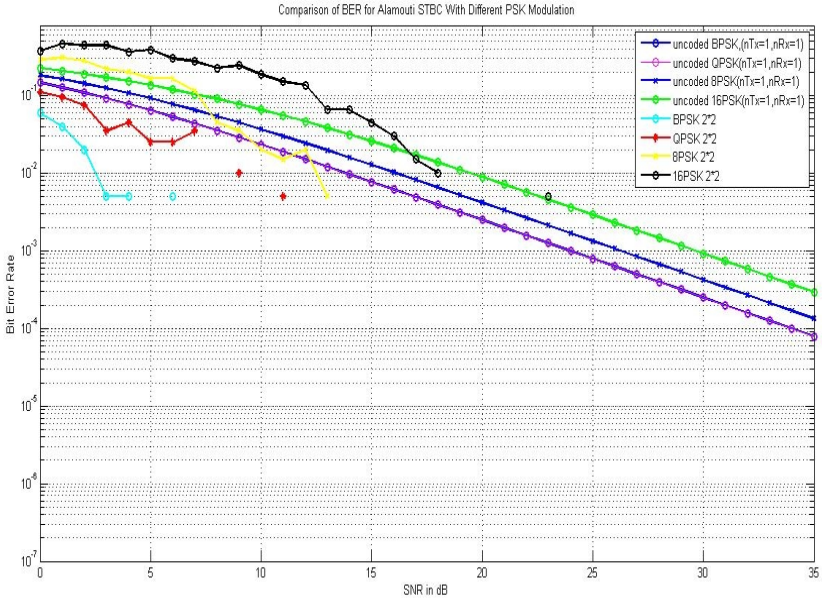


Fig. 3. Comparison of BER vs SNR for Alamouti STBC with different PSK modulation techniques.

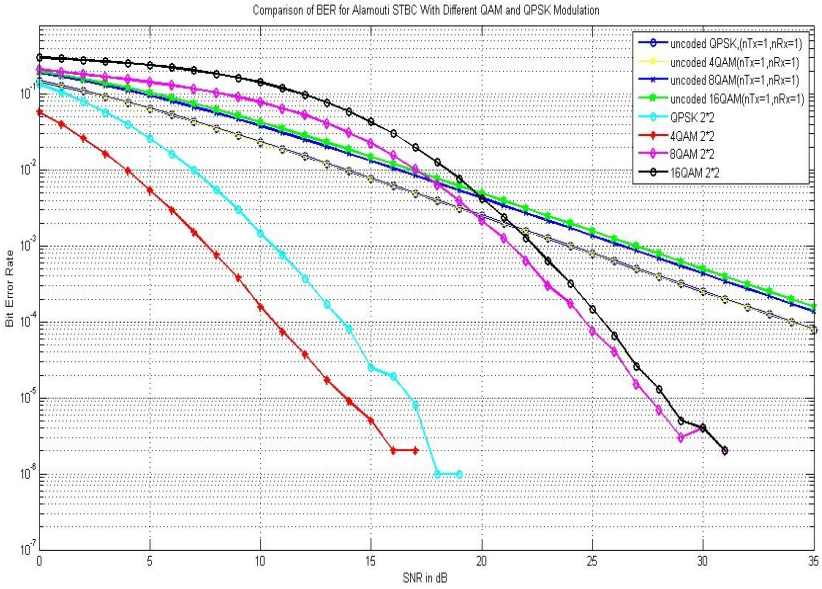


Fig. 4. Comparison of BER vs SNR for Alamouti STBC with different QAM and QPSK modulation techniques.

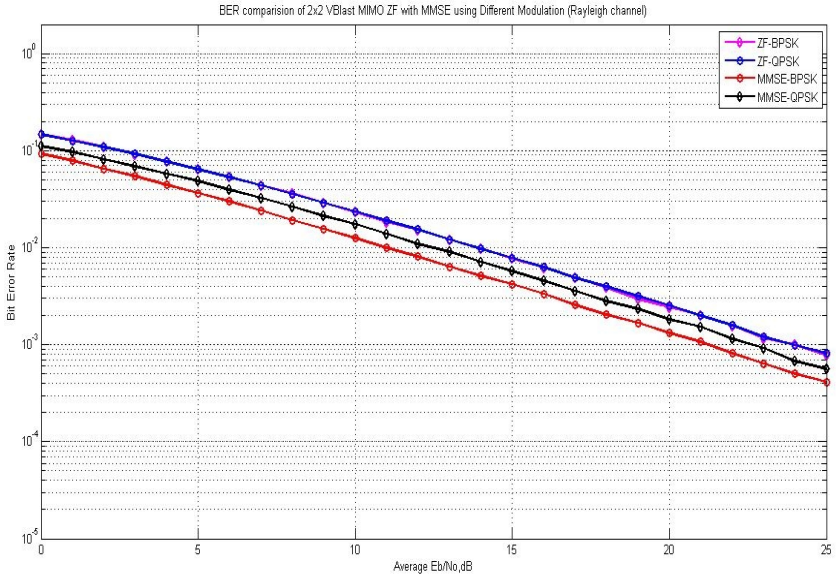


Fig. 5. Comparison of BER vs SNR for VBlast MIMO with BPSK and QPSK modulation techniques.

TABLES

Modulation	BER at 10 db	BER at 20 db	BER at 30 db	BER at 35 db
Uncoded BPSK	0.02326	0.00248	0.00024	0.00007
Uncoded QPSK	0.02326	0.00248	0.00024	0.00007
Uncoded 8PSK	0.03667	0.00416	0.00042	0.00013
Uncoded 16PSK	0.06559	0.00886	0.00092	0.00029
BPSK 2x2	0.00000	0.00000	0.00000	0.00000
QPSK 2x2	0.00000	0.00000	0.00000	0.00000
8PSK 2x2	0.02000	0.00000	0.00000	0.00000
16PSK 2x2	0.18500	0.00000	0.00000	0.00000

Table 1. Comparison of BER for Alomouti STBC with different PSK modulation techniques.

Modulation	BER at 10 db	BER at 20 db	BER at 30 db	BER at 35 db
Uncoded QPSK	0.02326	0.00248	0.00024	0.00007
Uncoded 4QAM	0.02326	0.00248	0.00024	0.00007
Uncoded 8QAM	0.03779	0.00425	0.00043	0.00013
Uncoded 16QAM	0.04237	0.00488	0.00049	0.00015
QPSK 2x2	0.00145	0.00000	0.00000	0.00000
4QAM 2x2	0.00015	0.00000	0.00000	0.00000
8QAM 2x2	0.07787	0.00214	0.00000	0.00000
16QAM 2x2	0.14158	0.00423	0.00000	0.00000

Table 2. Comparison of BER for Alomouti STBC with different QAM and QPSK modulation techniques.

VBlast 2x2 MIMO		BER at 10 db	BER at 20 db	BER at 30 db	BER at 35 db
ZF	BPSK	0.02898	0.00292	0.00028	0.00009
	QPSK	0.02886	0.00315	0.00033	0.00008
MMSE	BPSK	0.01558	0.00166	0.00017	0.00005
	QPSK	0.02126	0.00233	0.00024	0.00006

Table 3. Comparison of BER for VBlast MIMO with BPSK and QPSK modulation techniques.

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Hardness and Roughness analysis of WC-coated boiler steel for industrial application

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ABSTRACT

WC coating is now a day widely used on boiler steel to improve its overall performance. Coating plays major role to improve properties of boiler material. This work analysis the performance of WC coated stainless steel material which is used as for boiler application.WC-300 coating was sprayed on ASTM E384 stainless steel by high velocity oxy fuel (HVOF) coating. Coating introduced on stainless steel at 500^0C. Coating thickness were 400µm and 500 µm. It was observed that it possess very low porosity and improve micro hardness.

SUMMARY

WC-300 Coating on boiler steel material to improve hardness, surface roughness and decrease porosity.

Keywords: WC-300, HVOF, Hardness, Roughness

INTRODUCTION

High velocity oxy fuel (HVOF) Coating is widely used now a day for coating for various applications. HVOF is recommended due to its excellent properties like high adhesion strength, low porosity and high hardness. Most popular HVOF sprayed coatings are WC and Ni based coatings which also have high wear resistance. It was found some pathologies problem like skin diseases, asthma, cancer etc. related to use of nickel, WC coating was more popular [1-7].
Flame was created by combustion of fuel gas and oxygen mixture in the gas chamber of the thermal spraying gun. Powder is injected into the flame using inert compressed gas. It melts in the flame and is transferred in this state to the substrate where it cools down. This treatment is called re melting and is

performed using flame torch or high temperature furnace [8-10] .WC and Cr as well as its compound are most studied and applicable coatings. It improves strength as well as reduce porosity with better microstructure [11-20].
In present work HVOF technique used for WC-300 coating on stainless steel used for boiler application.

MATERIALS AND METHODS

A commercially available WC-300 coating powder was used for HVOF coating. ASTM E384 stainless steel is used as a substrate material which is used for boiler tubes.Coating was sprayed by using HIPOJET HYBRID gun for 400 µm and 500µm coating on stainless steel. Oxygen, LPG and Air gases used for coating with pressure 10bar, 6.5 bar and 6 bar respectively. Coating Temperature was 125^0 C with spray distance 7-8 inch. Travel speed of gun was maintained 0.3meter/minutes. Flow rate of Oxygen 255 lit/min, for LPG45 lit/min and for air 0.02 liter/minutes was maintained. Thickness of coating per path was < 25 micron.
Fig. 1 shows HVOF coating on boiler coating with the use of Gun. Atomic Force Microscope was used for finding surface roughness and microstructure. VHN hardness tester was used to measure hardness.

RESULTS AND DISCUSSION

Porosity of WC- 300 Coated boiler steel has been analysed. Table 1 shows porosity of boiler tubes after coating. It found that minimum porosity achieved was 0.99% and maximum 2.62%. Average value of porosity was 1.73%. Very lower porosity achieved at 500 µm thickness. At 400 µm coating thickness porosity range reported 0.92% to 1.25%(shown by Table 1 and Table 2) which is lower than porosity reported at 500 µm.Porosity is inversely proportional to thermal conductivity, and will also be susceptible to penetration by corrosive reagents during operation on account of their open structure. Consequently, the need arises to employ denser coatings which degrade less due to their more closely packed structure [21-22].
Microstructure of WC-300 coated boiler tubes with thickness 500 and 400 µm has been analysed to represent growth and surface roughness. Atomic force microscope was used for topographical representation. Fig.2 and 3 shows topographyof500 and 400 µm thickness coated substrate. From fig. 2 and 3 it is clear that good interface seen across the microstructure that strengthens the overall strength of material.
Surface roughness played important role on material structure. Here batter surface roughness achieved which improve hardness of boiler tube and also have better corrosion resistance. For both 500 and 400 µm thickness better surface roughness achieved which improve hardness of tubes.
Vickers hardness test was implemented to find out Micro hardness of WC- 300 coated ASTM E384 boiler tubes. HV0.3 Load was applied for micro hardness testing. Table 5 and 6 and Fig 4 provides VHN hardness of WC-300 coated ASTM E384 boiler tubes for 500 and 400 µm thickness. For 500 µm thickness hardness was 1033VHN, while for 400 µm it was 1248VHN which is higher than that of 500 µm thickness. If we compare hardness plain boiler steel with WC coated steel, before coating hardness of boiler steel is 670VHN and after coating there was tremendous increase in hardness from 670 VHN to 1033 and 1248 VHN.

CONCLUSION

It was found that very low porosity achieved which gives denser structure. Batter surface roughness achieved which improve hardness of boiler tube and also have better corrosion resistance. There is tremendous improvement in hardness after coating from 670 VHN to 1033 VHN for 500 µm and 1248 VHN for 400 µm thickness. Better surface roughness also played a role to achieve good hardness.

FIGURES



Fig 1 WC-300 HVOF Coating at 125^0 C for coating thickness 500 µm

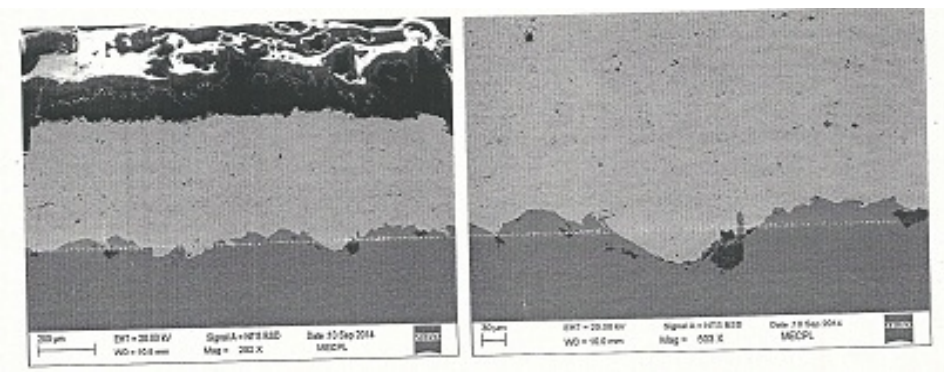


Figure 2 Microstructure of WC-300 coated boiler tube at 500 µm thickness

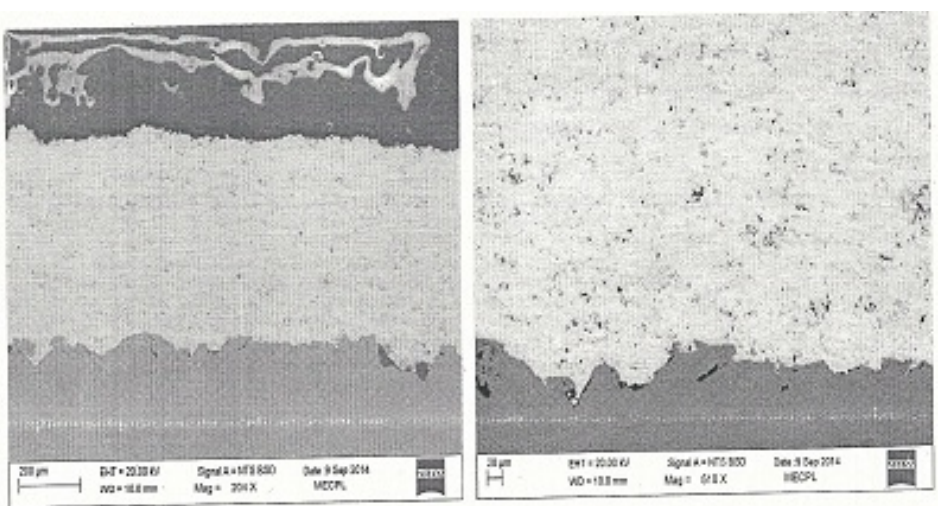


Figure 3 Microstructure of WC-300 coated boiler tube at 400 µm thickness

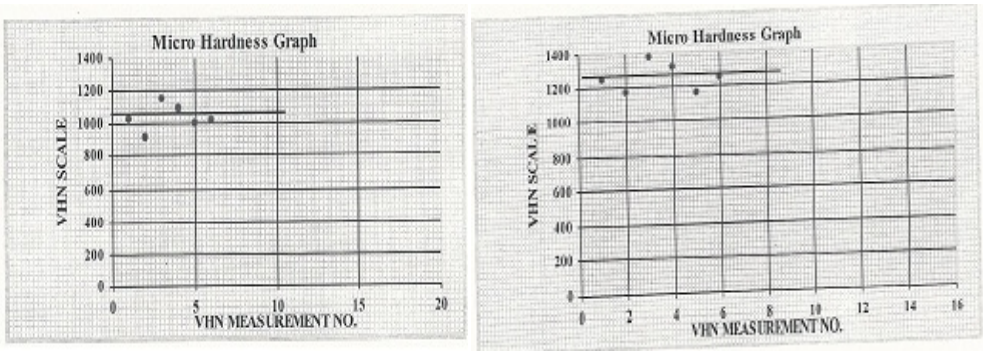


Figure: 4 Micro hardness for 500 µm and 400 µm thickness at 0.3HV load

TABLES

Boiler Tube	Tube 1	Tube 2	Tube 3	Tube 4	Tube 5	Average
Porosity	0.99	2.24	2.62	1.33	1.47	1.73%

Table1. Porosity of WC-300 coated boiler tube for 500µm thickness

Boiler Tube	Tube 1	Tube 2	Tube 3	Tube 4	Tube 5	Average
Porosity	0.92	1.25	0.87	1.14	0.93	1.02%

Table 2. Porosity of WC-300 coated boiler tube for 400µm thickness

Boiler Tube	Tube 1	Tube 2	Tube 3	Tube 4	Tube 5	Average
Surface Roughness	4.79	4.55	4.78	4.71	4.35	4.63

Table 3. Surface roughness of WC coated Boiler tube at 500 µm thickness

Boiler Tube	Tube 1	Tube 2	Tube 3	Tube 4	Tube 5	Average
Surface Roughness	4.11	4.44	4.87	4.35	5.21	4.59

Table4. Surface roughness of WC coated Boiler tube at 400 µm thickness

Boiler Tube	Tube 1	Tube 2	Tube 3	Tube 4	Tube 5	Average VHN
Microhardness	915	1150	1081	1004	1017	1033

Table 5. Micro hardness at load HV0.3 for 500 µm thickness

Boiler Tube	Tube 1	Tube 2	Tube 3	Tube 4	Tube 5	Average VHN
Micro hardness	1160	1374	1308	1153	1248	1248

Table 6. Micro hardness at load HV0.3 for 400 µm thickness

ACKNOWLEDGEMENT

Authors acknowledge Metallizing Equipment Co. Private limited, Jodhpur to allowing testing facility for present research.

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Harmonics mitigation and Power Factor Correction in BLDC motor using DC-DC Converter

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ABSTRACT

The Technique to improve the power Factor and harmonics reduction of Brushless DC motor by Power Factor Improvement converter serve an crucial role in the energy conservation ,saving and quality improvisation during power transformation. In BLDC motor the AC-DC transition of electrical power is generally required, while this conversion from AC main to DC power. In this Paper DC- DC converter such as Boost converter used as a single stage power factor improvement converter for a BLDC motor fed through a front end uncontrolled rectifier from a single-phase AC mains. The DC-DC converter, here Boost converter shows conformation to power quality international standards with improved performance of BLDC Motor.

SUMMARY

Harmonics mitigation and power factor correction in BLDC motor.

Keywords: Brushless DC motor, Boost Converter, Voltage Source Inverter, PFC.

INTRODUCTION

Brushless Direct current Motors are one of the best motor types rapidly gaining popularity. BLDC motor are used in various applications such as appliance, aeronautical engineering, consumer electronics,

Medical Science, Electrical and electronics automation instrumentation, automotive, equipment and instrumentality[4].Brushless are not used in BLDC motor for commutation. BLDC motors are electrically commutated motor. There are Many Advantages of BLDC Motor Compared to Brushed DC Motor and Induction Motor [4].

- Better operating life
- Reduced noise
- Speed Torque Characteristics
- Good dynamic response
- Better efficiency
- Better Speed range
- High Reliability
- Reduced Maintenance cost

They can compete on Cost alone Due to Three Major Factor [5]:

- Advances in Magnet Technology
- Improvement in Motor Control Electronics
- Capital investment By BLDC Manufacturers

As a result, brushless DC motors are utilized in a different range of cost sensitive Practical application. BLDC Motors are mentioned as Inside-Out Permanent Motors Because Their Speed-Torque Curves are very similar to the PM Motors. However, BLDC Motors have their Magnets on the Rotating Part of the Motor instead of on Stationary part and Winding on Stationary part. Working operation of a BLDC Motor is related except that the Winding Phases are Switched On and Off Electronically by Means of Control Device. Benefits of Lower Rotor inertia and more efficient Heat Dissipation than DC Brush type Construction. Due to the Assemblage of an Inner Permanent Magnet rotor and Outer Winding offers [4].

1. **Operation of BLDC motor with hall sensor :**

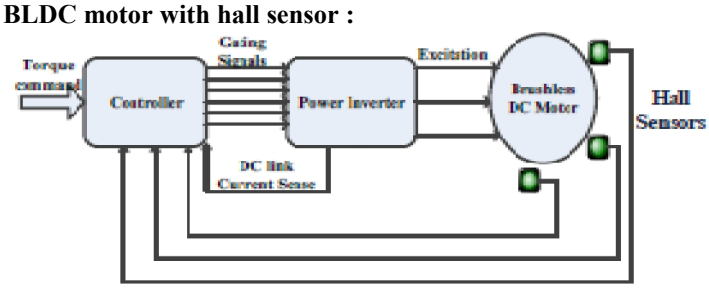


Fig. 1. Hall Sensor

The Hall sensors need a power supply. The Voltage May Range from 4Volt to 24Volt. And Current required is 5mAmp to 15mAmp [3].

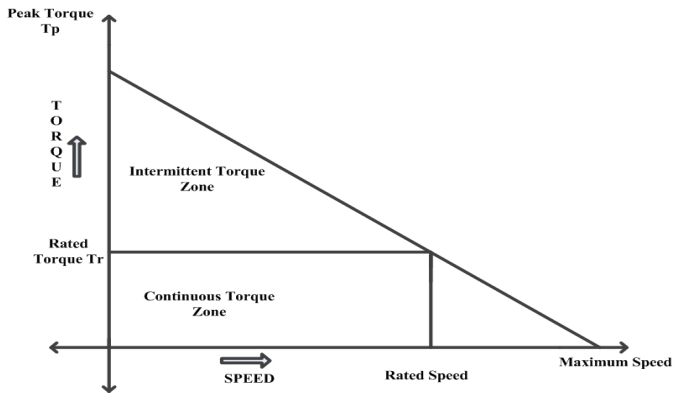


Fig.2. Torque/Speed Characteristics

When Speed Ranges up to the rated Speed then The Torque Remains Constant. The Motor Run Up to The Maximum Speed, Which Can is Up to 150% of Rated Speed but at that Time Torque Starts reducing [3].

2. Identification of problem :

A permanent magnet BLDC motor has the developed torque which is directly proportional to motors current and motors back-emf which is produced in motor is proportional to the speed of BLDC motor. Therefore, a constant current in BLDC motor stator windings with variable voltage across its terminals remain constant torque in a permanent magnet BLDC motor under variable speed condition.

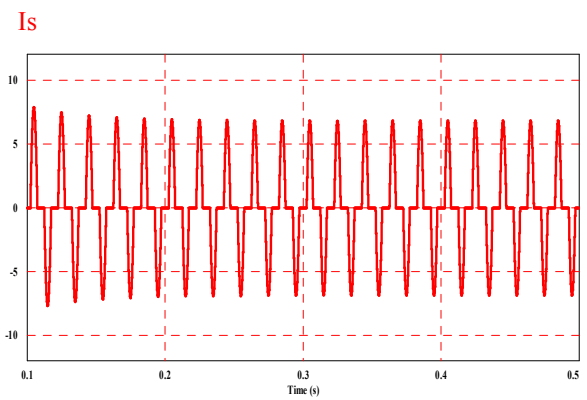


Fig. 3. Current waveform at AC mains without PFC

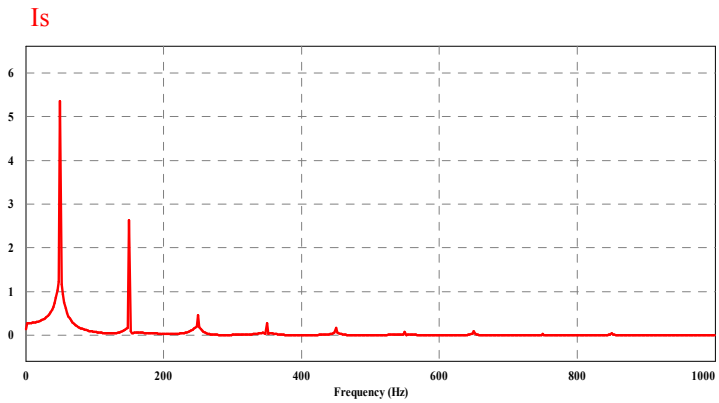


Fig. 4. BLDCM drive Harmonic Spectra without PFC

Table 1: Problem Identification Data

Supply Voltage(Vs)	230V
Supply Current(Is)	3.74A
Fundamental frequency	50Hz
(%)THD of Is	62.53%
Power Factor	0.77

3. Solution using boost converter:

Simulation is carried out in PSIM simulator. In which the DC link voltage of inverter is controlled by PWM as shown in figure no 5. In which the reference single is compare with actual signal and this output is given to MOSFET (switch of boost converter) In case of inverter control. It is done by PI controller. In which inverter is controlled by proportional gain block shown in Figure 5.

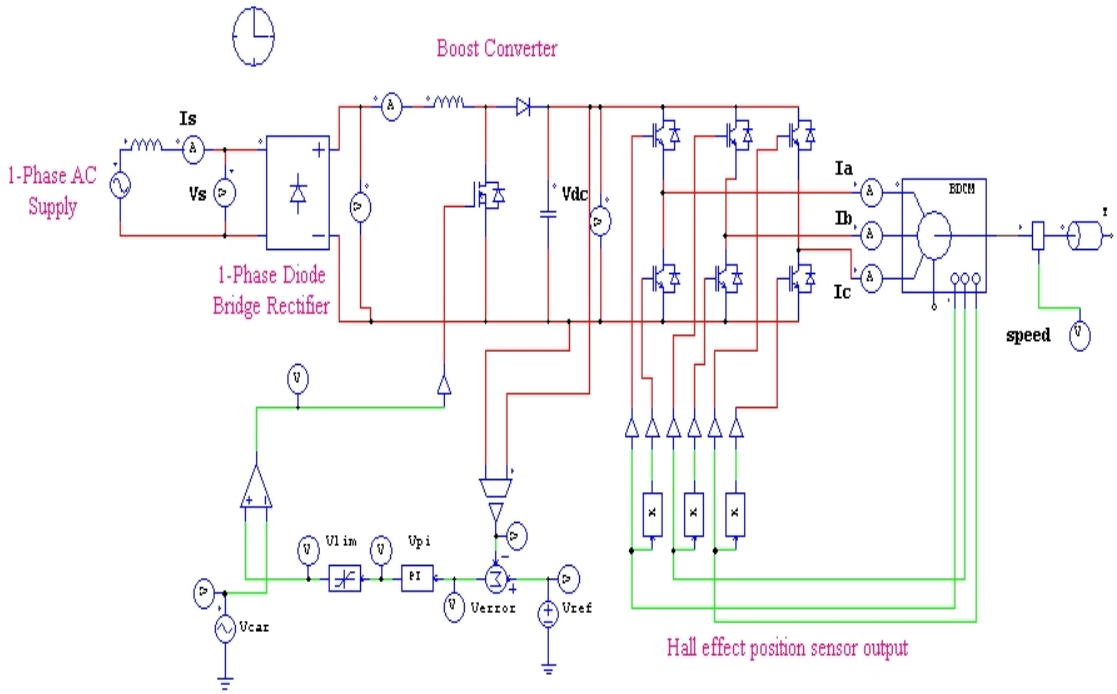


Fig. 5. Simulation of Boost Converter fed VSI Based BLDC Motor

- Phase Current Waveform

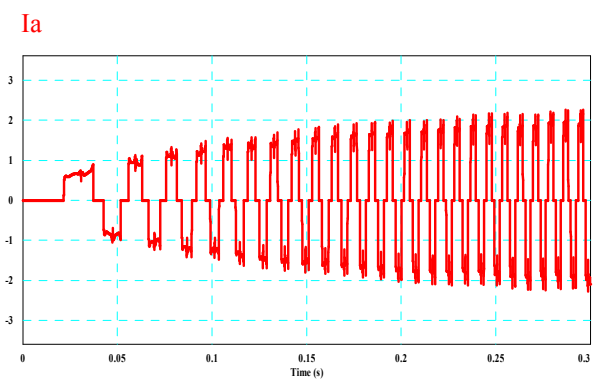


Fig. 5 (a). Current Waveform of Phase a (IA)

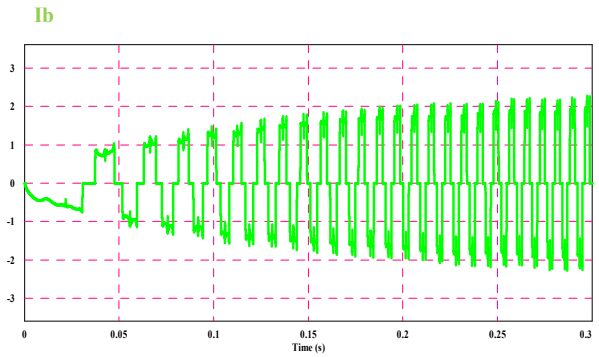


Fig. 5 (b). Current Waveform of Phase B (I)

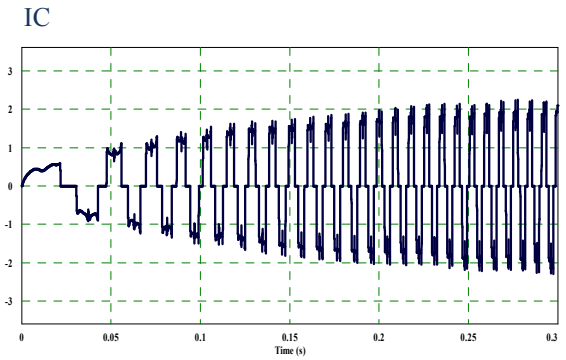


Fig. 5 (c). Current Waveform of Phase(IC)

- FFT Analysis of Supply Current

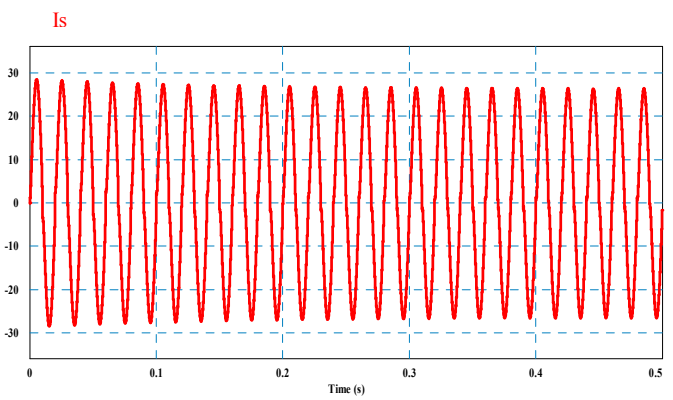


Fig. 5 (d). Supply Current

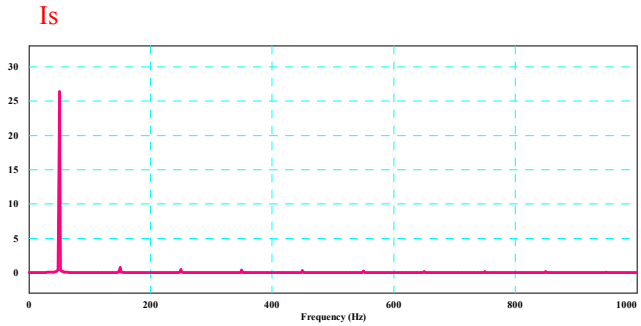


Fig. 5 (e). FFT Analysis of Supply Current

- Supply Current and Voltage Waveform.

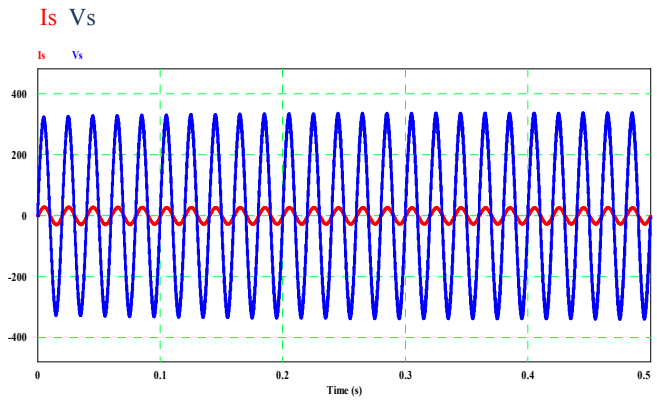


Fig. 5 (f). Supply Voltage and Current Waveform

Figure 5 (f) shows the relation between voltage and current. From the simulation result it is clear that power factor is near to unity (0.99).

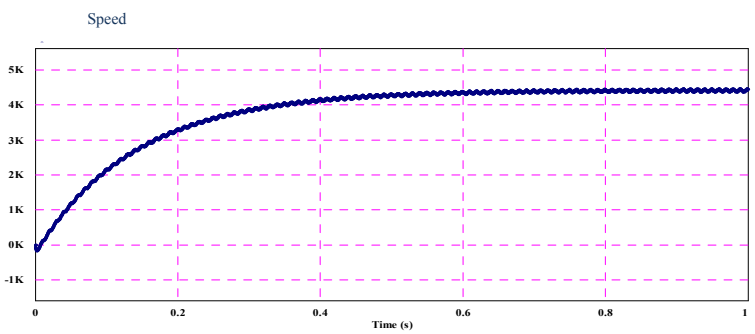


Fig.5(g). Speed Waveform of BLDC Motor

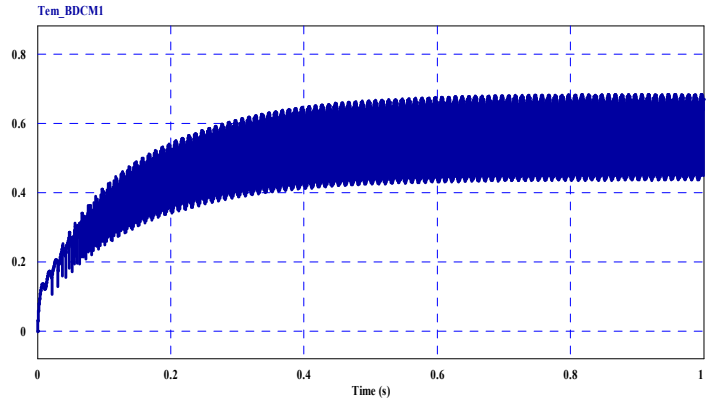


Fig. 5 (h). Torque Waveform of BLDC Motor

Table 2: Result Data

Supply Voltage(Vs)	230V
Supply Current(Is)	18.38A
Fundamental Frequency	50Hz
THD of Supply Current	**4.77%**
Power Factor	**0.99**

4. Conclusion :

In this paper, the DC-DC Converters like Boost Converter for BLDC motor is observed. The DC-DC converter for power quality improvement in Brushless DC Motor drives are design and their performance is simulated in PSIM. The speed of Brushless DC motor has been a directly proportionate to the DC link output voltages there for a linear speed control is achieved during controlling the DC link output voltage. The result of practical confirmed that THD in AC supply source current is minimized and power Factor is improved near unity with the help of DC - DC Boost Converter as per International power quality standards.

5. Appendix :

Power rating = 1500 Watt, Voltage rating = 300 Volt, Current rating = 5 Amp, No. of Poles-4, Stator Resistance(R) =11.9Ω, Stator Self Inductance (L) = 0.00207H, Vpk / krpm = 32.38, Vrms / krpm = 22.9.

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Experimental Analysis of Concentric flow Dry Wire Electric Discharge Machining (CDWEDM)

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ABSTRACT

Dry wire electric discharge machining process finds its application to cut intricate and complex profiles on difficult to cut thin materials. The process offers improved surface integrity, environmental and operator friendliness. However, low MRR and poor geometrical/dimensional accuracy are inherent drawbacks of this process. In this paper, the authors have proposed an innovative approach of concentric flow to supply gaseous dielectric fluid for dry WEDM process with a view to improve stability of wire travel, ensure uniform and effective sparking all over wire circumference and to obtain efficient debris flushing from sparking gap. Current, air pressure, pulse on time and pulse off time have been selected as process parameter and effects on cutting velocity, kerf width and MRR were investigated. ANOVA, regression and residual analysis have been performed to investigate the statistical behavior of process. Results suggest that proposed method can be an alternate to conventionally used side flow method.

SUMMARY

Performance enhancement of dry wire EDM is achieved by implementing novel approach of concentric flow for dielectric around wire.ne sentence.

Keywords: Concentric flow, Side flow, Wire EDM, Dry WEDM, Titanium alloy

1. INTRODUCTION

Wire Electrical Discharge Machining (WEDM) developed in late 1960 is widely accepted thermo electric spark erosion process for manufacturing industries, especially in production of dies, gauges, aeronautical and bio-medical parts (1,2). WEDM is a unique type of traditional electrical discharge machining (EDM) process in which a continuously travelling electrically conductive 0.05-0.3 mm diameter thin wire (copper, zinc coated brass, tungsten or molybdenum) used instead of electrode. The chain of repetitive and discrete electrical spark discharges between wire and workpiece cause high heat generation and results in melting and vaporization of minute amount of molten wok material in forms of debris as a part of fundamental mechanism of material removal in this process (3). Molten material is removed and flushed from gap in form of debris by dielectric fluid to perform one of its basic functions out of four (insulation, ionization, cooling, removal of waste particles), hence it plays important role in process. For different kind of dielectrics (hydrocarbon oil/ deionized water) having different composition, properties and cooling rates, it finally affects performance and control of the electrical spark (4). Although dielectric fluid is a critical element, but normally use of conventional dielectric liquid develops critical issues such as deterioration of dielectricity of fluid, air pollution, and carbon deposition on the work surface which are intolerable especially due to compulsion for ISO 14000 standards in manufacturing industries for minimizing environmental impact of process and its elements and for achievement of cleaner and greener process (5). As a same approach in EDM, first time in 1985, a novel approach of Dry EDM was developed and reported in NASA paper and dielectric liquid was replaced by dielectric gas (6). Especially in wire EDM, there is used demineralized water as dielectric fluid and normally substances generated like nitrogen oxide, carbon monoxide, metallic particles and chloride in emissions during process are harmful for health of the operator and also for the environment. At the end of the operation in wire EDM, disposal of the sludge (small workpiece particles in form of debris), degraded dielectric fluid waste, filter cartridges and resins used for deionization is a critical problem, to prevent pollution of both the land and water. Gaseous dielectrics became popular solution for reducing environmental impact and for higher safety of operator. Kunieda and Furudate investigated a new novel machining process Dry WEDM, in which air/gas was used to eliminate the use of dielectric liquid in 2001 (8). Dry WEDM also eliminates the serious problem of electrolytic corrosion due to deionized water. Wang et al. continuously worked in research related to Dry WEDM since 2002 to 2013 for process development (9, 10).

Since the development of Dry WEDM till now, in all research, air or gaseous dielectric supplied from side of wire by nozzle, called it as side dry WEDM (SDWEDM). Nozzle was inclined to wire, so that jet from nozzle reaches to spark gap for flushing of debris. But it causes blockage of gap, so it require to find effective way of removing debris generated to achieve better machining accuracy and process stability of dry WEDM. Spark gap flushing improvements leads towards the higher removal rate. Essential requirement for efficiency improvements necessitates innovative approaches for flushing. The present work for enhancement of material removal efficiency of Dry WEDM is incorporated by novel approach of concentric air flow in the working gap, concentric to wire. This paper presents concept of concentric flow dry WEDM (CDWEDM) as shown in fig. 1, for removing debris and renewing dielectric instead of inclined jet through side nozzle. The machinability of Ti6Al4V, titanium alloy (grade-5) was investigated by varying control parameters, i.e., air pressure, current, pulse on time and pulse off time and effect on response parameters, i.e., the cutting velocity, kerf width and MRR were found. The process parameters significance was identified by using ANOVA for concentric flow dry WEDM. The mathematical model has been developed for Cutting velocity, kerf width and MRR using regression analysis in terms of control parameters.

2. EXPERIMENTAL SETUP AND PROCEDURE

The reuse type CNC wire cut electric discharge machine (DK-7720 model) were used for performance of experiments. For reusable WEDM setup, wire is repeatedly wound on top and bottom drums through height adjustable tensioning pulleys system. Servo controller based table positioning was employed in the machine. An experimental setup for supplying compressed air through nozzle with concentric jet was developed. Flow control valve and the pressure gauge were mounted in the supply line to monitor the air pressure. For concentric jet system, specially designed nozzle shown in fig. 2 was fixture with machine column to ensure coaxial flow of compressed air over travelling wire.

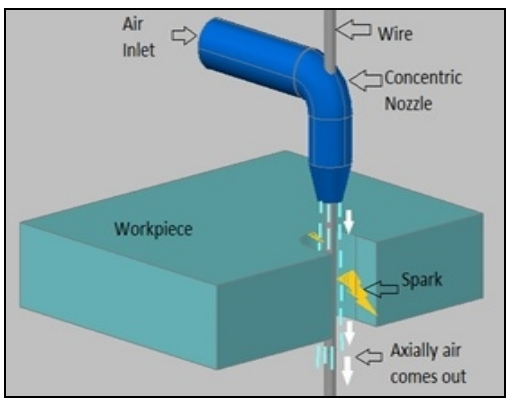


Fig. 1 Schematic diagram of Concentric dry

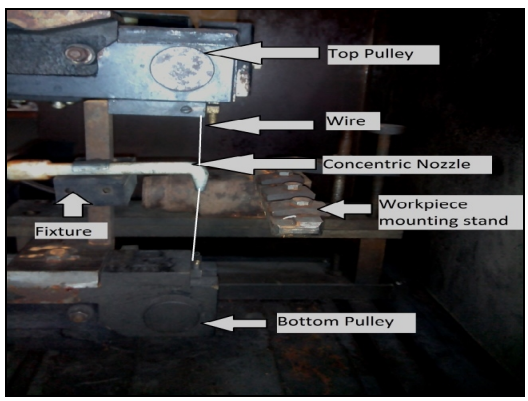


Fig. 2 Concentric dry WEDM setup

The machining of 0.6mm thin work piece sheet (titanium alloy grade-5) was carried by 0.18mm diameter molybdenum wire at 10.3 m/s wire speed. Compressed atmospheric air was used as gaseous dielectric fluid for present study. The chemical composition of thin work piece sheet material taken for experimentation work as follows:

Table 1 Chemical Composition of Work Piece Materials

Al	V	C	Fe	O	N	H	Ti
6.08	4.02	0.08	0.22	0.2	0.05	0.15	Balance

Ti-6Al-4V, Titanium alloy sheet (ASTM Grade 5), of 100mm ×25mm×0.6mm size, was used as the work-piece material. Ti-6Al-4V material finds applications in medical, aerospace and automobile industry. The four parameters i.e. air pressure, current, pulse on time and pulse off time were varied over range to investigate effects on response parameters i.e. the Cutting velocity, kerf width and MRR (material removal rate). Table 2 shows the levels of experimental parameters and Table 3 shows the constant parameters for used for the experiments.

Actual samples cut during the experiments are shown in the figure 3. Fig. 4 shows the wire cutting profile on workpiece during machining. The start point taken was A. The wire tool will trace the AB-BC-CD-DE. A Profile shown in fig. 4 was cut during each experimental run.

Table 2 Control parameters and levels

Variable Parameter	Level Value
Pulse-on Time(µs)	16,24,32,40,48,56
Pulse-off Time(µs)	5,7,9,11,13,15
Discharge Current(A)	2,3,4,5,6,7
Air Pressure(Bar)	2,2.5,3,3.5,4,4.5
Pulse-on Time(µs)	16,24,32,40,48,56

Table 3 Constants parameters and its values

Parameters	Value
Wire material	0.18mm diameter molybdenum
Work piece material	0.6mm thick Ti-6Al-4V
Wire winding speed	10.5 m/s
Machining length	25mm for each cut
Nozzle material	Acrylonitrile Butadiene Styrene

The cutting velocity was measured taking ratio of machining length to machining time for each profile cut. Digital profile projector was used to measure the kerf width. The kerf width was measured at the gap of CD of the profile shown in fig. 4. MRR is calculated by multiplying cutting velocity, workpiece thickness and kerf width.

MRR = CV x kerf width x thickness of workpiece

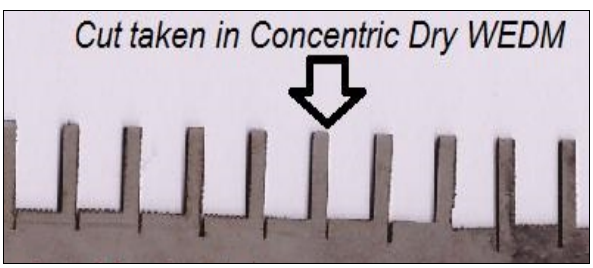


Fig. 3 Photograph of workpiece on which cut made

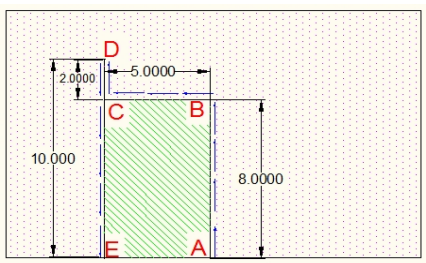


Fig. 4 Profile and path of wire

3. RESULTS AND DISCUSSION

3.1 Effect of control parameters

3.1.1 Current Effects

The response behaviour of concentric flow dry WEDM for cutting velocity, kerf width and MRR is depicted under the influence of current in Fig. (5a-5c). Figure 5(a) shows influence of current on the behaviour of CV. It is found that with increase in current, CV increased for all levels of current due to higher discharge energy at higher current from wire to workpiece. Spark dissipates higher thermal energy; it leads towards more workpiece material removal. Hence, the cutting velocity increases by increasing discharge current *(11)*. Figure 5(b) indicates influence of current on the behaviour of kerf width. It is found that with increase in current, kerf width increased for all levels of current due to higher crater depth. Summarize effect of cutting velocity and kerf width is reflected on MRR as shown in Figure 5(c).

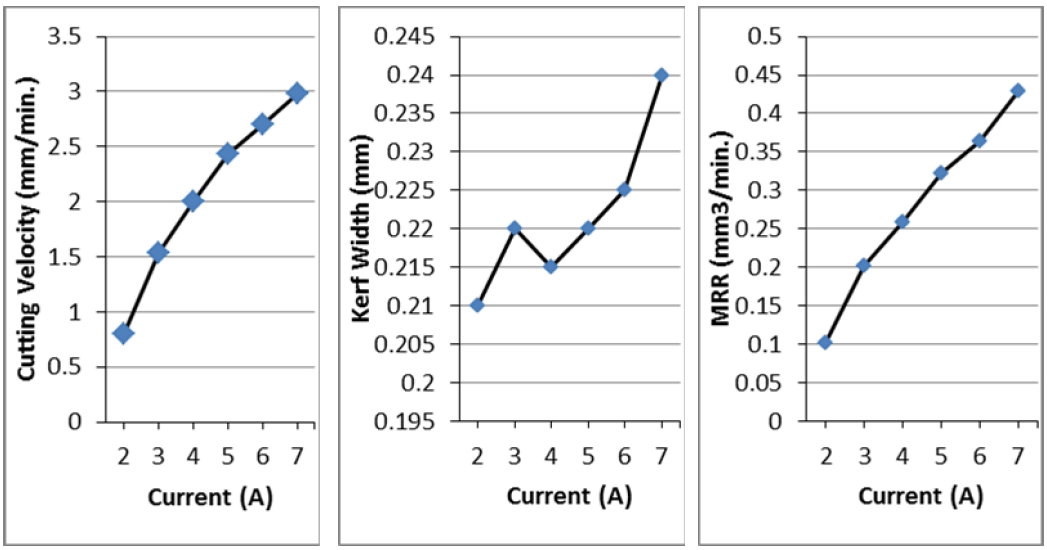


Fig. 5 Effect of Current on characterisitcs (a) Cutting Velocity (b) Kerf width (c) MRR

3.1.2 Pressure Effects

The response behaviour of concentric flow dry WEDM for cutting velocity, kerf width and MRR under the influence of pressure is depicted in Fig. (6a-6c). Figure 6(a) indicates influence of pressure on the behaviour of CV. It is found that with increase in pressure, CV decreased for all levels of pressure due to disturbance of spark at higher pressure. Hence, the cutting velocity decreases by increasing pressure. Figure 6(b) indicates influence of pressure on the behaviour of kerf width. It is found that kerf width decrease and increase and not follow any pattern. Summarize effect of cutting velocity and kerf width is reflected on MRR as shown in Figure 6(c).

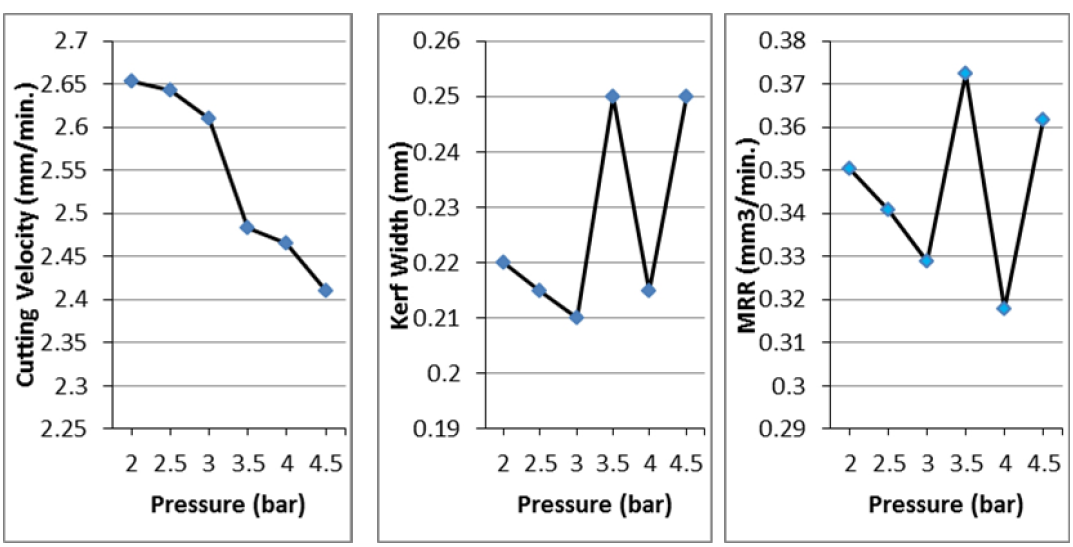


Fig. 6 Effect of Pressure on characterisitcs (a) Cutting Velocity (b) Kerf width (c) MRR

3.1.3 Pulse on Time Effects

The response behaviour of concentric flow dry WEDM for cutting velocity, kerf width and MRR is shown in Fig. (7a-7c). Figure 7(a) indicates effects of pulse on time on the behaviour of CV. It is found that at initial level with increase in pulse on time, CV, kerf width and MRR increased due to higher time for discharge energy for melting and vaporization of workpiece material. But after 32 micro second time, further rise in pulse on time causes reduction in CV, kerf width and MRR due to short circuit occurs in presence of highly accumulated debris.

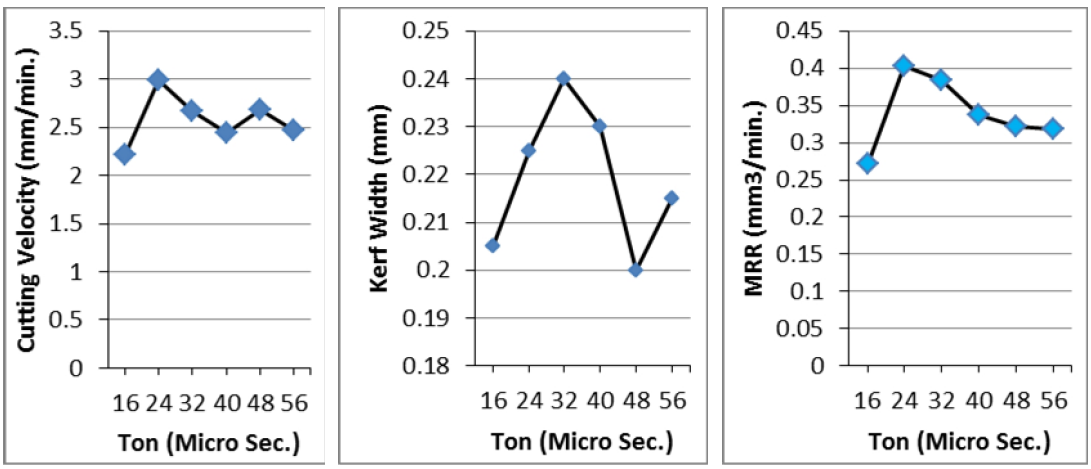


Fig. 7 Effect of Pulse on time on (a) Cutting Velocity (b) Kerf width (c) MRR

3.1.4 Pulse off Time Effects

The response behaviour of concentric flow dry WEDM for cutting velocity, kerf width and MRR under the effects of pulse off time is shown in Fig. (8a-8c). Figure 8(a) indicates effects of pulse off time on the behaviour of CV. It is found that with increase in pulsed off time, CV decreased for all levels of pulse off time due to higher idle time in which no material removal occurs. Hence, the cutting velocity decreases by increasing pulse off time. Figure 8(b) indicates influence of pressure on the behaviour of kerf width. It is found that kerf width increase as more time debris removal. Summarize effect of cutting velocity and kerf width is reflected on MRR as shown in Figure 8(c).

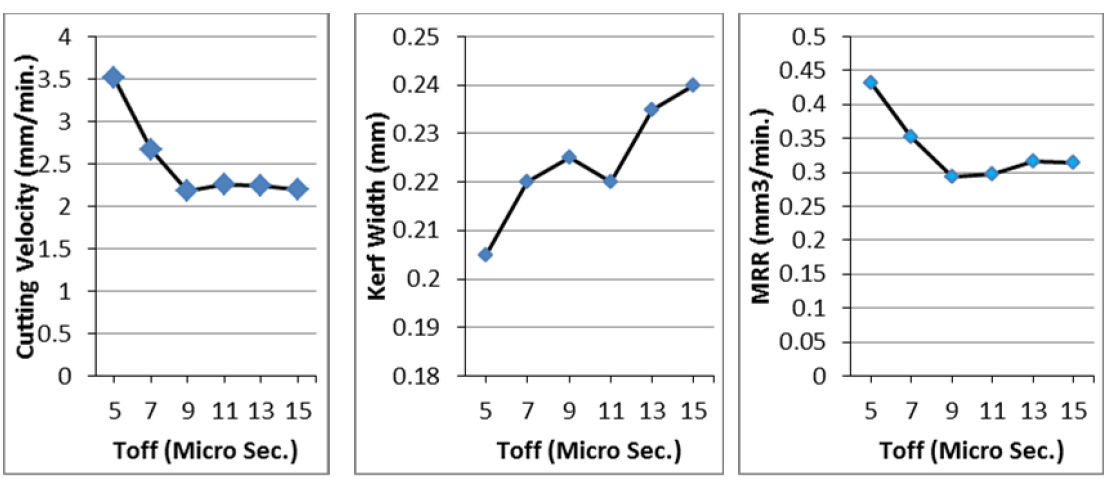


Fig. 8 Effect of Pulse off time on characterisitcs (a) Cutting Velocity (b) Kerf width (c) MRR

3.2 ANOVA Analysis

There are 24 experiments in total carried out according to the OVAT. For this purpose, ANOVA is performed. Results for Cutting Velocity, Kerf Width and MRR were evaluated using ANOVA as shown in Table 4, 5 and 6 respectively. The ANOVA table having columns (i) Degree of freedom (DoF), (ii) Sequential sum of squares (Seq SoS) (iii) Adjusted mean squares (Adj. MS) (iv) F-value and (v) P-values. The analysis was carried out by MINITAB software. The significant level and confidence level were 5% and 95% respectively. Value of F indicates significance of a control parameter over response by showing the mean square error to residual. Value of P reports level of significance. The determination coefficient R^2 is measure of the degree of fit and it is ratio of the explained variation to the total variation. Value of R^2 near to unity shows how better the response model fits to the actual results and indicates minor variation in actual values to predicated values. R^2 for ANOVA of Cutting Velocity, Kerf Width and MRR are 98.97%, 95.40% and 99.24% respectively, and all are more than 95%, hence experiment models are at best confidence level and created consistent responses. The results in ANOVA table for the CV and MRR, indicates significance of individual control parameters on the responses, and current is most significant as value of p for it in Table 4 and 6 are 0.008 and 0.004 respectively and below 0.05.

Table 4: Results of ANOVA for CV

Variance source	**DoF**	**SoS**	**Adj. MS**	**F-value**	**P-value**
Pressure	5	0.23530	0.04769	2.26	0.267
Current	5	4.15585	0.70386	33.38	**0.008**
Pulse on time	5	0.36354	0.07852	3.72	0.154
Pulse off time	5	1.33543	0.26709	12.67	**0.031**
Error	3	0.06325	0.02108	-	-
Total	23	6.15337	-	-	-
S = 0.145206 R-Sq = 98.97%					

Table 5: Results of ANOVA for kerf width

Variance source	**DoF**	**SoS**	**Adj. MS**	**F-value**	**P-value**
Pressure	5	0.0016669	0.0003144	4.72	0.116
Current	5	0.0005361	0.0001078	1.62	0.368
Pulse on time	5	0.0011373	0.0002100	3.15	0.187
Pulse off time	5	0.0008056	0.0001611	2.42	0.249
Error	3	0.0002000	0.0000667	-	-
Total	23	0.0043458	-	-	-
S = 0.00816497 R-Sq = 95.40%					

Table 6: Results of ANOVA for MRR

Variance source	DoF	SoS	Adj. MS	F-value	P-value
Pressure	5	0.0081725	0.0015456	5.25	0.101
Current	5	0.0826198	0.0146222	49.64	***0.004***
Pulse on time	5	0.0115301	0.0024234	8.23	0.057
Pulse off time	5	0.0128261	0.0025652	8.71	0.052
Error	3	0.0008837	0.0002946	-	-
Total	23	0.1160323	-	-	-
S = 0.0171628		R-Sq = 99.24%			

3.3 Regression Analysis

It is required to develop mathematical relationships to represent the exact characteristics of control parameters on response parameters. Regression equations for response are generated in terms of input control parameters for this purpose. The significance of regression analysis based mathematical model is found by using statistical ANOVA. The mathematical relationship for correlating the cutting velocity, kerf width and MRR with the considered control variables are obtained as bellows:

The regression equation is

Cutting Velocity for Concentric = 1.27 + 0.080 Pressure (bar)
+ 0.444 Current (Amp)
- 0.00271 T_{on} (Micro sec)
- 0.109 T_{off} (Micro sec) (1)

The regression equation is

Kerf Width for Concentric Dry = 0.137 + 0.0110 Pressure (bar)
+ 0.00476 Current (Amp)
- 0.000030 T_{on} (Micro sec)
+ 0.00278 T_{off} (Micro sec) (2)

The regression equation is

MRR for Concentric Dry = 0.0488 + 0.0266 Pressure (bar)
+ 0.0647 Current (Amp)
- 0.00050 T_{on} (Micro sec)
- 0.00980 T_{off} (Micro sec) (3)

Table 7, 8 and 9 reports estimated coefficient for cutting velocity, kerf width and MRR model and also indicates variable coefficient significance. The table 7 for CV indicates that current and pulse off time is significant except to other as their P-values are less than 0.05. The analysis for MRR was performed and Table 9 shows estimated coefficient and also indicates variable coefficient significance for MRR model. By observing P values of all parameters it is found that except the pressure and pulse on time, remaining parameters are significant as their P-values are less than 0.05.

Table 7: CV Estimated Coefficients

Predictor	Coefficient	SE Coefficient	T-value	P-value
Constant	1.2697	0.6877	1.85	0.080
Pressure	0.0801	0.1288	0.62	0.541
Current	0.44412	0.06441	6.90	0.000
Pulse on time	-0.002710	0.008051	-0.34	0.740
Pulse off time	-0.10945	0.03220	-3.40	0.003

Table 8: Kerf width Estimated Coefficients

Predictor	Coefficient	SE Coefficient	T-value	P-value
Constant	0.13733	0.02985	4.60	0.000
Pressure	0.011004	0.005590	1.97	0.064
Current	0.004761	0.002795	1.70	0.105
Pulse on time	-0.0000298	0.0003494	-0.09	0.933
Pulse off time	0.002775	0.001398	1.99	0.062

Table 9: MRR Estimated Coefficients

Predictor	Coefficient	SE Coefficient	T-value	P-value
Constant	0.04883	0.09120	0.54	0.599
Pressure	0.02657	0.01708	1.56	0.136
Current	0.064725	0.008540	7.58	0.000
Pulse on time	-0.000498	0.001068	-0.47	0.646
Pulse off time	-0.009795	0.004270	-2.29	0.033

ANOVA is used for validation of regression models and model significance is determined by using results of ANOVA. The results for cutting velocity, kerf width and MRR are mentioned in the Tables 10, 11 and 12, respectively. The P values for main performance measure CV and MRR are 0.000. It validates significance estimated regression model at the level of 0.05.

Table 10: CV regression model ANOVA

Source	DoF	SoS	Adj. MS	F-value	P-value
Regression	4	4.6873	1.1718	15.19	0.000
Residual Error	19	1.4660	0.0772	-	-
Total	23	6.1534	-	-	-

Table 11: Kerf width regression model ANOVA

Source	DoF	SoS	Adj. MS	F-value	P-value
Regression	4	0.0015845	0.0003961	2.73	0.060
Residual Error	19	0.0027613	0.0001453	-	-
Total	23	0.0043458	-	-	-

Table 12: MRR regression model ANOVA

Source	DoF	SoS	Adj. MS	F-value	P-value
Regression	4	0.090254	0.022563	16.63	0.000
Residual Error	19	0.025779	0.001357	-	-
Total	23	0.116032	-	-	-

3.4 Residual Analysis

The distribution of data is tested by Residual Normal probability plot *(12)*. The observed value from the regression is found from residual and predicted value. Distribution of data is called normal, if the standardized residual point values are closer to the straight line on plot *(13)*. Further, using the regression equation models, plots of experimental data over predicted data are shown in Fig. 4, 5 and 6 for CV, KW and MRR, respectively. ANOVA analysis and above plots for CV, KW and MRR indicated that the models equations (1), (2) and (3) reflect higher significance and suitable for depicting exact relationship between the control variables and responses, with determination coefficient (R^2) more than 95% and values of P less than 0.05. Additionally, the generated mathematical models for CV, KW and MRR have been validated by residual plot analysis.

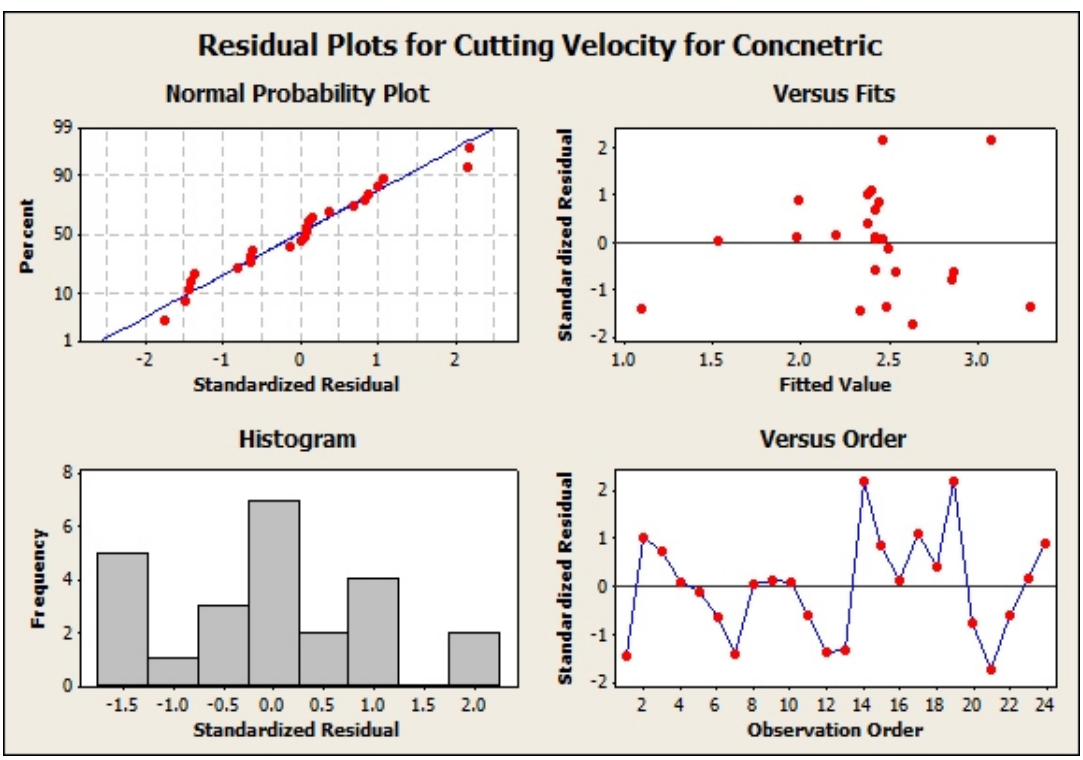


Fig. 9. Residuals plots for response Cutting Velocity
(i) Residual Normal probability plot (ii) Standardized residuals versus the fitted values,
(iii) Residual Histogram and (iv) Standardized residuals versus the order of the data

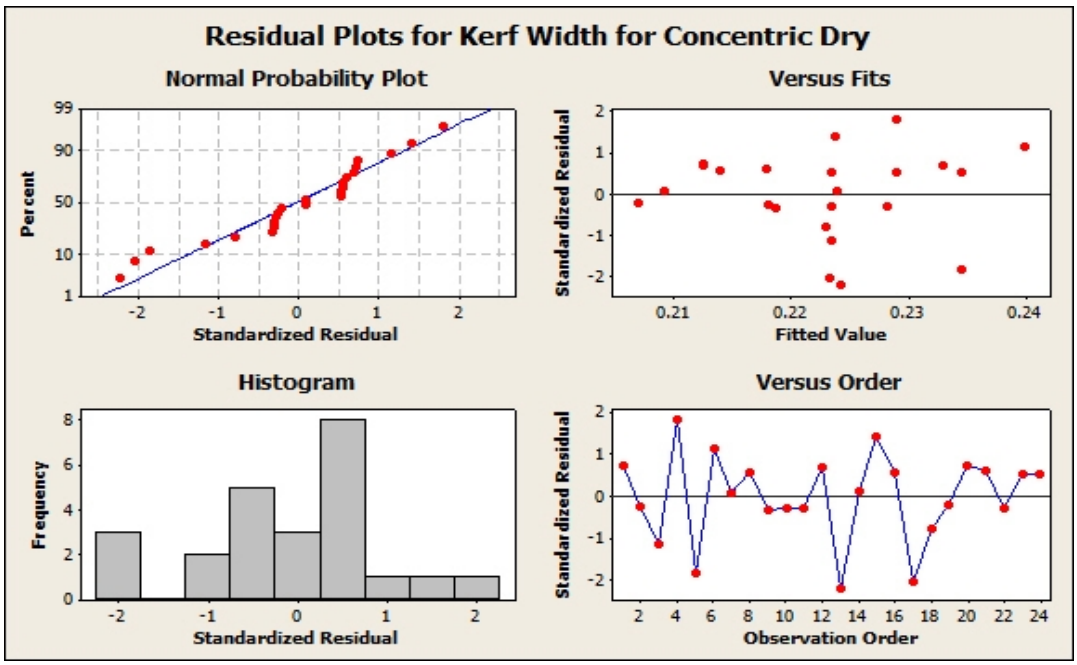


Fig. 10. Residuals plot for response kerf width
(i) Residual Normal probability plot (ii) Standardized residuals versus the fitted values,
(iii) Residual Histogram and (iv) Standardized residuals versus the order of the data

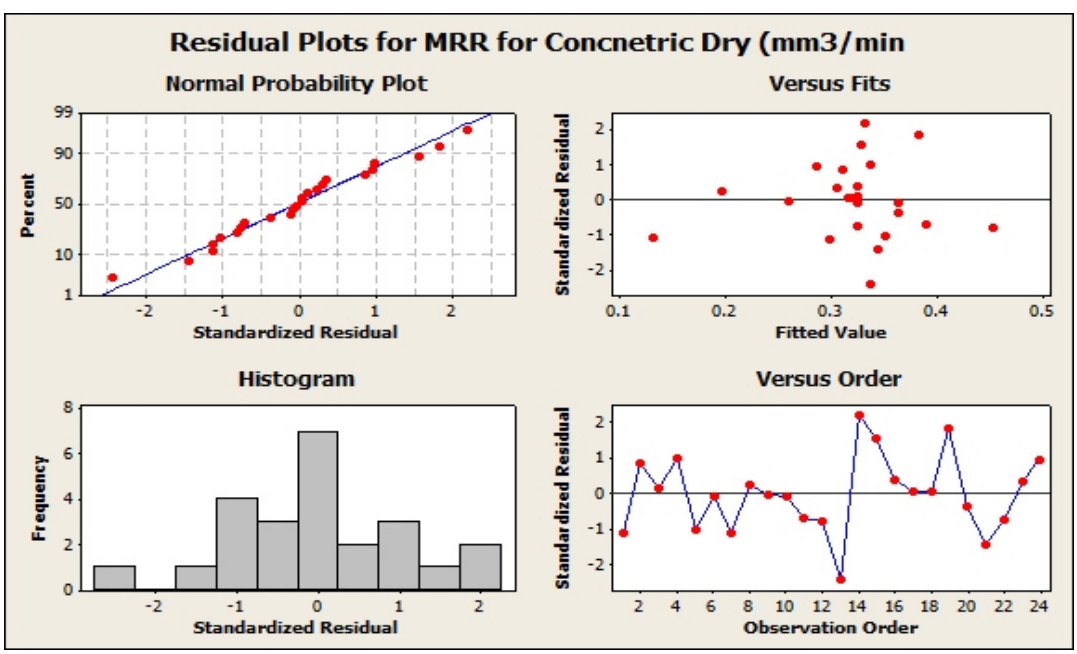


Fig. 11. Residuals plot for response MRR
(i) Residual Normal probability plot (ii) Standardized residuals versus the fitted values,
(iii) Residual Histogram and (iv) Standardized residuals versus the order of the data

Fig. 9, Fig. 10 and Fig. 11 shows the residual plots for CV, KW and MRR, respectively. Part (a) of all figures show normal probability plots, experimental response value are on good correlation with predicted value as plot having approximately straight line for the data and normal distribution for residuals. The residual versus predicted values are plotted in Fig. 9(b), Fig. 10(b) and Fig. 11(b). Part (c) of all figures show residual statistics in histogram plots. Residuals for all 24 experiments in order are shown in Fig. 9(d), Fig. 10 (d) and Fig. 11(d). The existences of definite correlation are affirmed by positive residuals and negative residuals pattern on plot. The models for all responses show adequacies throughout residual plot.

4. CONCLUSIONS

For improvement of performance of dry wire EDM process, authors in this paper have proposed an innovative approach of concentric flow of pressurised air jet in the sparking gap of WEDM process to supply gaseous media using. The proposed concentric flow method is likely to result minimal spark deflection due to impact of air jet, stable wire travel, uniform and effective sparking all over the wire circumference and better debris flushing from the sparking gap. The conclusions from experimental study and analysis are as below.

- Current is most significant parameter for CV and MRR and increase in current increases CV, KW and MRR.
- Lower air pressure results into higher CV.
- Lower pulse pause time results in better cutting CV, lower KW and higher MRR.
- Regression models developed for CV, KW and MRR have P values less than 0.05 indicating that the developed equations are significant.
- Residual plots obtained the regression models confirm the normality of the process indicating the validation of statistical performance of the proposed concentric flow method.

5. ACKNOWLEDGEMENTS

The authors acknowledge support of Prof. Janak B Valaki, Associate professor, Government Engineering College, Bhavnagar for his valuable remarks for improving the quality of the paper. The research project is financially supported by Gujarat Council for Science and Technology (GUJCOST). The authors like to acknowledge GUJCOST support and motivation from Commissonerate of Technical Education, Gujarat state, India for utilizing facilities for this research project.

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Determination of Cycling Speed using Close-form Solution

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ABSTRACT

Cycling speed is very important for customizing bicycle and it play crucial role in improvement of athlete's performance. If cycle frame can be modified such a way that it enhance cycling speed as well as it will add more comfort to the athletes. By changing the angles, length and weight of the frame such as that it is possible to get resultant force in forward direction. This paper represents a theory which is based on close form solution to enhance the speed of the cycling by means of resultant force. For the simulation purpose, here the rolling resistance, frontal area, drag coefficient, tractive force, cycling speed and power consumption studied. Analytical results show that cycling speed can be increased by varying resultant force, air drag coefficient, rolling resistance, frontal area and road grad. This research provides theoretical support to designer to improve bicycle design.

Keywords:Cycling Speed, Angle and length of tube, Resultant force, Tractive efforts,close-form solution

INTRODUCTION

Generally, the bicycle is used for the transportation purpose. Day to day various modifications are done for improving the efficiency of the bicycle and for withstand in a competition. (*Silberman, M.R.,2005 [8]*)

Now a days many options are available in transportation which require less input and make drive comfortable. For increasing the performance and comfort of bicycle modification are require (*Burke, E. 2003 [6]*). In the racing bicycle the speed of the bicycle, efforts and comforts of athlete's are most important (*Hsiao, S.W., Chou, J.R., 2005 [7]*). Many modifications are done on the frame like weight reduction, improvement in the strength of the frame, aesthetic view of the frame etc. Frame modification is done for increasing speed and increasing the comforts of the rider by changing the angle and length of the conventional frame. For understanding the concepts, first understand that, "less efforts are require in cycling on the sloppy road surface". It's happens because of the one component of the weight which is parallel to the road surface that helps to move the bicycle when the tractive reaction is less than this component of the weight. This concept used for modify the frame design. In present paper frame angle and the length are set such a way that resultant force will set in the forward direction. This resultant force helps to pull the bicycle and tractive effort increases. In conventional bicycle it is possible by changing the angle of the seat tube, length of the top tube and down tube in the conventional frame. Here the speed is calculated on the base of close form solution method. The nonlinear equation are formulated for the cycling and expressed in term of velocity (*Junghsen lieh, 2006[1]*).

TRACTIVE REACTION AND CYCLING SPEED

If the resultant force in the vehicle or bicycle is in the forward direction, it increases the bicycle speed. As the closed form solution suggested in (*Junghsen lieh, 2006[1]*) and as shown in fig.1.change in resultant force is depends on the frame design modification. Modification should be such that it gives the resultant force in the forward direction. For displacement of the bicycle, tractive effort should be increase by some percentage of the total resistance. The total resistance is addition of the gradient resistance (F_g), rolling resistance (F_r), air resistance (F_a) and acceleration force (F_i). But here air resistance was not considered.
Total Resistance (F_t) = Rolling Resistance (F_r) + Gradient Resistance (F_g) + Accelerating Force (F_i) – Resultant Force (R)

$$F_t = F_r + F_g + F_i - R \quad (1)$$

$$F_a = \frac{\rho}{2} C_d A_f V^2 \quad (2)$$

$$F_g = W \sin\alpha \quad (3)$$

$$F_r = f_0 W \quad (4)$$

$$F_i = m\frac{dv}{dt} \quad (5)$$

Tractive force is near peak value such that maximum the

$$Ft = \mu_p N \quad (6)$$

Where, W is gross weight (N) and m is mass (Kg) of bicycle including rider's, ρ is density of air, A_f is frontal area, C_d is air density coefficient, v is velocity, α is grade coefficient, g is gravity constant, $f_o$is rolling coefficient. Tire traction force can be calculated by following formula no. (6). Where μp is peak coefficient of tire or friction coefficient of the tire/rod, N is normal reaction load on the driving wheel. Here R_b and N both are same for our conveniences we use R_b.

Taking the moment about point oin fig.1.without considering airlift, so the normal load on the driving wheel is

$$R_b = \frac{WL_a + F_a h_a + mh\frac{dv}{dt}}{L} \quad (7)$$

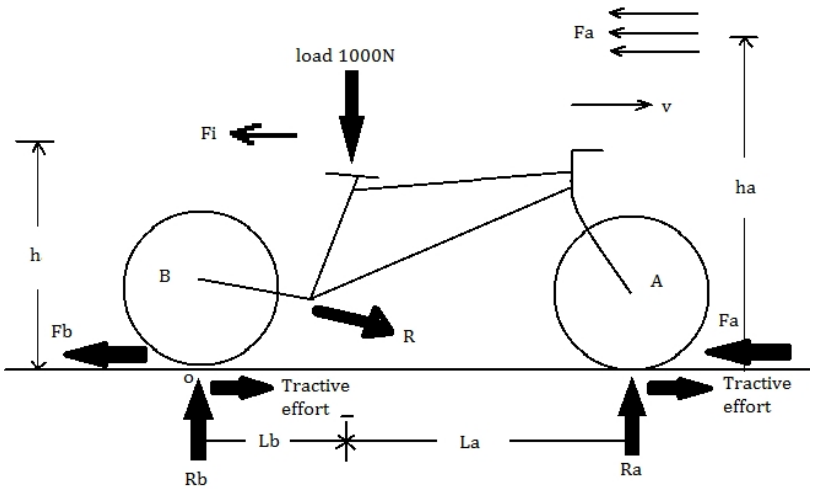


Fig.1. Balancing of force during cycling

Substituting equation of forces (3), (4), (5), (6) into Eq.(1), then the equation can written in following form:

$$x_1\frac{dv}{dt} = x_2 - x_3V^2 \quad (8)$$

Where, the expression for X_i are

$$x_1 = m\left(1 - \frac{\mu_p h}{L}\right) \quad (8i)$$

$$x_2 = W\left[\frac{\mu_p L_a}{L} - f_0\right] + R \quad (8ii)$$

$$x_3 = \frac{\rho}{2}C_d A_f\left(1 - \mu_p\frac{h_a}{L}\right) \quad (8iii)$$

Eq.(8) represents the tire traction capability of a bicycle when its power availability is unlimited. To determine the relation between speed and time, it is require to integrate the Eq. (8) with respect to time. By close- form integral equation of the speed obtain as follow:

$$v = \frac{\sqrt{x_2}}{\sqrt{x_3}}\tanh\left[\frac{\sqrt{x_2x_3}}{x_1}(t - t_0) + \tanh^{-1}\left(\frac{\sqrt{x_3}v_0}{\sqrt{x_2}}\right)\right] \quad (9)$$

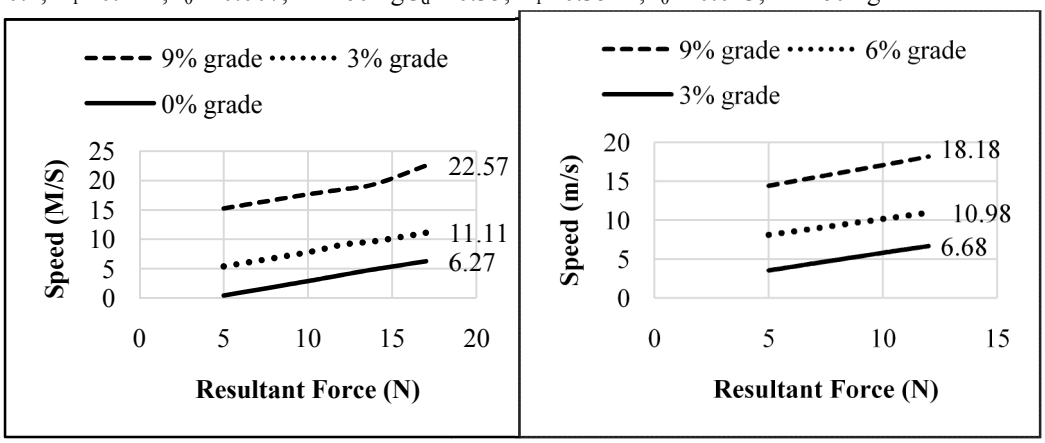
Where, V_0 is initial velocity at time t_0. Starting condition of the bicycle is rest, i.e. $V_0 = 0$ at t_0, the velocity can be expressed as:

$$v = \frac{\sqrt{x_2}}{\sqrt{x_3}} \tanh\left[\frac{\sqrt{x_2 x_3}}{x_1} t\right] \quad (10)$$

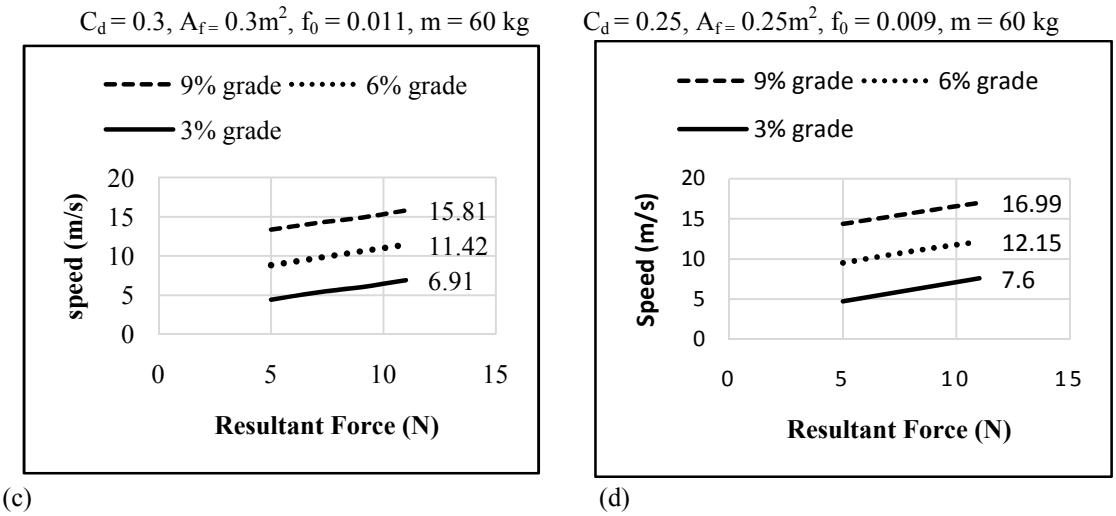
RESULTS AND DISCUSSION

Bicycle speed depends on the resultant force, air drag coefficient, rolling resistance coefficient, frontal area and rod grade. As weight of rider varies resultant force will also change. Here for the sake of simplicity it is assumed that rider weight remains constant and only resultant force will change. Varies graph can be obtained by putting the different values in Eq. (10). The results are as shown in Fig.2. As shown in Fig.2 (a) speed is calculated by considering the air drag coefficient $C_d = 0.2$, frontal area should be $A_f = 0.2m^2$, rolling resistance $f_0 = 0.007$, wheel base length take 1.3m and road grade consider 9%, for the 60 kg racer frame should modify such as it gives speed 15.25 m/s for 5N resultant force, and at 15 N resultant force it will rise the speed up to 20 m/s.

$C_d = 0.2$, $A_{f=} 0.2m^2$, $f_0 = 0.007$, m = 60 kg$C_d = 0.35$, $A_{f=} 0.35m^2$, $f_0 = 0.013$, m = 60 kg



(a) (b)



(c) (d)

Fig.2. Resultant force Vs Speed

Fig.2 (b) to (d) shows the different speed obtained by changing air drag coefficient, rolling resistance coefficient, frontal area and rod grade. Fig. 2 (a) and (b) shows that as the air drag coefficient andfrontal area consideredas 0.2, obtain speedcan increase up to 20 m/s. But if these values increase and consideredas 0.35 the highest speed can be obtained at 15N resultant force is 18.18 m/s. Bicycle speed is also depends on the road gradefor example as shown in Fig.2 (a) the variation in speed for different road grade is 5 to 10 m/s . As the quality of the road decrease i.e. road grade decrease it will decrease the considerable speed. Fig.2 (c) and (d) also predict the same result for different values of the same parameters.

CONCLUSION

Athlete performance mainly based on the proper bicycle frame design.To design appropriate bicycle frame it is necessary to consider various parameters affecting it. By selecting appropriate frame it is possible to increase the bicycle speed. This paper has outlined the variation in the speed of the bicycle by changing various parameters like air drag coefficient, rolling resistance coefficient, frontal area and rod grad. As the resultant force in frame increases, there is increment in speed of cycling for deferent road grade.Result predicts that for the same road drag it is possible that the desired speed can be obtain by changing the other parameters i.e. by changing bicycle frame design. Based on this solution it is easy to calculate the speed and modifying the frame. This paper could be useful for further modifications in bicycle frame and increasing the speed of the racer. It provides theoretical supports to develop and design the bicycle.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

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Adsorption Refrigeration System: Components and its design

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ABSTRACT

Cooling systems like vapour absorption or vapour adsorption refrigeration system ,which runs on low grade energy sources provide favorable alternatives to the conventional vapor compression refrigeration system. These system do not contribute to global warming, depletion of fossil fuels or ozone layer depletion as there is absence of conventional mechanical compressor and use of Eco-friendly refrigerant. Among heat operated systems, some vapour absorption systems have been in recent use in industries ,which is driven by waste heat ,engine exhaust ,solar energy etc. Adsorption refrigeration systems depend on successive adsorption and desorption of refrigerant in a cycle to produce the cooling effect. The work includes design of suitable adsorption refrigeration systems with defined adsorption cycle and adsorption material. In order to make it solar based system, heat flux absorber surfaces also need to be designed. As part of extended work, the system should be compatible to rural usage.

SUMMARY

Adsorption refrigeration system would steer towards eco-friendly way of cooling for rural area.

Keywords: Adsorbate, Adsorbent ,Adsorption chillers.

1. INTRODUCTION

The World are facing global climate change and day by day ,these Changes become more and more severe .There is a drastic change in seasonal variation even in Asian country also. Besides that need of air conditioning &refrigeration are increasing .Conventional cooling technologies are generally electricity driven customary vapor compression refrigeration systems which usually operated with artificial refrigerant[*1*] .Such as CFCs ,HCFs or HFCs, they causes the ozone layer depletion and/or cause

Nomenclature

P_{ev}	Evaporator Pressure(kPa)	ϱ_v	Vapour density(kg/m^3)
T_{ev}	Evaporator Temperature(K)	h_{fg}	Latent heat of vaporization(kJ/kg)
P_{con}	Condenser Pressure(kPa)	G	Gravitational force(m/s^2)
T_{amb}	Ambient Temperature(K)	h_i	Inside convective heat transfer Coefficient(W/mK)
T_{con}	Condenser Temperature(K)	h_o	Outside convective heat transfer Coefficient(W/mK)
T_{gen}	Regeneration Temperature(K)	$m_{a.c}$	Mass of activated carbon(kg)
T_{ad}	Adsorption Temperature(K)	$m_{adsorbent}$	Mass of adsorbent(kg)
L	Length(m)	K	Thermal conductivity(W/m^{2}K)
ϱ_l	Liquid density(kg/m^3)	U	Universal heat transfer coefficient(W/mK)
θ^*	Adsorbate volume fraction	W_0	Maximum adsorption capacity(kg/kg)
X	Adsorbate concentration ratio	D,n	Constants of D-A equation

greenhouse effect.[*2*] To control the emission of these refrigerants several protocol ,like the Montreal protocol or the Kyoto protocol, were established[*3,4*].due to increasing emissions it became necessary to find alternatives ways for refrigeration which can run on eco-friendly way.[*5,6*]. Moreover increasing energy consumption worldwide makes it vital to find new ways to use the energy resources in a more efficient and sensible way[*7*].In addition ,fossil fuel resources provides 80% of the energy on earth[*8*].

Most commonly used vapor compression refrigeration cycles are electrically powered ,which needs high grade source energy such as fossil fuel; which are the major causes for ozone layer depletion and greenhouse effect. In another view, there is large population lives in remote areas where there is shortage of electricity or still not existence. In order to fulfil need of refrigeration in these areas ,development of non-conventionally driven system is vital .Traditional refrigeration equipment cannot be used ,for instances ,in storage of food, medicines for its preservation ,ice making, or even for air conditioningTherefore location challenges like in remote areas, scarce of fossil fuel, energy saving makes it urgent to develop new Technology steered the human kind to look with better interest for ecological and renewable energy sources like solar ,hydropower , wind ,tidal, biomass and geothermal energies, or even thermal waste from various processes.

Currently solar energy is a subject of great interest, and refrigeration is a particularly attractive as adsorption system switch over adsorption and desorption period ,so it need energy in intermittent period (in case of intermittent cycle) and solar energy is also in intermittent nature. Recently, adsorption refrigeration processes have been scrutinized(theoretically and experimentally) and proposed as an substitute to conventional vapor compression refrigeration systems , while attempts are on to preserve the efficiency level and performance of system are same as traditional system ,and becoming one of the most promising solar refrigeration methods[*4*].Adsorption systems are costly ,and have some technical drawbacks,such as low coefficient of performance(COP),low specific cooling power(SCP), and poor heat and mass transfer on the adsorbent beds ,which makes the systems more massive[*10,11*].moreover ,Solar energy is highly irregular and intermittent in nature .However ,these systems saves significant amount of

energy in comparison with common mechanical vapor compression refrigeration systems ,and have artless control ,no noise ,no vibration lower operation and maintenance costs ,simpler and more robust and most importantly it has a lower environmental impact[*11*]. While Comparing with the absorption systems ,adsorption systems can be run over a large range of heat source temperatures for hot,cool, and cold water, low operational and maintenance costs , low carbon emissions ,no crystallization , no corrosion hazardous Leaks, no vibration or noise, or chemical disposal issues.[*11,12*].therefore, the adsorption refrigeration systems appear as a good alternatives to replace the traditional refrigeration systems by more environmentally friendly systems ,which can be powered by solar energy ,waste heat etc. these system paying more attention to the researchers worldwide to improve the performance of adsorption cooling systems in order to overcome its current technical and economic issues.

2.THE PROCESS

2.1 Principles of adsorption : The adsorption is a surface phenomenon, which result from the adhesion between a solid and a fluid (refrigerant) based on a physical or chemical reaction. In this process molecules of one substance stick to the surface of another substance .There is no penetration into the bulk of the base substance.The molecules which are in adsorbed state are called adsorbate and the solid surface on which the adsorption takes place is called the adsorbent. In physical adsorption molecules of adsorbate fix themselves at the surface of a porous solid element (adsorbent).Vander walls forces and electrostatic forces are responsible for their bonding. This process can be reversed in which adsorbate molecules can be released (which is called desorption process), which is happened on application of heat. In order, in the chemical adsorption ionic or covalent bonds between the adsorbate molecules and adsorbent are responsible for their bonding. In this chemisorption forces are much greater than that of physical adsorption.so in this case larger amount of physical adsorption .So in this case larger amount of heat is require for desorption .however in chemical adsorption , the process cannot be easily reversed. Besides ,this type of bonding promotes the chemical variation of the adsorbed substance ,thus the adsorbate and adsorbent molecules altered or get change after adsorption .Therefore ,most of the adsorption refrigeration systems mainly involve physical adsorption[*12,14*].These cycle alternating between the successive adsorption and desorption stages ,so provide the cooling effect intermittently. But, when continuous cooling effect is required ,two or more adsorbent beds must be operating out of phase, for that heat source that's is always available is required ,that is not possible in the case of solar based system because of its intermittent nature.

2.2 Basic solar adsorption refrigeration cycle

Phase 1: Isosteric heating (Process 1-2):
This is the first phase of the cycle. In this phase, the adsorbent bed (reactor) which is already saturated with the adsorbate (refrigerant) is heated. The temperature and pressure of the system rise. The concentration of the adsorbate remains constant.
Phase 2: Isobaric desorption and condensation (Process 2-3& 2-C):
In this phase, desorption of the adsorbate takes place and simultaneously, the condensation of the adsorbate in the condenser takes place at the condenser pressure.

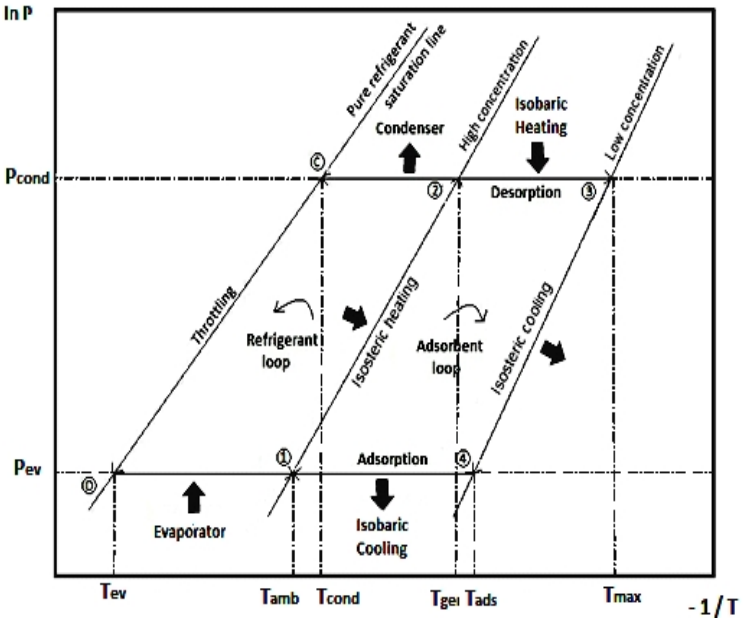


Fig.1:Theoretical adsorption cooling cycle

Phase 3: Isosteric cooling (Process 3-4& C-D):
In this phase, the adsorber bed starts to cool down. The system temperature decreases and due to this the internal pressure of the system also decreases.
Phase 4: Isobaric cooling and evaporation (Process 4-1& D-1):
In this phase, the liquid refrigerant enters into the evaporator and starts boiling at a low pressure (evaporator pressure). Simultaneously, the vaporized refrigerant is allowed to enter into the adsorber bed and it gets adsorbed in the adsorbent. This process completes the cycle.

2.3 Applications of the adsorption process:

•Solid adsorbents ,in combination with suitable adsorbate can be used in air separation systems to separate gases.

- The principle of adsorption is used in refrigeration cycle to provide air conditioning or for ice-making purposes. Several companies have successfully commercialized adsorption chillers.
- Desiccants such as silica gel and zeolite are also used in many systems to extract moisture from the air and prevent damage to products. Such as medicines,shoes etc.
- For vacuum preservation ,sugar clarification, In paint industry, In softening of hard water.
- In Petrochemical industries, In chemical plant ;CO_2 and water removal from air in air separation plant.

3.MATERIALS AND METHODS

3.1 Design of System components :

Design parameters:
Global solar radiation: 276 W/m^2
Adsorbent/Adsorbatepair :A.C.F/methanol

Condensing temperature : 35 ˚C
Generation period :6 h
Adsorption period :12 h
Ice temperature : -5 ˚C
Average ambient temperature : 30˚C
Maximum generation temperature : 100-150˚ C
Evaporating temperature :0 ˚C

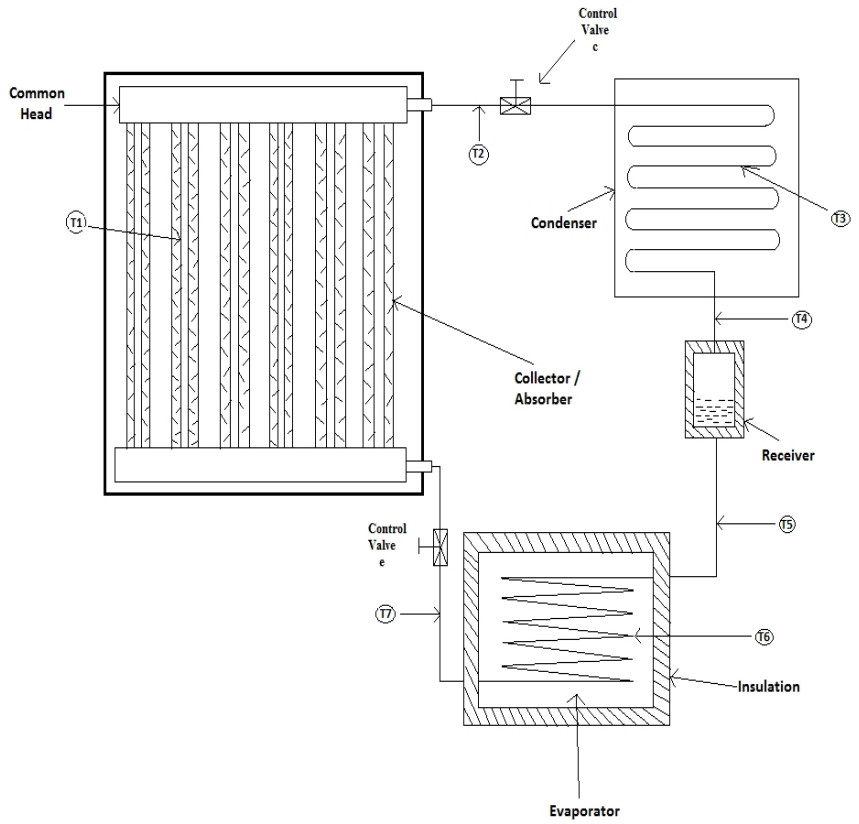


Fig 2.Schematic diagram & Experimental setup
of adsorption refrigeration

Heat load calculations :

Heat load or refrigeration load is the rate at which heat must be removed from the area to be cooled in order to produce and maintain the desired temperature conditions

Wall heat gain load: Heat will be lost from cold box by conduction through walls.
The wall heat gain is given by:
Q = AUΔ T
Where, A = Outside heat transfer area
T = Temperature difference between cold box Interior& exterior plus a
temperature allowance for solar radiation

U = overall heat transfer coefficient

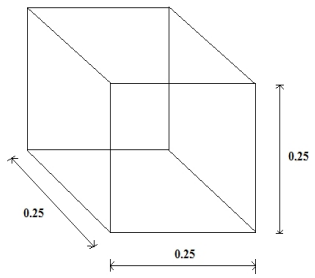


Fig. 3 Cold box design dimension

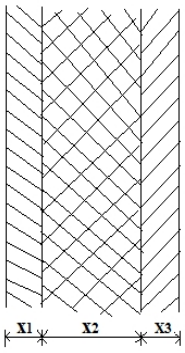


Fig. 4 Cross section of cold box

Now $\frac{1}{U} = \frac{1}{fo} + \sum_{i=1}^{n} \frac{X}{K} + \frac{1}{fi}$

$= \frac{1}{fo} + \frac{X1}{K1} + \frac{X2}{K2} + \frac{X3}{K3} + \frac{1}{fi}$

Where, X1,X2,X3 are the thickness of G.I, Insulation and Al respectively

h_o,h_i are cooling factor for outside and inside respectively

K is thermal conductivity

U = 1.2112 W/mK

T_{amb}= 35 ˚C

T_{design}= -5 ˚C

Refrigeration load allowance for solar radiation where 3, 4, 4, 4, are solar temperature allowances for N, S. E, W respectively.

Now according to **Q** = AU Δ T

Q_N= 3.2702 W

Q_S= 3.2551 W

Q_E =3.3308 W

Q_W = 3.3308 W

Q_{roof}= 3.2702 W

For floor, U need to be calculate separately as box will be held at bottom so h_0 will be neglected in this case.

U_{floor}= 1.2795 W/mK

Q_{floor}= 3.4546 W

So, Total heat conducted through wall can be given as

$Q_{walls} = Q_N + Q_S + Q_E + Q_W + Q_{floor} + Q_{roof}$

Product load: It is the load of product that is put inside box to achieve desired temperature .It is usually calculated for 24 hours period .With product ,container also itself cooled with it.

$$= \frac{\text{Amount of heat removed to cool the water from } 25\,^{\circ}\text{C to } 0\,^{\circ}\text{C}) + \text{Latent heat of fusion of ice at } 0\,^{\circ}\text{C} + \text{Amount of heat removed to to cool the ice from } 0\,^{\circ}\text{C to} -10\,^{\circ}\text{C}]}{\text{time period}}$$

Service load :The service load or heat usage load is the sum of the heat loads of : cooling the contents to cabinet temperature; cooling of air changes; removing field and inhalation heat from fresh produce; removing heat released from electrical lights and motors; and removing heat given off by people entering and/or working in the storage room per unit of time(usually 24 hours)

For current case service load is not much concerned, as purpose of this work is ice making in cold box.
Total cooling load can be given by,

Total cooling load $=Q_{wall} + Q_P + Q_{usage}$

3.2 Design of an evaporator :

The evaporator should be made from very high conducting material of copper.
For a copper pipe with standard dimensions:
Outer diameter D $= 9.375 \times 10^{-3}$ m
Outer Radius R $= 4.688 \times 10^{-3}$ m
Inner diameter, d $= 6.25 \times 10\text{-}3$ m
Inner radius, r $= 3.125 \times 10\text{-}3$ m

$Q_{evaporator} = Q_{total\ cooling\ load}$
$= 2\pi r L h_i[t_i - t_o]$
where, h_i = convective heat transfer coefficient
L = Length of evaporator
$t_i - t_o$=Temperature difference inside &outside of evaporator coil

Now according to chatoequation,nusselt number in the case of refrigeration given by

$$Nu = 0.555\left[g\rho_v \frac{(\rho_L - \rho_v)d^3 h_{fg}}{\mu_v k\ \Delta T}\right]^{1/4}$$

And Nu can be relate to hi by following equation

$$Nu = \frac{hid}{k}$$

Now according to newton law of cooling,
$Q = hA(t_i - t_0)$
$= hi\ (2\pi rL)\ (t_i - t_0)$

Now ,
$$L = \frac{Q}{2\pi r hi\ [ti - to]}$$

3.3 Design of an Condenser :

By applying heat balance equation,

$Q = 2\pi rLh_i[t_i - t_o] = 2\pi rLh_i[t_R - t_C]$

For film condensation inside horizontal tubes,chato equations holds,

$$h = 0.555\left[\frac{\rho_L(\rho_L - \rho_v)gk^3h_{fg}}{\mu d(T_g - T_w)}\right]^{1/4}$$

Similar to evaporation calculation,

$$L = \frac{Q}{2\pi rhi\,[ti - to]}$$

3.4 Quantity of refrigerant required :

Cooling load from the evaporator is taken in form of sensible + latent heat of circulating refrigerant.

$Q_{cooling\ load}$= Total heat (Sensible heat + Latent heat) of circulating refrigerant in evaporator coil
= $m_{ref}C_p\Delta T + m_{ref}$ (Latent heat)

So, $m_{ref} = \frac{Q * cooling\ time}{Cp\Delta T + (LH)}$

Adsorption capacity of Activated carbon is 30 %

$m_{adsorbent} = m_{ref} / 0.3$

3.5 Design of an adsorption unit:

Adsorption unit is the main heart of this system. Compared to conventional VCR system,this unit replace the compressor.So the pressure & temperature rise from evaporator to condenser is achieved in this unit .Cross section is shown in figure 4.In which adsorbent is packed in annular space, which is enclosed by metal casing. At the centre there is passage for refrigerant to flow.

Density of A.C = 400 kg/m^3

$M = \varrho V$

Volume of adsorbent = $\frac{M}{\varrho}$

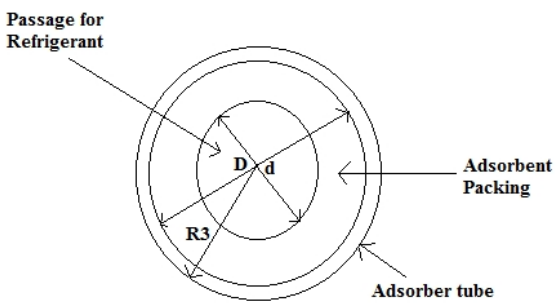


Fig. 4 Cross section of adsorption

Volume of each annular space given by

$V=\frac{\pi}{4}(D^2-d^2) L$

From literature ,unit gives best result when thickness of adsorbent bed is in between 1.5 to 2 cm.

So here we are considering 2 cm

So D-d= 4 cm

= 0.04 m

D = d+0.04

L = 1.5 m

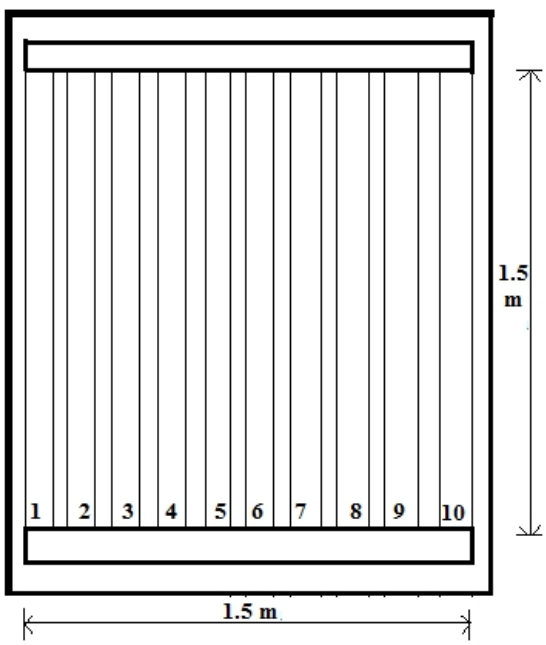


Fig. 5 Adsorption unit

4.RESULTS

Table 1.Design parameters

Parameter	**Value**	**Unit**
Wall heat gain load(Q_{wall})	19.8048	W
Product load (Q_p)	160	W
Total cooling load	180	W
Cooling period	12	Hours
Collector area	(1.5*1.5)	m^2
Average Solar irradiation	276.25	W
Cooling capacity(design)	10 (ice)	Kg
Refrigerant(methanol)	6.439≈ 7	kg
Adsorbent(A.C)	23.33≈24	kg

Length of condenser	11.22≈11	m
Length of evaporator	5.31≈5	m
Desorption Temp. (t_d)	100-130	˚C
Adsorption Temp.(t_a)	35-40	˚C
Condensation temp. (t_c)	40-45	˚C
Evaporation temp. (t_e)	-5 - 0	˚C
Condensation pressure (p_c)	20-25	kPa
Evaporation pressure (p_e)	2-5	kPa
Adsorption tube (O.D)	8.63	cm
Adsorption tube (I.D)	4.63	cm
Adsorption unit length	1.5	m
Adsorption unit width	1.5	m

5. CONCLUSION

Success of adsorption unit greatly depends on selection of working pairs .Different working pairs have been analysed and suitable pair is selected for stated application .As well as design of different components of these system have been calculated. All these works will help in developing a eco-friendly adsorption refrigeration system. In our opinion, the adsorption technology would steer towards development of refrigeration technology for rural usage.

ACKNOWLEDGEMENT

It is with immense pride and pleasure to express my sincere gratitude to my guides(Prof.) Dr. N.S.Mehta&JaspalDabhi, for his encouragement and constant help throughout This research work right from its inception. Authors also thankful to The principal and Head of the mechanical engineering department of L.D.R.P-ITR, for their kind support and necessary guidance.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

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RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

An Attempt to Enhance Data Security for the Digital Locker System (An Initiative by the Government of India) by incorporating the "Digital Locker Security Enhancement Model"

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ABSTRACT

The Digital India program envisions the creation of digitally empowered economy and e-governance and services on demand to improve access of information as well as resources for citizens. Any security breach in initiative will break moral development and also raise major concerns about privacy and security of confidential data. Prime Minister Shree Narendra Modi wants to make India - Digital India, so as to reduce the paper work anddigitalize it. There are several security issues which can be raised when implementing an application live in Digital India project. So, this paper will particularly focus on DigiLocker application and security mechanism implemented for it. To enhance the current mechanism of Data security,this paper has proposed one novel model for Data security of DigiLocker.

SUMMARY

The implementation of secure Digital India will need to adopt an end to end approach like never before. For security and privacy purpose, proposed model will enhance security features then now. As a nation, Government could not take risk for unsecure version. Security feature will make bolder then before.

Keywords: Digital India, Digital Locker, Data Security, OTP code, Random Generated Questions

INTRODUCTION

"Digital India is a Programme to transform India into a digitally empowered Society and Knowledge economy"*[1]*. Digital India is Umbrella program where a single project covers multiple departments. The Weaving together makes the mission transformative in totality. There are many schemes which is currently working under government of India. In this program every schemes are re-focused or restructured. High
Accessible digital resources, All documents and certificates should be available on cloud etc… are few of major functions in Digital India program.

MATERIALS AND METHODS

Digital India Program launched on 1st July 2015 by Prime Minister of India – Shree Narendra Modi. Digital India has three core components which are:

- Creation of digital infrastructure.
- Delivering services digitally.
- Digital Literacy.

This schema will be monitored & Controlled by Digital India Advisory Group which will be chaired by theMinistry of communications and IT. Some ofthe facilities which will be provided through this initiative are Digital Locker, e-education, e-health, e-sign and national scholarship portal.

DigiLocker:
DigiLocker is a service launched by Government of India in February 2015 to allow secure dedicated electronic space for storing personal documents. Currently it allows you to upload data up to 10MBonly. Later on DigiLocker will allow up to 1 GB. Here, you can store importantDocument like voterid, PanCard, Property documents, Marriage Certificate, EducationalCertificates etc... If we look theSecurity Mechanism then following is the Signup process for DigiLocker. Following are steps which described in Figure 1

Step 1: Input Aadhar Number

Step 2: If Aadhar number is verified then OTP will be received in Mobile or in Inbox of your mail id.

Or

If Aadhar number is verified then you need to give your thumb impression to finger print device.

Step 3: If entered OTP is correct then system will ask username and password.

Or

If your thumb impression to finger print device is match then system will ask username and password.

Step 4: Upload your data.

Above mentioned steps shows that the application is easy to use. So, there are several advantages of it. Following are Advantages of DigiLocker.

Advantages of DigiLocker:

- It will ensure authenticity of the e-documents so, now elimination of usage of fake document.
- All your e-documents are available 24*7 and anywhere in world.
- Architecture of this service is easy. So, it is easy to share documents across different departments.
- Personal storage space is enough for individuals to store their important data.
- People can see the list of documents issues to any company or department and requestors who accessed your documents.
- This service is free.

With above all above mentioned advantages, there is lack of Strong security feature. If anyone want to access any other person's data, then it is easy to access by unauthenticated person.Moreover, there are certain disadvantages related to Data security. Following are disadvantages of DigiLocker.

Disadvantages of DigiLocker:

- Trust is important factor while submitting your important documents on web. Here, your data will not be breached by Government or someone else is the only major problem.
- Sign up process is very simple because you only need an Aadhaar number. Here, for more security of your data Fingerprint is best option but what about OTP? If OTP is received by email then email id is known to anyone and password can be cracked by anybody. There are various software also available in market for cracking password. So, this is not good security method.

So, to overcome disadvantage of DigiLocker, We proposed following Model (shown in Figure 2)

Before using this model, Signup process should be performed. Information like First name, Last name, Middle name, DOB, Place of birth, Mobile number printed in an Aadhar card, Mobile number in which you want OTP code and randomly generated question and Email id Registered in an Aadhar card will be stored. If provided mobile number for getting OTP and answer of question is different than printed in an Aadhar card then confirmation mail will be delivered to user's registered id and OTP code also sent on that email id. Now, if user enters correct OTP code then system will send OTP code and Question to new mobile number. If entered information is correct then person can retrieve data and he can also upload files in this system.If entered information is not correct then again request for OTP and answer of randomly generated question should be correctly entered to access this feature.

RESULTS AND DISCUSSION

Proposed model have following advantages over currently applied model.

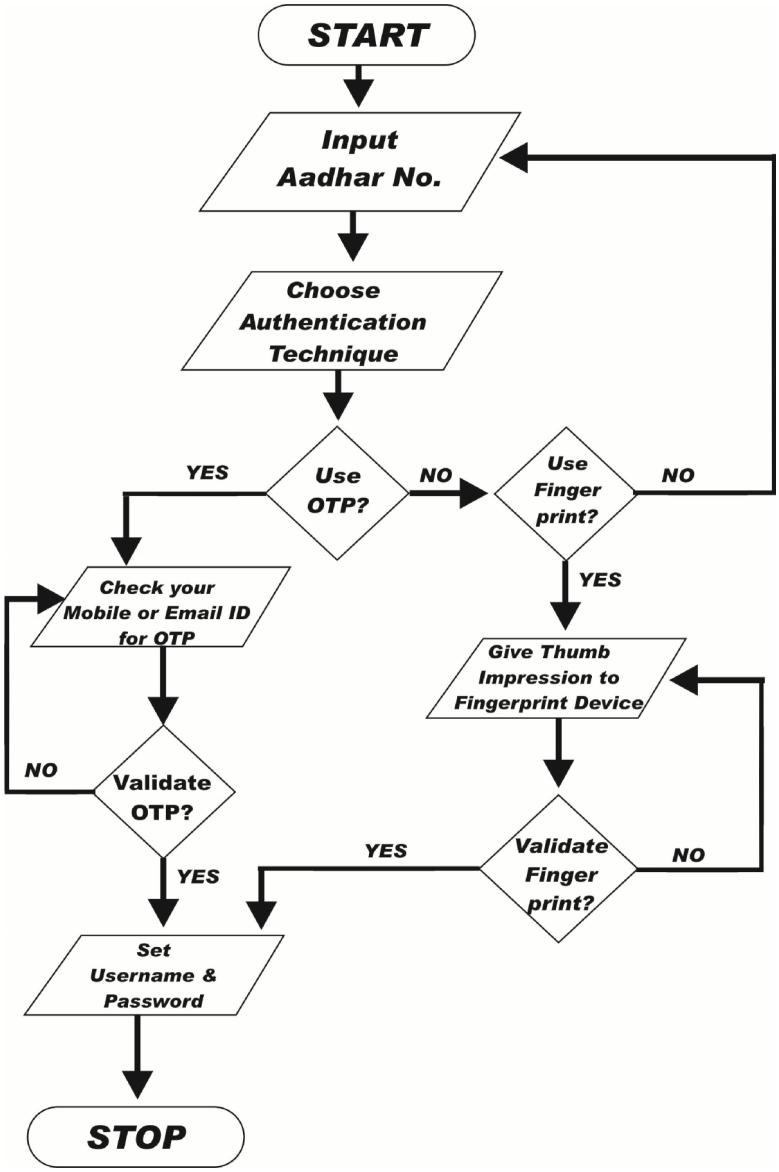
- OTP mechanism is applied with random question generation. So, now if user got OTP code by any way then also he can't predict answer of randomly generated question. So, here one more layer of security is introduced.
- Trust level will be increased because of more secure model will be implemented.

Main drawback of proposed model can be if any unauthenticated person knows you more closely then he/she can predict your answer of randomly generated question.

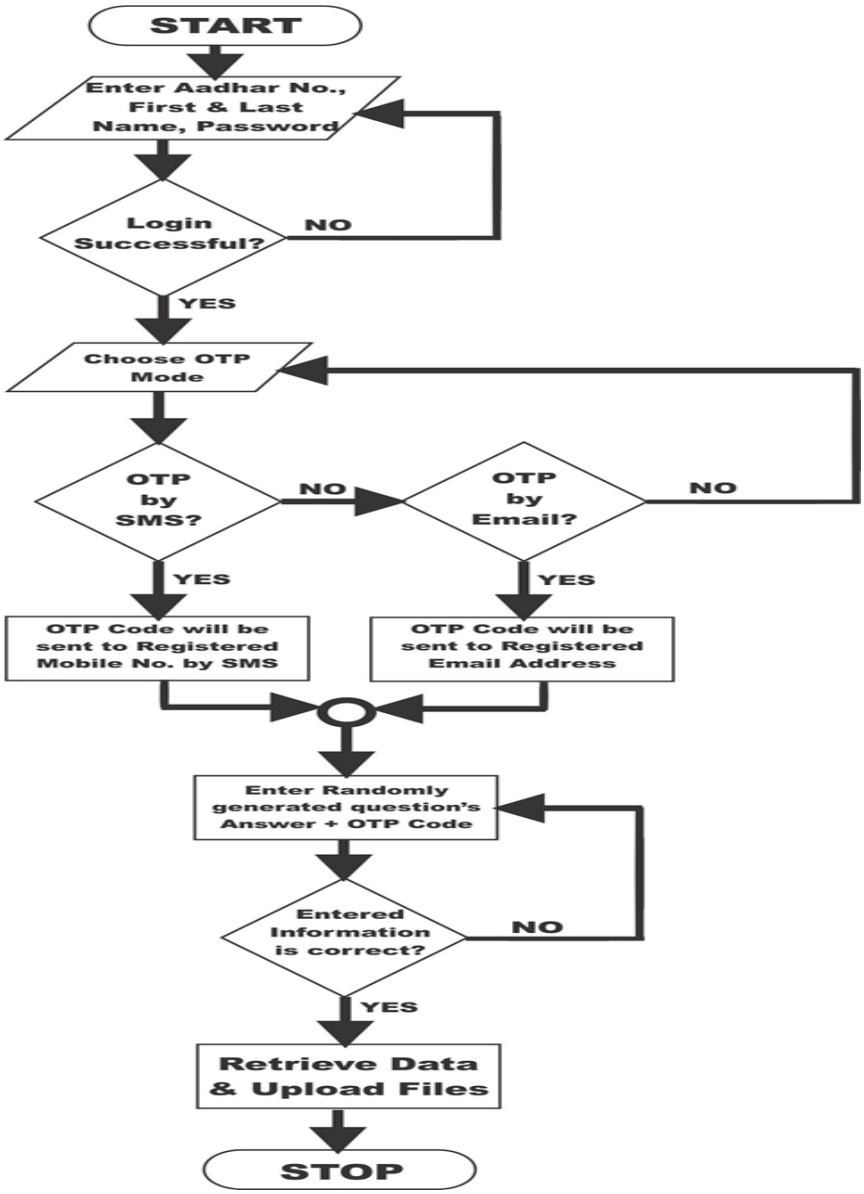
CONCLUSION

Government of India initiate to centralize data on cloud. Current mechanism to secure digital Locker and its document is good but enhancement is necessary. So, to secure strongly, "Digital Locker Security Enhancement Model" will enhance security mechanism. Here, randomly generated question is asked tousers. Only OTP is not sufficient. This random generation of question add one more layer towards data security. Proposed system is not costly and also easy to implement.

FIGURES



[Fig 1: Signup process for Digital Locker]



[Fig 2: Model for Enhanced Data Security mechanism for Digital Locker]

ACKNOWLEDGEMENT

We would like to thank Mr. Raghu Khimani – Cyber Crime Expert for his valuable suggestions.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

(Proceedings available for download at rku.ac.in/icre)

RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

Today's Security challenges on web and protection policy for secure web communication in Service Oriented Architecture

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ABSTRACT

Information security is a most challenging aspect during online transactions and communication. Organizations, Government, Social or Individual every persons are facing security risks. About 80% of transactions done through online web services, but it is not safe and reliable. Online services are managed automatically, without human interaction, developed by multiple stake holders on the base of SOA (Service Oriented Architecture). A new threat identification approach and guideline will be helpful to build a quantitative security risk model for information systems.

SUMMARY

To help in reducing cyber crime and providing safe online transaction.

SOA, TFA, VPN, SSL, SET

INTRODUCTION

Now a days, many people are attached with each other using web technology. Many service providers deliver facilities to exchange of ideas, information, videos, pictures, and graphics based on SOA. It also allows easy sharing and distribution of existing content to others, due to that professional work can be shared through on-line networks [10].
Using Social networking websites maximum people share or transfer images, video clips, text and personal details without any precautions and bothering about fraud. On-line transactions are done without

any security check because many of them do not have awareness about on-line fraud and cyber crime. Thus, hackers can easily hack and misuse their information. The issues include privacy issues, identity theft[6], social networks spam, social networks malware, and physical threats.[9] There are certain issues regarding on-line fraud are describe as below,

- **Hacking:** This is a type of common crime, in which a person's computer is becoming out of order so that his/her personal and sensitive information as well as the entire device can be accessed by unauthorized person. In hacking the criminal, uses variety of different software's to enter into a person's computer unknowingly without his awareness**.** [7][14]
- **Theft:** This crime occurs when person violence copyrights laws by downloading music, movies, games and software. Generally, license version software is costly hence culprit person can crack its license software and use for profit. To use cracked software, company's logo, domain name and idea of good name websites for misguide people is also consider as crime.[7][6]
- **Cyber Stalking:** This is a kind of online harassment wherein the victim is subjected to a bombardment of online messages and emails.[5] Typically, these stalkers know their victims and instead of alternative to offline stalking, they use the Internet to stalk. However, if they notice that cyber stalking is not having the desired effect, they begin offline stalking along with cyber stalking to make the victims' lives more depressed.[7]
- **Identity Theft:** This has become a major problem when people use the Internet for money transactions and online banking services.[11] In this cyber crime, a criminal accesses data of a person like bank account, credit & debit cards details, Social security and other sensitive information to draw off money or to buy things online on the victim's name. It can result in major financial losses for the victim and even spoil the victim's credit history also. [3][8]
- **Malicious Software:** These are Internet-based software's or programs which are used to disturb the entire network. The software is used to gain access to a system to steal sensitive information or data or causing damage to software present in the system. While surfing such websites these malicious software pop up and ask to download, as soon as downloading starts they start damaging victim's network and system.
- **Child soliciting and Abuse:** In this type of cyber crime wherein criminals solicit minors through chat rooms for the purpose of child pornography. Many Investigating companies or agencies has been spending a lot of time to monitoring chat rooms frequented by children with the hopes of reducing and preventing child abuse and soliciting.
- In general, most of the website developers are testing their websites using white box testing, black box testing and gray box testing for protection.[1] After web hosting, some web automated tools are provided in SOA for performance, load and security testing like Soap, Apache jmeter, Curl, Jconsole, Jprofiler, Jira, Bugzilla, Mantic, Redmine, SET, SSL etc. [15]

Challenges of web services

Functionality

- According to Asankav (2014), it is different from traditional software testing because in traditional testing GUIs, number of user, types of requirements and inputs are fixed. Mainly problem occurs for multiple types of GUIs and huge amount of various types of data.[4]
- These multi functionalities are managed by different service providers e.g. In the Nokia token machine website, some web pages or facilities are managed by other services providers/developers. So it is difficult for testing without testing rights and source code.

Publish, Find, and Bind

- According to Asankav (2014), before publishing and developing of websites they needs to think as customer, developer, service provider and stakeholder point of view. [4]
- It is also a major and important problem for binding and transportation because web services are managed by multi or distributed server and some time services are provides by third party like online payment services done by third-party bank or PayPal. [3][8][11]

Security

- Dolvara Gunatilaka was mention that there are many types of SOA related issues from customer side like Privacy issues, Identity theft issues, Span issues, Malware issues, Physical issues, etc. because of improper architecture, technology or security method of SOA.[7]
- As per report by US government (2013), online services provider collects many personal and bank information of customers. But, it may not be secured because provider sells our private data to other provider for marketing without any intimation of customer which increased spamming, phishing, etc.
- Fake and same domain name (with minor change in domain name) also misguide the victim. Many websites developed for collecting victim's personal information. This information's will further be used for frauds like spamming, spoofing, phishing etc by the culprits.

Performance

- Asankav (2014) pointed that it is also a big challenge or nearly impossible to develop user-friendly and error or bugs free system because after implementation it is difficult to do testing. Nowadays the information's collected from the websites are managed in distributed server or third-party server. These stored data also access by multi languages from multi platform. So, Huge and variety of data is difficult to manage load and performance testing. The automation needs to be done through programmatic interfaces. [4][6][12]

Current scenario of data transfer in web applications

Data will be exchanged between source to destination using different network and IP address which maybe varying. Because when router is switched off, public IP address of that router will be changed. Sometimes, range of IP address are allocate for a DNS (i.e www.google.com has IP address range like 64.233.160.0 - 64.233.191.255). Communication of data is shown in given figure as bellow:

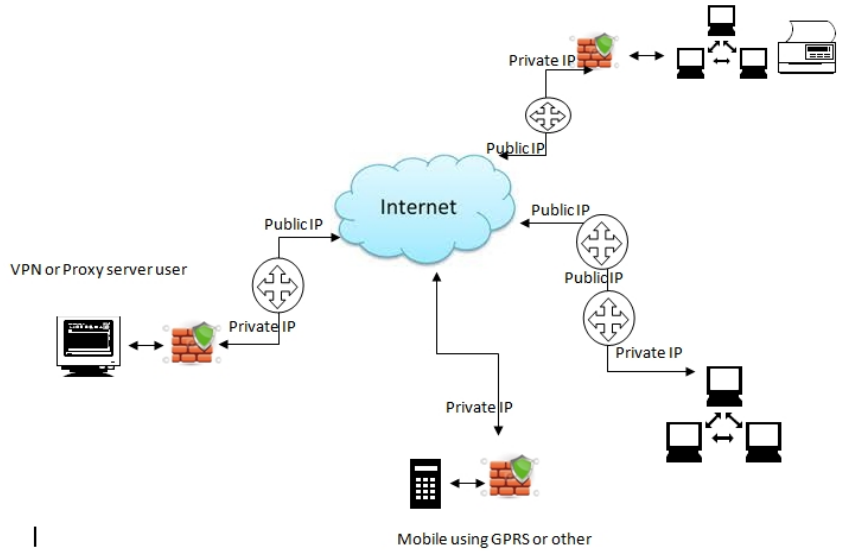


Fig. 1. "Data transfer over network."

In this communication, data will be generally transferred through HTTP/HTTPS protocols. Even if data is transferred using HTTPS which is secure connection, still hacking is possible because it also depends on OS, open ports, Security software, User's Password, ISP and Routers etc.

Provision of 100% protection from all aspects of different attacks is not possible. There are some techniques available which makes it possible as well as difficult to hack by any random fly-by hacker.

Problems with current scenario

How computer/system/data can be hacked?

1. Using a small script or a program, the hacker can scan any vulnerability loopholes of the victim's system by using tools like acunetix, skyboxsecurity, saintcorporation etc.
2. Open port is a big problem for individual system/device, because if the hacker can find out open ports of the target system easily then the hacker can send backdoors using this open port which may harm victim's system. This can be done easily by a hacker using readymade tools or commands. For example, using Nmap and netstat command of Linux operating system, hacker can get information about IP addresses of victim's system network and open ports of particular IP address.
3. Always check the domain address of a website, which maybe redirected automatically to some fake or cloned website while transferring the data. For example, a valid bank transaction maybe redirected to the web page which can function or perform operation without SSL or which maybe a cloned page of a valid bank website.
4. Always avoid website surfing using automatic text suggested in address bar of browser. For example, if a victim types "www.Fa "in address bar of browser, it automatically suggests text as "www.faceb00k.com". If the victim clicks on a suggested text, victim maybe redirected to a dummy DNS. (here "o" is replaced with "0").

5. If the victim gets any unknown hyperlinked text or image via an email or a message on computer or mobile, don't click on that hyperlinked text without verifying that email id or website. To verify details, websites that are already developed are available. For example, (a) victim can verify IP address using "whatsthierip.com" website; (b) victim should check the string at the end of the received email or link, For e.g "*.readnotify.com", if this is the string it redirect's to the Public IP address of hacker's.
6. Victim's at times, use other's system for internet surfing. Victim inserts their personal details, account details, username and passwords which maybe hacked because of the key logger software. Through key logger, data may capture information and store keystroke for hacking purpose.
7. Some websites and readymade tools maybe used for hiding original IP because, it does not maintain log of IP. Cyber ghost, hotspot shield, no-ip, open VPN, ProxyPN etc provide VPN for public IP management and hide original IP of hacker's.
8. Some websites provide facility to generate fake DNS as well as link to collect user's detail which is further used for phishing. Name of Websites are 000webhost.com, 110mb.com, t35.com and hostia.com.
9. Generally Kali Linux is use for penetration testing but it has many developed tools which is used to hack the victim. For example, metaspolit tool is used to hack the victim's device and send viruses, worms and Trojan. It will also take controls of victim's device.
10. Some software is providing dummy port numbers which bind with hacker's public IP address and send the information of victim through tools like NJRate, No-IP, Androrate and Prorate etc. When victim access his/her device, log will be display on hacker's computer. With this log details hacker can easily access victim's device.

Loss of data, during internet transaction is not only the problem of proper technology/testing methods of web server, web services (single/multi stakeholders) or websites, but it is the problems, mistakes, unawareness of client(user's).

MATERIALS AND METHODS

Protection of data is not only the responsibility of developers but it is a composite responsibility of client, website developer and service provider. Following are the course of action to be followed by client.

Protection steps followed by user:

Step 1: Make your passwords strong

Password is the first line of attack in this digital war between hackers and the potential victims. If hacker can get victims password, the rest is easy. So always use strong passwords. A **strong password** consists of at least six characters (and the more characters, the stronger the **password**) that are a combination of letters, numbers and symbols (@, #, $, %, etc.) if allowed. Passwords are typically case-sensitive, so a **strong password** contains letters in both uppercase and lowercase. Example: P455W0RD.

Step 2: Use TFA (Two-Factor Authentication)

Authentication means to identify valid user. To increase systems security two-factor authentication can be used. Which is describes as bellows.

1. Username and password factor
2. Biometrics factor
3. Secret questions factor (which victim have)

Step 3: Refuse click/select unknown and suspicious links

Do not click on unknown hyperlinked text or images unless verifying the sender email id or website.

Victim can verify unknown sender identification using "whatsthierip.com" tool. For example, If the email address of the sender is followed by "*.readnotify.com" then it redirects victim's Public IP address to hackers. So, Hacker can steal victim's information like passwords, bank account details, brokerage, email accounts, social security number, identity, etc. Information will be sells by hacker in marketing world.

If victim got email from any unknown email id, it will be is trace using following steps,

a. Copy of source content using show original option
b. Paste on header box in ip2location.com->email tracer
c. Then Click lookup button.
d. It will display IP root (source to destination).
e. Check IP root is valid or not.

Step 4: Generally refuse P2P File Sharing Networks

Peer to peer is an unsafe way to transaction. Malware or undetectable backdoor will be attached with music, movies, documents and other files using ready-made tools like NJRATE, PRORATE or ANDROIDRATE etc. When victim downloads the file, backdoor will install automatically in victim's device so, Hacker will get access of victim's system. The solution to this:

a. If victim found any unknown process in the start-up, the path should be accessed from the task manager.
b. Remove the process from msconfig file and delete it from target location.
a. Also delete the file from registry using run->cmd->regedit manually.

Step 5: Update your operating system, software and system regularly

New security vulnerabilities are discovered daily in operating system and applications. For example operating systems like Windows 7 or 8 and applications like Flash, IE8, and Adobe Reader. Hackers generate exploits for these vulnerabilities to attack.

Soon these "exploits" are passed around to other hackers and everyone is trying to use them against victim. This allows victim to install their software on victim system to control it and steal victim resources and information.

When the software developers such as Adobe, Microsoft, and Apple learn these vulnerabilities, then they develop "patches" to close these loopholes. They offer these patches as updates to the victim, Victim must update to be secure.

Step 6: Use latest and updated Antivirus

Everyone should have some form of antivirus software on their system. Antivirus software is not perfect, but it is certainly better than nothing.

Even the best Antivirus software is effective to approx. 95% of known malware. i.e. one in 20 pieces of malware will be missed. Some of the lower quality antivirus software will miss 1 in 2 pieces of malware. Antivirus software is only effective if it is activated and updated.

Antivirus software can't protect victim from silliness. A well-designed malware can embed itself into the Windows system files and victim's Antivirus software can neither detect nor remove it. In some cases, it can even disable victim's Antivirus software before found out.

Step 7: Do Not Use Adobe Flash

Adobe's Flash Player is available on nearly every computer, tablets, smart phone and even Android devices. It enables us to run those interesting videos as well YouTube, animations, etc. Without it, when victim visits a website with video or animations, he/she gets the warning messages to install Flash Player and a blank screen.

Flash Player is such a poorly designed and coded piece of software that it is known as "hackers best friend".

Step 8: Be secure using good Firewall

Although Microsoft ships a rudimentary firewall with its operating system, it is strongly suggested that victim should install a third-party firewall for better protection and block ICMP protocol.

One of the third-party firewall is Zone Alarm's Free Firewall. As the name says, it is free and very effective. It not only blocks outsiders from getting in, but also stops malware from accessing resources on victim's computer and talking out.

Protection steps for web service/website:

Step 1: Do not publish websites in shared DNS, because if any other website is hacked on shared web server, it can easily get access to victim's website.
Step 2: Purchase domain name with extra protection mechanism which will hide domain name details like providers, IP address, owner details etc.
Step 3: Use CDN (Content Delivery Network) which hides IP address of websites/web services provider.

CONCLUSION

The increasing popularity of service-oriented architectures has introduced the need for additional standards to help support the new security challenges involved in new model but still 100% secure transaction is a big question. In any online communications system, there are some challenges and these challenges are considered as an indicator of the security gaps which generate weakness in the system protection and are vulnerable to attacks. Some online fraud and challenges in web services are mentioned in this paper. Several guidelines for victim and developer has been put forward corresponding with the challenges for secure web transactions. Future work includes extending this approach to develop Security testing/technology/tools for multi stakeholder's website/web services.

ACKNOWLEDGEMENT

I thank my colleagues of Vivekanand college For BCA, Surat who provided insight and expertise that greatly assisted the research, although they may not agree with all of the interpretations/conclusions of this paper.
I thank Dr. Sanjay Buch, 2Assistant Vice President Of Reliance Industries Ltd for guidance in this subject that greatly improved the manuscript.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

(Proceedings available for download at rku.ac.in/icre)

RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

Various Methodologies for Power Quality Improvement

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ABSTRACT

Any type of Power Quality (PQ) difficulty in current, voltage and frequency variation results in collapse of consumer equipment. PQ problems are defined by different terms like, harmonic distortion, unbalance load and reactive power. By using the proper technology, PQ problems can be solved. Now a day various methodologies are available for PQ improvement, i.e. UPQC (Unified Power Quality Conditioner), DVR (Dynamic Voltage Restorer), DSTATCOM (Static Synchronous Reactive Compensator), DPFC (Distributed power flow controller), SAPF (Shunt Active Power Filters), Series APF (Active Power Filters), PWM (Pulse Width Modulated). Main focus of this paper is to give brief idea about all above mentioned methodologies for PQ improvement and comparison between all techniques based on construction, application, reliability and cost.

SUMMARY

To Learn about Various Methodologies for PQ Improvement and their comparison.

Keywords: *Power Quality, Unified Powr Quality Conditioner, Dynamic Voltage Restorer, Static Synchronous Reactive Compensator, Distributed power flow controller, Shunt/Series Active Power Filters, Pulse Width Modulated*.

INTRODUCTION

A PQ problem has different types of disturbances like voltage sags, voltage swells, harmonics deformation, impulse transients and flicker. By voltage sag and swell are result to trip the breakers and sometimes it may damage the equipments. To solve these problems, PQ improvement methodologies which are commonly used are (i)UPQC (ii)DPFC (iii)DVR (iv)DSTATCOM

(v)SAPF (vi)Series APF (vii)PWM. The main focus of this paper is to study about PQ improvement methodology as mentioned in [i-vii].

In [i-ii], the authors has presented artificial controlled based UPQC which is used to reduce the trouble that change the shows of the significant load in the power system and DPFC method that helps to improve power quality with less cost plus more reliability. In [iii-iv], the authors has presented DVR methodology which reduce the harmonics distortion of sensitive load and improve power quality, DSTATCOM technique is also use to improve the PQ by solving the problems like, voltage sags/swell, low power factor and harmonic deformation in distribution system. In [v-vii], the authors has presented Shunt/ Series APF which commonly use to compensate load current and source voltage helps PQ improvement. Now to add more on this, PWM techniques are also very useful for harmonic reduction and improve power quality.

This paper presents a complete review on all the available methodologies for PQ improvement.

DIFFERENT METHODOLOGIES FOR PQ IMPROVEMENT.

As discussed in introduction, the complete descriptions of different topologies are follows,

1. Unified PQ Conditioners (UPQC):

In power system, UPQC is helpful methodology for PQ improvement. The function of UPQC is to reduce the conflict that affect the act of significant load in electrical network. Actually, UPQC is very helpful method of solving for very high capacity loads receptive to source voltage and current flicker. A UPQC is consists of collective series and shunt active power filters (APFs). UPQC controller has comparatively high cost and average reliability.

The basic function of a UPQC controller is given bellow, (see fig. 1)

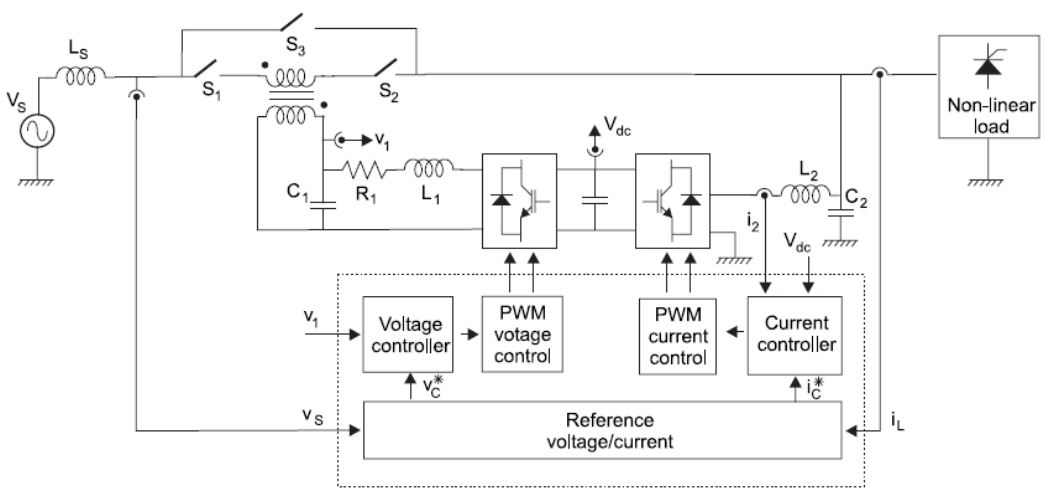


Figure 1: UPQC controller

In this figure the reference signals for voltage (V_C) and current(I_C) compensation are solved from the instant calculation of load current (I_L), Source Voltage (Vs) and DC-bus Voltage (V_{dc}). Given reference signals are comparing with the measure feedback signals V_1 & I_2 applied to the decoupled voltage as well as current controllers that make sure that reward signals and match up to the reference value. Now gate signals of power converters are obtain by affecting to Pulse Width Modulator (PWM) to the controller output. Switches S_1, S_2 & S_3 will control recompense position of UPQC. A power converter switch at tall frequency generates a PWM o/p voltage waveform that is low pass filter. In this circuit L_1, R_1 and C_1 is used as a series APF & L_2 as well as C_2 are for the shunt APFs.

In feedback structures, it permits good stationary response during forward structures and it generates fast responses throughout voltage transients. In given case reference signal is produce within the voltage controller and it is not permit selective harmonic compensation. To the circuit, hysteresis controllers are implementing by way of easy analogy circuits other than, as weakness, the range of the output current isn't correctly contained and this problem can dissolved by means of generalized integrator. For that fuzzy logic and Artificial Neural Network (ANN) may be used as a current controller as a reference signal.

2. **Distributed Power Flow Controllers (DPFC):**

The DPFC is an advanced modification of UPFC. It can capable for manage the entire network stricture i.e. line impedance, bus voltage and transmission angle. The DPFC has less cost and high reliability. In DPFC, it removes the common DC link which is in between the shunt & series converter.

DPFC had total 3 way of controllers system: central control, shunt control & last series control. Let's see figure of DPFC.

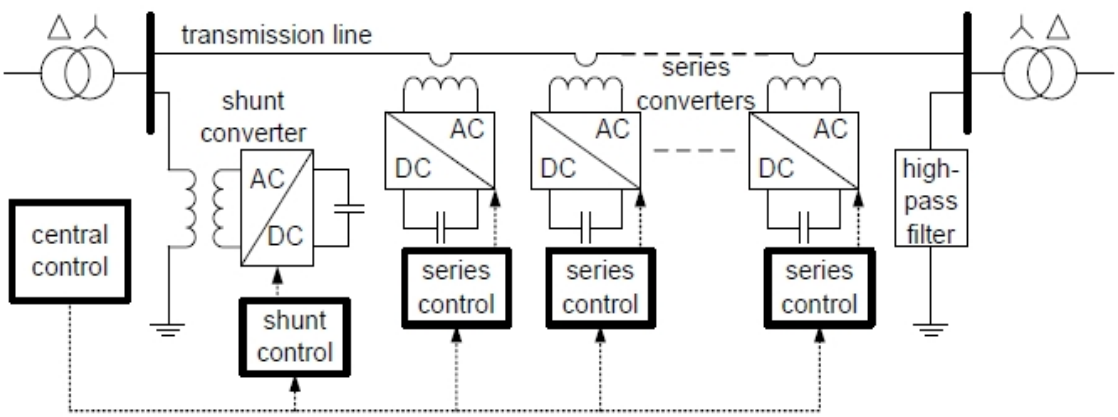


Figure 2: DPFC controller

2.1 Central Controls:

It requires a reference signal which comes from shunt as well as series converter of DPFC. It can be control the function depend on a purpose of DPFC. A purpose may be such as power flow control and balancing of asymmetrical components. Central control also gives equipment voltages to reactive current signals for the shunt converters.

2.2 Series Controls:

Every series converter possesses series control. A series controller is helped toward sustain capacitor. It can be done through 3rd harmonic frequency apparatus. For generation of series voltage control at the primary frequency we essential central control.

2.3 Shunt control:

Main purpose of shunt control is toward adding a constant 3rd harmonic current to system. During that time, it's required to sustain the capacitor of shunt converter at a stable value via the gripping active power supply from network and insert essential reactive current on primary frequency keen on network.

2.4 Advantages:

a) High controlling power: DPFC can concurrently control every factor of the transmission line.
b) Tall consistency: An idleness of the series converter provides elevated consistency lacking lift the price.

c) Less cost: No phase to words phase voltage division required, so cost is reduced.

3. Dynamic Voltage Restorers (DVR):

Generally, DVR is useful for improving PQ improvement & it's also helps to reduce the harmonics distortion of sensitive load. As we know, the most important PQ issue is voltage drop. By increase custom of voltage concern device, it has complete industrial process added liable to deliver voltage sag. Voltage sag can be minimized by the help of energy storage DVR Load voltage will balance by adding voltage through DVR. Just because of energy restrictions, it's required to reduce power addition on or after DVR.

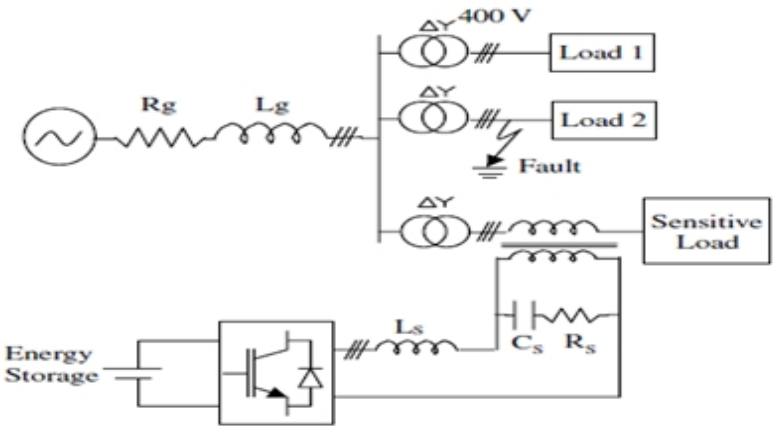


Figure 3: Circuit diagram of DVR.

The main purpose of a DVR is to safety of receptive load due to fluctuation of voltage. Now see in Figure 3, DVR is on sensitive load side. When fault will done on other line then DVR will active and voltage will balanced. In figure no. 4 main device of DVR is energy storage facility, LC filter, coupling transformer and voltage source.

In thyristor controlled load which helps to utilize supply voltage and phase angle. These method will helps to balance the sag and pre-sag voltage as per given circuit. But this circuit will also provide real power which is not required. In this given circuit (figure 4) has voltage source (Vth), thevenin resistance (Rth) and thevenin Inductance (Lth).

Now, if fault will done then in DVR then load voltage and source voltage subtraction gives DVR voltage, like VL – VS = Vdvr.

Let's see equivalent circuit of power system as figure 4,

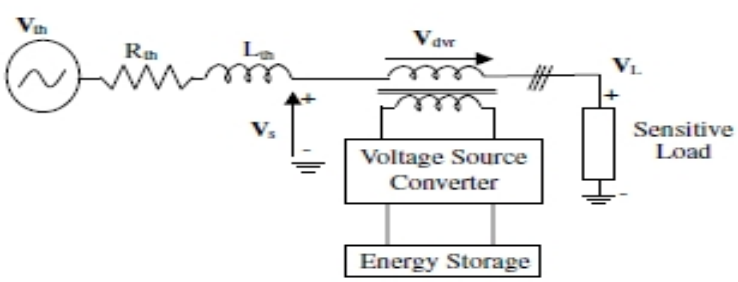


Figure 4: Equivalent circuit of power system.

In this system magnitude of added voltage is very less. Due to that voltage and current sag apparent power will be reduced. Now active power of is added as per this equation; $3(V_L - V_S) * I_L * \cos \phi = P_{dvr}$.

A capacity of adding voltage to the DVR system is around 50%. It will be done with only 0.1 second. So it is very fast and sensitive. By help of DVR reimburse voltage sag is easy and quick.

4. Distribution Static Compensator (D-STATCOM):

The D-STATCOM may very efficient devices for PQ improvement in power transmission and distribution systems. It has supplementary potential to maintain reactive current at low voltage. D-STATCOM may develop a voltage as well as frequency hold up by changing capacitors & batteries used for energy storage. We can develop PQ used for voltage sag or voltage swell, harmonics deformation and small PF (power factor) into transmission line.

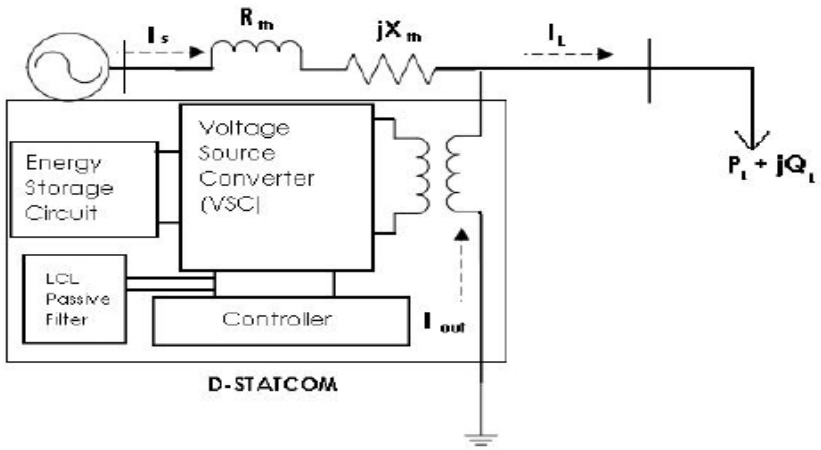


Figure 5: Main circuit diagram of DSTATCOM

The D-STATCOM has two-level Voltage Source Converters (VSC), DC energy storage apparatus and a pairing transformer which is linked in shunt with distribution line by pairing transformer.(see figure no. 5) In this circuit, VSC renovate DC voltage diagonally into storage tool in place of three-phase AC output voltage. Sometimes appropriate adjustment is require for the phase and magnitude of the D-STATCOM output voltages which can be permits the useful manage of active as well as reactive power connections involving D-STATCOM and AC power system. Sometime configurations agree to device to take up controllable active & reactive power. As per the circuit, VSC is linked in shunt with the ac system which gives a multi-tasking method for the following purposes:

1) Voltage ruling & reward of immediate power.
2) Improvement of PF.
3) Removal of current harmonic.

As per the circuit, the importance of Ish may prohibit with regulating the output voltage to a converter. So Ish may be written as,

$$I_{sh} = I_L - I_S = I_L - \frac{V_{th} - V_L}{Z_{th}}$$

Where, I_L = Current of given load, Vth = thevenin voltages, I_S = source current, Zth = Impedance, V_L = Voltage of given load. Now as per formula, Output current may accurate the voltage sag with regulating the voltage fall diagonally the line impedance, i.e .Zth = Rth + jXth.

4.1Advantages:

a) DSTATCOM is used in voltage regulation in distribution line.

b) It may also used to improve PF up to unity.
c) It can also used to moderate harmonics in distribution system.
d) DSTATCOM can be used for load balancing.

5. **Shunt Active Power Filter (SAPF):**

As we know, SAPF can use to balance the load current harmonics with introducing equivalent excluding reverse harmonic compensate current that results improve PQ of the system. A SAPF is also produces the same amount of harmonic by the load but it can be shift on 180^{o}. Now onwards that harmonics will be added with line at common coupling point whenever the load current harmonics can eliminated & usefulness supply due to sin wave.

The SAPF circuit has active power filter, line impedance, dc voltage and coupling coil which is connected to the parallel with non –linear load that balance the load current harmonics. (See figure 6)

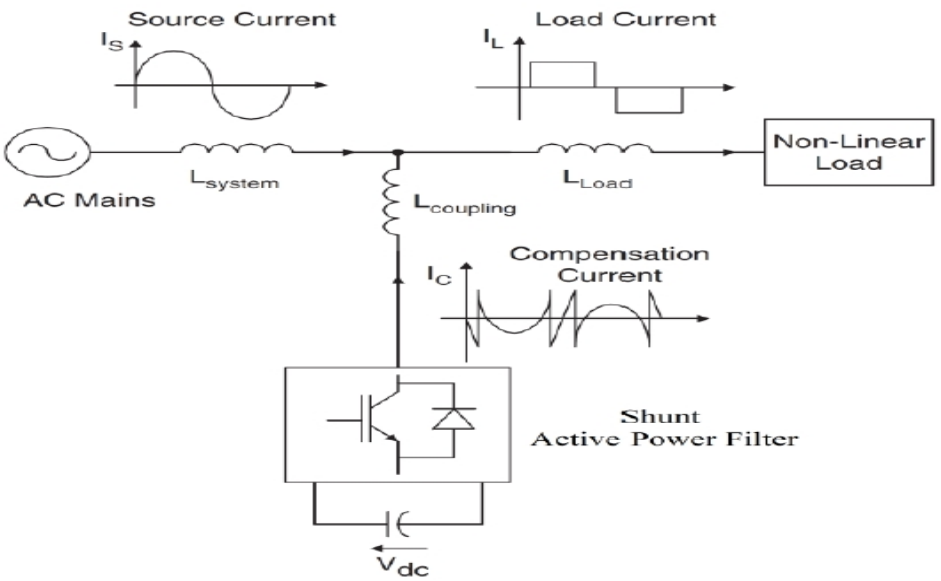


Figure 6: Basic scheme of SAPF

In given figure, the basic idea of SAPF that balance the load current harmonics with introducing harmonic compensating current. Normally SAPF run with current source which inserts harmonic apparatus created with load of 180° phase shift. As per given scheme the capacitor is use to improve the PF same way in this circuit capacitor is in passive filters to balance for current harmonics.

5.1Advantages:

a) The Shunt APF is very reliable and money-spinning resolution for PQ problems.
b) In given scheme of SAPF, the filter presents gives a very active and steady-state reply and it may be a greatly solution for PF improvement also.

6. **Series Active Power Filters (Series APF):**

The Series APF (Active Power Filters) is a device which inserts a voltage module in series with supply voltage also eliminates harmonic apparatus of voltage. Series APF is look upon organized voltage supply; balanced voltage sag or swell to load, Due to this, PQ of the system may improve.

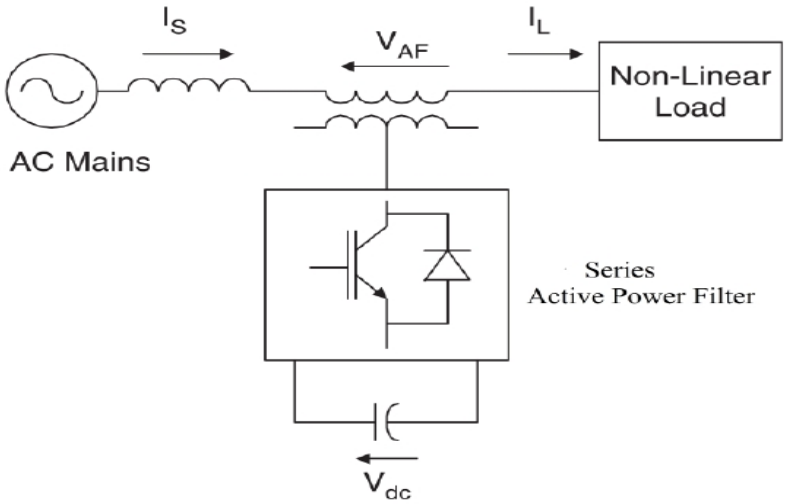


Figure 7: Basic scheme of series APF

As per scheme of Series APF which include of APF, dc voltage and coupling transformer connected in series with non-linear load. Coupling transformer is use to is insert a voltage module in series with the supply voltage & its helps to eliminate harmonic apparatus in voltage waveform. (Look at the fig.: 7)

The series APF works mostly like harmonic isolator between main supply and nonlinear load. It may also useful to stable voltage sag as well as swell on load. Sensibly series APFs are more valuable and low cost device compared to shunt APFs. But series APF requires sufficient protection scheme.

6.1Advantages:

a) Series APF is low cost solution for PQ improvement.
b) This method consist much faster input/output response which helps to improve power quality with less time period.

7. **Pulse Width Modulated (PWM):**

The PWM technique is used for harmonic diminution and development of primary peak voltage & PQ is improved. In PWM method, total harmonic effluence in the power system would be decreased and as a result, the PQ will be increased.

By survive with these harmonic troubles many alternatives are planned in the past. But PWM method is very famous technique. The main benefit of PWM control method may get includes sine wave input as well as output current and voltage waveform. It's also helps to increase PF, better transient response, removal of the tidy harmonics. Moreover, manage by control is often come with additional failure because of the controlling victims.

A major issue for harmonic creation in AC supply is fluctuation of supply voltage. By using this technique the discontinuity of the main voltage has been reduced. Duty cycle of PWM is straight comparative to whole conveyance angle for AC voltage. Suppose, when total conduction angle goes 45°, then duty cycle of PWM could be 25% (i.e. 45°/180° X 100); but when angle goes 90°,then duty cycle will be on 50% (i.e. 90°/180°X 100). For the outcome, when conduction angle will improved from 0° to 180°, same way duty cycle of PWM could improved from 0% to 100%. Now graph of O/P voltage wave of the projected PWM-AC controller through ohmic load are presented as in Figure 8 & 9.

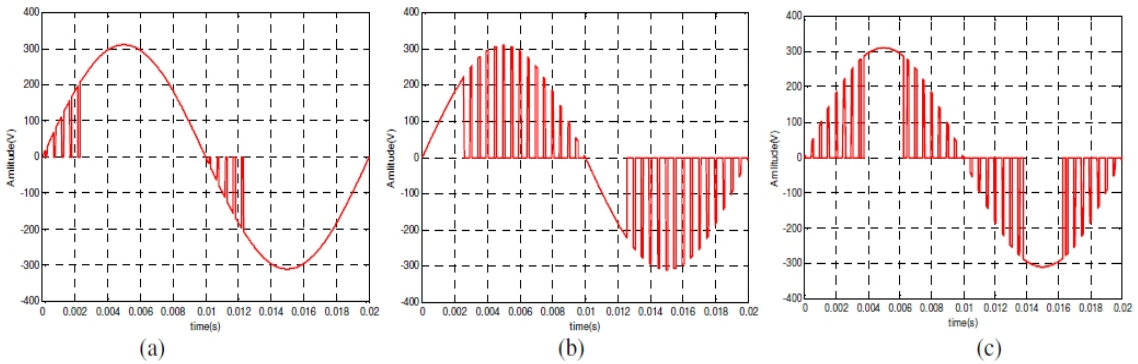


Figure 8: O/P voltage for PWM-AC controller with angle of 45 degree.

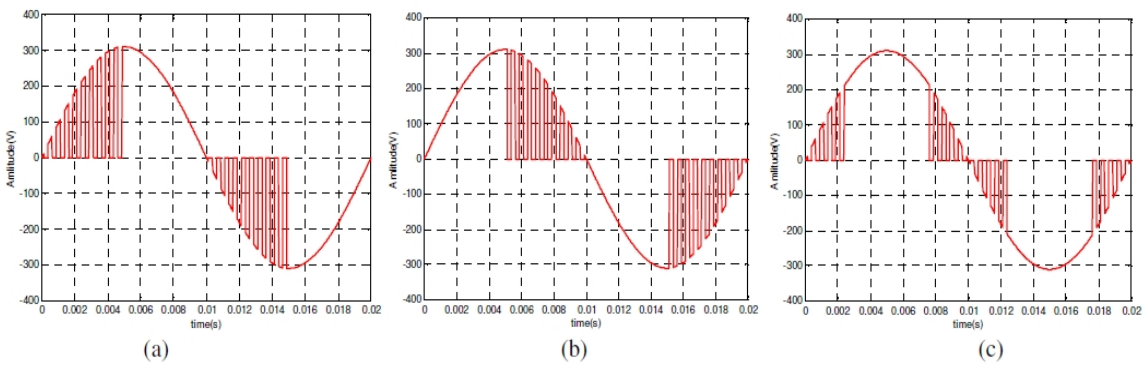


Figure 9: O/P voltage for PWM-AC controller with angle of 90 degree.

Let's see the circuit for PWM-AC controller as mention below Figure 10.

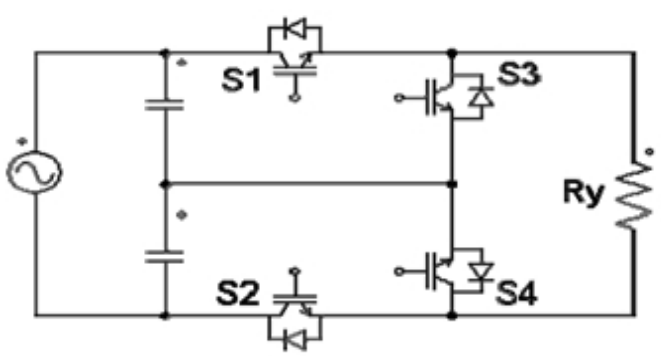


Figure 10: The diagram of power circuit used in planed PWM controller.

As per the above figure, circuit may act from the single-phase AC supply; the voltage crosswise every switch will restricted to system voltage. Now series switches S_1 and S_2 attach with load terminals to the supply, it will control the power which sends to the load. Now parallel switches S3 and S4 are offer a permissive path for the load current which helps to release their stored energy during series switch is turned off. In this diagram switching mode of the controlled switches are

determined with the help of polarity of the source voltage. If the supply voltage is positive, then the switches S_1 and S_3 will be driven complementarily but at those time switches S2 and S4 will fully turned on. If supply voltage polarity may change that time switching mechanism could goes inverse.

7.1 Advantage of PWM:

a) Trouble-free to implement and simple control.
b) There is no any temperature deviation.
c) No ageing origin drifting in linearity.
d) Well-matched with latest digital microprocessors.
e) Minor power dissipation.
f) PWM will allow linear amplitude control to the output voltage or current.

COMPARISON BETWEEN ALL ABOVE METHODS:

Table 1: Comparative Analysis

Name of methods	Required elements and its usage.	Applicability for PQ improvement.	Reliability and cost.
UPQC	UPQC is made by mixture of series as well as shunt APF, which helps to elimination of harmonics.	It wills use for harmonic removal and synchronized reward of voltage as well as current which improves the PQ.	It has comparatively high cost and average reliability.
DPFC	DPFC reduce general DC link involving the shunt as well as series converters due to that we can minimize the voltage variation.	It is very useful for moderating the voltage deviation and in results it improves PQ.	It has comparatively Less cost and high reliability.
DVR	As we know DVR has few important elements which are energy storage system, LC filter and voltage source converter which helps to reducing the harmonics distortion.	DVR is very valuable device for PQ improvement and it will be done by dipping the harmonics deformation of sensitive load.	It consist high cost and more reliability.
D-STATCOM	It is made by some important elements like 2-level VSC, DC energy storage apparatus also pairing transformer which is joined in shunt. So these elements help to improve PQ of the system.	D-STATCOM can absorb or else deliver reactive current against voltage trouble such as voltage sag or swell, which helps to improve PQ.	It consist very high cost and good reliability.
SAPF	SAPF has few important components i.e. active power filter, DC voltage and coupling coil which is connect in parallel with nonlinear load. Now these components help to balance load current.	SAPF can easily balance the load current harmonics which could be done by adding equal but reverse harmonic equilibrium current and improve PQ of the system.	It has comparatively less cost and average reliability.
Series APF	Series APF has some elements like active power filter, dc voltage and pairing transformer linked in series with non-linear load its help to remove harmonic from voltage wave.	It will help to insert the voltage segment with series to supply voltage, that gives result of eliminate harmonic apparatus in voltage waveforms. So that PQ will improve.	It consist medium or less cost and average reliability.
PWM technique	PWM is consists different switching mold (i.e.S1, S2, S3, S4 etc.) which used for harmonic decrement and perfection of primary peak voltage.	This technique may improve PQ by decrementing of harmonics and developing of primary peak voltage.	It consist medium cost and good reliability.

CONCLUSION

In this paper we have been studied about various methodologies for PQ improvement like UPQC, DPFC, DVR, D-STATCOM, SAPF, Series APF and PWM. This paper also presented comparative analysis between all above mentioned techniques. Now a day's DPFC techniques is most popular due to less cost and good reliability.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

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A Comparative Study Of The Pre- And Post-Reform Performance Of Gujarat's Power Distribution Sector

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ABSTRACT

In past three decades, the Electricity Industry throughout the world is undergoing restructuring and adopting the deregulated structure for better utilization of the resources and for providing choice and quality services to consumer at economical prices by improving the efficiency in the operation of the power system.

This paper examines the impact of governance reforms on efficiency, performance and service delivery in order to identifying the factors responsible for the success of reforms in the power sector in Gujarat. The study is aimed to know the gap between ARR & ACS and generation capacity for pre reform period, thrust for reform and to evaluate the business parameters of the distribution company like distribution losses, AT&C losses, collection efficiency, consumer mix, sales mix for the pre and post reform period.

SUMMARY

By the process of reform implementation, Gujarat power distribution sector has achieved remarkable improvement in performance.

Keywords: G.E.B., Distribution Reforms,Gujarat Power utility,Power distribution

INTRODUCTION

Along with the formation of Gujarat State in the year 1960, the Gujarat Electricity Board (GEB) was established under Section 5 of the Electricity (Supply) Act 1948. The generation capacity was 315MW and a consumer base was 1.40 million consumers and nearly 50000 employees working with the organisation. The GEB bearing investment of billions of rupees. In the first two decades after establishment the prime concern of the GEB was on electricity in the rural areas, resulting Gujarat became the first state to achieve the landmark of '100% Electrification of Villages'.

As per the 1991 Census, 17,940 out of 18,028 villages were electrified -which was notified as close to 100 %. *(1)*

Before the reformation i.e. during 1992 to 2000-01, GEB was facing huge financial losses and spoiling the image among the public due to unsatisfactory services. The first step for the reformation was initiated in the FY 2001-02 as a corrective measure like renegotiation of power purchase agreement, curtail the rate of interest on loans, detection of malpractice and mischief with power, reduction in T&D losses prior to unbundling the GEB. The government of Gujarat has implemented reformation process by unbundling the GEB in seven companies.

MATERIALS AND METHODS

For performing exploratory study, primary data was collected by experience survey and the secondary data was collected for the study from reports, published government documents, newspaper clippings, websites, books, journals and magazines. The supportive reports and presentations and meeting proceedings collected from holding company i.e. Gujarat Urja Vikas Nigam Ltd (GUVNL).The reports made available from GERC website like RIM and SOP as well as Annual reports of discoms' and GUVNL. The MYT submitted by DISCOMS' and Tariff order and Petitions from gerc website utilised for gathering the data.

RESULTS AND DISCUSSION

LITERATURE REVIEW

(Baijal, 1999) summarises the experience of many countries on power sector reforms. Further, he appeals to government to retain its role as a policy maker. As urged by him to set-up national and state level regulatory commission to supervise the working of the different players. As conclude by him, unbundling of SEBs would allow the formation of transmission, distribution and generation Companies.

(Morris, 2000) makes an appeal to address the various issues of power sector reforms such as enormous leakage of revenue of the SEBs, privatisation of distribution and generation of electricity business and changes in the institutional mechanism for the success of power sector reforms. He emphasizes on a need to consider a financial capacity of the state and SEBs before signing an agreement by IPPs. Further, he points out that hastily crafted structure of IPPs and fast track power

generation projects have further increased the financial burden on SEBs.

Confederation of Indian Industry (CII) — *(A.T. Kearney Report, 2009)* "Sustaining Growth– Future of Indian Power Sector" Message from R S Sharma Chairman - CII's National Committee on Power and Chairman and Managing Director NTPC Ltd. A robust and thriving Power sector is central to India's sustained economic growth India's power sector has responded strongly to the reform measures undertaken by the government with a wide spread participation across Public and Private sector, Indian and multinational companies

(Madhavan,2012) before 2001, GEB was passing through the huge financial loss facing dissatisfaction from the consumers, hated by farmers, full of mismanagement, bureaucracy, huge line losses, heavily accumulated debt, political interference in day to day working, difficulties in implementation due to wrong vertical structure- all these collectively brought GEB on the verge of bankruptcy.Year 2012 onwards an unbundled GEB – GUVNL and subsidiaries became a model public utility having clear vision, mission and core values. Excellent in services by adopting modern technology and IT enabled services. The GUVNL and subsidiaries won several global awards for competency, achieved award for best distribution utilities (PGVCL) and A^+ rating.

OBJECTIVE OF STUDY

The Gujarat power sector has achieved remarkable achievement after 2004-05, i.e. after reforms. How power sector could achieved this is always be a learning steps for other sector of the state.

The objective of the study is to evaluate the Power sector reforms process of Gujarat state vis a vis objective for reforms, early reform actions, comparative study of pre and post reforms business parameters affecting distribution utility like Distribution losses, AT & C losses, Collection efficiency, Consumer Mix and Sales (Revenue) mix

SCOPE OF THE STUDY

The scope of the study is to

1) Review of various performance parameter for pre reforms period of the Gujarat Electricity Board and to identify the reasons for reforms of Gujarat Electricity Board
2) The objectives (reason) for reforms of Gujarat power sector
3) Study & evaluation of implementation process of reforms in Gujarat
4) Impact of Distribution reforms in Gujarat
5) Comparative analysis of Gujarat Distribution sector for pre and post reforms period

1) Performance of Gujarat Electricity Board for pre reform period:

The prime concern and focus of the GEB (during pre-reforms) was to electrify the rural areas

by erecting new transmission and distribution lines, supplying quality power to consumers by minimising interruptions and updating the generation capacity. The GEB was unfocused for effective revenue realisation and T&D loss levels. The heavy financial losses caused due to ignorance of revenue aspect and high subsidy level to agriculture consumers.*(4)*

A) Generation efficiency of Gujarat Electricity Board

The strength of the power sector of the state is always being measured by the generation capacity of the generating stations, plant load factor. As shown in *(Fig.1)* the plant load factor remained 60% to 65% on an average from year 1995 to 1999. It indicates the need of improvement in generating efficiency of the generating stations under Gujarat state.

B) Transmission Losses of Gujarat Electricity Board

As sown in *(Fig. 2)* the transmission loss level of GEB was reduced from 7.14% to 5.95% from year 1995 to 1999. High transmission loss indicates the lack of vigilance on high tension consumers, improper maintenance of high tension lines.

C) Distribution losses of Gujarat Electricity Board

The high level of distribution loss directly indicates the high revenue loss. The distribution loss mainly comprising of commercial losses, theft of power, non-metering, non-payments, unbilled cases and illegal use of electricity. Till 1999 the GEB was losing 7272MUs of electricity as a distribution loss as shown in *(Fig. 3.)*.

D) Financial performance of Gujarat Electricity Board

During 2000-01 a revenue realisation of Rs. 0.15/kwh only. This is because of extremely low tariff of 0.5 million agriculture consumers i.e. Rs. 350 per hp per year. To set right the financial deficit minimum RS.2 per unit subsidy was required for each unit power sale to agriculture consumers. The agriculture consumption was boosted from 16.7% of total units sold in the year 1970-71 to 43% in 1999-00. In the year 1999-00 the estimated loss of the GEB was Rs. 14 billion due to the low tariff power to agriculture consumers, lead to larger GAP between ARR and ACS *(3)*.

The gap between ARR and ACS is shown in *(Fig. 4.)***.** The larger gap leads to recurring financial deficits and not able to raise resources for investments. Average Revenue Realisation (ARR) of Rs.2.05/kwh and Average cost to serve (ACS) of Rs.2.49/kwh for pre reforms period was recorded as shown in *(Fig. 4.)*.

E) Power availability to consumer of Gujarat Electricity Board

In the year 1998-99 there was acute shortage of power due to insufficient generation capacity

against demand of power, which lead to load shedding between 50 MW to 1,450 MW was experienced on 362 days of the year.*(4)*

F) Political Interference in day to day functioning of the Board

The organisational structure of GEB was overlapping between planning commission and state ministry of power. Due to that many a time interference of either body affected the performance of GEB. The unpopular activity like detection of power theft and malpractices performed by GEB was directly affecting the public which indirectly invited the pressure from local leaders.

2) Reasons for reforms of Gujarat Electricity Board

Listed below are the main reasons for urgent reforms of the GEB.

1. In adequate generation capacity and poor generation efficiency
2. High level of Transmission and Distribution losses
3. Poor performance in distribution of energy
4. Recurring financial deficits
5. Political interference in day to day functioning of the board
6. Power sector reforms initiation in India

3) Process of Reforms implementation in Gujarat

As per the recognisation in Indian Electricity Act 2003, the electricity industry in gujarat needed to be reformed and establishing the Electricity Regulatory Commission in the state.

On the base of this, the organisational restructuring of the GEB took place by unbundling the vertically integrated structure into seven different companies. *(Fig.5.)*

One for generation and transmission each, four distribution companies (Discoms) and a holding company known as Gujarat Urja Vikas Nigam Limited (GUVNL).

All companies became fully operational from April'05 and started their business activities independently. The power distribution in the cities of Ahmedabad and Surat had been with private sector entity viz Torrent Power through it's subsidiaries as AEC and SEC.

<u>Key features of reform implementation:</u>

A) Full Support from staff

From the initial stage of reforms the extensive support of representatives of the unions and associations

of the staff was a favourable feature in the process of reforms as it had been convinced that the GoG and GEB were not pursuing any hidden agenda. During entire process of transition from the GEB to companies no incident of strikes/protests took place from employee.

B) Transition support by the state government

The GoG took over the liability of debt payment of GEB, settled outstanding dues of Rs. 1627.72 crores payable to CPSUs up to September'01 and in lieu issued bonds to these CPSUs.

C) Financial support by the state government

At the time of reform there was liability of approx Rs.624 crore as loan, which had been converted in to equity shares in GUVNL by GoG. For remaining outstanding loan of Rs.842 crore moratorium periods of six years was allowed.*(1)*

Pre reform initiatives in Gujarat

Unlike the other state, Gujarat had started the process of reforms in early 2000 during the GEB days.

A brief of the measures undertaken by GEB is as below.*(5)*

A) Revenue enhancement measures
- Effective monitoring of the revenue situation
- Fixing the performance parameters for review
- Regular and systematic review of performance parameter and strict implementation there of
- Feeder level revenue monitoring by assigning the feeder manager

B) Efficiency enhancement measures

The GEB took massive actions to curb theft of power and dealt the theft and non paid bills cases sternly. The initiatives were like

- Setting up a vigilance department with 500 retired army personal headed by IPS officer to check power offenders
- Incentive scheme for power theft information from the public
- Formation of vigilance squad
- Creation of dedicated police stations at Surat, Baroda, Sabarmati, Rajkot and Bhavnagar which were created only to deal with cases of power and power property theft
- Sealing of Installation to stop the energy leakages

4) Impact of Distribution Reforms in Gujarat

The key areas of focus:

- ✓ Distribution loss reduction
- ✓ Reduction in commercial losses
- ✓ Revenues enhancement measures
- ✓ Enhancement in customer services and satisfaction

✓ DISTRIBUTION LOSS REDUCTION

The distribution loss includes Technical & Commercial loss. The discoms have focused on curbing of power theft, pilfering of installation, strengthening of network and fine tuning of processes and procedures to reduce the distribution loss level.

✓ REDUCTION IN COMMERCIAL LOSSES

Power theft was the only reason of high level of commercial loss. GEB had started actions for massive installation checking and same had been continued by the GUVNL and discoms in Gujarat. The actions were not acceptable and opposed by the people initially. Apart from installation checking other steps to avoid future power theft occurrence following steps were taken by discoms.

1. Replacement of old meters by new meters
2. Strengthening the cash collection services
3. Replacement of bare conductor by Insulated/Aerial bunch conductor to prevent hooking.

✓ REVENUE ENHANCEMENT MEASURES

Following revenue improvement measures were taken by the GUVNL to improve the financial position of Gujarat state's discoms:

1. Reduction in power purchase cost: In the FY 2003-04, savings of Rs 4.95 crore took place due to renegotiation of PPAs. In the subsequent negotiations in 2005-06, they managed to get a further reduction of Rs 64 crore.*(1)*
2. Centralized purchase cell: Formation of CPC under direct control of GUVNL leads to cost effective procurements of materials and inventory planning. This step lead to savings of almost Rs. 137.93crore over the period of 2002-06.*(1)*
3. Releasing new connections: Camps were arranged in poor areas and slums for on spot release of connection. A number of schemes, like TASP (Tribal Area Sub Plan), KutirJyoti, Zupadpatti, were started to provide connections to poor people.
4. Settlement of Old dues: By implementing Voluntary Disclosure Schemes (VDS) and one time settlement scheme were also started to clear old dues. One time settlement scheme was availed by 28793 consumers.*(6)*

✓ ENHANCEMENT IN CUSTOMER SERVICE AND SATISFACTION

At the time of unbundling of GEB, consumers were facing problems like delay in getting new connection, delay in resolving power complaints, inadequate bill details like address, name, etc. As a solution to all these problems Discoms in Gujarat took following measures:

1. Formation of Customer Care Centres (CCC) at all sub-divisions, divisions and circle offices as well as at corporate level for all Discom. These centers took care of all the customer queries related to
    a. Release of new connection
    b. Queries related to wrong billing
    c. Change of consumer master details like name, address, etc. related process
    d. Faulty/ fast meter and other technical parameter related queries
2. Central Trouble Call Management (TCM) centre: Over and above CCC at subdivision level, central Trouble call management center was setup to register and resolve power supply related complaints through telephone.
3. Private cash collection agencies for bill collection: For providing better facilities to pay the energy bills by the consumers, discoms has started private cash collection centres by outsourcing the contracts, acceptance of payments at post office, Any Time Payment Machine (ATP) for 24Hrs acceptance of payment and online payment facility.
4. Geographical Information System (GIS): The mapping of distribution network and consumer database was done using GIS software. This lead to easy and accurate availability of consumer and network details for attending complains as well as system improvement works.
5. Jyoti Gram Yojana (JGY): The big issue faced by the rural consumers was non availability of power during load shedding hours of feeders. The consumers in rural areas hardly getting power supply for 8 to 10 hours in a day which lead to migration of people from rural to urban areas and non establishment of industries as well as no availability of proper education.

    By implementing JGY scheme the combine feeder catering power supply to villages and agriculture was made separate by installing huge HT/LT networks and transformers. The pilot for the scheme was done in 8 districts in Gujarat in September'03. In October/November'04 the scheme was extended to the entire state after successful completion of pilot.

5) Comparative analysis of Gujarat Distribution sector for pre and post reforms period

Majority of the parameters found improving after reforms took place in Gujarat. The Gujarat having adequate power generation capacity and qualitative services were availed to the consumers of discoms

after reforms.

The parameter like generation capacity of electricity, distribution losses, cash collection, AT&C losses and consumer mix of all four discoms after reforms is tabulated below:

1. Generation of Electricity :

As shown in *(Table 1.),* the generating capacity of the Gujarat state found strengthening after reforms. The total Installed capacities of all Sources were 8761 MW at the time of reforms which reached to 19212 MW in the month of March-2015.

2. Distribution losses :

The distribution losses of GEB were 30.64 % *(Table 2.)* at the time of unbundling. After formation of Distribution companies' exhaustive loss reduction efforts were put up as elaborated above and resulting the overall distribution losses of GUVNL were 18.47 % *(Table 2.)* as on March-2015. This clearly shows the improvement in loss level and realisation of more revenue after reforms. The graphical trend of reduction in distribution loss is shown in *(Fig. 6).*

The distribution losses of all four discoms' also found reducing in the span of time after reforms. *(Table 3.)* Shows the statics of distribution loss of the discoms.

3. Cash Collection :

The Distribution Company collecting the amount of energy bills from their consumers after issuing the Monthly/Bi Monthly bills for use of Electricity. The amount realised against the bills named as collection or cash collection. Non realisation will be booked under Arrears in the book of account. The Gap between Assessment and collection reflects poor collection efficiency which ultimately affects to the AT & C Loss picture of the company.

At the time of reforms the collection amount yearly was Rs.10204 crore (2004-05) which increase to Rs.34548 crore which shows the increase in business by the discoms. *(Table 4.)*
Considerable cash in flow increased in all discoms as shown in *(Table 5.)* and the graphical representation of overall cash inflow is represented in *(Fig.7)*

4. % AT & C Losses :

Aggregate Technical & Commercial loss of the Discom's indicates the overall performance of distribution utility (Billing as well as collection efficiency). The AT & C Loss level was 35.20 % in 2004-05 which

reduce up to the level of 19.48 % in the year 2013-14 *(Table.6)* Still the loss level is high but distribution utility had put up all efforts in bringing down in last decade. The data for individual discom AT & C loss represented in *(Table.7).* The trend of reduction in AT & C loss clearly understood from *(Fig.8).*

5. Consumer Mix (Pre and Post reforms- GUVNL) :

The statics shows the 60 % rise in nos of consumers under all categories. *(Table. 8)* represents the consumers of GUVNL (All Discom's)

(Table. 9) represents the consumers of DGVCL, *(Table.10)* represents the consumer mix of MGVCL, *(Table.11)* represents the consumer mix of UGVCL and *(Table.12)* represents the consumer mix of PGVCL.

The category wise nos of consumers increase in all Discom's in last decade. It shows the increase in Business of Discom's after reforms.

RESULTS :

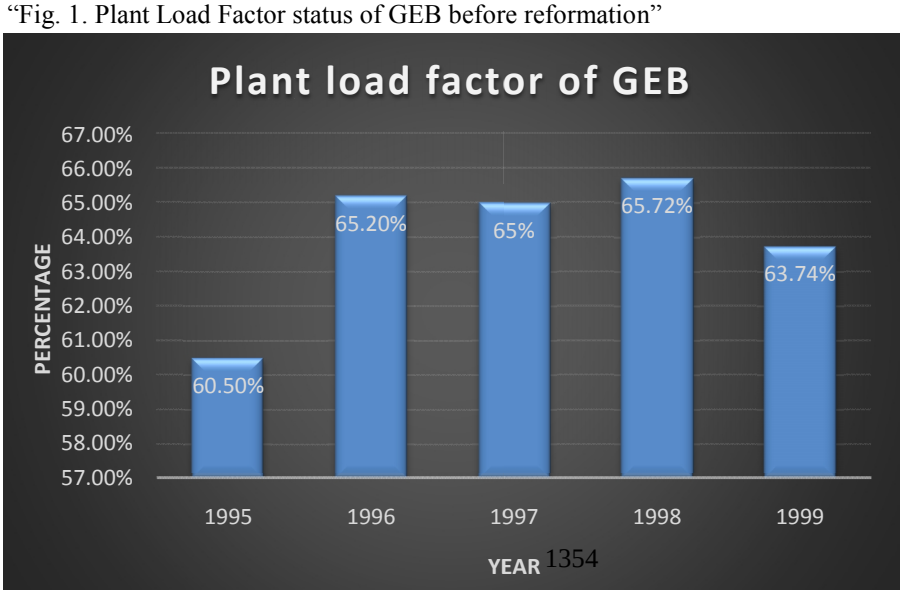
All the business parameters of post reform period indicate the improvement in performance of Power sector in Gujarat.
(Table.13) shows the comparison of parameters for pre and post reform periods. The parameter values reflects the total value for Gujarat (Sum of all discoms)

CONCLUSION:

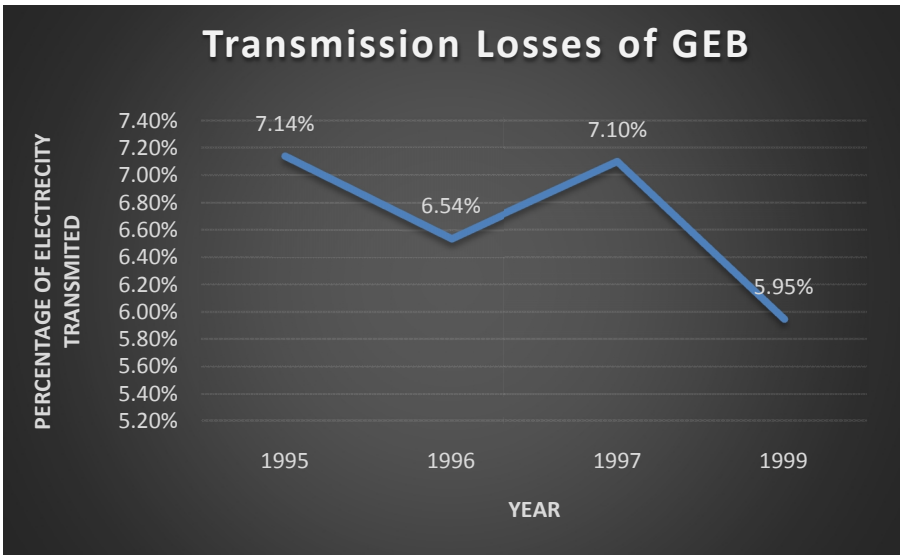
By implementation of reform in power sector-Gujarat, the consumers get benefits in terms of quality power and prompt services. The achievements of all four companies are different because of geographical and other constraint. Still there are scopes for further improvements. On the base of historical data of business parameters, trend of performance of the discoms can be studied further and concrete actions in the direction of better results can be planned.

FIGURES

"Fig. 1. Plant Load Factor status of GEB before reformation"



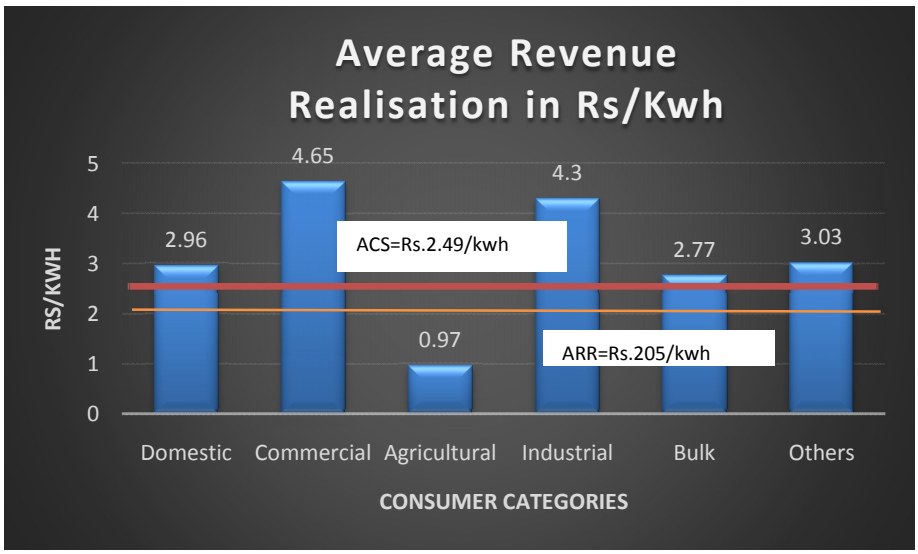
"Fig. 2. Transmission Losses of GEB"



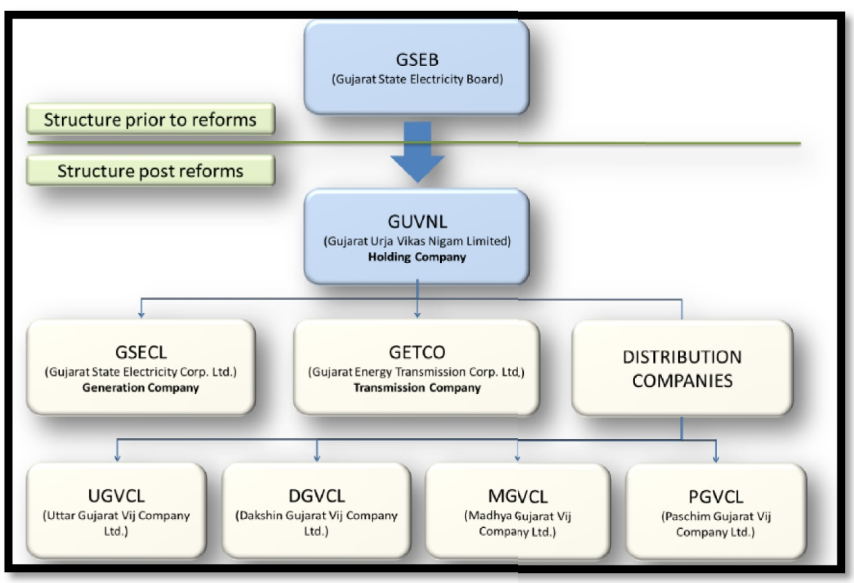
"Fig. 3. Distribution Losses (MUs) of GEB before reformation"



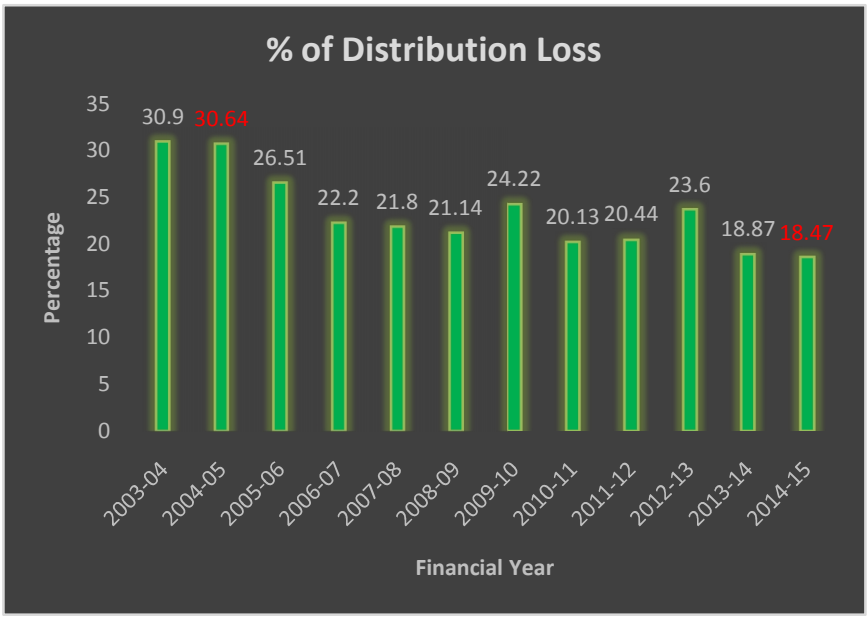
"Fig. 4. Gap between ARR & ACS of GEB before reformation"



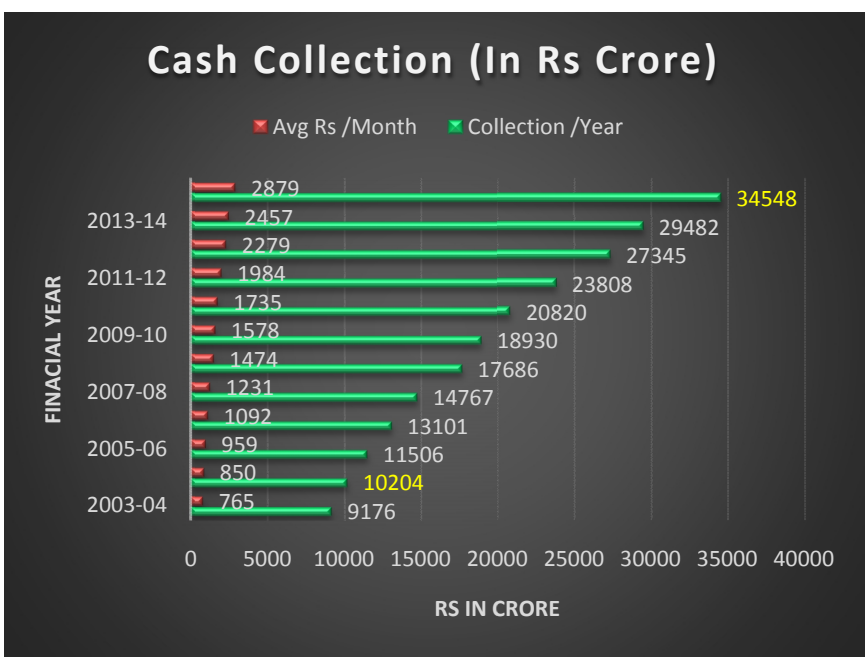
"Fig.5. Structure of GEB before and after reforms"



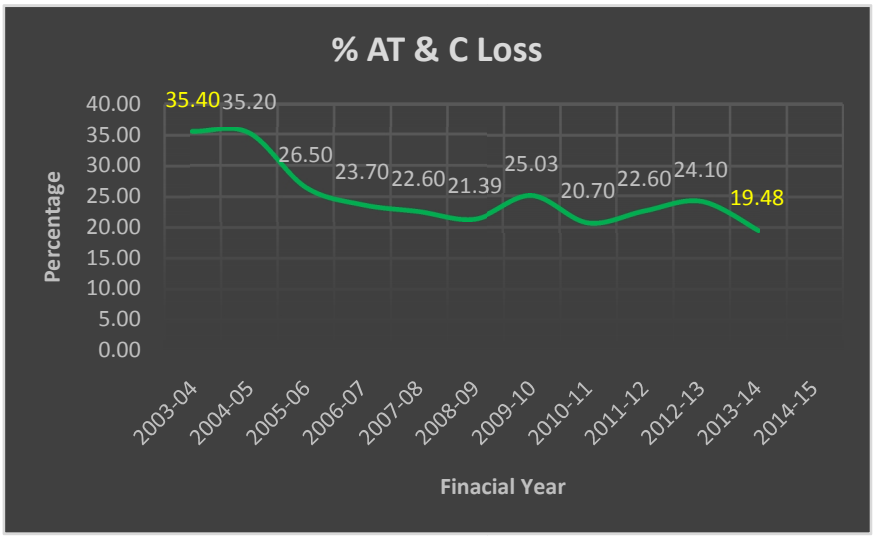
"Fig. 6. Representation of Distribution Losses Pre and Post Reforms"



"Fig. 7. Representation of Cash collection Pre and Post Reforms"



"Fig. 8. Representation of % AT & C losses Pre and Post Reforms"



TABLES

"Table 1.Sector wise Installed Capacity (MW)" (Source: GUVNL Annual report).*(7)*

Sectorwise Installed Capacity (MW)															
Particulars	**Pre Reforms**					**Post Reforms**									
	2000-01	**2001-02**	**2002-03**	**2003-04**	**2004-05**	**2005-06**	**2006-07**	**2007-08**	**2008-09**	**2009-10**	**2010-11**	**2011-12**	**2012-13**	**2013-14**	**2014-15**
GSECL (GEB+GSECL)	4960	4933	4888	4995	4995	4968	4968	4766	4766	5216	5216	4996	5496	5496	5496
IPPs (Public.Sect)	850	946	946	946	946	956	961	966	966	966	1216	1567	1567	1567	1567
IPPs (Pvt. Sect.)	1210	1210	1210	1210	1210	1210	1455	1455	1455	3102	4102	5563	7607	7607	8309
Central Sector	1568	1568	1538	1538	1610	1843	2177	2310	2677	2724	2820	3180	3600	3840	3840
TOTAL	**8588**	**8657**	**8582**	**8689**	**8761**	**8977**	**9561**	**9497**	**9864**	**12008**	**13354**	**15306**	**18270**	**18510**	**19212**

"Table. 2 GUVNL Dist. Losses" (Source: GUVNL Annual report).*(7)*

Year	% Dist Loss
2003-04	30.90
2004-05	**30.64**
2005-06	26.51
2006-07	22.2
2007-08	21.8
2008-09	21.14
2009-10	24.22
2010-11	20.13
2011-12	20.44
2012-13	23.6
2013-14	18.87
2014-15	**18.47**

"Table 3. DISCOM's Dist. Losses" (Source: GERC website)*(9)*

DISCOM	2005-06	2006-07	2007-08	2008-09	2009-10	2010-11	2011-12	2012-13	2013-14	2014-15
UGVCL	23.0	15.8	17.3	14.61	17.24	10.98	9.81	17.83	11.99	14.26
DGVCL	20.0	16.5	15.5	14.74	15.2	15.95	10.24	15.84	13.83	13.69
MGVCL	20.2	15.1	15.9	13.86	13.84	18.58	12.18	17.59	17.56	17.09
PGVCL	38.7	32.5	32.8	30.68	31.88	29.61	27.87	33.61	27.02	28.66

"Table 4. Cash collection (Rs.Crore)" (Source: GUVNL Annual report).*(7)*

Year	Collection /Year	AvgRs /Month
2003-04	9176	765
2004-05	**10204**	**850**
2005-06	11506	959
2006-07	13101	1092
2007-08	14767	1231
2008-09	17686	1474
2009-10	18930	1578
2010-11	20820	1735
2011-12	23808	1984
2012-13	27345	2279
2013-14	29482	2457
2014-15	**34548**	**2879**

"Table 5. DISCOM's Cash collection (Rs.Crore)" (Source: GERC website)*(9)*

DISCOM	2005-06	2006-07	2007-08	2008-09	2009-10	2010-11	2011-12	2012-13	2013-14	2014-15
UGVCL	2110	2494	2804	3814	4176	6064	6064	7383	8107	8415
DGVCL	3338	3566	3745	4574	4923	6637	6637	7632	8591	11546
MGVCL	1683	1924	2109	2768	2910	4092	4092	4621	4565	5428
PGVCL	2890	3671	4158	5376	5698	8319	8319	10130	10572	11804

"Table 6. % AT & C Losses" (Source: GUVNL Annual report).*(7)*

Year	% AT & C Loss
2003-04	35.40
2004-05	35.20
2005-06	26.50
2006-07	23.70
2007-08	22.60
2008-09	21.39
2009-10	25.03
2010-11	20.70
2011-12	22.60
2012-13	24.10
2013-14	19.48

"Table 7. DISCOM's % AT & C Losses" (Source: GERC website)*(9)*

DISCOM	2005-06	2006-07	2007-08	2008-09	2009-10	2010-11	2011-12	2012-13	2013-14	2014-15
UGVCL	23.0	15.8	17.3	14.61	17.24	10.98	9.81	17.83	11.99	14.26
DGVCL	20.0	16.5	15.5	14.74	15.2	15.95	10.24	15.84	13.83	13.69
MGVCL	20.2	15.1	15.9	13.86	13.84	18.58	12.18	17.59	17.56	17.09
PGVCL	38.7	32.5	32.8	30.68	31.88	29.61	27.87	33.61	27.02	28.66

"Table 8. Pre & post Reform Consumer Mix of GUVNL" (Source: GUVNL Annual report). *(7)*

Consumer Category	Pre reforms	Post reforms
	2004-05	**2014-15**
Residential	6400602	10315525
Commercial	947530	79630
LT Industrial	157691	1497713
HT Industrial	5194	12032
Agriculture	664059	1184303
Water works	31793	66581
Street light	18583	30388
Other	43	13
Grand Total	**8225494**	**13186185**

"Table 9. Pre and Post reform Consumer Mix of DGVCL" (Source: GERC website) *(8,9)*

Historical Trend in Category wise Consumers DISCOM : DGVCL											
Category	**2004-05**	**2005-06**	**2006-07**	**2007-08**	**2008-09**	**2009-10**	**2010-11**	**2011-12**	**2012-13**	**2013-14**	**2014-15**
Residential	1239716	1307843	1371201	1472747	1564107	1664164	1795059	1907301	2013974	2121964	2228197
Commercial	173605	189917	201337	212551	223121	233585	246472	252034	11928	12844	13586
LT Industrial	43790	44967	46471	48215	49667	51624	54043	56655	300389	308832	329371
HT Industrial	1799	1773	1827	1932	2060	2256	2419	2608	2803	2970	3156
Agricultural	74917	77184	79101	81279	84317	89080	92135	95177	104646	116541	131941
Waterworks	5780	6329	6759	7373	8315	9879	11438	13056	14813	17081	20008
Street Light	4398	3306	3463	3701	3976	4897	4564	4930	5314	5870	6511
Railway	4	5	5	5	5	5	5	6	6	6	6
Military	0	0	0	0	0	0	0	0	0	0	0
Licencee	1	1	1	1	1	1	1	0	0	0	0
GRAND TOTAL	**1544010**	**1631325**	**1710165**	**1827804**	**1935569**	**2055491**	**2206136**	**2331767**	**2453873**	**2586108**	**2732776**

"Table 10. Pre and Post reform Consumer Mix of MGVCL" (Source: GERC website) *(8,9)*

Historical Trend in Category wise Consumers DISCOM : MGVCL											
Category	**2004-05**	**2005-06**	**2006-07**	**2007-08**	**2008-09**	**2009-10**	**2010-11**	**2011-12**	**2012-13**	**2013-14**	**2014-15**
Residential	1525144	1575838	1642434	1754484	1955446	2064337	2158484	2254446	2323621	2378126	2408189
Commercial	199725	205057	209188	213892	219412	232418	243560	268520	19364	271387	20905
LT Industrial	21466	21853	22973	23751	24638	25714	26724	6679	262273	7145	273644
HT Industrial	862	872	940	994	1123	1186	1293	1439	1595	1684	1741
Agricultural	56229	57332	58738	60147	62585	67320	69859	75533	82289	94682	110267
Waterworks	6013	6304	6711	7110	7330	8112	9114	10363	11622	13178	15133
Street Light	4416	4469	5501	5733	5878	6049	6472	5856	6133	6424	6662
Railway	6	6	6	6	6	6	8	6	6	7	6
Military	0	0	0	0	0	0	0	0	0	0	0
Licencee	0	0	0	0	0	0	0	0	0	0	0
GRAND TOTAL	**1813861**	**1871731**	**1946491**	**2066117**	**2276418**	**2405142**	**2515514**	**2622842**	**2706903**	**2773871**	**2836547**

"Table 11. Pre and Post reform Consumer Mix of UGVCL" (Source: GERC website) *(8,9)*

Historical Trend in Category wise Consumers DISCOM : UGVCL											
Category	**2004-05**	**2005-06**	**2006-07**	**2007-08**	**2008-09**	**2009-10**	**2010-11**	**2011-12**	**2012-13**	**2013-14**	**2014-15**
Residential	1450169	1510239	1574925	1727765	1870033	1998494	2134121	2226817	2298488	2368434	2432337
Commercial	179842	188812	198606	210030	221980	236621	253486	264090	19209	19410	20483
LT Industrial	24684	26068	27366	29317	30350	32004	36029	28742	267930	281019	295632
HT Industrial	1212	1292	1445	1599	1734	1867	2082	2268	2551	2754	3001
Agricultural	201974	205478	208184	213639	217668	221836	225754	233396	245079	261281	283395
Waterworks	9466	9957	10455	11037	11703	12282	12994	13650	14511	15412	16540
Street Light	5844	6194	6514	6998	7455	7944	8449	8992	9693	10274	10923
Railway	1	1	1	1	1	1	1	1	1	1	1
Military	19	4	19	19	19	20	20	5	0	4	0
Licencee	4	4	4	4	4	0	0	0	0	0	0
GRAND TOTAL	**1873215**	**1948049**	**2027519**	**2200409**	**2360947**	**2511069**	**2672936**	**2777961**	**2857462**	**2958589**	**3062312**

"Table 12. Pre and Post reform Consumer Mix of PGVCL" (Source: GERC website) *(8,9)*

Historical Trend in Category wise Consumers DISCOM : PGVCL											
Category	**2004-05**	**2005-06**	**2006-07**	**2007-08**	**2008-09**	**2009-10**	**2010-11**	**2011-12**	**2012-13**	**2013-14**	**2014-15**
Residential	2185573	2251332	2448989	2425826	2566565	2773828	2896718	2987617	3072070	3169624	3246802
Commercial	394358	409830	446593	449142	468382	501466	518134	460768	22552	23340	24656
LT Industrial	67751	70029	75916	75600	77980	80650	83773	158906	568821	583364	599066
HT Industrial	1321	1540	1814	2014	2224	2492	2741	3064	3417	3756	4134
Agricultural	330939	349799	364427	394131	411590	451394	457992	504221	559171	601456	658700
Waterworks	10534	10536	11759	10703	11053	12068	12339	12718	13342	14326	14900
Street Light	3925	3985	4306	4308	4539	4976	5096	5269	5647	6008	6292
Railway	0	0	0	3	6	6	6	10	0	0	0
Military	5	5	6	7	8	8	8	7	0	5	0
Licencee	2	2	1	1	1	1	1	1	0	1	0
GRAND TOTAL	**2994408**	**3097058**	**3353811**	**3361735**	**3542348**	**3826889**	**3976808**	**4132581**	**4245020**	**4401880**	**4554550**

"Table 13. Summary of Parameters for All Discoms (GUVNL)" (Source : Summary of all above)

Parameter	Unit	Pre reforms	Post reforms
		2004-05	2014-15
Generation capacity (Installed capacity)	MW	8761	19212
Distribution loss	%	30.64	18.47
Collection Amount	Rs.Crore/Annum	10204	34548
Aggregate Technical & Commercial loss (AT & C)	%	35.2	19.48 (2013-14)
Total nos of Consumer	Nos	822494	13186158

ACKNOWLEDGEMENT

This research paper is made possible through the help and supports from everyone, including family, friends, guide, colleagues, seniors, in essence all sentient beings. The product of this research paper would not be possible without all of them.

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Optimizing Expanding Ring Search for Multicast Routing in MANET

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ABSTRACT

Expanding Ring Search (ERS) is one of the widely used techniques to reduce broadcast overhead in wireless networks .ERS start its successively searching process with TTL= TTL_START and end if TTL>= TTL_THRESHOLD reaches. Based on TTL_INCREMENT value, ERS process starts by searching successively larger areas in network keeping the source as centered of broadcast. When TTL cross the value of TTL_THRESHOLD, network-wide broadcast is initiated. To take advantage of ERS, we propose a better algorithm named Optimized Expanding Ring Search (OERS) which executes an effectual route discovery scheme by reducing total number of transmissions of route request packets (RREQ) during route discovery process for multicast in MANET. Paper also compares the proposed method with original ERS technique used by known Multicast Ad-hoc On demand Distance Vector Protocol (MAODV) using various parameters like End to End Delay, Goodput and Packet Delivery Fraction for varied number of receivers TTL_Increment values.

Keywords: Control Overhead, Mobile Ad hoc Network, Multicast

INTRODUCTION

Type here. References should be cited in parentheses with an italic number *(1)*. Multiple references are separated by commas *(2, 3)*;an en dash is used for a series of references *(4–8)*.

The goal of the Expanding Ring Search (ERS) technique during route discovery process is to find mobile nodes that have the required route information to the destination mobile node in their route table or the destination mobile node by flooding of route request packets (RREQs) in a controlled manner *(1-4,13)*. To restrict the flooding of RREQs, TTL based ERS is used by various routing protocols in MANET. The ERS based on TTL restricts its searching range by RREQs with predefined TTL value. TTL value specifies the radium of a searching space. For each time if it fails to find any destination node or any node that has route information to the destination node, the source/sender node rebroadcasts the RREQ with an augmented TTL number to allow the RREQ to cover a larger area. Even with the controlled manner of flooding, expanding ring search still suffers from high control overhead because each time route discovery process starts from the source/sender node. Researchers says that when a larger area of the network needs to be searched, the cost drastically increases.

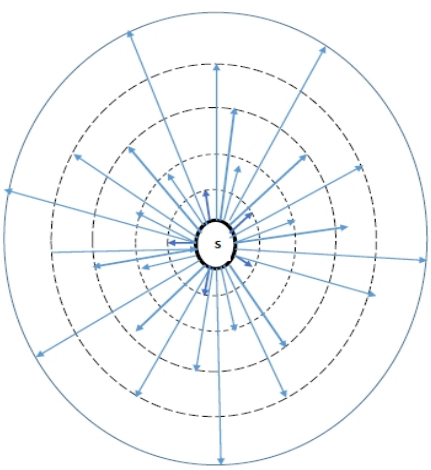


Fig. 1 TTL based ERS

In figure 1, we consider that source/sender node S wants to discover the destination node. Then it will start its searching process by broadcasting the RREQ to all nodes which are one hope away in the case of TTL_START=1. If target node is not found then TTL will be incremented based on TTL_INCREMENT value. Based on updated TTL value, again node S will broadcast the RREQ. Node S will repeat the same process until TTL crosses the TTL_THRESHOLD or in between destination node is found. From the figure 1, we can see that more number of RREQ packets are required during route discovery process.

PROPOSED WORK

We suggest an alternative ERS scheme to support reactive types of protocols in MANETs, which reduce the control overhead during route discovery. The basic route discovery structure of Optimize Expanding Ring Search (OERS) is similar to that of the TTL based ERS. Main differences from the TTL based ERS is that the OERS does not broadcast the RREQ every time during its route search procedure from the source/sender node. Instead of that it unicast the RREQ, if target node is not found then it broadcast the RREQ and find the target node. Still target node is not found then it unicast the RREQ and find the target node. In this way, alternatively unicasting and broadcasting of RREQ will be done instead of every time broadcasting of the RREQ to find the target node during route discovery process. Because of unicasting/broadcasting of RREQ, many number of control packets required during route discovery process.

In figure 2, we have optimized the ERS based on modifying the forwarding RREQ packets. Instead of broadcasting the RREQ packets, we are unicasting the RREQ, then broadcasting the RREQ, then unicasting the RREQ, then broadcasting the RREQ and so on, until target node is found. Blackarrow shows the unicasting the RREQ and blue arrows show the broadcasting the RREQ. During unicasting, RREQ will be forwarded to furthest node from the neighbor list based on current TTL value. From figure 1 and figure 2, we can compare the required RREQ for original ERS and proposed ERS technique *(8)*.

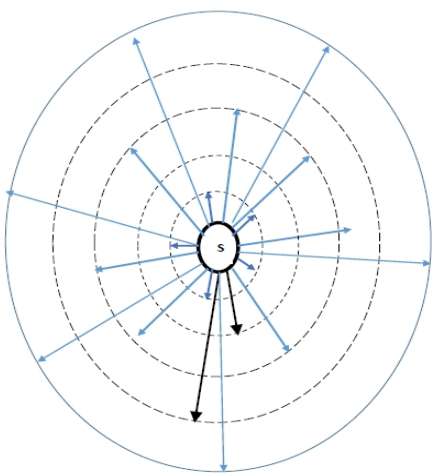


Fig.2 Optimized ERS

To justify the proposed concept, assume ad hoc network with network diameter five and consider total five mobile nodes in radio range of every nodes, where network diameter is the lengthiest path between any two mobile nodes. To find out the route to the farthest node located five hops away from the source node, broadcasting RREQ will start with TTL equal to 1 and increasing by TTL_Increment with each re-broadcast of RREQ up to the predefined network diameter or TTL_Threshold. Now for designed network, total 4 +8+20+32+68=132 transmission of RREQ will be required by OERS instead of 4+16+52+160+484=716 transmissions of RREQ done by TTL based ERS which shows the effective

reduction in control overhead during route discovery process. And this difference in reduction will go on increasing for bigger values of network size.

The proposed technique uses a fast and improved route discovery by modifying the expanding ring search technique which is used by MAODV, which is state of the art tree-based multicast routing protocol *(12, 14)*.

PERFORMANCE EVALUATION

The performance of OERS based protocol is compared with the known multicast routing protocol MAODV *(5-7)* which uses the TTL based ERS. For the purpose of the simulation of OERS based protocol, NS-2.26 simulator has been used. Data traffic was generated using constant bit rate (CBR) UDP traffic with 32 network size with one sender and 2, 3, 6,9,12 varying mobile nodes acting as receivers in the multicast group. All wireless mobile nodes are randomly distributed in a square area of 500m x 500m. The nodes use the IEEE 802.11 radio and MAC model provided by the CMU extensions. Each simulation executes for 200 seconds. The number of receiver mobile nodes is varied from 2 to 12 nodes to observe the effect of the number of receiver nodes on the performance on the system performance. Also other parameter, TTL_INCREMENT value is varied to see the performance of proposed concept. TTL_INCREMENT value is varied with 2,3,4,5 and each time proposed concept is compared with original approach for various performance metrics. With that based on node speed value, performance of OERS with MAODV compared.

Various metrics used for performance evaluation are: (i) Packet Delivery Fraction (PDF) — the ratio obtained by dividing the number of data packets correctly received by the destination by the number of data packets originated by the source. (ii) Goodput (iii) E2E (End To End) delay of data packets - this includes all possible delays caused by queuing delay at the interface, buffering during route discovery, retransmission delays at the MAC, propagation and transfer times *(9-11)*. Figure 3 shows the Packet Delivery Fraction for 2,4,6,8, 10 and 12 number of receivers.

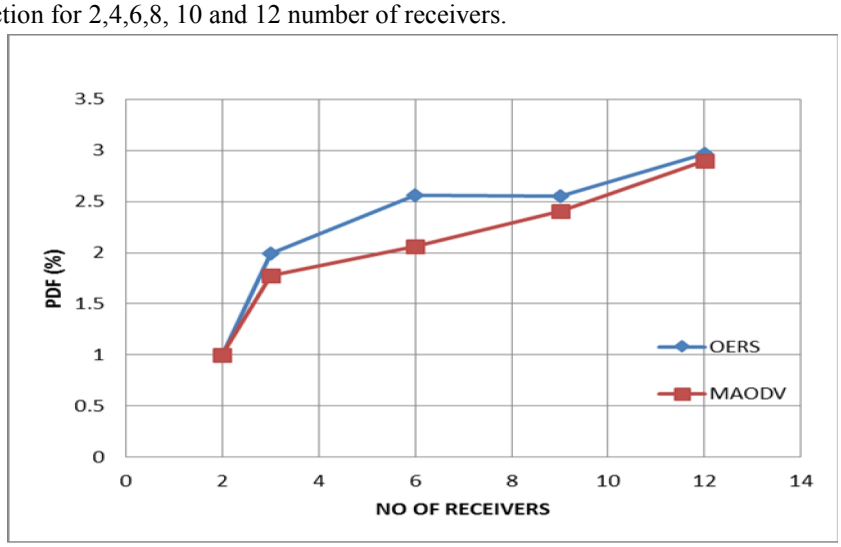


Fig. 3 Packet Delivery Fraction

Figure 4 shows the throughput and figure 5 shows the End To End delay for same scenarios. From that we can compare the performance of MAODV and OERS and also analyze the better performance of proposed method compared to original one. Considering all these results, we can see that OERS give its good performance compared to MAODV even though number of receivers are increased.

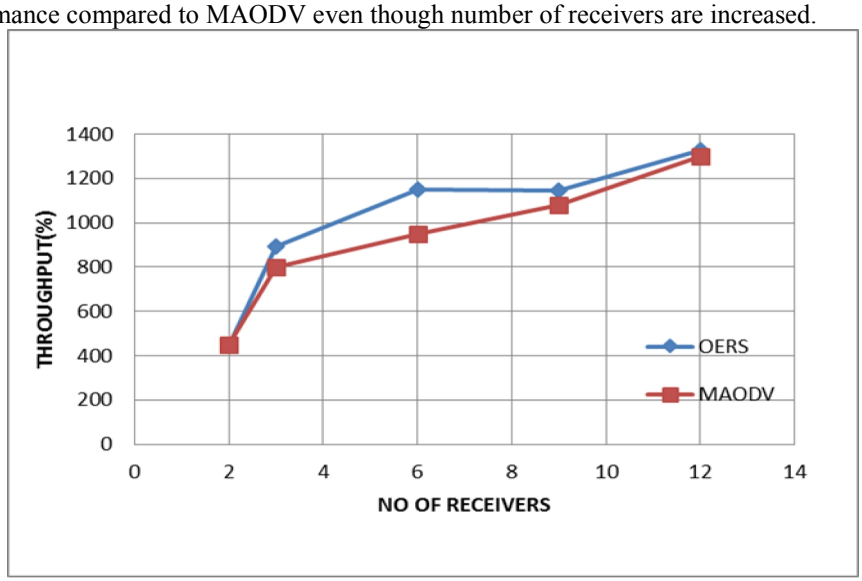


Fig.4 Throughput

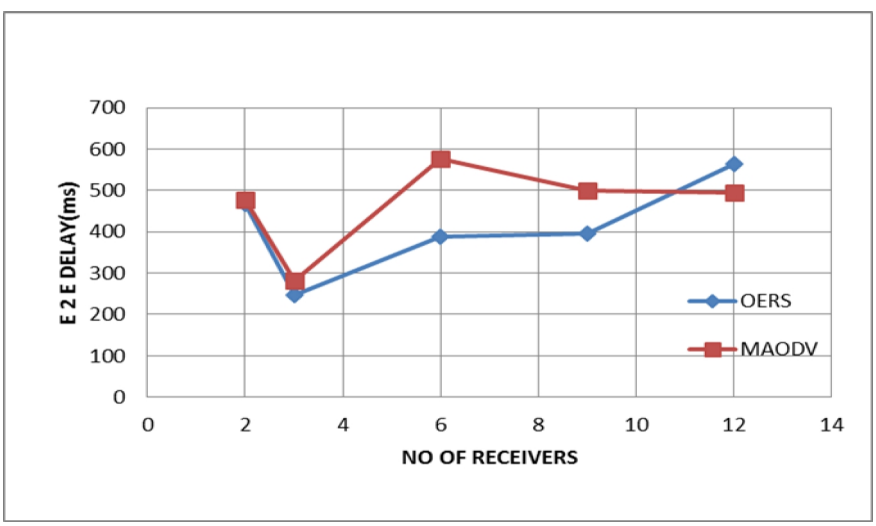


Fig.5 E2E Delay

To analyze the effect of TTL_INCREMENT parameter value on the performance of MAODV and OERS, we change the value of TTL_INCREMENT from 2 to 5 with TTL_START=1. From figure 6 and figure 7, we can see the better performance of OERS compared to MAODV. Figure 8 also

support the better outcome of OERS compared to MAODV. As and when we increase the TTL_INCREMENT value, number of generated rings during packet forwarding process will be reduced and also load on intermediate node will also be reduced.

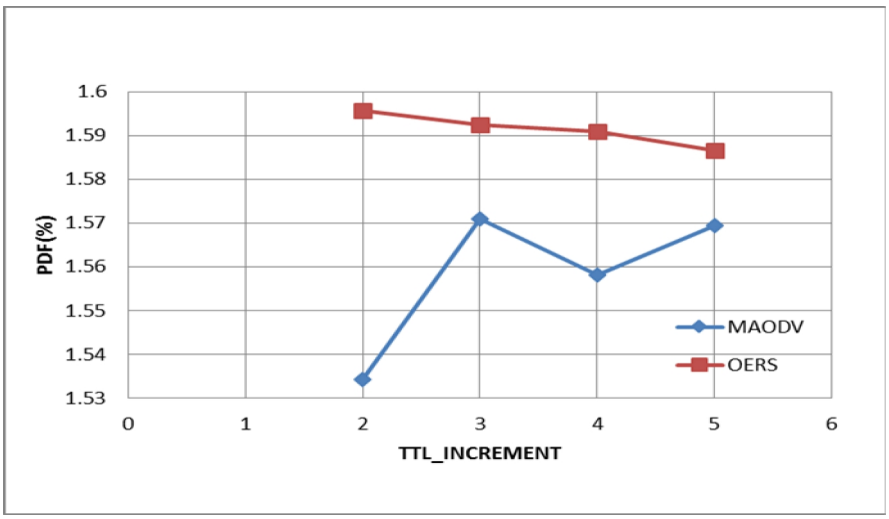


Fig.6 Packet Delivery Fraction

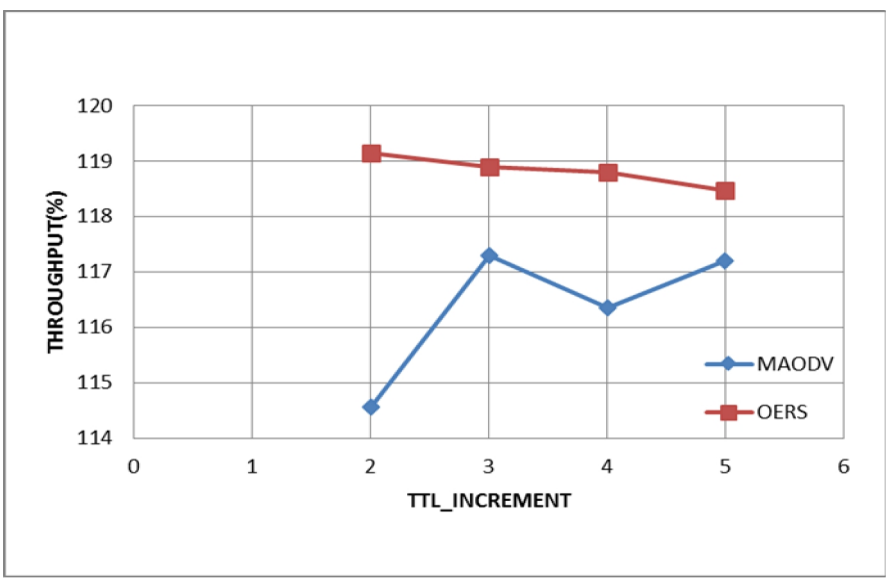


Fig.7 Throughput

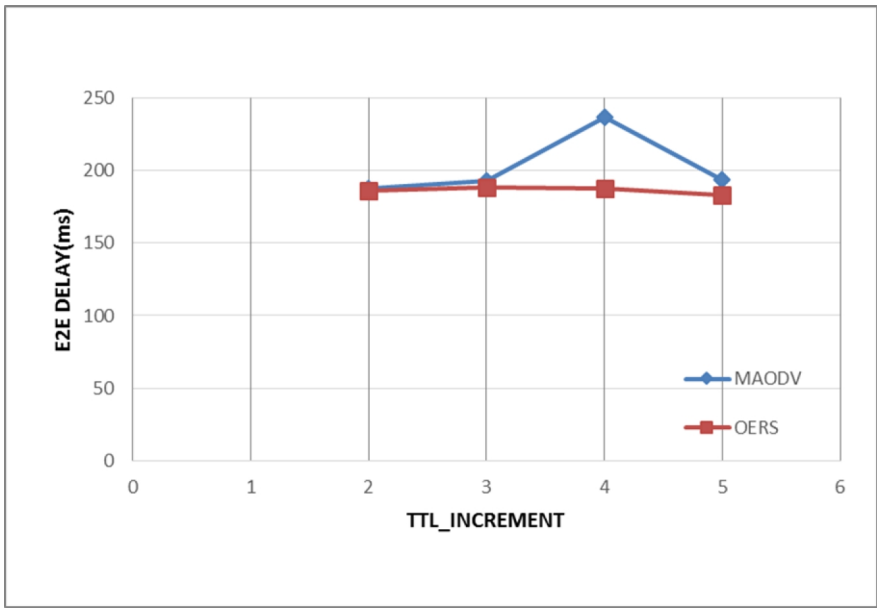


Fig.8 E2E Delay

CONCLUSION

The proposed methodology uses a fast and improved route discovery by modifying the expanding ring search technique which is used by MAODV, which is state of the art tree-based multicast routing protocol. Because of reduction in required transmissions of RREQ during route discovery process, the lifetime of mobile nodes and of Adhoc network is also sustained. It also leads less chance of network clogging and less overheads as well.

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MANCAF: A Framework for Building Collaborative Applications in Mobile Ad Hoc Networks

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ABSTRACT

This paper presents the MANCAF framework that helps the application developers to design a collaborative application for mobile devices. With this framework application developer can easily develop a collaborative application. The framework provides the different services like managing, joining of new device and lost device. The framework also provides the service to exchange the message between devices. Currently the framework has been implemented using J2ME and tested in simulator. The paper elaborates the architecture and describes main features of framework which application developer has to use.

SUMMARY

This paper describes the framework to develop collaborative application for mobile devices.

Keywords - Collaborative Application Framework, Framework Design, MANET

INTRODUCTION

Now days, Mobile devices are most popular because of their functionality from to dial a number to digital helper. Mobile applications are more flexible and can be integrated with the existing services by using web interface. Collaboration supported by mobile devices has offered more advantages to users and also more challenges for application developers.

From most recent couple of years, the regions of Computer Supported Cooperative Work (CSCW) (*11*) and Mobile Computing (*12*) were intrigued field from the individual user and business group. CSCW is characterized as the investigation of how an individual cooperates by utilizing innovation of Computer systems administration, and related equipment, programming techniques and strategies (*1*).

Wireless devices support personal area network (PAN) technologies like IrDA and/or Bluetooth (*2*). This technology can be used to transfer the data between mobile devices. It can be pictured as an advanced circle around the mobile phone which bolsters a collaborative network for clients in an extent. A MANET is a self-configuring network, which can be utilized to exchange the data between people with mobile devices. In MANET, devices can connect to and disconnect from the network dynamically, making the topology unstable and random. Mobile devices in MANET can communicate with each other by initiating direct request by user, automatically by mobile device or hybrid of them (*3*).

The proposed MANCAF framework is introduced to support collaborative application development for mobile device in short span of time. The Main idea behind the framework is to present an advanced programming framework to developers so that they only need to use simple methods to manage the different task of MANET. The primary aim of this paper is to describe requirement of Collaborative application framework and framework itself.

The rest of the paper is ordered as follows. Related work section describes the other similar framework. Material and methods describes the requirement finding of framework, architectural design of framework. Result and discussion section describes how the framework can be utilized by application developer for application development. Finally, Conclusion section concludes the paper with direction about future work.

RELATED WORK

In this section, we describe the related project that have developed a framework to design and develop collaborative application.

Mobile Chedar(*7)* is middleware allowing mobile devices to access central chedar network and exchange the information with each other. The purpose of Chedar is to provide flexible API to application developer for developing collaborative application. It is implemented using J2ME and Bluetooth is used for communication. But the communication is based on Mobile Chedar gateway device which runs on PC. This approach has a problem of single point of failure.

Proem (*8)* is framework with an objective to provide a framework to develop collaborative application for ad-hoc network. It is implemented on Java and runs of PDAs and other wireless devices. Proem design is independent from network protocols. It can be implemented on top of TCP/IP, HTTP and others. J2ME version of Proem has not been succeeded.

JXTA (*9, 10*) provides an APIs for PC-to-PC communication which are independent from platform and communication medium. JXME is a light weight implementation of JXTA for mobile devices. It provides full JXTA services through relay host. No stable implementation is currently there.

MATERIALS AND METHODS

A. Defining Scenario

The process of identifying the user requirements is the major challenging task for application developer. The collaborative applications are also among them. Sutcliffe (*4*) shows that scenario is best used to extract the pattern in real world element and as design inspiration. For MANCAF framework design requirement, we have proposed the different scenario. There are different approaches available to write the scenario. We have used unstructured free-format narrative text because it is best when we considering the completeness of the scenario (*5*). The main reason of choosing unstructured free-format narrative text is that we want to extract the all possible action so that we can design a good framework. Following is the short description of three different scenario proposed by us.

A.1 Strategy Game

This scenario is based on the multiplayer game. Group of people standing nearby at some place and start the playing of game. Each user's movement is reflected on other user's screen. Each player does their movements when they get the turn. This continues, turn after turn, until one of the players has fulfilled its mission. At last final scores are displayed before exits the game.

A.2 Exchanging information between users

This scenario is related to explore the possible usages of the combination mobile phones and wireless network in large scale environment. The key aspect for this scenario is exchanging information between users who are connected to the network. This scenario proposes an application using which user can connect with network to exchange the information with other users of the network.

A.3 Visiting Card Exchange

This scenario deals with the task of exchanging some kind of file between two users using their mobile devices on wireless medium. This scenario proposes an application which can be used to exchange the visiting card between two users.

A. Scenario Analysis

The scenario describe above is the examples of possible collaborative applications which can be developed using MANCAF framework. In (6), Sutcliffe present a method to extract requirement from scenario scripts. Different steps are performed based on Figure 1 which is described follow.

B.1 Goal Analysis

Each goal of the described scenario is assessed to see if they are sustaining by scenario description. After this process, the requirement specification is sketched.

B.2 Inbound Event Analysis

Inbound events are the events triggered by user or object to the system. The system is checked to verify that it can handle all required incoming events.

B.3 Characterize System Output

In this step, different kinds of outputs are described. It also ensures that output is generated whenever it is needed.

B. List of Requirement for Framework Design

Table 1 shows the important list of requirements which framework needs to fulfil. These requirements are identified by the analysis of scenario.

MANCAF Design

The design of MANCAF is based on layered architecture to provide features like modularity and simplicity. Each layer is assigned a specific responsibility and it is based on the below layer. The drawback of this approach could be slower execution of application if it contains many layers to perform operation. MANCAF framework can be used in mobile devices which have limited resources so we limit the number of layers used in the architecture. Figure 2 shows the compact design of framework and Figure 3 shows the high-level design of framework.

The MANCAF architecture contains the following part:

Device: It represents the mobile devices which run the application develop using framework. Two or more devices create ad-hoc network.

Group: It is a logical collection of devices within communication range and aware of each other's existence. All devices can communicate with each other in a group.

Service: It is a unique ID for the device application running the MANCAF framework. The application which runs the same service can able to communicate with each other.

Network: It represents the communication medium used in the framework to communicate with neighbor devices.

Message: It is an entity which represents the different kinds of data transfer between devices. It can be sent to single device or a group of devices. It can contain text, objects or binary data like images, video or audio.

Session: It represent the duration of all communications between devices in a group. It maintains information about devices, group and communication medium.

Framework: It is core entity between application and system. It provide interface to developer and hides the complexity.

Application: It is application developed on some platform using MANCAF framework. It controls the framework.

RESULTS AND DISCUSSION

This section describes the implementation of MANCAF. The main purpose of MANCAF framework is to design a framework for collaborative application. In this section, we describe the main parts of MANCAF framework through code examples. Currently MANCAF framework implemented using J2ME platform with Bluetooth as communication medium.

The first step is to import all necessary part of MANCAF framework into the project. The application MIDlet must implement interface named FrameworkSubscriber interface and then framework variable must be created as shown in Figure 4. Then framework initiated with different parameters like group name, service name and a network medium used. The mobile device which runs the application is now ready to discover the service of other devices running the same service.

After initializing the framework, the application needs to implement the methods which provide the information regarding status of devices, devices search and exchanging the message between devices. The deviceFound is invoked when a new device is found in the network. MANCAF do not intimate about the new device found in a network once the initial search process is completed. Application developers need to implement the functionality of searching a new device in application. The deviceLeft is invoked when background thread detected that one of device from the group is not reachable. The sample code for implementation of methods is shown in Figure 5.

CONCLUSION

We have presented a MANCAF framework to develop collaborative application for mobile devices. The implementation of MANCAF is very simple and contains a few lines of code to initialize the framework. The MANCAF provide different methods to handle the events. The future work is to develop many applications using MANCAF framework and test them with specified requirements.

FIGURES

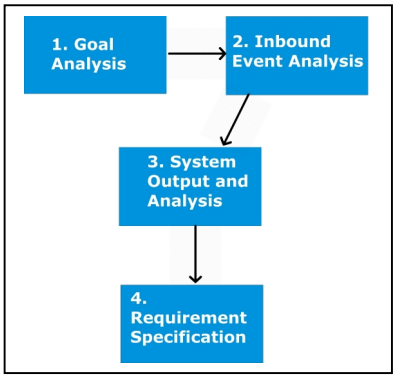


Fig 1. Steps for Scenario Analysis

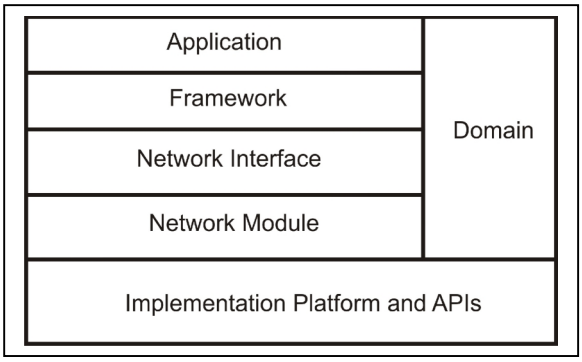


Fig 2. Design of MANCAF Framework

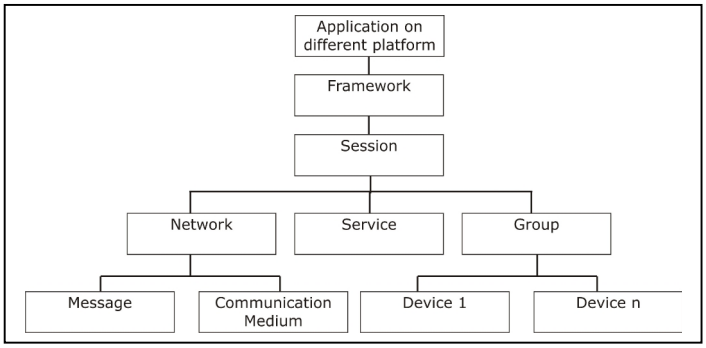


Fig 3. High level design of MANCAF Framework

```
Public class MANCAFChat extends MIDlet implements FrameworkSubscriber, Commandlister
{
    private Framework frameworkObject;
    frameworkObject = Framework.getInstance("ChatGroup","MultiChat",new Bluetooth(),this);
    frameworkObject.initialize();
    frameworkObject.search();
}
```

Fig 4. Sample code for Framework initialization

```
public void deviceFound(Device device)
{
    dialog.append(device.getDeviceName() + "has joined",null);
}

public void deviceLeft(Device device)
{
    dialog.append(device.getDeviceName() + "has left",null);
}
```

Fig 5. Sample code for method implementation

TABLES

Table 1. Framework requirements

ID	Requirement description
R1	The framework must support mobile devices
R2	The framework must be able to work with different network mediums
R3	The derived application must not be dependent on what network medium is used by framework.
R4	The framework must be able to create a network without any central device
R5	The framework must be able to create a group of two or more devices
R6	The framework must be able to transfer the data to different connected devices
R7	The framework allows the application to form a new group or join existing group
R8	The framework must provide searching facility to find the existing group
R9	The framework able to query the application for any incoming or outgoing connection
R10	The framework must notify about new device joining notification to every connected devices.
R11	The framework must be able to limit the number of devices in a group
R12	The framework must be able to identify the source which originate the transfer
R13	The framework must be able to send the same data to multiple devices
R14	The framework must allow the continuous searching of group
R15	The framework must support different resource management technique to balance the load

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Petrographic Examination of Hardened Concrete Mix with Blast Furnace Slag

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ABSTRACT

Concrete petrography is one the latest and newly adopted technology to understand how concrete internally in its present condition. It is not only used for assessment work of old concrete but also useful in assessing long term behaviour of concrete mix with blast furnace slag (BFS). In this paper, petrography examination is consists of concrete core with specific exposure condition converted into slices for selected grade of concrete wherein BFS is used as sand in production of concrete. These slices are observed with scanning electron microscope (SEM) to identify basic components in concrete and cementitious materials, study micro cracks, examine coarse and fine aggregate distribution in concrete mass, bond between cement paste and aggregates, bond between reinforcement and concrete mass, condition of reinforcement with respect to its corrosion etc. This study ultimately provides detail information regarding concrete in most accurate manner related to its durability and long term behaviour.

SUMMARY

Petrographic examination attribute concise information regarding concrete with BFS for its durability and long term behaviour and commercial use of BFS can be initiated in the production of concrete.

Keywords: Petrographic Examination, Hardened Concrete, Blast Furnace Slag, Sand (Fine Aggregate)

INTRODUCTION

Concrete petrography is one of the latest and newly adopted technology for hardened concrete. The petrographic examination of concrete is, in many respects, more qualitative than quantitative. Petrographic examination of concrete is visual examination and analysis in terms of properties of individual particles. By petrographic examination, the relative abundance of specific types of aggregates is established along with physical and chemical attributes of particle shape, surface texture, pore characteristics, hardness, chemical resistivity etc. are described*(2)*. The presence of contaminating substance of aggregate used is determined in relation to proposed waste material utilized in concrete mix. The petrographic examination consists of preparation of concrete specimen mix with BFS of grade M40 and converted cylindrical specimen into slices. The crushed BFS is utilized as full replacement of fine aggregate (sand) in concrete production and cured the prepared specimen of concrete for 365 days of curing before it prepared for petrographic examination. These slices are observed in scanning electron microscope (SEM) at different magnification level to examine and analyze the sample for various parameters. The plates or images obtain from SEM, indicates the positive output of various parameter of concrete sample mix with BFS and provide much information related to durability and long term behavior of concrete with waste material like BFS utilized for production of concrete. Thus, this paper shows the advantage of employing petrogrphic procedure to analyse the concrete microstructure obtained in BFS induced concrete and possibility to predict its behavior after course of time *(10)*.

MATERIALS AND METHODS

2.1 Materials used:

2.1.1 Cement:

OPC 53-grade satisfying the requirements of IS:12269 having Specific Gravity-3.15, Fineness- 3.25% on 90μ IS sieve has been considered.

2.1.2 Coarse Aggregate: *(5, 6)*

Locally available angular crushed stone of normal size 20 mm down confirming to IS:2386-1963 12 having Specific Gravity -2.77, Water absorption-0.6% and Fineness modulus (F.M.)-7.18 has been selected for investigation.

2.1.3 Blast Furnace Slag: *(5, 6)*

Air cooled blast furnace slag obtained from industrial area of Rajkot city, Gujarat (India) of Size-4.75 mm down up to 150micron (i.e. as fine aggregate) having gradation shown in figure-1, Specific Gravity -2.72, Water absorption-1.35%, Fineness modulus (F.M)-2.82 and confirming to zone-I as per IS:383-1970 has been used. Table 1 shows the chemical properties of blast furnace slag used.

2.1.4 Water:

Ordinary tap water was used for both mixing and curing of concrete specimens.

2.1.5 Concrete Mix: *(7, 8, 9)*

The method of mix design as per IS: 10262-2009 and IRC: 44:2008 has been employed to design the mix for M40 grade of concrete. This design mix is prepared for concrete with BFS as fine aggregates. The details of design mix for 1 cubic meter of concrete produced is shown in Table-2 for M40 grade of concrete selected which is most common grade of concrete utilized for pavement construction. The

appropriate dosage of super plasticizer is also utilized for concrete mix to achieve desire strength & workability which is required for higher grade of concrete like M40.

2.2 Equipment used: *(11)*

The main equipment required for petrographic examination is the microscope along with some other miscellaneous apparatus like Diamond blade watering saw, Ultrasonic bath, Drying/curing oven, Glass plate, Abrasive papers, Polishing cloths, Lint-free cloths, Compressed air etc. Scanning electron microscope (SEM) has been used in this study because the application of the SEM in petrographic examination and analysis of concrete microstructure is becoming increase day by day. SEM imaging provides detailed images of the microstructure that augment those from stereo and optical microscopy. The primary advantages are the high-contrast images of the microstructure, the high spatial resolution of the images, and the ability to perform simultaneous imaging. The SEM scans a focused beam of electrons across the specimen and measures any of several signals resulting from the electron beam interaction with the specimen. The most commonly used imaging modes are secondary electron, backscattered electron and x-ray out of which backscattered electron mode is best suited for higher magnification examinations of microstructure features.

2.3 Specimen detail for petrographic examination: *(1, 11)*

The specimen used for petrographic examination is cylindrical mould of size 100mm X200mm which is casted for M40 grade of concrete. Two types of cylindrical specimen are prepared for petrographic examination. Specimens with plain concrete and embedded reinforcement are used for study to see the effect of reinforcement with concrete mix with BFS. This cylindrical specimen is cured for 365 days of curing period to study the long term effect of BFS on concrete mix as well as effect due to embedded reinforcement. These specimen are use to prepare thin millimeter-sized sections (slices) for petrographic examination with help of diamond blade watering saw machine as shown in figure-2. These samples are prepared as per ASTM C-856 guidelines.

RESULTS AND DISCUSSION

Based on the various specimen prepared for petrographic examination as per ASTM guidelines, images and observations are taken using scanning electron microscope (SEM) at different magnification level to ensure maximum understanding can be achieved related to concrete mix with BFS. Following Plate or images illustrated the long term effect of BFS mix concrete along with embedded reinforcement in concrete mass.

Plate 1 represents arrangement of BFS in thin sections and is quite distinctive in appearance. Unhydrated BFS is primarily glassy in nature and, therefore, almost completely lacking in birefringence. Distribution of BFS particle used as fine aggregates in concrete mass is well arranged and describes the shape of the particles. At one of the places micro-cracks are present adjoining to BFS particle in cement paste. As such no entrapped air is shown in cluster of cement paste.

Plate 2 represents typical image between embedded reinforcement and cement paste. The reinforcement is shown as light reflecting nature while concrete mass is brownish in colour. There is slight edge like gap between reinforcement and concrete mass which is not predominant for formation of any type of crack. As such the image indicate very good bond between reinforcement and concrete mass. Course aggregate with dark brown colour very near to the reinforcement also shown rigid bond with cement paste without any fractural crack, indicate good bonding between three different materials attached together.

Plate 3 represents the very important area where the paste meets the aggregate surface is sometimes called the interface transition zone (ITZ). The quality of the ITZ or the paste-aggregate bond has obvious implications for the strength and durability of concrete. Narrow separations between this aggregate and the paste are sometimes referred to as bond cracks. *(11)* Image indicates very good bond between the paste and the aggregate and no cracking or porosity found within the aggregate and the distribution of the cement particles. At one of the place bubble like texture shown the unhydrated cement particle present. Some surface void shown in the texture of BFS particle is basically the characteristic of BFS itself as it forms during crystalline process of molten slag.

Plate 4 represents the general microscopic arrangement of BFS made concrete at magnification level of around 25x. The course aggregates are well spreads with grayish to blackish colour shown good bonding characteristic with other material of concrete. BFS used as fine aggregates in concrete are blackish in colour well distributed in concrete mass showing sharp angular texture facilitate good bonding between cement paste and BFS. Cement paste in concrete mass grayish to whitish appearance shown good hydrated paste without any entrapped air present.

Plate 5 represents the reinforcement steel embedded in concrete at magnification level of around 30x. No corrosion effect is shown in the sample after 365 days of curing. The periphery of reinforcement along the concrete mass is very light in colour shown no evident of corrosion. Indirectly it is also proposed the good condition of concrete with embedded reinforcement ensures better water tightness.

Plate 6 represents bond between course aggregates, fine aggregates and cement paste in which cement paste is rigidly attached with course aggregate surface angular in nature and no micro-crack is found in microstructure. There are some air void are present in paste structure as entrapped air in centre of concrete mass. BFS utilized as sand material in prepared concrete is shown as amorphous and reflected glassy in nature firmly attached to paste formation. Course aggregates are shown as dark brown colour while BFS is light brown colour. The colour of cement paste is very light brown colour indicate the fresh concrete in terms of age. As the concrete gets older paste colour becomes darker.

CONCLUSION

The developed technique for petrographic examination of concrete produced with BFS has demonstrated that this waste material used as fine aggregates in concrete fulfils almost all parameter just like natural river bed sand.*(10)* Petographic examination have confirm good bond between cement paste and BFS. The specimen prepared with embedded reinforcement shown no corrosion as well as no adverse effect due to chemical reactivity of BFS particles with reinforcement. This examination also promises about leachate and no formation of leachate is notice on specimen which is observing after long period of time. This examination reveals no micro cracks are found in the particle of BFS and superior rigidity is shown between BFS and course aggregates as well as BFS and cement paste. Petrographic examination envisages good cluster of concrete mass among all material present along with specimen of embedded reinforcement. By this examination and analysis, commercial use of BFS can be initiated in the production of concrete in general and for pavement concrete in particular.

FIGURES

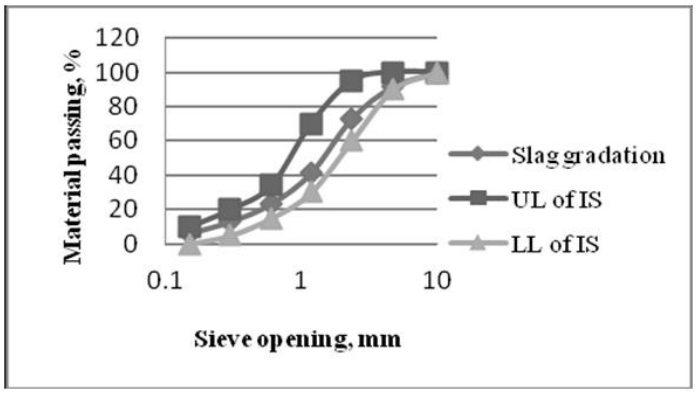


Fig. 1 Gradation of blast furnace slag *(4)*



Fig. 2 Sample Preparation

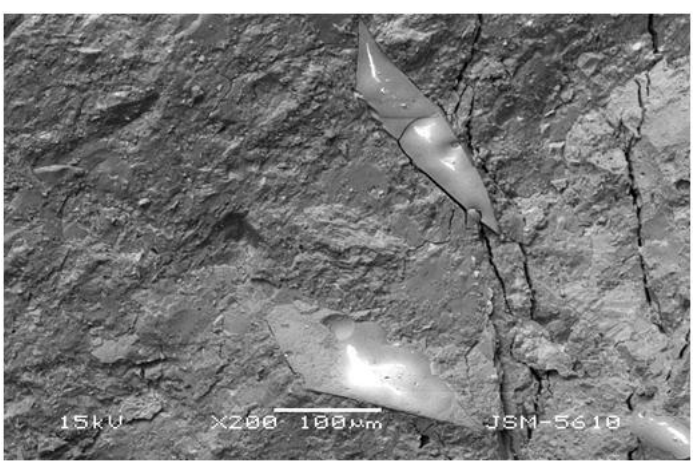


Plate 1 Arrangement of BFS

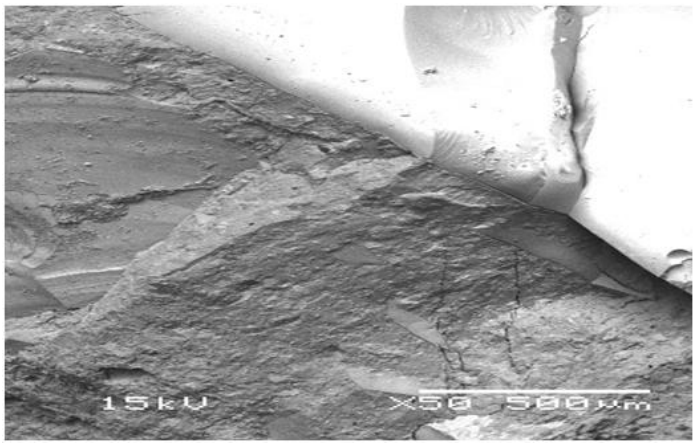


Plate 2 Bonding with reinforcement

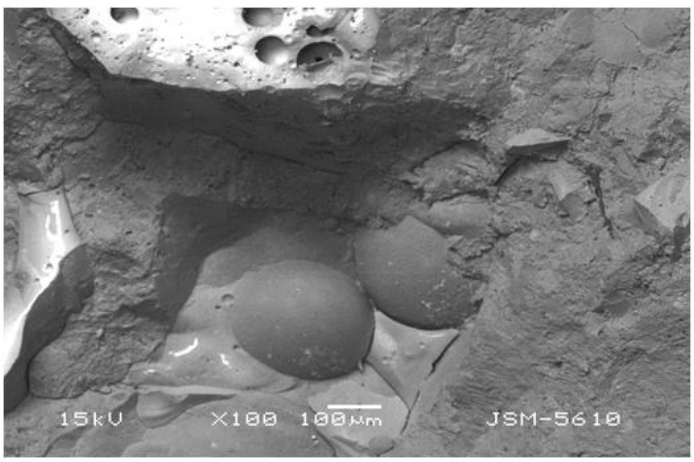


Plate 3 Bonding of BFS



Plate 4 Microscopic arrangement of BFS made concrete



Plate 5 Reinforcement without corrosion

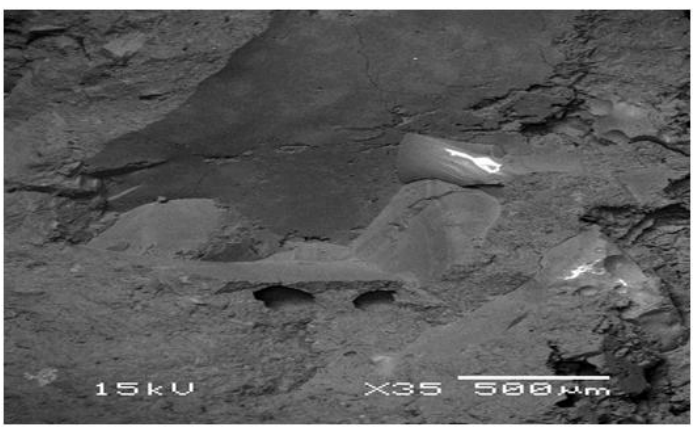


Plate 6 Bonding between the ingredients

TABLES

Table 1 Chemical Properties of Blast Furnace Slag *(3)*

Chemical Properties of BFS in %	CaO	SiO_2	Al_2O_3	MgO	Fe_2O_3	SO_3
	26.5	34	17.25	7	18.25	0.5

Table 2 Mix proportions for 1 cubic meter of concrete with BFS *(4)*

Grade of concrete	Cement (Kg)	BFS (sand) (Kg)	Coarse aggregates (Kg)		Water (Kg)	Admixture (Kg)
			10mm down	20 to 10mm		
M40	416	684	498	750	175	2.5

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th& Jan. 6th, 2016)

(Proceedings available for download at rku.ac.in/icre)

RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

Performance Evaluation of Scheduling and Clustering Algorithms for Dynamic VM Allocation in Cloud Computing

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ABSTRACT

Cloud computing is flexible and scalable virtualized technology. Cloud computing provides services on demand over the internet. Services provided by the cloud are categorized based on purpose. The cloud provides services like Software as a service (SAAS), Platform as a service (PAAS), Infrastructure as a service (IAAS).VM allocation allows sharing of virtual machines over the data centers. In this paper dynamic VM allocation policy is there that takes VM as per user requirement and allocate them in cluster form to the available data centers. For allocation of VM there are various scheduling algorithms are there with their advantages and disadvantages. Clustering of VM is done by using clustering algorithm. Using this policy efficient load sharing and CPU utilization can be achieved.

SUMMARY

Dynamic VM allocation using clustering approach will provide efficient load balancing and resource utilization among the data centers and improve the performance.

Keywords: Virtualization, VM allocation, Cloud computing.

1 . INTRODUCTION

Cloud Computing provides services on demand over the internet. Cloud infrastructure consists of a shared pool of hardware and software commodity resources that distributed geographically and provides on demand services *(1)*. Cloud computing basically provides three types of services:

1) **Software as a service (SAAS):** Consumers based on the need can subscribe to applications hosted on cloud infrastructure. Such applications can be subscribed by more than one consumer. The consumer is not concerned about application updation and development functions *(1)*.

2) **Platform as a service (PAAS):** Customers looking for development and deployment of services can use PAAS. The overhead of software licensing, version configuration will be handled by the PAAS *(1)*.

3) **Infrastructure as a service (IAAS):** IAAS provider provides all computing resources like hardware and software. Users are free to use that. The user has only paid for the usage of the resources *(1)*. The grid/cluster architecture provides high performance to the organizations *(1)*. Advantages of this model are elasticity, reduce operational cost and provide the high level infrastructure *(1)*. For this model system must be powerful failure tolerant, selection of hardware is very important otherwise it leads to high damage *(1)*.

Cloud supports various types of deployment models to cater the needs of all classes of cloud customers.

1) **Private Cloud:** Cloud infrastructure will be provided to only specific customer. This approach does not support multi tenancy and is a costly model among all the deployment models *(2)*. Examples of private cloud are Amazon virtual private cloud (VPN), Eucalyptus cloud platform, IBM smart cloud foundation, Microsoft private cloud *(3)*.

2) **Public Cloud:** In this model cloud infrastructure is shared among various customers. This mode widely supports multi tenancy and is the cheapest among all the deployment models *(3)*. public cloudexamples are Amazon elastic cloud, blue cloud by IBM, Google App Engine and Microsoft Azure by Windows *(3)*.

3) **Hybrid Cloud:** This model is a combination of public and private cloud. Hybrid cloud is used for cloud bursting. This approach is economical when compared to the private cloud. Organization's secure data are stored in private cloud infrastructure and non prioritized data is stored in public cloud *(3)*.

4) **Community Cloud:** This model is used when organizations require more security and privacy. The infrastructure is shared by organizations having similar requirements*(3)*. Example of community cloud is Google's Gov.cloud *(2)*.

1.1 Scheduling: Scheduling is a process in which resources are assigned to the tasks as per requirement. Efficient scheduling mechanisms will improve work load management in cloud computing and resource utilization can be optimized *(4)*. Scheduling of resources is a very complex task in cloud computing.

1.1.1 Scheduling In Cloud Computing: Cloud computing is mainly the virtualization.It provides the commodity services. For virtualization various virtual machine scheduling is required. Scheduling in cloud computing works in following manner [1] Discovering resources and filtering of resources*(5)*, [2] Select the target resource*(5)*, [3] Allocate the resource to the target task *(5)*.

There are several scheduling algorithms that are supporting work load management in traditional systems. They are also used for scheduling cloud computing resources. Various scheduling algorithms are following :

1) **FIFO (First In First Out)**: FIFO means the task that inserted first will be executed first. FIFO is easy and simple to implement. It allows the currently running task to complete it work first means there is no preemption. Disadvantages of this algorithm are there is no preemption in this method so if large task is there than small task have to wait for completing large task and throughput degradation.

2) **Round Robin Scheduler**: In this algorithm time is divided into time slice first and then given to the resources. The first request for the resource is given to any random resource then other respective request is given in circular queue order. Advantages of this algorithm are each task have fixed time slice so the small task has no need to wait for completion of large task and the workload is equally distributed *(6)*. Disadvantages are that currently running task taken off the processor and next task put on that so context switching reduces the overall CPU utilization and there is no equal running time for all tasks so throughput is lower.

3) **Shortest Job First (SJF)**: SJF is same as FCFS but the difference is that in SJF first job will not execute first, but when the complete cycle will complete the job will execute. This algorithm minimize the average waiting time is a benifit of this algorithm. Disadvantages of this algorithm are that it is difficult to predict the CPU burst time and large task has no chance of execution if smaller task executes continuously.

4) **Min-Min Algorithm**: Min-Min algorithm selects the smaller tasks to be executed first *(5)*. This same process works until all tasks are assigned *(6)*. This algorithm is simple and easy to assign. Disadvantage of this algorithm is that in this existing load is not consider so proper load balancing is not done *(6)*.

5) **Max- Min Algorithm**: In this algorithm bigger tasks are executed first. Jobs that have the larger execution time or compile time are executed first *(6)*. Disadvantage is that small task has to wait for a long time for execution of the larger task.

1.2Virtualization In Cloud Computing: Virtual machine is a software that behaves as a separate computer within the same system. Virtual machine provides its own hardware, CPU, memory, hard disk and network interface. Various purposes of virtual machines are for server consolidation, cloud computing, quality testing, failure-recovery. Virtual creation of operating system, memory, network, CPU is known as virtualization.

1.3VM Scheduling: In cloud computing VM allocation is used to schedule the VM request to the physical machine of any particular data center as per the requirement. Allocation of VM divides into 2 parts : first is admission of new requests for VM provisioning and second is optimization of the current allocation of VM *(7)*.

VM allocation algorithms works in following manner:

- For the set of VMs first of all find the appropriate physical machine.
- Find the appropriate provisioning scheme of VM.
- Schedule the task on particular VM.

VM allocation works in following manner.

- First cloud user request for the VMs to cloud broker.

- Cloud broker looks for the service provider who can fulfil the VM request.
- When cloud service provider is chosen by broker it submits the VMs list to the data center which holds the physical computing server.
- VMs are placed on the host as per allocation algorithms and policies *(4)*.

1.4 Clustering:

Clustering means the group of objects which have similar characteristics. In another way clustering is a group of similar objects and dissimilar to the objects in the other clusters. Researchers have proposed various clustering methods come on each method have their own set of advantages and disadvantages.

1.4.1 Partition Clustering Method: It is the simplest and easiest method in clusters. In this method data points are splitting into k partition where each partition represents a cluster *(5)*. In a partition clustering method various algorithms like K-means, K-Mediods (PAM) are included.

1) **K-Means Clustering Algorithm:** K-means works in following manner: [1] Randomly choose k objects from the data set as initial cluster center. [2] Assign each object to the cluster by considering closet center point. [3] Compute new position for each centroid by computing the mean value of each object *(8)*. Advantages of this algorithm iseasy and simple to implement, improves the performance and takes less time for execution comparable to other algorithms *(8)*. Some drawbacks of this algorithm are that there is no specific answer for finding the minimum number of clusters for a given data set. *(8)*. Randomly selected k value is provided and it is very sensitive to outlier.

2) **K-Mediods Algorithm :** In K-Mediods or Partition Around Mediods (PAM) cluster is represented by mediods. The mediods is the most centrally located object *(9)*.K-Mediods works in following manner: [1]Select object as mediods for each K cluster [2] Objects which are similar to that mediods are grouped into one cluster [3] For each medioids and non medioids point compute the cost. If total cost is increased than previous step, then undo that process *(10)*.Theadvantage of this algorithm is that there is no need to choose randomly any centre point. Disadvantage is that Works effectively for small dataset not for large dataset.

1.4.2 Hierarchical Clustering Method: Hierarchical clustering method assigns objects into tree like structure. It can be divided into 2 categories, namely Agglomerative and Divisive. [1] Agglomerative : It follows a bottom-up approach. It starts from each object and consider as one cluster, then it merges clusters into large clusters, until all objects are not merged into one cluster. AGENS (Agglomerative Nesting) is an example of Agglomerative approach. [2] Divisive: It follows a top-down approach. It starts from large cluster and divide large cluster into smaller cluster until cluster is represented as one object. DIANA (Divisive Analysis) is an example of Divisive method. BIRCH, CURE, CHAMELEON are the examples of hierarchical clustering method.

1) **CURE (Clustering Using Representative):** It is an agglomerative method. In this instead of any single point cluster is represented by a scattered points and representatives are moving toward their centers. In each iteration pair of clusters are merged which are closest to a representative. CURE helps to reduce the noise, but this algorithm is not working for large clusters.

2) **CHAMELEON:** In this algorithm, a graph, is there which is created by links between every point and its nearest neighbour. After that graph partitioning algorithm splits the graph into many small graphs which are not connected. After that this each sub graph is considered as initial cluster and then agglomerative approach is applied to merge two sub cluster which are similar. Two sub-clusters are merged based on resultant inter-connectivity and closeness of two individual clusters. CHAMELEON is more effective in finding arbitrary shape cluster.

3) **BIRCH (Balanced Iterative Reducing and Clustering Using Hierarchies):** In this data is compressed and sub-clusters are created. This sub-clusters are known as Cluster Features (CF). Clustering is applied on summary of data instead of raw data. This CF are stored into leaf. The non–leaf node stored the sums of CF children. Object is inserted into closest leaf. After the CF tree created, any partition or hierarchical algorithm can apply to perform clustering in main memory. BIRCH is fast in execution. But this algorithm allows to produce the only non-spherical cluster.

1.4.3 Density Based Clustering Methods: Density based method grouped neighbouring objects into clusters based on density of data points in a region *(11)*. Density is measured by the number of objects which are nearest to the cluster *(12)*. It means that each point has a minimum no of points within its neighbourhood. It finds the spherical shape cluster. Following are the density based clustering algorithms:

1) **DBSCAN (Density Based Spatial Clustering of Applications with Noise)**: This algorithm defines the cluster with a maximum of density connected points. In this cluster are created by checking E- neighbourhood points in the database. In the E-neighbourhood if any point P contains more than MinPts. Then new cluster P is created as a core object. Then DBSCAN iteratively process and collects objects which are directly density reachable to core object. This process continues until new point is added into any cluster. Advantages is that it can handle large noise in the dataset and insensitive to ordering of data objects in the dataset and insensitivity in ordering of data objects. Disadvantage of this is that quality of cluster depends on distance measure and it is very sensitive to input parameters and MinPts.

2) **DENCLUE (Density Based Clustering):** DENCLUE works based on distribution approach. This algorithm works effectively with dataset containing high level of noise. It works faster than DBSCAN algorithm. It is good for investigating a random shape cluster. It works on small or medium level dataset *(11)*.

1.4.4 Grid Based Method: Grid based clustering approach uses multi resolution grid data structure. *(11)*.it considers an object space into finite no of cells that forms the grid structure. *(11)*. Examples of these approach are followings:

1) **STING (Statistical Information Grid)**: STING algorithm checks the statistical information stored in grid cells. There are several levels of rectangular cells corresponding to different levels of resolution. These cells create one hierarchical cluster. For low level, each cell of high level is partitioned. Statistical information in each grid cell is precomputed and stored. STING is faster in processing. Drawback of this algorithm is that quality is depending on no of cells in each dimension *(11)*.

2) **CLIQUE (Clustering QUEst):** It is a grid based approach for high dimensional data. This algorithm firstly portioned the data into grid known as units. This algorithm automatically finds

the dense units. It finds the maximum density region in the dataset in the subspaces. Then it uses a depth-first search algorithm to find all clusters that dense units in the same connected component of the graph are in the same cluster. Then it will generate clusters. It is useful for finding clusters in high dimensional space. Drawback of this algorithm is that it depends on size of cells in each dimension *(11)*.

1.5 Virtual Machine Clustering:

Clustering is a group of similar types of objects. Virtual Machines clusters are built with VMs installed on distributed physical server. Creation of virtual machine clusters is done by logically connecting virtual network to a physical network. Each VMs cluster is formed with physical machine. No of nodes within clusters can increase or decrease dynamically as the network is changed.

Using virtual machines clustering, we can achieve various benefits like one time configuration of software,drivers,VMs shifting when a physical node fails, backup and fast recovery, minimization of downtime, highly scalable platform and applications.

2. RELATED STUDY

Karan D. Prajapati *(4)* has discussed about the comparison between various virtual machine scheduling algorithms in cloud computing. He discussed that VM scheduling algorithms are used to schedule the VM request to the Physical Machine (PM) of particular Data Center (DC) as per the requirement. He obtained that determination of best scheduling algorithm for cloud computing is depends on various factors like time, power, cost, security and memory.

Sonam Rathore *(13)*has discussed about efficient allocation of virtual machines in a cloud computing environment. Cloud computing provides dynamic provisioning of computing services. She defines that for IAAS layer scheduling of resources and virtual machines are one of the major issues. She obtained the algorithm in which efficient VM scheduling is done in resource utilization rate. The objective of her research work is to find allocations of VMs an available physical machine that satisfies the VMs resource requirements and increases overall resource utilization.

Anand D. Khandare *(14)* has discussed about modified K-means algorithm for emotional intelligence. As we know that K-means is the most popular and simple algorithm. But as we know that k-means algorithm has some problem like produce empty cluster, complexity in large clusters, sensitive to outlier and noise, etc. so in this paper problem of K-means algorithm is overcome by modified K-means algorithm. Modified K-means algorithm is applied on emotional intelligence data set. In this data set dimension of person like emotional, social, personal are considered and survey of 200 people is done. Then on data set K-means and modified K-means algorithm applied. From comparison of result it is shown that modified k-means does not produce empty cluster like k-means.so it is better than k-means algorithm.

Subhadra Bose Shaw, Dr. A.K. Singh *(6)* has discussed about A Survey on Scheduling and Load Balancing Techniques in Cloud Computing Environment. Load Balancing balances the workload and resources in an efficient manner. In this paper various load balancing algorithm is there to resolve the issues in load balancing. This scheduling algorithm is there with its advantages and disadvantages. They also discuss about VM migration for over utilization and under utilization of resources. In this various algorithms are used to increase overall throughput and proper utilization of resource.

D.Kiran Kumar (15)has discussed about review on virtualization in cloud computing. As we know that virtualization is the process of creation of virtual of something like hardware,os,network.In this paper cloud computing and virtualization are discussed in terms of their use in real world and how cloud computing and virtualization are worked in future.In this paper 3 sections like virtualization technologies, dynamic scaling of web application and I/O bottlenecks in cloud computing are discussed.

3. OBJECTIVES

The main objective of this research is to provide efficient VM allocation (scheduling) on available data centers using a clustering algorithm. The major objectives of this study are:

- To provide dynamic VM allocation on available datacenter by clustering algorithm.
- Using VM allocation policy as per user requirement allocation of VMs is done.
- Using this reallocation of VM's load on data centers will be reduced.

4. RESULTS AND DISCUSSION

We are proposing an optimized algorithm for scheduling of virtual machines with efficient resource utilization. The proposed algorithm supports dynamic scheduling of virtual machines using clustering techniques. As we all know, clustering is the best method to find similar types of objects. We will be grouping similar virtual machines using clustering. The Scheduled algorithm can dynamically allocate virtual machines grouped by a using clustering approach for user requests. This approach will provide efficient CPU utilization, resource sharing and load balancing. This approach will reduce the overall power consumption of the data center.

We will be simulating the proposed model using cloudsim environment.

5. CONCLUSION

This paper explores the comparative study of various clustering algorithms with their advantages and disadvantages. The best algorithm will be chosen by as per required environment.Scheduling algorithm are discussed with their advantages and disadvantages.In the proposed method using a scheduling algorithm virtual machine allocation is done. Dynamic allocation of virtual machines using clustering algorithm is done by clustering algorithm which shares resources among virtual machines and provides load balancing.

6. TABLES:

Table 1. Comparison Between Clustering Algorithms.

Clustering Technique	**Clustering Algorithm**	**Pros**	**Cons**
Partition Based Clustering	K-Means *(9)*	It is easy and simple to implement. It takes less time for execution compare to other algorithms.	There is no specific answer to find the minimum number of clusters for given a data set. Randomly selected k value is provided. It is very sensitive to outlier.
	K- Medioids	No need to choose a centre point.	Works effectively for small dataset not for large datasets.
Hierarchical clustering	CURE *(9)*	Helps to reduce or avoid the noise.	Not used for large datasets.
	CHAMELEON *(9)*	More effective in finding arbitrary shape cluster	Splitting and merging of graph required.
	BIRCH *(9)*	Fast in execution	Data ordering sensitivity and enable to deal with non spherical cluster.
Density Based Clustering	DBSCAN *(11)*	Can handle large noise in the dataset and insensitive to ordering of data objects in the dataset.	Quality of a cluster depends on distance measure.And it is very sensitive to input parameters and min points.
	DENCLUE *(11)*	good for a investigating random shape cluster.	It works on small or medium level datasets.
Grid Based Method	STING *(11)*	Fast processing time	Depends on no of cells in each dimension.
	CLIQUE (11)	Independent of no of data objects.	Depends on no of cells in each dimension.

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Implementation of Improved Swarm Intelligence Technique for NP-Complete Problems

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ABSTRACT

In computation paradigm NP problems are always difficult to solve, especially optimal solution for the NP-Complete problems is a nightmare. Many researchers have already done by tradition techniques on NP-Compete problems. TSP is one of the NP-Compete problems and swarm intelligence techniques like ant colony optimization algorithm leads to the promising results then the previous tradition methods. Stagnation behaviour of available swarming algorithm leads the poor results many times. A new swarm component cockroach reduces the limitation of the earlier swarming algorithm and gives promising results for the future aspects.

SUMMARY

The new swarming component cockroach avoids the stagnation behaviour of ant colony algorithm and leads to the comparatively better results for NP-Complete problems.

Keywords: Swarm Intelligence,Ant Colony Optimization, NP-Complete Problems

1. INTRODUCTION

In computational theory, problem which can be solved in non-deterministic polynomial time are considered as NP problem. The problems which are hardest to solve are known as NP-Complete (NPC) problems (*1*). The specialty of NPC problems is that the time required to solve the current problem is increase as the size of the problem increases. The quick solution of the NPC problem is almost never

known that is the main characteristics of such problem. But the optimum or nearer to optimum solution is desirable in many cases. Travelling Sales Person (TSP) problem is considered as most fundamental NP-Complete problem in which each salesperson has to visit each city exactly once. TSP is always researcher's priority to test it on different algorithm. TSP is a problem where salesperson can start the journey from the any of the node specified and he needs to visit the each and every city of the graph area exactly once and returning to the same city from where journey started. The target of the salesperson is to visit each city exactly once with minimum distance travelled. Here distance can be cost, money, energy or another parameter depending upon the problem.

If we use any traditional technique like exhaustive search technique then its take immense amount of time to get possible paths as solution. Every tour is represented as 2n different way for symmetric TSP. Since exhaustive search technique based on finding total distance with all possible permutations of N cities, the number of permutations can be very large as number of cities goes large. To avoid this limitation many approximation algorithms based on nearest neighbor, greedy algorithm, 2-opt 3-opt algorithm (*2*), simulated annealing, lin-kernighan, tabu search, neural network and genetic algorithm have been developed to get good solution in fair time. However, every algorithm found difficulty in finding optimal or nearer optimal solution.

Then there arise a need to find a heuristic based search method to solve TSP with optimal solution for the given sets of data set. Swarm intelligence is one of the promising approaches to solve such NP- Complete problems. Paper discusses one of the main methods namely ant colony optimization (ACO) and implementation of its basic algorithm on symmetric TSP.

The rest of the paper discusses these techniques, implementation and limitations of TSP on ACO. The modified version of the technique is implemented on self developed simulation tool by taking the random parameter of TSP. The successful results are displayed in tabular form.

2. SWARM INTELLIGENCE

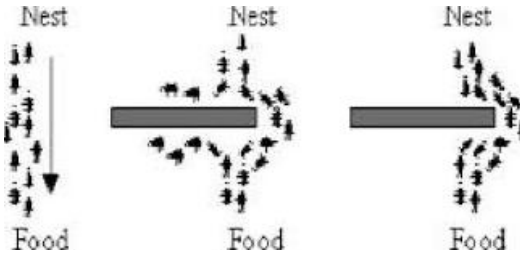
In the early 20th century, the French entomologists Pierre-Paul Grasse (*3*) observed that some species of termites react to what he called "significant stimuli". Grass's used the term *stigmergy* (*3*) to describe this particular type of communication. The term stigmergy can be defined as (i) an indirect, non-symbolic form of communication mediated by the environment (ii) stigmergic information is local means it can only be accessed by those insects that visit the locus in which it was released. In more simplified manner one can say that it is the communication where one particular insect follows the path of its successors to reach the destination area. Such behavior can be seen to the insects such as ants, birds, bees, etc which are weak in their individual behavior but in group they can be stronger than the strongest. Scientifically we call it their swarming behavior.
A swarm is a large number of homogenous, simple agents interacting locally among themselves and their environments, with no central control to allow a global interesting behavior to emerge (*4*). The study of swarm includes the behavior of organisms like ants, bees, termites, fish and birds. Swarms are stochastic, random and self organizing in their nature.There exist a tight relationship between the collective behaviour of individual and the individual behaviour of a particle (*5*). As swarms are stochastic by nature their behaviour cannot be viewed as an independent entity. Instead their behaviour is with respect to the interactions among themselves. Such natural intelligence of swarm can be termed as swarm intelligence. The mathematical form of available swarm intelligence techniques are Ant Colony Optimization (ACO), Particle Swarm Optimization (PSO) and Artificial Bee Colony Optimization (ABCO). In this paper the focus is on Ant Colony Optimization and performance analysis of its basic algorithm on symmetric TSP.

3. ANT COLONY OPTIMIZATION

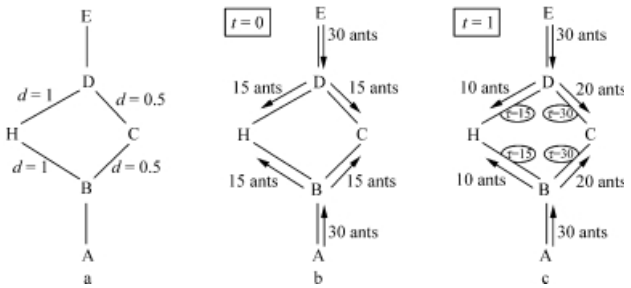
This Ant Colony Optimization is introduced by Marco Dorigo and his co-authors in the early 1990's (6 – *8*). The development of these algorithms is done based on ant colonies. Ants are type of insects and their behavior of searching for the food is the encouragement for the optimization and its related tasks.They live in the form of colonies and their behaviour is survived by the goal of colony.

The behaviour for ACO is such that the ant's tries to find shortest paths between food sources and its place. When searching for its food ants initially explore the area surrounding their nest in a random manner. Ants leave a chemical pheromone trail on the ground while moving. They smell pheromone and while choosing their way, they tend to choose, in probability, way is marked by strong pheromone concentrations. After finding the food source by ants, it evaluates the quantity and the quality of the food and carries some it back to the nest. While returning, the capacity of pheromone that an ant leaves on the ground may depend on the capacity and its flavour of the food. The other ants will follow the same pheromone to the food source.

The following figure shows the swarming behavior of ants:



"**Fig.1** Ant's biological behaviour"



"**Fig. 2** Ant's artificial behaviour (*9*). (a) The initial graph with distances. (b) Initially, at time t = 0 there is no trail on the graph edges; (c) After pheromone update, at time t = 1 trail is stronger on shorter edges, which are therefore, in the average, preferred by ants.

ACO is successfully applied to solve many combinatorial problems (*10*) which are NP-Hard and NP-Complete in nature (*10*). It is used in the applications like Quadratic Assignment problems (*11*), Travelling Sales Person problem (*11*), Graph Coloring (*12*), Job Shop Assignment and Scheduling (*13*), and other scheduling problems.

The Ant Colony Optimization (ACO) algorithm is a metaheuristic that has a combination of distributed computation, autocatalysis (positive feedback), and constructive greediness to find an optimal solution for combinatorial optimization problems. This algorithm tries to mimic the ant's behavior in the real world

The metaheuristic presented by Dorigo et al. was given below:

Algorithm ACO Meta heuristic ();

While (termination criterion not satisfied)

Ant generation and activity ();

Pheromone evaporation ();

Daemon actions ();

"Optional"

End while

End Algorithm

Ant colony algorithm has the following variations which can be utilized depending upon the problem. Detailed discussions of the methods except Ant System are not given as it is out of scope of this article.

1. Ant system
2. Elitist AS
3. Ant-Q
4. Ant Colony System
5. Max-Min AS

6. Rank Based AS
7. ANTS
8. Hypes cube AS

This paper focuses on the implementation of Ant System (AS) on symmetric TSP.

3.1 ANT SYSTEM (AS)

Various ant system algorithm were introduced like ant density, ant quality and ant cycle etc. AS algorithm is divided into two phase. First is solution construction by ants and other is updating of pheromone. The amount of pheromone value deposited by ants in one tour is depends upon the number of ants as well as length of the tour generated using any nearest neighbour heuristics approach.

3.3.1 Solution Construction by Ants

As per the previous description of TSP, in AS algorithm n_1 artificial ants currently execute a symmetric tour. Based on ants philosophy every ants visit the next not is based on the probability. Hence, Ant k uses probabilistic action choice rule to decide which city to visit next at each solution construction step. The rule can be described as follows:

$$\rho^{k}{}_{ij} = \frac{[\mathrm{Ŧ}ij]^{\alpha}[\eta ij]^{\beta}}{\sum_{l \in \mathrm{N}i^{k}}[\mathrm{Ŧ}ij]^{\alpha}[\eta ij]^{\beta}} \qquad \text{if } j \in \mathrm{N}^{k} \qquad \textbf{(1)}$$

Where,

$\eta_{ij} = 1/d_{ij}$ which specifies a heuristic value decided before start of the algorithm. It is the inverse of the distance between the two cities. Algorithmically it is initial pheromone value for the ant.

α, β = Used determines the relative effect of the pheromone value and the heuristic data. These are artificial parameter added for optimize the algorithm.

N_i^k = probable neighborhood of ant k when being at the current city. It is a set of city that k^{th} ant has yet not visited. It maintains the list of unvisited cities.

Using the stated rule in Eq. (1), the probability of choosing a particular edge increases the value of the pheromone trail associated with that edge. It also affects the information related to heuristics that is ηij.

Every ant maintains a memory, which is a collection of cities that is already visited. It also maintains the order of their visit. It helps to decide neighborhood which is feasible for the above rule.

3.3.2 Updating the pheromone

When all the ants have constructed their individual tour the next step should be the updating of pheromone which ants has deposited during the tour specified. Pheromone evaporation will decided the

next tour of the ants. Pheromone update can be calculated by the rule known as pheromone evaporation rule.

$$\mathbb{T}_{ij} \leftarrow (1 - Þ)\, \mathbb{T}_{ij} \qquad \textbf{(2)}$$

Where, $0 < Þ \leq 1$ is the rate pheromone evaporation.

$\mathbb{T}_{ij}$ is the pheromone value between the two cities.

After the above phase the ant's needs to deposit pheromone on the edges they have passed in their entire tour. The formula used for this purpose is as follows:

$$\mathbb{T}_{ij} \leftarrow \mathbb{T}_{ij} + \sum_{k=1}^{n1} \Delta\, \mathbb{T}^{k}_{ij}\,, \forall (i,j) \subset L \qquad \textbf{(3)}$$

Where $\Delta\, \mathbb{T}^{k}_{ij}$ is the value of pheromone that ant k has deposited on the edges during the visit. $\Delta\, \mathbb{T}^{k}_{ij}$ can be specified as follows:

$$\Delta\, \mathbb{T}^{k}_{ij} = \begin{cases} \frac{1}{C^k}, if\ edge(i,j) belongs\ to\ \mathbb{T}^k \\ 0, Otherwise \end{cases}$$

Where, k is the total distance covered by the k^{th} ant during the tour $\mathbb{T}^k$.

3.3.3 Implementation of AS on TSP

The AS algorithm discussed above is implemented on self developed simulation tool by taking 20 iteration of 10 cities. The simulation tool is developed on PHP 6.0 which compares the existing swarm approach as well as proposed approach.

"**Table 1.** Ant System (Number of Ants: 20, Alpha: -1.5, Beta: 1.1, PDF: 0.1, PBF: 0.15)"

Run	Iteration																			
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20
1.	107	107	101	107	114	96	107	96	107	107	96	101	107	107	109	107	128	96	107	96
2.	97	97	97	97	97	97	97	97	97	97	97	97	97	97	97	97	97	97	97	97
3.	135	135	135	134	135	135	135	135	135	135	135	135	135	134	135	135	135	134	134	135
4.	72	72	72	72	72	72	72	72	72	72	72	72	72	72	72	72	72	72	72	72
5.	64	64	64	64	64	64	64	64	64	64	64	64	64	89	64	64	64	64	64	64
6.	77	77	77	77	77	77	77	77	77	77	77	77	77	77	77	77	77	77	77	77
7.	105	105	105	105	105	105	105	105	105	105	105	105	105	105	105	105	105	105	105	105
8.	111	111	111	111	111	111	111	111	111	111	111	111	111	111	111	111	111	111	111	111
9.	137	137	137	137	137	137	137	137	137	137	137	137	137	137	137	137	137	137	137	137
10.	113	94	94	94	94	94	100	100	100	100	94	94	94	94	94	94	94	94	94	94

3.3.4 Limitation of AS on TSP

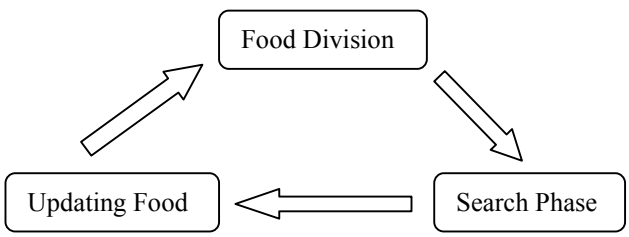
As shown in the tables 1, as the numbers of ants are less algorithm shows the behavior in which each and every ant follows the same path. It can be termed as stagnation behavior of ants. We can say that solution

falls into local optimum area where it ruins the possibility of better solution than the previous one. From the above results we can see that in all 20 iterations it shows the static results only. The problem can be that the better results other then this value cannot be possible in such cases. Technically, one ant follows the other ants and when such behavior is repeated; all the ants follow the same path and there is no other choice for the dynamic result.

Another problem with the algorithm is it is highly dependent on probability rule and constant. Such parameter makes behavior of the algorithm predictable at some sense. Hence the need of new swarm component is highly desirable where biological behavior has more roles to play than the predefined parameters.

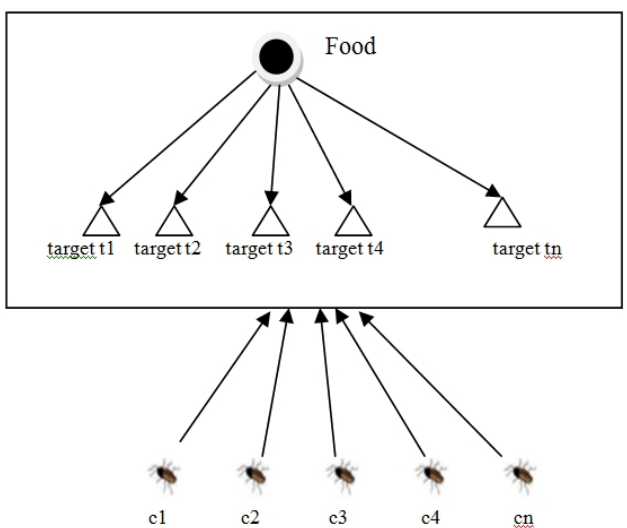
4. SWARMING BEHAVIOUR OF COCKROACH

The swarming and mating behaviour of cockroach are similar to ants. They leave chemical trails in their faeces. Similar to ants, other cockroaches follow the trails to find the food and water. The cockroach has another interesting behaviour to find the location of where the other cockroaches are hiding. According to Havens (*14*) behaviour of cockroaches are categorised into three parts: (i) they search for the darkest location in the search space, (ii) they socialise with the nearby cockroaches and (iii) they periodically become hungry and leave the friendship to search for food. Graphically it can be shown as follows.



"**Fig.3** Model of swarming behavior of cockroach"

The food division strategy of the cockroach can be depicted in the following figure.



"**Fig.4** Food searching strategy of cockroach"

The another interesting behavior of cockroach is that while searching for food in night or dark places ,whenever light focuses on it they disperse themselves for the sage passage. This nature of cockroaches helps us to avoid the limitation of previous ACO algorithm. Because of this nature of cockroach algorithm will never fall into local optimum or it never gets stagnation phase.

The behavior of cockroach is divided into three parts and they can be characterized mathematically as follows.

Swarming Behavior: The new cycle of all the strongest cockroaches form small swarms and follow the global solution and other cockroaches will follows the strongest. However there is a possibility that the cockroach follower in a small swarm will be strongest if it finds a better solution.

Mathematically it can be represented as follows:

If $X(i) \neq P(i)$ Then,

$$X'(i) = X(i) + step \cdot rand \cdot (P(i) - X(i))$$

Else

$$X'(i) = X(i) + step \cdot rand \cdot (P_g - X(i))$$

End IF

Where,

$X(i)$ is each individual cockroach chase another individual $P(i)$ with its visual scope.

$X'(i)$ is the new modified position of each individual cockroach.

Escape from the Light: Cockroach has a biological nature that whenever light appears they disappears. Dispersion of the cockroach means that at a time each individual cockroach will be randomly dispersed in order to maintain the current individual diversity. This is significant because using this we can prevent the algorithm to stuck in to the local optimum and we can find the global best solution. It also guarantees the continuous movement of the cockroaches. After certain amount of time each individual cockroach disperses randomly.

Mathematically, it can be shown as follows:

$$X'(i) = X(i) + rand(1,D) \quad Where, \ i=1,2,....,N$$

D is the dimension of the search space and rand() show the random behavior of cockroaches in search space.

Ruthless Behavior: We need to deal with the ruthless behavior of cockroaches, when the current best replaces a randomly chosen individual. In case of shortage of food the stronger eats the weaker. So, Current best individual replaces an individual selected at random. The mathematical form is,

$$X(k) = P_g$$

Where P_g is the random selection of individual cockroach.

"**Table 2.** Swarming Behaviour of Cockroach (No. of cockroaches: 50, Visibility Range: 3, Step Length: 2)"

Run	Iteration																			
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20
1.	138	124	137	123	128	139	123	117	131	**95**	128	126	126	109	131	133	121	122	109	128
2.	114	119	126	**96**	114	121	139	117	139	112	133	102	114	114	127	112	121	138	119	126
3.	162	142	145	145	138	154	138	140	148	151	147	147	147	141	160	**133**	137	144	154	147
4.	95	78	95	109	78	93	**71**	105	90	91	89	96	78	104	72	95	84	114	77	76
5.	**52**	76	74	68	84	83	68	74	64	77	70	66	74	67	74	65	72	60	71	68
6.	102	**77**	82	96	104	96	111	77	96	117	111	78	128	102	105	109	117	114	113	108
7.	117	119	124	125	**102**	130	**102**	115	116	118	136	110	125	115	114	132	116	138	119	119
8.	109	141	**104**	111	127	122	109	107	133	132	132	115	119	127	112	129	116	151	117	113
9.	176	153	**127**	165	150	152	133	162	**127**	159	167	150	164	152	144	157	137	138	164	173
10.	105	108	93	108	94	110	111	104	104	117	111	115	109	106	124	118	111	117	118	

From the above table we can see that for the same input values of the previous algorithm the new approach gives the promising results and it also avoids the limitation of previous algorithm. Because of ruthless behaviour of cockroaches the output result cannot remain in stagnation phase and it gives better results as well.

4. FUTURE ASPECTS

The problem with the proposed technique is time complexity. It takes more time than its previous swarming techniques. So time complexity can be minimized. The other enhancement can be done on the implementation side, is that the programmer can define the database instance in terms of standard file instead of taking the input randomly. By doing this the result verification process can be standardized. The proposed algorithm can be applied to many other NPC problems like job scheduling, vehicle routing, graph colouring etc.

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SVM- and K-NN-Based Hybrid Approach of Sentiment Analysis for Camera Product Reviews

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ABSTRACT

Sentiment analysis help organizations and individuals an effective way to monitor the publics' feelings towards them and their competitors. Authors presented the novel study on the hybrid model using multiple classifiers as SVM and K-NN in sentiment analysis which performs several experiments over different camera reviews datasets from the highlight sites such as Amazon and eBay. After several experiments perform with different approach on SVM classifier, we identified the Hybrid approach which is applied on specific "Camera Review Comments" to get product review. An experiment result also contains the performance of hybrid approach reached up to approximate 81% accuracy, which is actually very promising with compare to other models. The major focus of this research paper is to reinforce the sentiment analysis accuracy mistreatment in hybrid approach.

SUMMARY

SVN & K-NN based hybrid approach to analyze the publics' feelings towards them and their competitors in the field of sentiment analysis.

INTRODUCTION

With the rapid development and people's ever growing interest in social networking, blogging, web applications and other information-sharing channels brought about by Web 2.0, more and more time is spent online in people's daily life. This has also contributed to the flourishing online shopping market. However, the problem is that nowadays things are many and all the product are best of each other As we know that now a market is kind of place where we can find the same product with different specification . Now problem is: how the user gone a choose one of the best among them. Here data is larger than before days . we have to find the solution approach that will allow us to find easily what we want from the large dataset that almost impossible to analyse manually.

In current search application societies to examine for other publics approximation from the net previously buying a item, when we are not aware with a exact product, we inquire our consistent sources to reference. Various Websites suggested to employer remarking, online expression and ratting services, and that analyses help to users opinion about precise product, and existing essential information helpful for professional to market and organizations to make choice in a improved way.

Also now a days we have seen that so many large website like Amazon and eBay getting large numbers of data within comments or as product reviews. The website makers nowadays building the system which are really do sentiment analysis easy and faster than human. Sentiment analysis is the ways of future to analysis the large number of reviews. Large numbers of comment review website using CMS (Content Management System) which allow them to analysis the user opinions. From that point forward, a gigantic measure of information has been delivered by clients, an important substance that can be to a great degree helpful both as an integral, additionally by and large as an essential and interesting wellspring of information. Because of the way of this substance, which is unstructured information in type of free content, the recuperation and extraction of important data relies on upon specific procedures. There are a few difficulties in Sentiment examination. The main is a feeling word that is thought to be sure in one circumstance may be viewed as negative in another circumstance. A second test is that individuals don't generally express assessments in a same manner. The third one is mixed Review in that we got mixes review from the user like this phone is good but price is so costly that's kind of review called Multi-theme documents.

In our work we have present the hybrid approach on Camera review Documents. An Empirical study on hybrid approach is present the accuracy of sentiment analysis combining the different dependency classifiers such as SVM & K-NN. Both of these have its own advantages in sentiment analysis. The paper also includes architecture & description about the actual sequence of experiments.

RELATED WORK

Sentiment analysis method will be consummate at several levels as facet primarily based i.e. word level, sentence level and document level. In major products review, customer categorical mixed opinions on variety of aspects of the merchandise even though they rated product beautifully or bad. This analysis exhausted the facet level which can be able to mine mixed opinions and to get a lot of useful results. Thus facet based SA is the one of the accepted during this work.
In[11], the makers joined minimum-cuts in graph to remove the individual fragment of compositions and used machine learning frameworks to thought examination on works just. In[13], inventors analysed

organizing works into polar and unprejudiced first before making sense of if a positive or negative inclination is conveyed through the substance. In[7], the makers made methods that algorithmically distinguish hundreds of modifiers, each with a doled out score of extremity, from around twelve of seed descriptors. Their systems extend two bunches of modifiers ($+^{ve}$ & $-^{ve}$ words) by recursively questioning the equivalent words and antonyms from WordNet. Since recursive inquiry rapidly unites words from the two groups, they executed a few pre-cautionary measure measures, for example, appointing weights which diminish exponentially as the quantity of bounces increments. This affirms that the calculation produced modifiers are very precise by contrasting them with the consequences of physically picked word records. It merits calling attention to that this work utilizes Lydia as the spine to process vast measure of news and online journals. In [3], the creators gave a decent study of different methods grew in online opinion investigation. It covers idea of feeling in composed content (examination theory), various strategies which can be comprehensively separated into two gatherings:

(i) typical systems that spotlights on the power and heading of individual words (the alleged ”bag of words” methodology) and (ii) machine learning procedures that portrays vocabularies in connection. In light of the review, the makers found that run of the mill methods fulfil precision lower than 80% and are all around poorer than machine learning strategies on movie review supposition examination. Among the machine learning systems, they considered three controlled procedures: SVM, NBM, and most great Entropy (Maxent). They found that each one of them pass on commensurate results on distinctive segment extraction (unigrams, bigrams, et cetera) with high precision at 80-87. Another discriminating effort for conclusion gathering on Twitter data is driven by [2]. The inventors use limit estimates from three locales as noisy names to set up a model and use 1000 physically checked tweets for tuning and another 1000 physically stamped tweets for testing. They however don’t say how they assemble their test data. They propose the use of sentence structure segments of tweets like rewets, hash marks, association, emphasis and clamour stamps in conjunction with components like previous furthest point of words and POS of words. In [5], the makers perform estimation examination on feedback data from Global Support Services review. One purpose of their study is to dismember the piece of phonetic segments like POS marks. They perform wide component examination and highlight determination and demonstrate that theoretical phonetic examination components add to the classifier exactness. Current research offers several interesting approaches to the challenge posed by SA Abstracting away from specific implementations; these approaches can be classified into 3 Categories: the lexical - phrasal approach, the compositional semantics approach, and the Machine Learning approach. Clearly, these approaches are not mutually exclusive. Indeed, systems Employing all three have been implemented to varying degrees of success [20]. The strategies by which these approaches are implemented may vary significantly from system to system. For example, feature classification in Machine Learning based systems can be based on the authors’ self-assessment [21] corpora tagged by human judges [22] and even on exogenous sources. Likewise, the Compilation of relevant lexicons and the definition of compositional semantic rules are subject to significant variation [23]. The advances in sentiment analysis lured researchers to explore the possibility of a hybrid approach which could collectively exhibit the accuracy of a machine learning approach and the speed of lexical approach. In [24] authors use two-word lexicons and an unlabeled data, dividing these two-word lexicons in two discrete classes negative and positive. Pseudo documents encompassing all the words from the set of chosen lexicons are created then computed the cosine similarity amongst the pseudo documents and the unlabeled documents. Depending upon the measure of similarity, the documents were either assigned a positive or a negative sentiment. This training dataset was then fed to a naïve bayes classifier for training purpose. Another approach presented by [25], derived a “unified framework” using back-ground lexical information as word class associations. Authors renewed information for particular areas using the available datasets or training examples and proposed a classifier called as Polling Multinomial Classifier (PMC) (also known as the multinomial naïve

bayes).Manually labelled data was incorporated for training purpose. They claimed that making use of lexical knowledge improved performance. Another variant of this approach was presented by [26]. But so far only [26] have been able to claim good results. The hybrid method is a combination of the two previous methods. It takes as input the output of the other two methods and calculates according to the condense indices for each result, an average which will b e translated into positive or negative. The exact weighting varies with several factors, including the accuracy of the manual annotation and the size of the corpus. The statistical method allows making an initial mining in the texts to get positive and negative messages. Then the user who set up the grammar can modify and improve it for better results. The work takes the form of a cycle where results are constantly improving. The analysis of the thread is done at the level of sentences and allows improving the result by adding or removing words in the lexicons. This has the advantage of showing exactly what sentences of the do cement express a feeling. It is an approach that keeps the robustness of the machine learning of the statistical method and orients at the same time the base of the training on a manual configuration of the symbolic method. This helps to correct significantly errors of aching learning and to integrate the specie project specifications, i.e. the particularities of each corpus (using glossaries and lexicons of different terms depending on the domain of application) [27].

PROPOSED METHODOLOGIES

Our technique of sentiment analysis relies upon machine learning. we tend to make a case for what sources of information we tend to utilized in A and the way we tend to performed classification in this data-set.

A. Dataset

The data set which we have choice for this hybrid model us from the reviews websites .we took this review set from the Amazon, eBay and other popular sites for training the hybrid model. We have choice the different cameras with different features, so we can easily show the enhancement in a tabular form. The reviews of Canon A2300, Canon Powershot SX260, Sony DSC-RX100, Nikon D5100 and Canon A70 were used for training purpose.

B. Pre-processing

In this block we have done the pre-processing task in which we have used different pre-processing techniques. These are used to cleaning the dataset and remove the noise from the datasets. Our pre-processing task involved four main steps. Which are tokenize, filter tokens, stem and filter stop words.

Tokenize: This operator is basically do splitting the text of documents into a sequence of tokens. There are units of measurement several decision some way to specify the ripping points. Either you will use all non-letter character, that unit of measurement the default set-tings. This may result in tokens consisting of one single word, what is the foremost applicable selection before finally building" the word vector.

Filter Tokens: Filters tokens supported their length its accustomed operator filters tokens supported their length (i.e. the amount of characters they contain).

Stem (Porter): This operator stems English words victimisation the Porter algorithm applying AN unvarying, rule-based replacement of word suffixes assuming to cut back the length of the words till a minimum length is reached.

Filter Stop words: This operator filters English stopwords from a document by removing each token that equals a stopword from the intrinsic stopword list. For this operator to figure properly, we've to recollect that each token ought to represent one English word solely. to get a document with every token

representing one word, you'll tokenize a document by applying the Tokenize operator.

C. Future Extractions (validation)

This extraction is performed to appraise the factual execution of a learning administrator it does a cross-acceptance more often than not on inconspicuous information sets. It is basically used to gauge how precisely a model that has been learnt by a specific learning administrator will perform practically speaking. The Future Extractions (X-Validation) administrator is a settled administrator. It has two sub-forms, specifically (1) a preparation sub-procedure and (2) a testing sub-process. The preparation sub-procedure is utilized for preparing a model picked. The prepared model is then connected in the testing sub-process. The testing stage is additionally focused to gauge the model's execution. In this approach, the data Example Set is divided into k equivalent estimated subsets. Out of the k subsets, a solitary subset is held as the testing information set (i.e. information of the testing sub-procedure), and the remaining (k-1) subsets are utilized as preparing information set (i.e. information of the preparation sub-process). The cross-acceptance procedure is then rehashed for k times, considering every subset once as the testing information. The single estimation is produced by utilizing k results from the k cycles which can be joined or found the middle value of. The number if division (k) can be balanced utilizing the quantity of approvals parameter. The learning procedures typically advance the model to make it t the preparation information and additionally conceivable. This model performs modestly on the off chance that we think of some as free arrangement of information as testing information; however it performs well on the information that was utilized to produce it. This marvel is called 'over-tting'. The Cross-Validation administrator predicts the t of a model to theoretical testing information.

D. Support Vector Machine (Linear)

SVM is basically one of the classifiers that can categorize by constructing hyperplanes means that splits into several categories. In experiment, the SVM classifier should first be trained on a set of user reviews so that the machine gains knowledge for effective categorization. For the application, we have an iterative training algorithm to define a hyperplane that" effectively separates two classes. We adapt the SVM classifier to our terminologies as below: Let X Y be a mapping. Here X corresponds to the extracted opinion of the aspect of a product; Y can take real values" and they are +ve / -ve scores. These scores are either from Sentiwordnet or manually assigned as described in the previous step. The simplest case of classification is the binary classification. In addition to this, we can also include the 3rd class which includes reviews with neutral polarity. In the mapping X Y is a single feature value pair, where Y R: Y can take +ve and –ve polarities. The training of the system can be done by using the feature set $x_1 : y_1$, $x_2 : y_2$, ..., $x_m : y_m$. Once training process is completed, system learnt a classifier, separate of the +ve and –ve opinions by SVM classifier and represented in graphic form. Different hyperplanes are used to break up the text-review into two categories. The SVM classifier must be trained to find out that hyperplane which considerably classifies samples. In our work, we used SVM [14] a type of kernel used is linear. All the parameters of the classifier are set to their default values. The result of the classification is the polarity assignment to each feature of the product.

E. K-NN (k-Nearest Neighbour)

The k-Nearest Neighbour calculation is in view of learning by relationship. It contrasts a given test case and preparing cases that are practically equivalent to it. The essential k-Nearest Neighbour calculation is made out of two stages: Find the k preparing illustrations that are nearest to the inconspicuous sample.

Take the most generally happening classification for these k samples (or, on account of relapse, take the normal of these k name values). The preparation samples are depicted by n-characteristics. Every illustration is demonstrated by a point in a n-dimensional space. Accordingly, the greater part of the preparation illustrations are in charge of era of a n-dimensional example space. At the point when given an obscure case, k-closest neighbour calculations hunt the produced example space down the k preparing samples that are nearest to the obscure illustration. These k preparing illustrations are the"k-closest neighbours" of the obscure sample. For the most part separation lattice, for example, the Euclidean separation is utilized to characterize"Closeness". The k-closest neighbour calculation is amongst the least complex of all machine learning calculations: an illustration is classified by a lion's share vote of its neighbours, with the case being doled out to the class most basic amongst its k closest neighbours (k is a positive number, ordinarily little). In the event that k = 1, then the illustration is basically appointed to the class of its closest neighbour. By essentially appointing the name esteem for the sample to be the values' normal of its k closest neighbours, the same system can likewise be utilized for Regression. It can be helpful to weight the neighbours' commitments, so that the closer neighbours contribute more to the normal than the more inaccessible ones. The neighbours are chosen from an arrangement of illustrations for which the right classification or estimation of name is known. This can be considered as the preparation set for the calculation; however no unequivocal preparing step is n.

F. Apply model

A Model is prepared over an Example-set; so as to make the model learn data identified with the Example-set. At that point that model can be connected on another Example-set as a rule for forecast. Every required parameter are put away inside of the model article. In the event that properties of metadata like the number, request, sort and part of qualities are distinctive or not predictable for both Example-sets, then it may prompt genuine lapse. So they ought to have precisely same properties. On the off chance that you need to apply a few models consecutively; for instance you need to apply a couple pre-preparing models before applying an expectation model; then you may gather models.

G. Performance improvement model

Rather than the other execution assessment administrators like performance (Classification) administrator, Performance (Bi-ostensible Classification) administrator or Performance (Regression) administrator, this administrator can be utilized for a wide range of learning undertakings. It naturally decides the learning errand sort and ascertains the most widely recognized criteria for that sort. For more advanced execution counts, you ought to utilize the administrators specified previously. On the off chance that none of them meets your prerequisites, you can utilize Performance (User-Based) administrator which permits you to compose your own particular execution measure. The accompanying criteria are included for binominal classification errands: Accuracy, Precision, Recall, AUC (optimistic), and AUC (neutral).

RESULTS AND DISCUSSION

To evaluate the performance measurement of system, we have used the factors i.e. precision, recall and accuracy on a sentence basis. An experiments performed on several dataset of specific products and concluded that different dataset give depicted values as mentioned in the table-1. And in this research work, we have study that by using the hybrid model we can improve the result in terms of accuracy and precision. we can easily examine that average result of hybrid model is above the 81.11% that is quite impressive and also using the validation model and apply model in hybrid model we have improve the

result of precision as well.

We have use the mainly there dataset within this experiment result. Each dataset contain the more than 10,000 of comments and reviews. we have use the Apex AD2600 Progressive-scan DVD player as dataset1, Canon G3 as for dataset2 and the last one Nikon coolpix 4300 as dataset3 based open this three dataset we have analysing our hybrid approach which is provide us below table result . We can also see that we have impressively improved the result in our hybrid approach.

We have developed the system which combines the three classification algorithms. All are have them own functionality and regular expression pattern rules. Our hybrid method depended upon the balance operator which we have design and developed in eclipse .which main functionality is to be improve the hybrid approach more accurate . All classification has its own merits and demerits. Some of classification algorithm is work more efficient on small dataset and other like SVM and more works more efficient on large dataset. Sometimes large data comments like multiple document combination comments make some classifier less efficient in accuracy. So problem is how do we identify that "which classifier is better and which is going to use & when". For that we have develop the balance operator that help us to know that when the multiple document text comes and when is single document comes. So based on this, the balance operator will allow different classifier. So we have one basic key rationale that offers data to classifier in view of the report and we can enhance the more proficiency of our mixture technique by including the balance operator.

As shown in the table-1, the method gives promising result with compare to other models discussed in the related work that means this result that we can see in the table in quite impressive and accurate than single classifier. However results further improved (precision, recall & accuracy) using hybrid model shown in Fig-1.

CONCLUSION

We have presented hybrid approach (using SVM and K-NN) for determining sentiment analysis of Camera review document. The performance of the hybrid approach achieved 81.11% accuracy which is quite good. This is attributing to fact that many opinionated words have good discriminative power. Further improvement can be achieved into this research by improving the pre-processing task which is important input to the hybrid classifier. Also, we can improve the result in future using more different combination of algorithm in hybrid approach. Another way to improve the classification is that we can try to use specific genre of camera review because the sentiment resulted from the word in different camera genre can bring different sentiment. For example the words crazy in the someplace it is seems to be as positive words. But as sentiment classifier consider it as negative word. So for specific domain approach will be a good to improve the sentiment analysis approach.

FIGURES

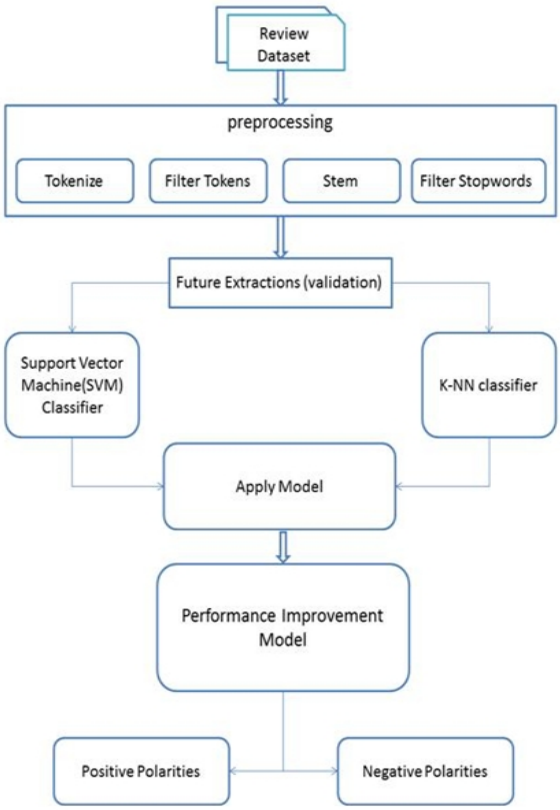


Fig. 1. Hybrid Model

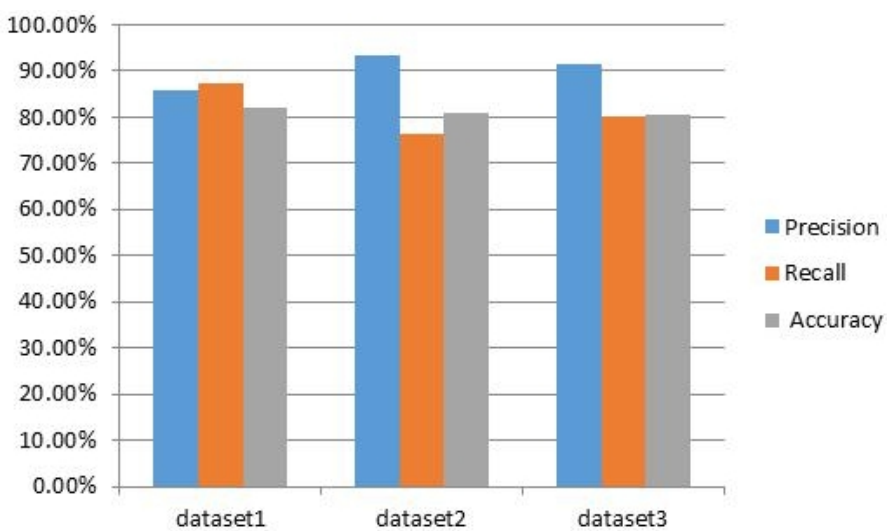


Fig-2: Simulation results with precision, recall & accuracy

TABLES

Table-1 : SIMULATION TABLE				
Product	**Dataset1**	**Dataset2**	**Dataset3**	**Average**
Precision	85.94	93.30	91.30	90.18
Recall	87.30	76.38	80	81.23
Accuracy	81.95	80.72	80.65	81.11
No of comments	10,000	9,854	10,201	10018
No of Positive	8200	8000	7850	8016
No of Negative	1800	1854	2351	1402

ACKNOWLEDGEMENT

Authors are thankful to the department of Information Technology & Computer Engineering of School of Engineering, RK University, Rajkot, Gujarat, India, for providing infrastructure facilities during progress of the work. Also Head of the department of my institution to make completion of this work successfully. Authors are grateful to everyone who contributed with data to make this project successful.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

(Proceedings available for download at rku.ac.in/icre)

RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

Performance Analysis of Job Scheduling Algorithms with Resource Reservation in Cloud Computing Environment

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ABSTRACT

The "cloud" is a combination of various hardware and software that work jointly to bring many aspects of computing to the users as an online service. Few uniqueness of Cloud Computing are pay-per-use, elastic capacity, misapprehension of unlimited resources, self service interface, virtualized resources etc. Various applications running on cloud environment would expect better Quality of Service (QoS) from Cloud environment. Improvement in Quality of Service (QoS) is possible through better job scheduling and reservation of resources in advance for execution of jobs. In this paper effects of Reservation Rate and Time Factor on the performance parameters like Resource Utilization, Waiting Time, Minimum Execution Time and Success Rate of Reserved jobs have been studied for various job scheduling algorithms and their performance have been calculated in resource reservation environment in Cloud.

SUMMARY

In this paper, performance of three job scheduling algorithms with resource reservation have been analysed and compared with one another in Cloud Computing Environment.

Keywords: Cloud Computing, Resource Reservation, Min-Min, Max-Mix, Priority Scheduling

INTRODUCTION

Cloud is a parallel and distributed computing system consisting of a collection of inter-connected and virtualized computers that are dynamically provisioned and presented as one or more unified computing resources based on service-level agreements (SLA) established through negotiation between the service provider and consumers. Main goal of cloud is to give access to assorted resources to users whenever and however they need. Various resources of cloud are processing power, data storage system, operating system, application software, infrastructure etc (*8*).

When resources are physically scattered and owned by variety of service providers or service consumers, resource administration plays very crucial role in achieving QoS. Scheduling is assigning set of jobs to set of resources. Output of almost every scheduling algorithm depends on efficient scheduling. Resource reservation is a scheduling technique for reserving a single or group of resources for a particular time for access only by a specified user or group of users (*1*).

Scheduling can be categorized in two types: static scheduling and dynamic scheduling. In static scheduling resources are allocated prior to execution of jobs and in dynamic scheduling scheduler keeps allocating the resources as jobs keep arriving for execution (*8*).

RELATED WORK

Resource Broker or scheduler maintains two separate queues. One for the jobs which need advance resource reservation and another for jobs which do not need any resource reservation. So first resources will be allocated to the jobs which are having reservation on the required resource while in the free slots resources will be allocated to other jobs which do not require any reservation. In the case of online scheduling, if job with reservation finishes its execution before predicted time then resource will be allocated to next job in queue immediately. In rigid resource scheduling, if job finishes its execution before time than next job in queue will have to wait till its pre defined starting time which leads to poor resource utilization.

Comparison of various job scheduling algorithms is given in below Table 1.

SIMULATION RESULTS AND DISCUSSION

Simulation has been done with single resource environment in Cloud. Each resource is having one processor. Capacity of processor is 200 MIPS (Millions of Instruction per Second). Simulation has been performed for 3000 jobs having random execution time.

Definition of scheduling algorithm simulation variables:

1. **Reservation Rate**: It is the ration of jobs which require resource reservation to total number of jobs.
2. **Time Factor**: It is the time at which jobs need to be submitted to scheduler in advance.

Definition of scheduling algorithm performance parameters:

1. **Resource Utilization**: This is ratio of running time of processor of resource to total time. Total time also includes idle time of processor.

2. **Waiting Time**: This is the time from which user submit job which does not require reservation to scheduler to it actually starts its execution. It is waiting time of non reservation jobs.
3. **Minimum Execution Time**: It is total execution time of all the jobs i.e. with/without reservation by respective scheduling algorithm.
4. **Success Rate of Reserved Jobs**: It is ratio of total successfully executed reserved jobs to total no of scheduled job.

Effect of Reservation Rate and Time Factor on job scheduling algorithms like Priority Scheduling, Min-Min and Max-Min have been calculated and analysed with performance parameters Resource Utilization, Waiting Time, Minimum Execution Time and Success Rate of Reserved jobs.

As shown from Fig. 1 to Fig, 3, initially Reservation Rate is 0; it means there is not any job which requires any resource to be reserved. With increase in Reservation Rate, Resource Utilization is also increased. When Reservation Rate increases i.e. more no of jobs with requirement of resource reservation, increase in waiting time has been observed. Here jobs which require resource reservation will get prior chance to be executed. So, non reserved jobs need to wait for resource till it gets ideal. Hence increase in Waiting Time is observed with increase in Reservation Rate. Delay in execution of non reserved jobs will affect overall completion time. So more the reserved jobs, more delay in overall completion time. So, increase in Minimum Completion Time observed with increase in Reservation Rate.

Now one interesting observation is, till some increase in Reservation Rate, Success Rate of Reserved job showing positive results as it keep increasing. After some increase, negative effect is observed in performance of reserved jobs. Reason for this negative effect is, when there are more number of reserved jobs, requirements of such jobs get conflicted with one another and as a result overall performance of reserved job get negatively affected.

To summarize, up to some Reservation Rate, we are observing positive effect on all mentioned parameters in all three scheduling algorithms. But beyond some acceptable Reservation Rate, due to conflicting requirements of reserved jobs, negative effects have been observed.

As shown from Fig. 4 to Fig, 6, when we are increasing Time Factor, Resource Utilization decreases. The reason behind this decrease is, where we are submitting jobs earlier to the scheduler, it will get more time to schedule the jobs. So scheduler can schedule the jobs with minimum scheduling overhead and optimize resource utilization. Other parameters like Waiting Time, Minimum Execution Time and Success Rate of Reserved jobs are showing negative effect with increase in Time Factor. Earlier the submission, reserved jobs will be scheduled prior to non reserved jobs. So it will affect overall performance of scheduling algorithms with respect to mentioned parameters.

CONCLUSION

In this paper effects of Reservation Rate and Time Factor on the performance parameters like Resource Utilization, Waiting Time, Minimum Execution Time and Success Rate of Reserved jobs have been studied for various job scheduling algorithm like Priority Scheduling, Min-Min and Max-Min, and their performance have been calculated in resource reservation environment in Cloud. Up to some Reservation Rate, we are observing positive effect on all mentioned parameters in all three scheduling algorithms. But beyond some acceptable Reservation Rate, due to conflicting requirements of reserved jobs, negative effects have been observed. We are observing decrease in utilization of resources by increasing prior submission time of jobs because scheduler will get more time to schedule jobs for available resource. For

the other parameters, in all scheduling algorithms, negative effect has been observed with increase in Time Factor.

FIGURES

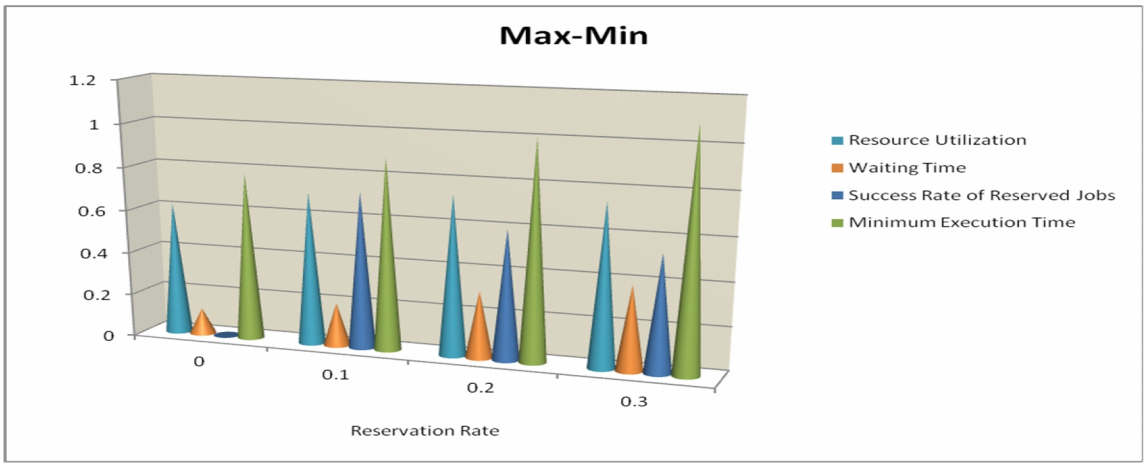


Fig. 1. Effect of Reservation Rate on All Performance Parameters for Max-Min Algorithm

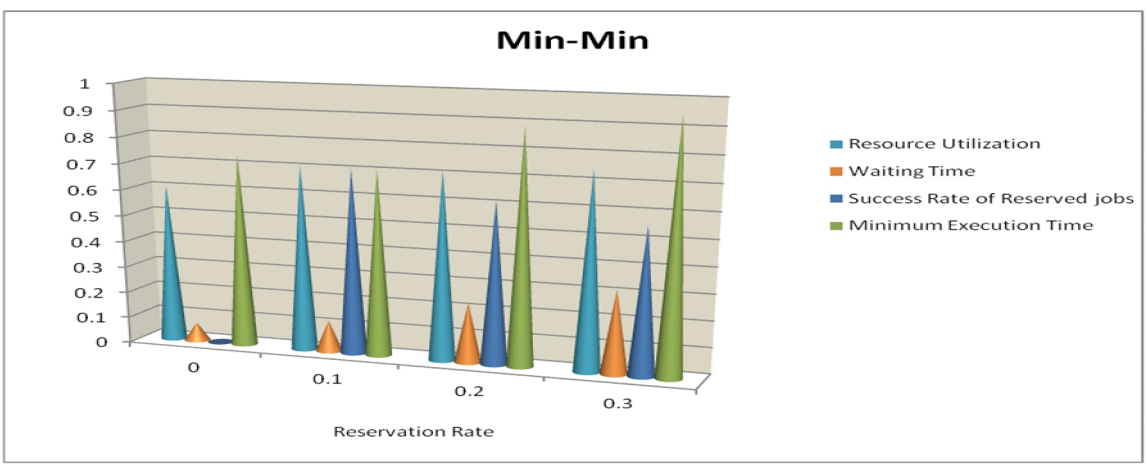


Fig. 2. Effect of Reservation Rate on All Performance Parameters for Min-Min Algorithm

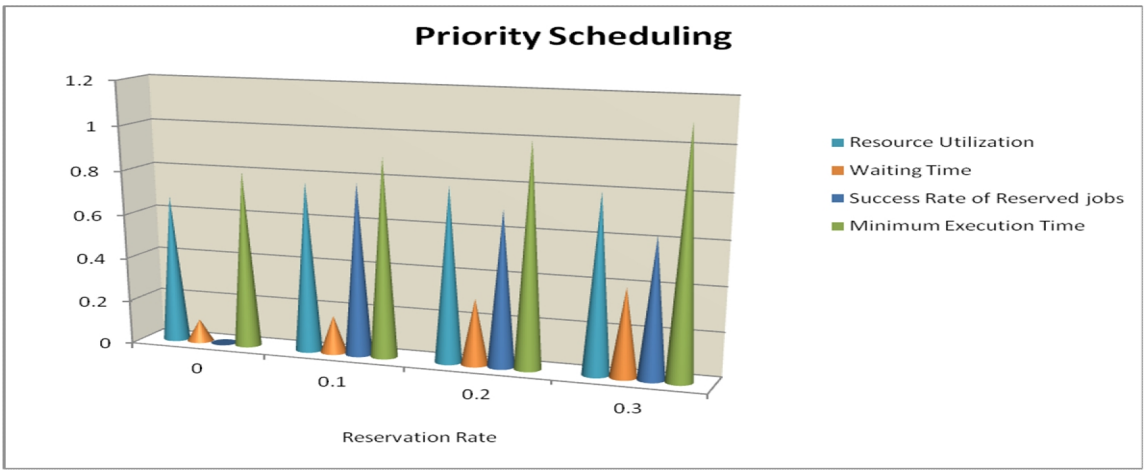


Fig. 3. Effect of Reservation Rate on All Performance Parameters for Priority Scheduling Algorithm

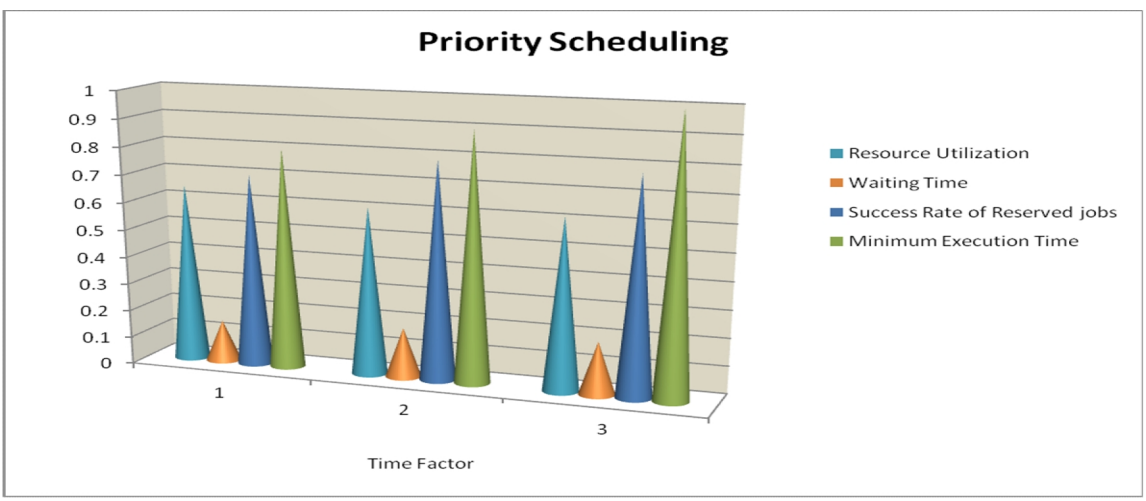


Fig. 4. Effect of Time Factor on All Performance Parameters for Priority Scheduling Algorithm

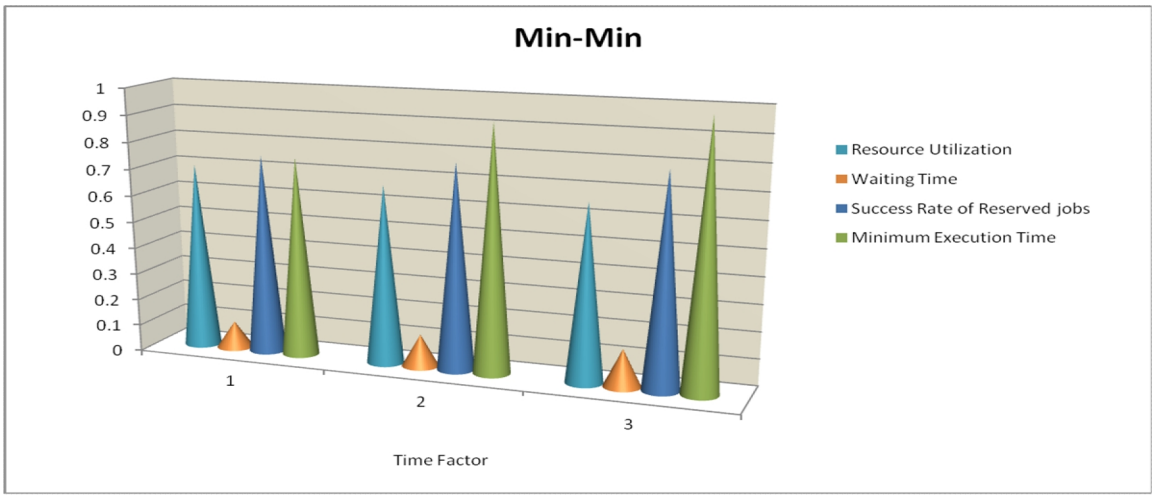


Fig. 5. Effect of Time Factor on All Performance Parameters for Min-Min Algorithm

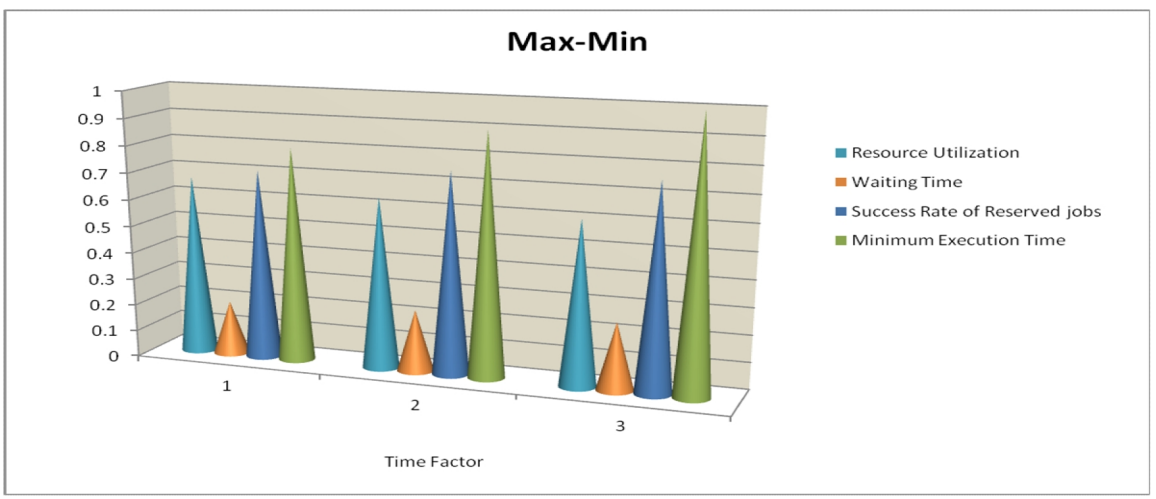


Fig. 6. Effect of Time Factor Rate on All Performance Parameters for Max-Min Algorithm

TABLES

Table 1. Comparison of Various Scheduling Algorithms

Sr. No.	Job Scheduling Algorithms	Advantage	Disadvantage
1	Opportunistic Load Balancing (OLB) [2].	Implementation is simple	Expected completion time will not be considered. Poor execution time
2	Minimum Execution Time (MET) [2].	Job is allocated to machine with best execution time for that job	Few machines may be over utilized and few will be underutilized, which may lead to load misbalancing
3	Minimum Completion Time (MCT) [3].	Combine few benefits of OLB and MET	Causes few jobs to be allocated to machines which do not have the minimum execution time for those jobs
4	First Come First Serve (FCFS)	Very simple to implement. Fair for shorter jobs	Long jobs make short jobs wait and unimportant jobs make important jobs wait
5	Shortest Job First (SJF)	Better for batch jobs	Execution time should be known in advance
6	Longest Job First (LJF)	Better for batch jobs	Execution time should be known in advance
7	Priority Scheduling	Urgency of the job will also be taken in to consideration.	Priority should be known in advance.
8	Min-Min [2].	Considers all tasks which are yet to be matted while taking each mapping decision	Execution time should be known in advance
9	Max-Min [2]	Considers all tasks which are yet to be matted while taking each mapping decision	Execution time should be known in advance
10	Duplex [2]	Combination of the Min-Min and Max-Min heuristics	Overhead of combining Min-Min and Max-Min.

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Thermal Analysis of Open Cycle Surface Condenser of120 MW Coal Based Power Plant for Investigation of different Tube Parameters for Optimum Performance

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ABSTRACT

The conventional steam power plant working under the Rankine cycle and the steam condenser as a heat sink and the steam boiler as a heat source having the vital importance for the power plant operating process. Due to highly corrosive sea water cooling water tubes of surface condenser are getting failure which was notified during the case study of reference plant at GSECL-Sikka, 120 MW power plant, Gujarat. The present work involves the analysis of various tube related parameters such as Tube outer diameter and Tube wall thickness taken from TEMA standards while as Tube material Cupro-Nickle, Admiralty brass, SS-304 and Titanium are selected. It is observed that Tube material as Titanium having outer diameter is 22.25 mm with 0.3 mm wall thickness and plain tube is most suitable for sea water condenser tubing. The predicted results through Matlabcan be used to determine the design consideration of new condensers.

SUMMARY

Different Condenser tube related parameters are compared and best set of parameter is suggested to obtain the optimum performance of surface condenser.

Keywords: Surface Condenser, Thermal Power Plant, Tube Material

1. INTRODUCTION

GSECL, Sikka plant having two unit of each capacity is 120 MW named Unit-1 and Unit-2. Both the units working under the open cycle cooling water supply system for condenser cooling.The power house consists of three steam turbines units at 100% load. The power plant uses Coal and Lignite which is obtained from a nearby query. Now a days due to lower quality of Indian coal and higher cost, imported coal is used for power generation. The need for electrical energy will certainly continue to grow, and it has become imperative to lower the cost of electricity and enhance the operational economy of the turbine unit.This paper elaborates the analysis and thermal design of tube related parameters of surface condenser of GSECL-Sikka of Unit-2, with comparison of Plain and Corrugated Tubethrough Matlab programming based on Overall heat transfer coefficient, Surface area, Tube length, Pressure drop and pumping power.

Nomenclature

C_N	Correction Factor For Inundation	m	Flow constant
C_p	Sp. Heat At Constant Pressure, kJ/Kgk	N	No. of tubes
C_u	Correction Factor For Vapor Shear	n	Flow constant
d	Diameter, m	Nu	Nusselt no.
f	Friction coefficient	P	Generator Output ,kW
g	Gravitational force, N	P_L	Longitudinal Pitch, m
HP	High Pressurized Turbine	P_t	Transverse Pitch, m
h	local heat transfer coefficient, W/m^{2}K	Pr	Prandlt no.
h_1	Local Heat Transfer Coefficient Of Top First Tube, W/m^{2}K	Re	Reynolds no.
h_G	Gravitational heat transfer coefficient, W/m^{2}K	$T_{s,}$	Saturated steam Temperature, °C
U	Overall heat transfer coefficient, W/m^{2}K	$X_{t,}$	Lockhart-Martinelli Parameter
V	Velocity, m/s	w	Velocity (based on mean flow width), m/sec

2. BACKGROUND

2.1 NussultCo-relations (5)

Generally two approaches are used in designing heat exchangers, especially two- phase flow heat exchangers such as condensers. In the first approach, the heat exchanger is taken as a single control volume with an average overall heat transfer coefficient and two inlets and two outlets (Lump Analysis). In second analysis, the heat exchanger is divided into segments or multiple control volumes, with the outlet of one control volume being the inlet to an adjacent control volume. This is a local analysis. The heat transfer rate for heat exchanger is obtained by integrating the local values. The first approach is more common and simpler and it generally provides acceptable results for design (5). For thermal design of surface condenser generally Nussult Correlations are used.

No. of tubes is calculated based on tube velocity and coolant mass flow rate.

$$v_t = \frac{m_c}{\left(\frac{\pi}{4}\right) d_0^2 \mathrm{gt}} \left(\frac{n}{N_t}\right) \quad (1)$$

(2)

Reynolds number= Re $= \frac{\mu_c \rho_c d_i}{\mu_c}$

Based on Reynold's no. type of flow is noticed. Mostly inside the condenser cooling water tubes, the flow is Turbulent.

$$N = \frac{\frac{f}{2}R}{1.07 + 12.7\left(\frac{f}{2}\right)^{\frac{1}{2}}\left(P^{\frac{2}{3}} - 1\right)} \quad (3)$$

Where, friction factor $f = (1.58ln(R) - 3.28)^{-2}$

Inside i.e. tube side heat transfer coefficient is found out by following relation,

$$h_i = \frac{N K_c}{d_i} \quad (4)$$

$$L = \frac{\Delta T_{i} - \Delta T_o}{ln \frac{\Delta T_i}{\Delta T_o}} \quad (5)$$

$$\frac{1}{u} = \frac{1}{R_t} + \frac{1}{h_o} \quad (6)$$

Where, Rt is the total fouling resistance which is found out by following relation,

$$R_t = R_f + \left[\frac{1}{h_o} + R_f\right]\frac{d_o}{d_i} + \frac{t_w}{k_w}\frac{d_o}{D_m} \quad (7)$$

Where, mean diameter = $D_m = \frac{d_0 - d_i}{ln(d_0 - d_i)} = \frac{1}{2}(d_0 - d_i)$

And t_w is the wall thickness; and do and di is tube outside and inside diameters.

Outside heat transfer coefficient is also needed to fine overall heat transfer coefficient,

$$h_o = 0.728\frac{k_l}{d_o}\left[\frac{\rho_l^2 g \, ln \, d_o^3}{\mu_l \Delta T_w k_l}\right]^{\frac{1}{4}} \quad (8)$$

$$h_o = \frac{18739.71}{\Delta T_w^{\frac{1}{4}}} \quad (9)$$

The temperature difference ΔT_w is given by,

$$\Delta T_w = \Delta T - R_i q''$$

Where, q'' = local flux = uΔT

$$\Delta T_w = \Delta T(1 - R_t u) \quad (10)$$

i. Gauss the value of ΔT_w

ii. Calculate h_0 from equation ……..(8)

iii. Calculate u from equation …… (6)

iv. Recalculate ΔT_w from equation …(10)

v. Repeat the calculations from step 2 and continue the iterations until u converges.

The mean overall heat transfer (U_{mean}) coefficient can then be determined by taking the average of the inlet and outlet heat transfer coefficients.

Heat load on condenser = $Q = m_s \times h_f$

2.2 Pressure drop:

In the heat exchanger there is a close physical and economical affinity between pressure drop and heat transfer. This pressure drop is the static fluid pressure which may be expanded to drive the fluid through heat exchanger. Increasing the flow velocity will cause a rise of heat transfer coefficient which results in compact heat exchanger design and lower investment cost. However increase of flow velocity will cause more pressure drop in exchanger which results in additional running cost. For this reason when designing a heat exchanger pressure drop must be considered with heat transfer[6,7].

$$\Delta P_t = \frac{\rho_t v_t^2}{2}\left(\frac{L}{d_i} f + p\right) N_p$$

where, p is a constant and its different values are considered by different authors, Generally for surface condenser application it is considered as 2.3.

2.3 Pumping power:

For steam surface condenser, only tube side is a running cost of condenser, while shell side steam expands in a condenser from turbine exhaust directly. That's why total pumping power is computed from tube side pressure drop only. This power is computed by following formula [6,7];

$$\Delta E_p = \frac{1}{\eta}\left(\frac{m_c}{\rho_c} \Delta P_t\right)$$ Where, η=Pump efficiency

3. PRESENT WORK

3.1 Tube outer diameter:

In reference plant Unit-1 Condenser is splitted into two different shells shown in fig. (1)(To minimize pressure drop) having equal size and shape with inside configuration which contains tube outer diameter is 25.40 mm with 1.22 mm tube wall thickness and material is Cupro-Nickel (Cu:Ni-90/10). This work focuses on one condenser shell because both the shell is totally same and as per one shell design, same as second shell can also be design [10]. For better design scope the

outer diameter values have been varied as per different materials and TEMA standards for availability of manufactured tubes in market. The table no. 1 to 6 indicates the different tube parameters and materials.

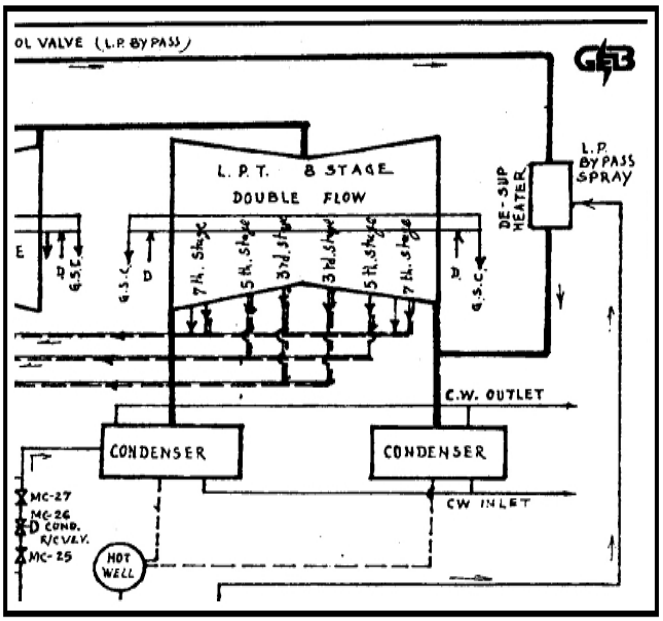


Fig. 1. Enlarged view of LP turbine and Condenser arrangement from steam cycle (Source: GSECL, Sikka)

Table 1. Tube diameter range selected from TEMA: (23)

Sr.No.	1	2	3	4	5	6	7	8
Tube outer diameter (mm)	15.87	19.05	22.25	25.40	31.75	38.10	50.80	63.50

Table 2. Tube materials are selected from TEMA: (23)

Sr.No.	1	2	3	4
Material	Cu:Ni	Admiralty Brass	SS-304	Titanium

The values of Overall heat transfer coefficient, Area, Length, Pressure drop and pumping power for different material have been obtained from Matlab program. Results and plots are listed belowed.

Table 3. For tube material as Cupro:Nickle

Sr.No.	Tube outer diameter (mm)	U_{mean} (W/m^2K)	Area (m^2)	Length (m)	Pressure Drop (Pa)	Pumping power (kWatt)
1	15.87	3009.6	5876.5	4.2307	20835	123.400
2	19.05	3010.7	5881.8	5.3937	20675	122.440
3	22.25	3002.6	5903.9	6.5841	20545	121.670
4	25.40	2990.1	5933.8	7.7977	20436	121.030
5	31.75	2960.2	6002.5	10.2836	20264	120.020
6	38.10	2928.9	6073.5	12.8353	20132	119.230
7	50.80	2870.4	6208.4	18.0973	19938	118.080
8	63.50	2732.6	6329.7	23.5316	19800	117.260

Table 4. For tube material as Admiralty Brass

Sr.No.	Tube outer diameter (mm)	U_{mean} (W/m^2K)	Area (m^2)	Length (m)	Pressure Drop (Pa)	Pumping power (kWatt)
1	15.87	3140.1	5639.0	4.0597	20475	121.260
2	19.05	3139.0	5648.3	5.1795	20327	120.390
3	22.25	3128.7	5673.1	6.3268	20208	119.680
4	25.40	3113.9	5705.1	7.4973	20108	119.090
5	31.75	3079.8	5776.9	9.8971	19951	118.160
6	38.10	3044.9	5849.9	12.3628	19830	117.440
7	50.80	2980.3	5987.5	17.4536	19653	116.390
8	63.50	2830.6	6110.6	22.7171	19527	115.650

Table 5. For tube material as SS-304

Sr.No.	Tube outer diameter (mm)	U_{mean} (W/m^2K)	Area (m^2)	Length (m)	Pressure Drop (Pa)	Pumping power (kWatt)
1	15.87	3140.1	5639.0	4.0597	20475	121.260
2	19.05	3139.0	5648.3	5.1795	20327	120.390
3	22.25	3128.7	5673.1	6.3268	20208	119.680
4	25.40	3113.9	5705.1	7.4973	20108	119.090
5	31.75	3079.8	5776.9	9.8971	19951	118.160
6	38.10	3044.9	5849.9	12.3628	19830	117.440
7	50.80	2980.3	5987.5	17.4536	19653	116.390
8	63.50	2830.6	6110.6	22.7171	19527	115.650

Table 6. For tube material as Titanium

Sr.No.	Tube outer diameter (mm)	U_{mean} (W/m^2K)	Area (m^2)	Length (m)	Pressure Drop (Pa)	Pumping power (kWatt)
1	15.87	2742.8	6432.8	4.6311	21679	128.390
2	19.05	2747.6	6429.0	5.8955	21489	1272.70
3	22.25	2743.7	6444.6	7.1871	21334	1263.50
4	25.40	2735.4	6469.6	8.5018	21205	1255.90
5	31.75	2713.2	6531.3	11.1896	20999	1243.70
6	38.10	2688.9	6597.5	13.9427	20840	1234.30
7	50.80	2642.0	6726.1	19.6065	20606	1220.40
8	63.50	2527.5	6843.4	25.4413	20439	1210.50

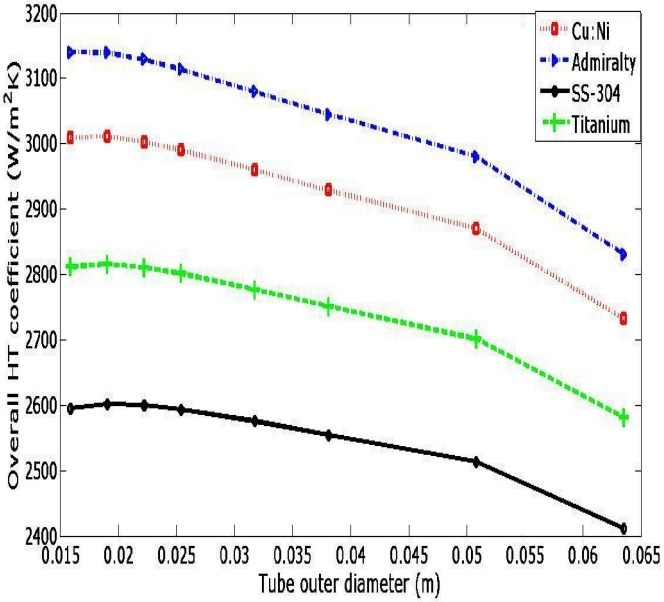


Fig. 2. Overall HT coefficient v/s Tube outer diameter

From the above plot it can be visible that Admiralty brass having very high thermal conductivity compare the all other three materials and highest overall heat transfer coefficient can be achieved. But when tube diameter increases, no. of tube decreases and due to less surface area which leads lower heat transfer coefficient. In case of other materials as per the thermal conductivity, heat transfer coefficient will be noted.

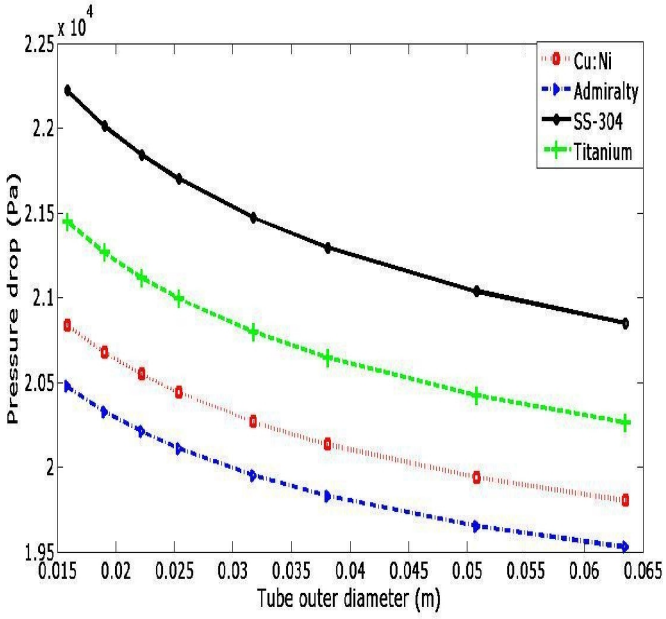


Fig. 3. Pumping power v/s Tube outer diameter

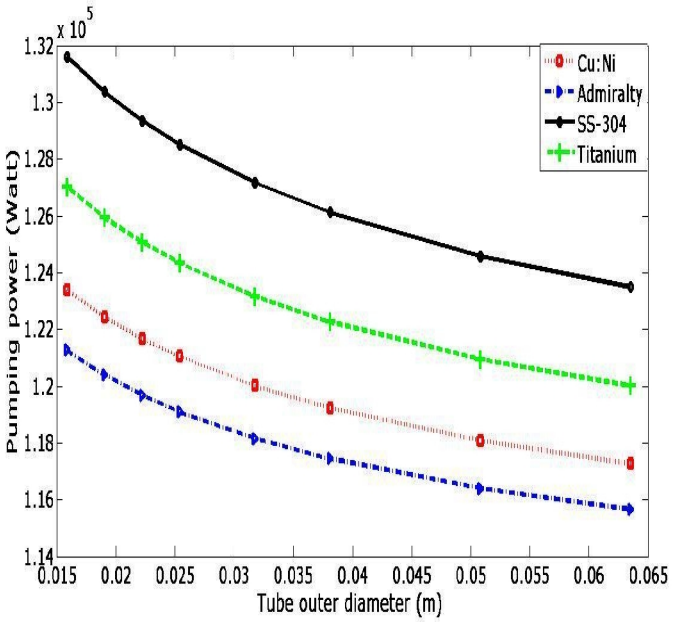


Fig. 4. Pumping power v/s Tube outer diameter

From above plots, it can be notified that nature of both the plot for different material is same, because pressure drop is directly proportional to pumping power. When diameter increases, pressure drop decreases because of length decreases. Highest pressure drop is noted in case of SS-304 because when outer diameter changes, inner diameter also changes and it affects no. of tube length and pressure drop. Similarly when conductivity increases, no. of tube decreases, due to more length pressure drop increases.

3.2 Diameter Selection:

To select the best diameter among the TEMA standard range from the above tables and plots, Superimposing has been done in Matlab i.e. two different scale graph is shown on one graphical layer for better and ease of comparison to select the best value of tube outer diameter which is shown below:

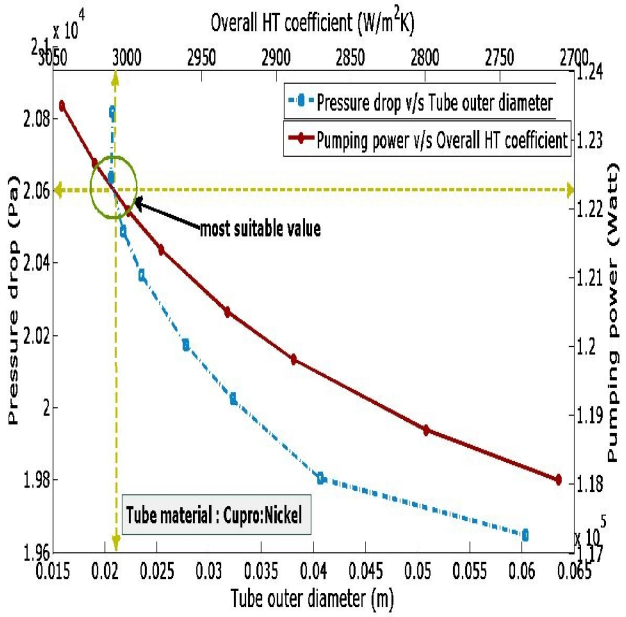


Fig. 5. Superimposed plot for Cupro: Nickel

This plot includes left side and right side Y-axis pressure drop and pumping power respectively and bottom and top X-axis indicates tube outer diameter and overall heat transfer coefficient respectively. Currently plant having outer tube diameter is 25.40 mm and tube material is Cu:Ni. But as per this work when both the line merges, at that point the tube outer diameter is 22.25 mm which is one of the value from TEMA standards also. When diameter decreases, no. of tube increases and surface area also increases. So, it affects on overall heat transfer coefficient and finally heat transfer increases. At that point the value of four parameters can be read as following:

Table 7. Tube Parameters

Parameters	U_{mean}(W/m^{2}K)	D_o (mm)	ΔP (kPa)	$E_{\Delta P}$ (kW)
Value	3010	22.25	20.600	122.100

3.2.1 Admiralty Brass:

Same superimposed plot has been made for Admiralty Brass as a tube material which is shown belowed. From the plot it is notified that both the lines intersects at the point includes diameter is 55 mm which is not standard value as per TEMA and at this diameter no. of tubes decreases and it decreases the oveall surface area. So, due to poor condensation heat transfer coefficient, heat transfer will be less which is not advisable. Next both the lines are nearer to intersection at the diameter value of 19.05 mm which is standard value as per TEMA and at this value surface area and heat transfer is also very high. At that point the value of four parameters can be read as following:

Table 8. Tube Parameters

Parameters	U_{mean}(W/m^{2}K)	D_o (mm)	ΔP (kPa)	$E_{\Delta P}$ (kW)
Value	3030	19.05	20.300	122.800

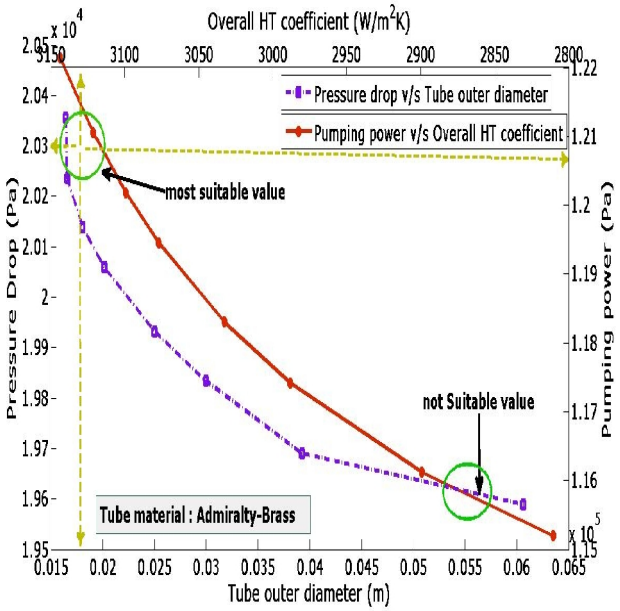


Fig. 6. Superimposed plot for Admiralty brass

3.2.2 SS-304

In case of stainless steel having 304 grade, the lines intersect at two different points. One is indication the diameter value is 60 mm and nearer to it one value 63.5 mm is the standard value as per TEMA. At this point due to large diameter , tube count will decrease and it affects adversely upon overall condensing heat transfer coefficient and heat transfer, which is not advisable. The next value is 31.75 which is one of the standard value from TEMA. At this diameter due to more surface area heat transfer coefficient will be high. At that point the value of four parameters can be read as following:

Table 9. Tube Parameters

Parameters	U_{mean}(W/m^{2}K)	D_o (mm)	ΔP (kPa)	$E_{\Delta P}$ (kW)
Value	2580	31.75	21.500	127.100

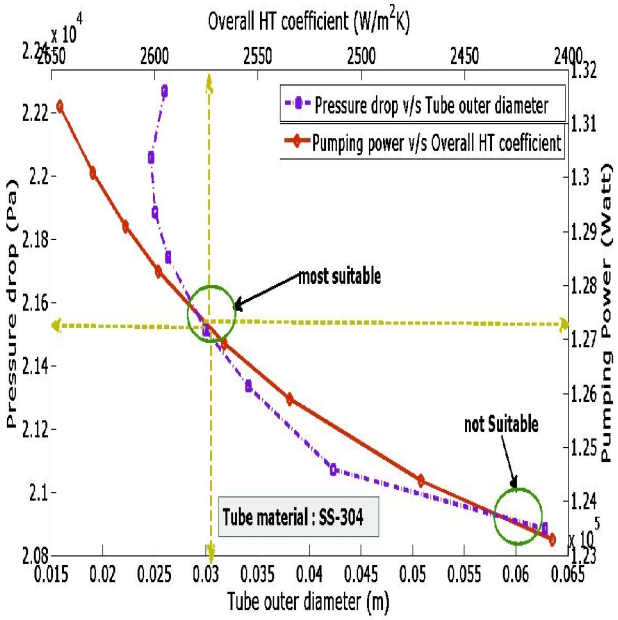


Fig. 7. Superimposed plot for SS-304

3.2.3 Titanium

In case of Titanium metal both the lines intersect at the point on which the tube outer diameter having value 22.25 mm. Less diameter means more overall heat transfer coefficient and more heat transfer inside the condenser. At that point the value of four parameters can be read as following:

Table 10. Tube Parameters

Parameters	U_{mean}(W/m^{2}K)	D_o (mm)	ΔP (kPa)	$E_{\Delta P}$ (kW)
Value	2820	22.25	21.200	126.000

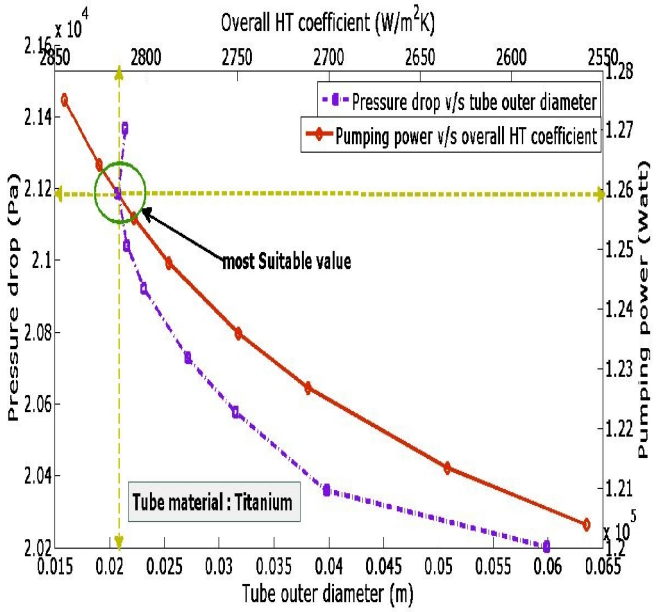


Fig. 8. Superimposed plot for Titanium

From above plots and tables it is clear that tube outer diameter can be selected as per the tube material and based on major rating parameters.

3.3 Tube wall thickness

Tube wall thickness of condenser tubing is very important parameters which are responsible for latent heat removal and conduction heat transfer. When tube thickness increases, due to more resistance of material heat transfer decreases and material cost increases. On other hand when tube thickness decreases beyond certain limit due to fouling and corrosion tube gets punctured and permanently chance of failure. So, tube thickness and condenser tube material must be set at its optimum level.

3.3.1 Cupro:Nickel:

Cupronickel or copper-nickel is an alloy of copper that contains nickel and strengthening elements, such as iron and manganese. Cupronickel is moderate resistant to corrosion in seawater, because its electrode potential is adjusted to be neutral with regard to seawater. Because of this, it is used for piping, heat exchangers and condensers in seawater systems, as well as marine hardware. But while working with sea water after a long time this alloys undergoes failure and many tubes got punctured which allows to leakage the cooling water from the tube and those tubes have to plugged out permanently. When no. of tube decreases in condenser, the required surface area also decreased to condense the steam property and its leads to poor heat transfer and tends to decrease in overall efficiency of power plant [13].

In case study plant i.e. GSECL-Sikka thermal power station having tube material Cupro:Nickel-90:10 and many tubes got punctured due to sea water fouling and corrosion effect. Those tubes are plugged out by operators and desired condensation coefficient cannot be obtained due to lower the surface area and tube failure. By communicating plant engineers it was noticed that for sea water application the tube wall thickness should not less than 1.22 mm because of highly corrosion and biological fouling due to sea water. Still many tubes got punctured due to failure after the long term use. It is required to think about the another very good material for condenser tubing [14,23].

The below graph indicated the current situation in plant. All the values of required parameters is found out as following:

Table 11. Tube Parameters

Parameters	U_{mean}(W/m^{2}K)	t_w(mm)	ΔP (kPa)	$E_{\Delta P}$ (kW)
Value	3020	1.22	20.350	122.700

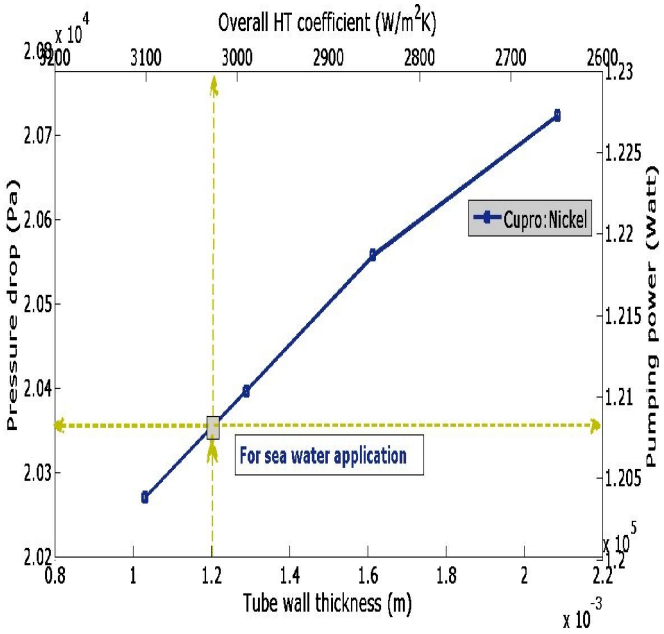


Fig. 9. Cupro:Nickel tube having wall thickness 1.22 mm

3.3.2 Admiralty Brass

In case of Admiralty brass, it contains very high thermal conductivity but not works effectively with sea water. As per Technical letter of Olin fineweld tube (USA) the tube thickness is 1.22 mm is kept for normal fresh water and 0.2 mm should be kept as a allowance for bio-fouling and corrosion effect. So, thickness must be 1.42 mm while using the tube Admiralty brass. All the values of required parameters is found out as following:

Table 12. Tube Parameters

Parameters	U_{mean}(W/m^{2}K)	t_w(mm)	ΔP (kPa)	$E_{\Delta P}$ (kW)
Value	3060	1.42	20.110	119.150

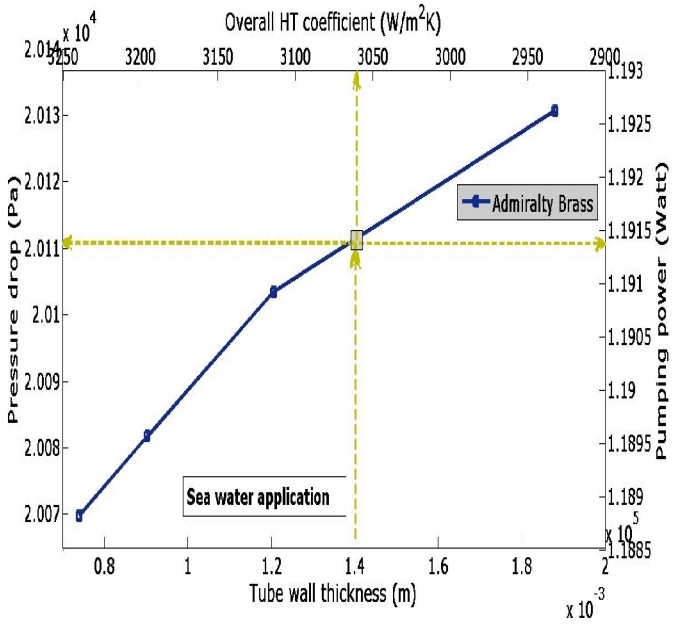


Fig. 10. Admiralty brass tube having wall thickness 1.42 mm

3.3.3 SS-304

In case of Stainless Steel with grade 304 contains highly corrosive resistance property with sea water and biofouling problems. The tubes made from SS-304 having very long life and very good sustainability against tube failure. As per the literature by John M. Burns ,P.E.Burns Engineering Services, Inc., "Modular Steam Condenser Replacements Using Corrosion Resistant High Performance Stainless Steel Tubing" now a days using SS-304 as tube material, wall thickness can be kept upto 0.51 mm which reduces the material cost and highly corrosion resistivity with long life. All the values of required parameters is found out as following:

Table 13. Tube Parameters

Parameters	U_{mean}(W/m^{2}K)	t_w(mm)	ΔP (kPa)	$E_{\Delta P}$ (kW)
Value	3000	0.51	20.800	123.000

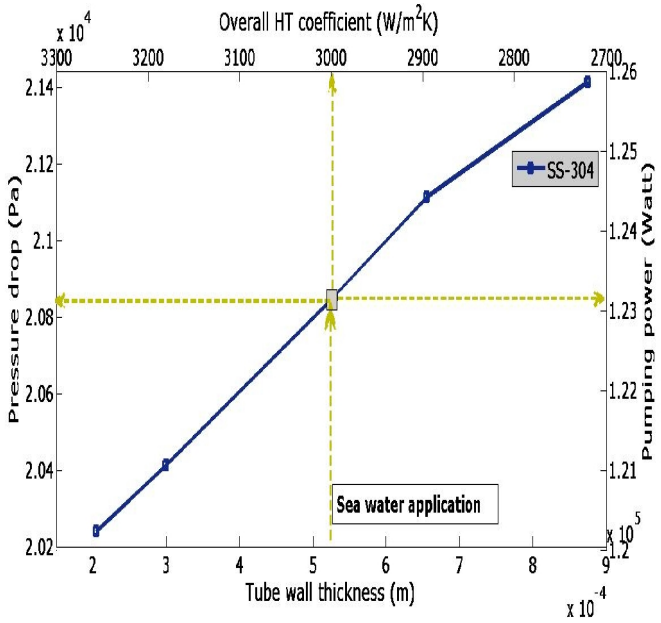


Fig. 11.SS-304 tube having wall thickness 0.51 mm

3.3.4 Titanium

Titanium tubing is a lightweight material whose density is approximately 60 percent of steel tubing and 50 percent of nickel and copper alloy tubing. Typical applications included marine, refinery, pulp and paper, chlorine and chlorate production, hydrometallurgy, and various other oxidizing and mildly reducing chemical services. Commercially pure titanium and its alloys provide excellent resistance to general and localized corrosion attack under most oxidizing, neutral and inhibited reducing conditions in aqueous environments [12]. Titanium is also notable for its outstanding resistance to chlorides and other halides generally present in most process streams. In addition, titanium resists other malicious phenomenon including steam and particle erosion, crevice corrosion, galvanic attack and MIC. Given this general corrosion immunity, designers have increasingly applied thin-wall condenser tubing in pursuit of cost savings and performance enhancement. From the literature Dennis J. SchumerthValtimet, Inc. Tustin, CA, Thin-Wall Titanium Condenser Tubing The Next Plateau, International Joint Power Generation Conference, Paper No. IJPGC2002-26121, Phoenix, AZ, USA, June 24-26, 2002[21]. It is notified that typically, these thin-wall applications have, over the past several years, been limited to 0.5mm walls or heavier. According to this article now a days tube wall thickness can be kept 0.3 mm only with titanium. Because titanium having very good mechanical properties at high temperature only and excellence corrosion resistant properties against sea water and bio-fouling. All the values of required parameters is found out as following: [23,24]

Table 14. Tube Parameters

Parameters	U_{mean}(W/m^{2}K)	t_w(mm)	ΔP (kPa)	$E_{\Delta P}$ (kW)
Value	3170	0.3	20.300	121.000

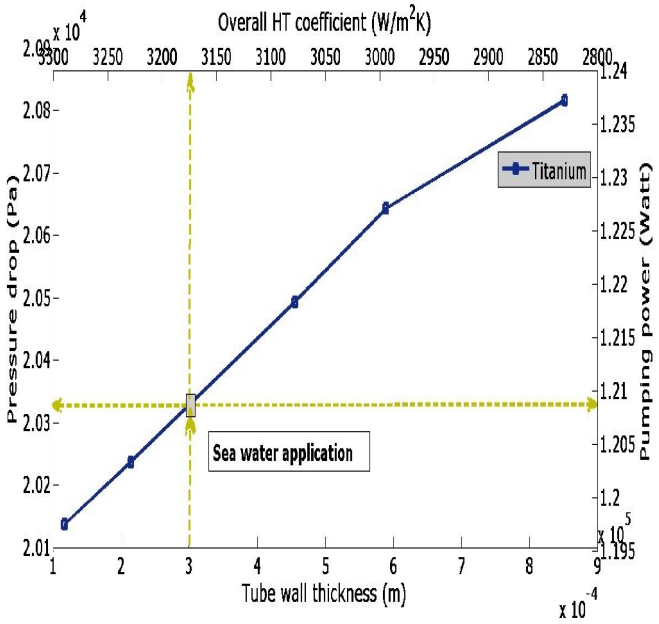


Fig. 12. Titanium tube having wall thickness 0.30 mm

4. RESULTSAND DISCUSSION

4.1 Based on tube outer diameter, tube wall thickness and tube material:

From the above discussion and superimposed plots it is clear that Cu:Ni and Admiralty brass having very good thermal conductivity and lower cost compare to other materials, but the major drawback is these tubes cannot work properly with highly salty and corrosive sea water application with long life. At the middle span of condenser life, these tubes get failure and need to retrofit it, which increases the overall cost and decrease the plant efficiency [8]. So, next option is SS-304 and Titanium having successful working with sea water throughout the entire life of condenser with maximum efficiency.

- With tube material SS-304, tube outer diameter and wall thickness should be 31.75 mm and 0.51 mm respectively. (From fig. 12)
- With tube material Titanium, tube outer diameter and wall thickness should be 22.25 mm and 0.30 mm respectively.

The final selection of tube material can be done from cost comparison.

Table15. Cost Comparison between SS-304 and Titanium

Sr. No.	Parameters	Tube material	
		SS-304	**Titanium**
1	Tube outer diameter (mm)	31.75	22.25
2	Tube wall thickness (mm)	0.51	0.3
3	Tube inner diameter (mm)	30.73	21.65
4	Tube length for above parameter (m)	11.77	7.18
5	No. of tubes for above parameter	5852	12840
6	Density (kg/m^3)	8000	4506
7	Cost of material as per Indian market/kg(Rs.)	140	350
8	Cross sectional area/tube	$50 \times 10^{-6} m^2$	$20.68 \times 10^{-6} m^2$
9	Volume/tube	$588.5 \times 10^{-6} m^3$	$148.53 \times 10^{-6} m^3$
10	Weight/tube	$4.7\ k$	$0.70\ k$

11	Tube cost/condenser shell	Rs. 38,50,616	Rs. 31,45,800
12	Total tube cost	Rs. 7 ,0 ,2	Rs. 6 ,9 ,6

As per above Comparison table it may be noticed that in case of Titanium tube due to less diameter, no. of tube increases, but due to less density overall cost decreases even cost of material is more compare to SS-304. Also by using Titanium material, due to less thickness heat transfer coefficient increases and tube having more life with sea water application.
From above discussion it is clear that tube material should be Titanium, outer diameter and tube wall thickness should be 22.25 mm and 0.3 mm respectively.

4.2 Tube type

Most of the conventional power plants uses plain tubes for condenser application. Currently case study power plant i.e. GSECL-Sikka having plain Cu:Ni tubes. To increase heat transfer rate one of the best option is to use spirally corrugated tubes for condenser. Now a days corrugated tubes are widely manufactured in various diameter and different material as per TEMA standards[23,24]. Due to corrugated rough surface, the flow inside the tubes become more turbulent and when turbulences increases, heat transfer also increases and area become more compact. But the major drawback is it increases pumping power also. By selecting best material with spirally corrugation, surface area, heat transfer, pumping power and cost can be optimized for best design and long life of surface condenser. Matlab program is generated for different tube material with same corrugation profile and compared with the same. (25)

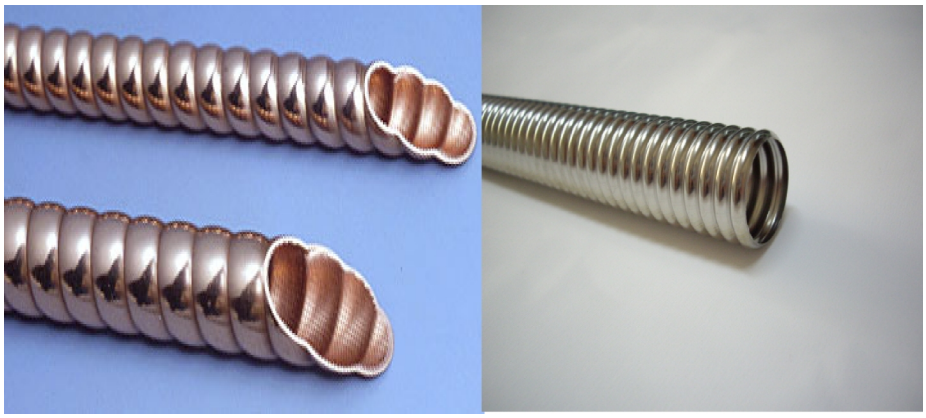


Fig. 13. Corrugated tubes of Cu:Ni and Stainless steel for surface condenser

- Using corrugated tube for any material the heat transfer rate should be increased up to double, but for heat exchanger application it is also required to check about pumping power also.
- Smaller pitch and highly corrugation increases pumping power very high which is not advisable. From engineering data book-III by Wolverine Tube Inc., there are two conditions is follow while using corrugated tube for condenser.
- MHT (Maximum heat transfer)
- LPD (Lower pressure drop)
- We can theoretically compare the friction factor for both Plain as well as Corrugated tube.

$$\text{Plain tube, } f = (1.58ln \quad - 3.28)^{-2}$$

$$\text{Corrugated tube, } \sqrt{\frac{2}{f}} = -2.46ln\left[r + \left(\frac{7}{R}\right)^{m}\right]$$

Due to corrugation, friction factor and turbulence increases. In the formula of corrugated tube the constant r and m is define as per the above two conditions as per data book-III by Wolverine Tube Inc.[25].

Table 16. Tube Parameters

Sr. No.	Constant	Condition for Corrugated tube	
		MHT	**LPD**
1	r	0.00595	0.0008
2	m	0.44	0.61

For this work i.e. condenser designing case, LPD (Lower pressure drop) condition is followed because to install corrugation tubes is one time investment, but pumping power is running cost which should be remain minimum. The Comparison of plain and corrugated titanium tube is given as follows:

Table 17. Comparison of plain Titanium tube and corrugated titanium tube

Sr. No.	Parameters based on d_o = 22.25 mm and t_w = 0.3 mm	Titanium	
		Plain tube	**Corrugated tube**
1	Overall HT coefficient (W/m^2K)	3333	3425
2	Area (m^2)	5189.3	5052
3	Length (m)	6.92	6.7
4	Pressure drop (kPa)	10.011	10.550
5	Pumping power (kW)	59.335	62.540

From above Comparison table it is notified that by using Titanium corrugated tubes as an adverse effect, pressure drop and pumping power slightly increases, but on other hand overall heat transfer coefficient and surface area decreases.

5. CONCLUSION:

Due to less cooling water flow rate inside the condenser, major variables are affected adversely and plant overall efficiency decreases. Due to this plant input increases and loss is approximated as Rs. 1,07,64,288 per annum.

To elucidate the problem marine growth should be cleaned and new circulating pump is to be installed with required discharge capacity with 2 year payback period.Nusselt theory is implemented to design the condenser major parameters thermally and for this Matlab codes has been made after validation with related literature with negligible variation.Major parameters like tube outer diameter, tube wall thickness, tube material and tube type is taken from TEMA standards and availability of material and manufacturing possibilities with reasonable cost. For this following major points are highlighted:

- **Tube material:**
  Among four present materials, according to this work titanium is most suited material for condenser tube working with sea water.
- **Tube outer diameter:**
  Among choices of diameter form TEMA, as per the tube material, 22.25 mm diameter value is most suited for 120 MW surface condenser.

- **Tube wall thickness:**
  Among choices of thickness form TEMA, as per the tube material, 0.3 mm thickness value is most advantageous.
- **Tube type:**
  Plain titanium tubes are easily manufactured by flowforming process with different diameter and thickness at reasonable cost. While on other hand corrugated titanium tube having some advantages compare to plain tube on overall heat transfer coefficient and exchanger area. But manufacturing cost of corrugated titanium tube is very costly. So, most suitable tube type for condenser is Plain titanium tube.

ACKNOWLEDGEMENT

The authors wish to thank Er. P. G. Modha (Deputy Engineer, GSECL-Sikka, Unit-II) for his great help to take permission for regular visits at GSECL-Sikka Plant and provide all necessary data with valuable guidance whenever required.

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Performance Optimization using FLS based Load Distribution Techniques in Cloud Computing

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ABSTRACT

As technology industry is growing, new computing ideas and concepts are to be needed. Very big amount of data is to be allowed to be store into particular storage. So that for such type of usage is to be done, more computing equipment should be available in the particular organization. By creating many virtualized models an organization can build more flexibility for the storage areas. Various algorithms are proposed and design in order to maintain standard and good resource provisioning, but due to certain problems all are not efficient. Flexible Load Sharing (FLS) algorithm creates clusters of resources and identifies and schedule task to respective resource. FLS is efficient in order to provide good QoS and by minimizing response time. Hence the performance of the system can be increased and we can utilize resource. These free resources can be used for other task and hence scalability can be increased.

SUMMARY

Flexible Load Sharing (FLS) is a Cloud Computing load distribution technique to optimize performance of system and to utilize cloud resources.

Keywords: Flexible Load Sharing (FLS), Performance Optimization, Load Distribution Technique, Cloud Computing

INTRODUCTION

Cloud computing is the most recent interesting technology. For the flexibility of the storage purpose the utilization of the hardware and load balancing information are to be shared in to the cloud computing technology (13). The users of the cloud services increased then at the same time the load balancing concept to be available and the utilization of the hardware are to be done. So that the load balancing is very important issue into the cloud computing. This type of traffic situation must be handled by the various load balancing techniques. To overcome this type of load balancing situation many researchers have been implemented the algorithms and various techniques for this. This paper shows the glimpse of the different algorithm techniques for the load balancing techniques. The ideas of each algorithm is discussed and finally summarized as an overview.

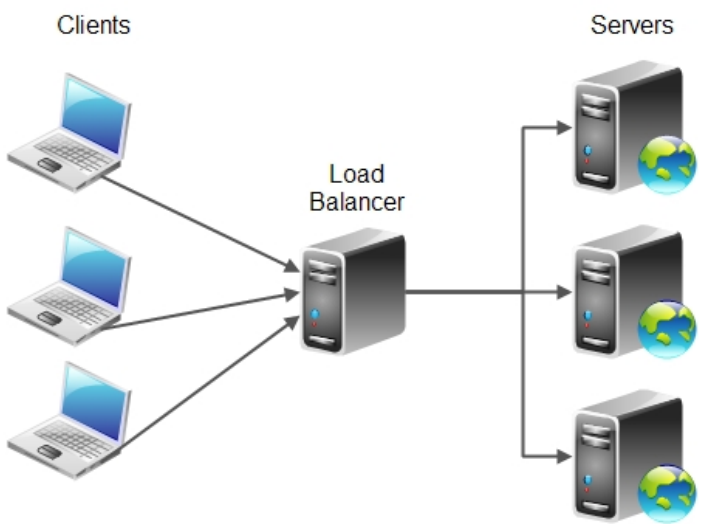


Fig. 1. Load Balancing Technique

ISSUE EFFECTING LOAD BALANCING

For the concept of the nodes distribution the performance is to be measured for the real time systems in to the cloud computing systems. The node distribution is to be done in to the form of the well balanced nodes in to the particular system organizations (4).

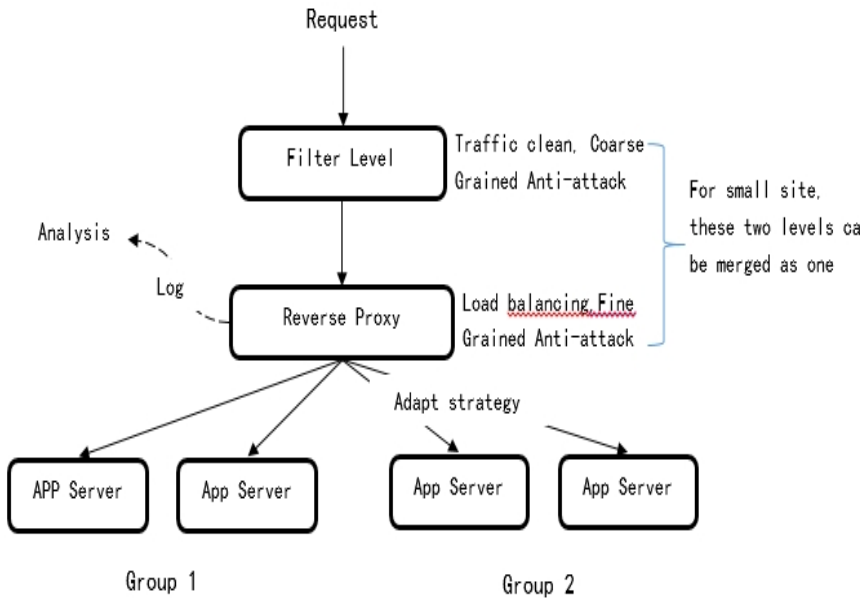


Fig. 2. Load Balancing Technique

Any load balancing algorithm will be having their complexity that may affect to the overall performance (Fig.2).If an algorithm is poor in complexity then it might down the overall performance of the systems. There for it might use for the system requirements and performance for the virtualization of the various hardware in to the systems.

SYSTEM ANALYSIS

This section describes the scheduling algorithms which can be used in CloudSim 3.0 for load balancing. Ke Liu, Hai Jin, Jun Chen, Xiao Liu, Dong Yuan, Yun Yang (2) presented a unique approach on compromised-time-cost in Compromised-Time-Cost Scheduling Algorithm. Scheduling algorithm have been concept of the balancing the various requests from the user to the server at the different time duration. CTC (compromised-time-cost)algorithm(2) can get lower cost compared to others during user-designated deadline meeting or decreasemean execution time compared to others within the user-designated execution cost. This system has been used for the presentation of the balancing the load situation and to analyse various cloud computing factors.Suraj Pandey, Linlin Wu, Siddeswara Mayura Guru, Rajkumar Buyya(3) presented a particle swarm optimization (PSO) based heuristic to this scheduling application are to be generated for the well balanced of the servers. It is also used for the cost effect generation and the analysis of the chart flow and optimization. The experimental results define that PSO can save cost and also provide efficientworkload distribution on resources. Mrs.S.Selvarani, Dr. G. Sudha Sadhasivam (1) have provided well mapped algorithm for the proper utilization and cost effect to the various virtualization users in Improved Cost-Based Algorithm for Task Scheduling. The improvement for the new taskscheduling strategy for cloud have been defined in Improved Cost-Based Algorithm (1).There will not be any overhead and well maintained. According to the priority based the tasks are to be well analysed and generating and handled the tasks as per the incoming priority

situations.Saeed Parsa and Reza Entezari-Maleki have proposed a task scheduling algorithm RASAResource-Aware-Scheduling algorithm(11). Two type of the scheduling algorithms are to be available: Max-min and Min-min. The requests are to be available for the particular servers. AS per the priority the requests are to be analysed .one by one and it will be proceed further for the new one. RASA covers the prose of Max-min and Min-min algorithms and overcome their cones. The requests migration time, throughput time, delay time, arrival time. For the distributed systems the RASA shows the overall performance for the distribution of the nodes. Yun Yang, Ke Liu, Jinjun Chen have proposed a scheduling algorithm "Innovative transaction intensive cost-constraint scheduling algorithm"(5) based on cost and time. Simulation results describe the efficiency by achieving lower cost than others during meeting the user designated deadline. Cui Lin, Shiyong Lu have proposed an SHEFT workflow scheduling algorithm to schedule a workflow elastically on a Cloud computing environment in Scalable Heterogeneous Earliest-Finish-Time Algorithm (SHEFT) (6). The experimental results conclude that SHEFT performs several workflow scheduling algorithms in optimizing workflow execution time and also enables resources to scale elastically at execution time. Meng Xu, Lizhen Cui, Haiyang Wang, Yanbing Bi have proposed Multiple QoS Constrained Scheduling Strategy of Multi-Workflows (MQMW)(7). Work load and work flow analysis of the distributed requests are to be well maintained. Such that the CloudSim (8) to be used for the well cloud simulation which can identify the real work system analysis.

EXECUTION OF TASKS IN CLOUDSIM

We don't have the benefits for the utilization of the cloud computing technology and their various techniques that may be used in to the load balancing purpose. Such that an efficient load balancing algorithms are to be required for the well balanced utilization of the loads on to the servers (12). There are various load balancing algorithms are available but that may be only utilized by their complexity and real time running application. According to the migration time and delayed in to the requests receive time those may be utilized. For the work load distribution the load migration algorithm may be affected in to the various challenges. For the virtualization and the migration purpose the various service oriented data centres are to be generated. Such that the system will be generating the any number of the requests and that will be analysed by the scheduling algorithm. Such type of the service oriented data centres are to be available for the scheduling algorithms and virtual machines to be migrated for the existing algorithms. So that the load balancing mechanism to be used for the completely service oriented data centres.

With earlier systems, it was not unable to be used the load balancing and to be analysed the scheduling system. Such that present environment is to be used for the various factors too be determine and the priority based work is to be done. For this paper we will be having many migration concept for the load balancing indicators and also the load balancing history values. The migration is going to be trigged and the virtualization is to be done (9). This approach avoids the problem of peak load count once the virtual machine is migrated. As a simulation scenario in CloudSim various parameters are considered. 1. CPU load indicator is measured as CPU, 2. Memory load indicator represented as Memory, 3. Bandwidth load indicator Band (10). We can identify that requests has come then according to the priority the requests are to be handled and the virtualization is to be done in good manner. We can also go for the nodes distribution concept such that virtualization is to be done in good manner and performance is to be increased such that memory size of the CPU and the through of the flexibility of the load sharing is to be increased well. The CPU will be having both the migration time as well as the memory resources such that in compare of the both memory resources can utilize the sensitive effect and the VM resource utilization. While increasing reserve CPU resource will increase total migration time. It occurs because with the increase of virtual machine memory size resultant into total migration time. Even the resource reservation in destination machine improve the efficiency of migration but it's important to avoid migration failure due inefficient resources at destination machine. Resource available at source machine

made high parallel migrations. During comparison with sequential migration, parallel migration get high efficiency in case source machine had enough and proper resources, otherwise parallel migration performed worst in case the source machine had no sufficient and proper resources. The workload-aware migration strategy can efficiently increase the migration performance. Numerous techniques have been invented and developed for migration as a whole virtual cluster. This approach also describes various challenges for live migration of virtual clusters like huge volume of data, Limitation of network bandwidth and Communication between VMs. Experimental results Experimental results conclude that Average migration time was sensitive to main memory, i.e., by increasing memory size there was considerable increase in total migration time. Downtime remained unaffected by change in memory size as downtime is affected by the rate of dirty pages and transferred pages.

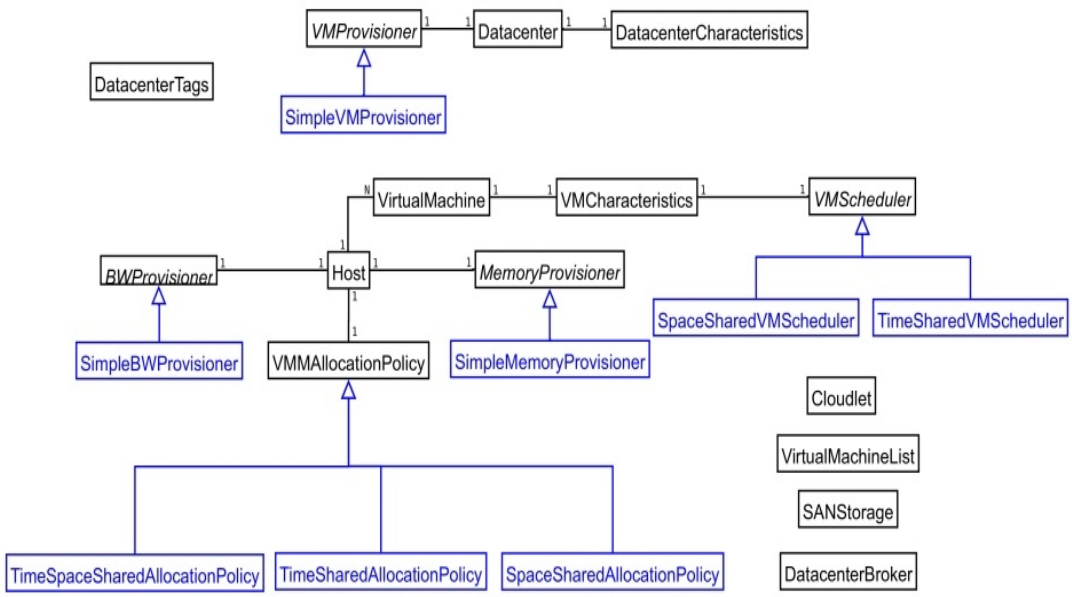


Fig. 3. Class Diagram for CloudSim Entities (14)

CONCLUSION

For load balancing and load maintenance in cloud computing, numerous algorithms have been created, implemented and simulated in CloudSim environment. Many researchers have focused and developed algorithm based on Virtual Machines (VMs), Number of Hosts and execution time or response time for load balancing in cloud computing. Aim of this paper's research work is to improve response time activity in CloudSim. Here in this approach, instead of sending requests on individual base, we first group the request into cluster based on their characteristics and required resources. Then secondly, map created cluster to subsequent VMs, but here if any tie or 2 VMs configurations are same then in that case this FLS based policy first choose nearest VMs or resource to execute tasks. FLS based approach normally works and executes clusters instead of individual requests, hence it will automatically improve response time and the required execution time will be much lower than traditional approaches. FLS based approach also reduce the usage of simultaneous resources so it can be used for other execution process and provide

efficient resource utilization in cloud environment. Add on cluster – VM mapping is based on configuration matching, hence always resultant in best efficient processing and maintains QoS for requested tasks.

This paper concludes that FLS is best to improve response time, provide QoS to the requested tasks and also better utilize resources in cloud computing. Ultimately, it optimize the performance of the system by utilizing existing resources.

ACKNOWLEDGEMENT

I am heartily thankful to my Head of the Department, Dr. Amit M. Lathigara, for his continuous motivation and support to always look for innovations and to carry out research work. I am also thankful to RK University for providing me great and precious infrastructure to carry out my research experiments. Last but not least, I appreciate the efforts provided by my colleagues, which helped me a lot to focus on my research experiments through their knowledge and experience sharing.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th& Jan. 6th, 2016)

(Proceedings available for download at rku.ac.in/icre)

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A survey on ETL strategy for Unstructured Data in Data Warehouse using Big Data Analytics

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ABSTRACT

In today's world the Digital Data generated is of 3 types, namely Structured, Semi-Structured and Unstructured. Standard techniques and tools are available to handle structured data. 90% of data generated in today's digital world is either semi-structured or unstructured. During the initial days of data analysis, users used to convert unstructured data into structured data and perform analysis. Nowadays people started developing tools and techniques to handle unstructured data. We can closely associate unstructured data with the term Big Data which refers to very large data sets that are difficult to analyze with traditional tools. Because unstructured data is typically large, dirty and noisy, it requires more computing power. I will improve ETL process on unstructured data in data warehousing using map reduce paradigm. The proposed method will process the data in parallel as small chunks in distributed clusters and aggregate all the data to obtain final processed data.

SUMMARY

Improving ETL process for unstructured data into data warehouse for Big Data Analytics.

Keywords*: HDFS, Map-reduce, Pig Latin, Hadoop, Data Cleansing, Profiling*

INTRODUCTION

Now a days bulk of data is being generated during use of internet. That data need to be managed properly so that we can use it further for more research purpose. Main challenge of big data is that some of that data is of no interest so it requires more filtering time. And also there is automatically generated data in some systems so it is difficult to manage and record it. We need to sort that data based on various requirements so that it can be reused again for analysis purpose.*(1)*

Digital data is of three type that are unstructured, semi-structured and structured. Among this unstructured data is the data that does not follow any data model, it means that it has no structure or little structure and also it is difficult to extract information from this type of data and store that into data warehouse. Unstructured data is generated everywhere. For example online forms, word document, PowerPoint presentations, images, videos, company records and all social media is generating unstructured data. Semi-structured data is also having no data model but have some kind of structure. For example emails, zipped files, HR records and XML data is semi-structured data. Structured data is totally organized kind of data, this type of data is used directly for business analytic.*(2)* Table 1 shows the comparative study of all the 3 types of digital data.

Big data analytics is used to get space for storing and processing large data sets. It is also used to work with distributed stored data sets with faster processing. Data warehouse is used for storing data for further data analysis. The data in the warehouse is read-only and the update or refresh occurs only on a periodical basis.*(3)*It has main 4 characteristics namely Subject-oriented, integrated, non-volatile and time variant. Subject oriented means data is collected according to subjects. Integrated means that the data must be consistent. Time variant says that generally historical data is taken into account for the data warehouse applications. So they allow access to more detailed information, as required. Non-volatile states that data warehouses are static.*(4)*

In our daily usage about 80-90% of the data is unstructured data and it cannot be directly processed so we need to convert unstructured data to structured data for business analysis. To deal with data from various sources ETL process is used.*(4)* ETL is a 3 stage process namely Extract, Transform and Load that allows integration and analysis of data stored in different sources.*(5)* As shown in figure 1, Extraction phase includes collecting data from multiple sources of database. This process is used to build data warehouse. Then next is the transformation phase in which the data is reformatted and cleansed to detect and rectify errors to meet the information needs as all the data from various sources are in different format and then loading phase will do sorting and load that data into the final target database.*(6)* There are many ETL tools available to manage data warehouse with some capabilities and advantages.

As data grows exponentially, we require ETL to work with high scalability so we use map reduce to perform distributed computing. Map reduce framework is having two phases : map phase will split all the data and subdivide into small parts and process all parts in parallel. Then reduce phase will shuffle data and sort according to the requirements. The output of this phase will be stored into HDFS file. As shown in figure 2, Map reduce framework uses JobTracker and TaskTracker to complete this task. Map reduce is used for searching, indexing and tokenization. It is used due it's feature of scalability in distributed environment.*(7)*

HDFS is a Java based file system used to store data and access scalable and fault tolerant data. HDFS is data management layer of Hadoop. It is a distributed file system used to store bulk of data. HDFS uses

NameNode and DataNode to store metadata and application data accordingly. Clients directly contact to NameNode and then it is directed to appropriate DataNode to read required content.*(8)*

RELATED STUDY

A research work says that generated volume of data is increasing day by day. Among these 90% of the data is unstructured and need to be managed properly for our required needs. For huge data older systems are not sufficient. So we use map-reduce to process data in key-value pair with Hadoop. With the concern of big data, the three main challenges being faced are volume, velocity and variety. It is very difficult to perform the operation on unstructured data. So the unstructured data is structured and processed by using Map-Reduce technique and collaborative filtering is used to generate recommendations based on user preferences. The sentiment analysis technique is used to analyze the sentiments of a user based on Text Analysis. The resulting data set is structured with a particular order according to the user requirements.*(3)*

Data Warehouse is different from operational database for handling large amount of data. To deal with this data we have ETL process with some framework. ETL process will map data from different data sources and load into data warehouse. Briefly ETL process will extract data from various sources, transform it, clean it and then load into data warehouse or data mart. But due to its difficulties and lack of formal model we propose new ETL model with some advance features that represent all activities and understood by Data Warehouse designer in all environment.*(4)*

ETL process is used to extract the data from multiple sources then transform it to fit your analytical needs, and load it into a data warehouse for further analysis. Apache Hadoop has been used as the primary standard for managing big data. When the source data sets are large, fast and unstructured, traditional ETL can become the bottleneck, because ETL becomes too complex to develop, too expensive to operate, and also it takes too long to execute. According to a study, 80% of the development effort in a big data project goes into data integration whereas only 20% goes towards data analysis.*(7)*

ETL is responsible for the extraction of the data, its cleaning and then loading it into the the desired target. Building ETL processes is expensive regarding time,money and the effort taken into account. ETL tools extract data from several sources such as database tables, flat files, ERP, internet, etc. and then apply complex transformation to them. Finally in the end, the data is loaded into the target database. These target databases are either fact tables or dimension tables in the context of the Data Warehouse. There are 2 types of ETL tools available. On one side, there is a collection of payable ETL which includes Data Stage and Information while on the other side, there is a collection of commercial ETL which are available free of cost.*(10)*

Hadoop is provided by Apache foundation which almost satisfies most of the goals of Big Data Analytics and supports HDFS (Hadoop File System) as its file system. Using map reduce paradigm it deals with the large data sets. The components of Hadoop like HDFS, Pig, Hive all are available from apache foundation as open source license. On a dedicated server meta data can be stored in the HDFS and termed as NameNode and the data regarding application can be stored to other servers which are known as DataNode. Other than NameNode and DataNode there is a CheckpointNode and BackupNode which are used at the time of performing operations on files. HDFS can be accessed by user application using HDFSclient. Supported operations by HDFS are namely reading file, writing file, deleting file and updating file. For operations to be performed HDFS uses the pipeline which connect the end nodes. For balancing the clusters balancer is used which works with predefined threshold input. Blocks with

uniqueID are there in HDFS which are allocated by NameNode which specifies the DataNode list that replicates the blocks. Blocks are placed in such manner so that efficient utilization of bandwidth can be done. Blocks scanning is performed by the block scanner. Losing data is very low if we uses the HDFS as it replicates the same blocks of data for three times and stores it at different storage backup.*(11)*

RESEARCH OBJECTIVE

Nowadays more amount of data is being generated with use of technologies, it is getting complex to handle such a large amount of data with RDBMS. So we are using DW for storing data and this is mostly unstructured, so for a specific user it is time consuming to get required data from data warehouse. ETL process is used along with map reduce and HDFS. Using this we can process data in parallel and fast manner for further business analysis.

PROPOSED MODEL

With more use of internet and services, more amount of log data is generated and it is very complex to handle that data. So we perform ETL process on that data. As shown in figure 3, Extract phase is performed to do data cleansing and profiling. Then transform phase is done with Map-Reduce so that data will be partitioned and processed parallel as well as fast. Node failure can not affect the executing task. Result of transform phase will be stored in HDFS. For further processing Pig Latin is used for querying data, output will be loaded in Data Warehouse for Business Analysis.

CONCLUSION

The research work discussed focuses on the problems of handling large volume of data for Big Data Analytics. The proposed model helps us in overcoming the overhead of converting unstructured data into structured data. In the proposed model, ETL process is performed with HDFS and Map Reduce. Map Reduce being parallel programming model optimizes the performance of ETL process. We run the Pig Latin scripts on the result data and perform the required Big Data Analytics. The proposed model is the need of the hour for various data intensive organizations.

FIGURES

Figure 1. Three phases of ETL process[7]

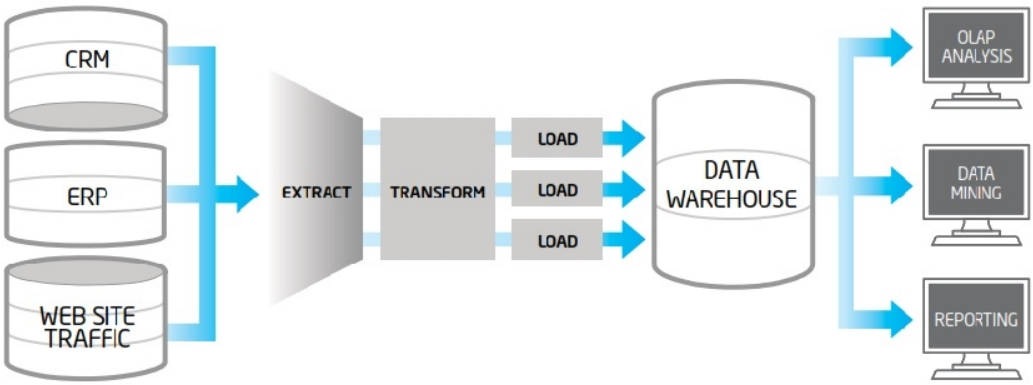


Figure 2 . Map Reduce procedure

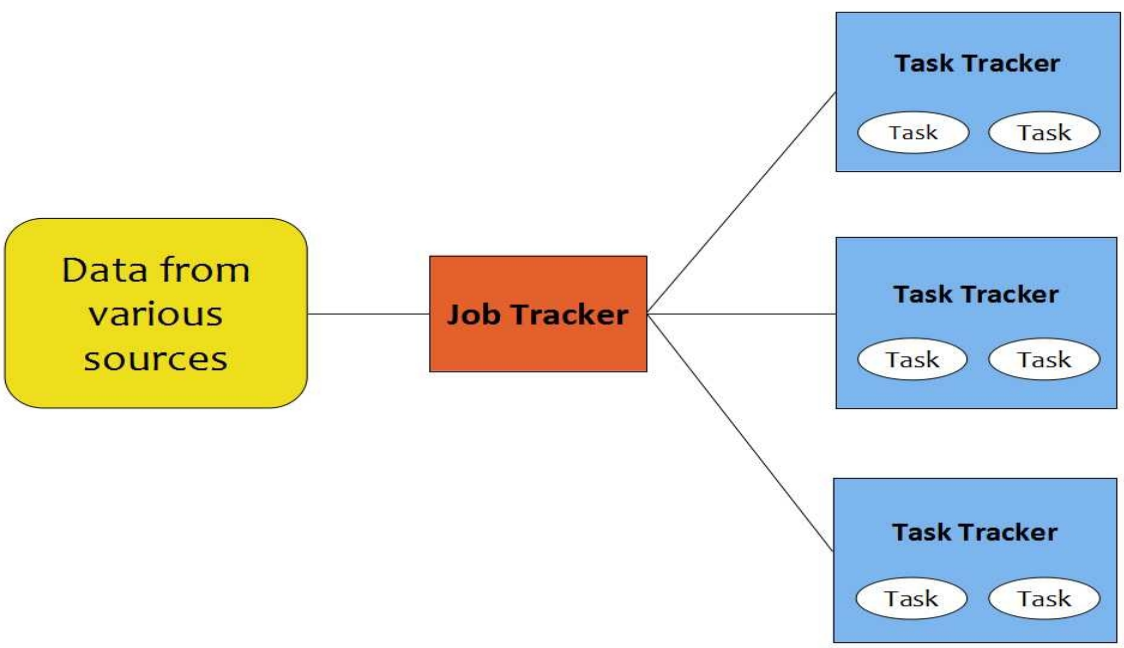
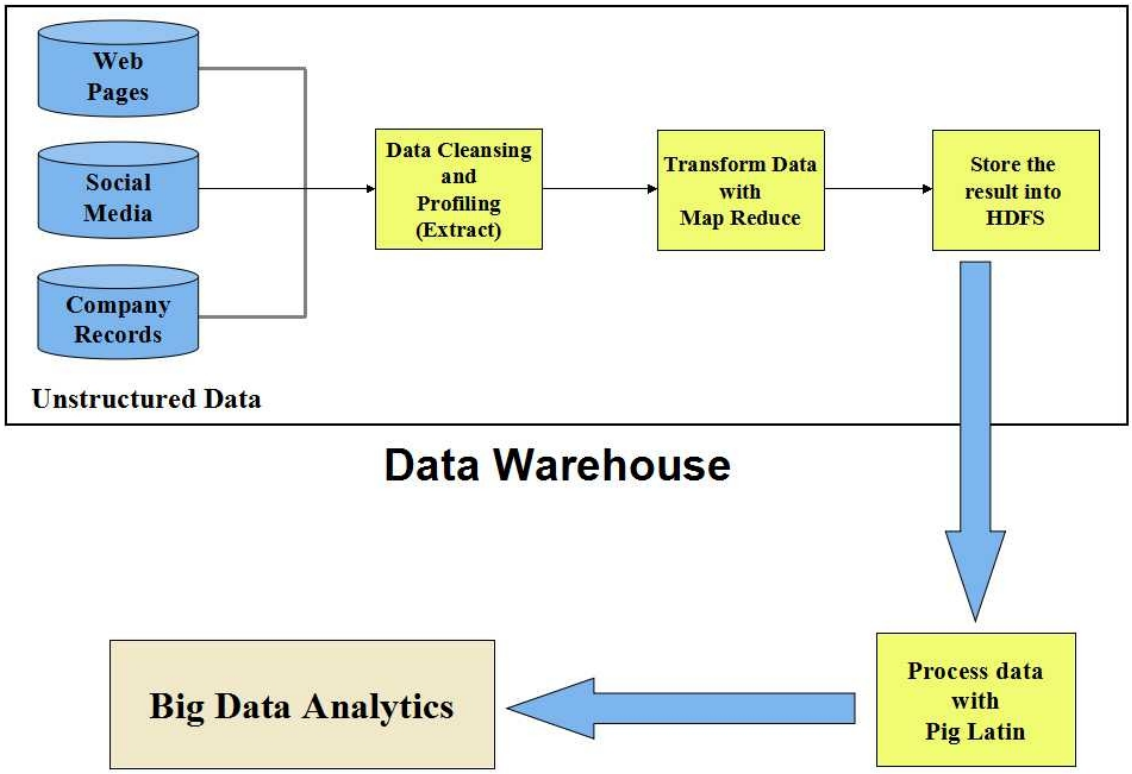


Figure 3. Proposed System


Web Pages
Social Media
Company Records
Unstructured Data
Data Cleansing and Profiling (Extract)
Transform Data with Map Reduce
Store the result into HDFS
Data Warehouse
Process data with Pig Latin
Big Data Analytics

TABLES

Table 1: Comparative study of digital data

	STUCTURED DATA	**SEMI STRUCTURED DATA**	**UNSTRUCUTRED DATA**
CHARECTERISTICS	- Data is stored in the form of rows and columns - Conforms to a data model - Attributes in a group are the same	- Does not conform to any data model but contains tags and elements (metadata) - Attributes in a group may not be the same - Similar entities are grouped	- Not in any particular format or sequence - Does not conform to any data model - Not easily usable by a program - Does not follow any rules or semantics
SOURCES	- Databases - Spreadsheets - SQL - OLTP systems, etc.	- E-mail - XML - Zipped files - Mark-up languages, etc.	- Web pages - PowerPoint presentations - Videos, Images - Reports - Surveys, etc.
CHALLENGES FACED	- Limited storage - Contains only homogeneous data	- Storage cost - Limited tools available - No ready tool available for querying. - Data heterogeneity.	- Indexing and searching - Security (varied sources of data) - Retrieve information - Lack of technical expertise

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th& Jan. 6th, 2016)

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RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

A Proposal of Resource Allocation Scheme based on Auction for Green Cloud Computing

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ABSTRACT

Cloud computing is internet based computing, which provides on demand services to the end users. IaaS (Infrastructure as a Service) is one of the cloud service among the different services. In that client gets VM (Virtual Machine) as a host for different computation purpose. VM allocation is toughest task for service provider for proper utilization of resource.Resource allocation task consume time as well as huge amount of energy. For time optimization, virtual machine organizer model which schedule data centre request based Quality of Service (QOS). To reduce energy consumption there are three proposed auction based algorithms. For the optimal solution the proposed algorithm is Exhaustive Search Algorithm (ESA), for the NP-hard problem Linear Relaxation based Randomized Algorithm (LRRA) and for reducing energy consumption of reserved resources there is Green Greedy Algorithm (GGA) in green cloud computing. This paper gives three auction based algorithms and comparative study between that.

Keywords: cloud computing, time optimization, auction, resource allocation, green computing

I. INTRODUCTION

Cloud computing is set of network enable services, which provides scalable, on demand, inexpensive computing infrastructure which could be easily accessed in simple way and provide guaranteed Quality of Service (QoS). Cloud computing is nothing but practice of using a network remote servers hosted on internet to store, manage and process data rather than local server or a personal computer.

In Cloud computing, Information Technology (IT) resources as services offered by data centre, software systems such as operating system, management software, different applications and hardware system such as server, data center network systems, storage systems etc. provides Platform as Service (PaaS) and Infrastructure as a Service (IaaS) respectively*(1)*.Applications, such as social networking, web search, computation etc. are hosted as Software as a Service (SaaS). These applications run on virtualized resources namely Virtual Machines (VMs), based on user request cloud service provider allocates VM to the request. These service provided by PaaS and IaaS *(2)*. According to need of request by user different types of VMs allocated.

Characteristics

1. **On-demand self-service**: Without any human interaction, user can provision cloud services.
2. **Broad network access**: Resources can be accessible over network, it supports different client platforms like mobile, tablet etc.
3. **Resource pooling**: Serve the multiple users from same physical resources with securely differentiation of resource on logical level.
4. **Rapid elasticity**: Resource are provisioned and released on-demand as per requirement of customer.
5. **Measured service**: Data resource are monitored and measured and billed as per utilization*(5)*.

Cloud computing can be separated into two distinct model, *(1)* first one is Deployment models and second is Service models as per shown in Fig. 1. The U.S. National Institute of Standards and Technology (NIST) has defined as follow four deployment models (I) Public cloud (II) Private cloud (III) Hybrid cloud (IV) Community cloud and mainly three service model (I) Infrastructure as a Service (II) Platform as a Service (III) Software as a Service.

Deployment Models

Purpose of cloud and nature of how cloud is located can be define by deployment model.

1. **Public Cloud:**
   In this cloud service model, the service provider offers different services like data storage, memory power, processing power, application hosting to the regular user. The infrastructure, which is available for public use alternatively for large scale industry

group is owned by an organization sell or provide cloud services. Simplest model to setup is public cloud.

2. **Private Cloud:**
   The cloud infrastructure is opened for exclusive use of an organization. It can be managed by organization or a third party. This cloud network is created for the private organization's purpose. The main purposes behind developing private cloud is security concerns that the data of any private organization should accessed in that network.

3. **Hybrid Cloud:**
   The combination of multiple clouds like public cloud, private cloud, community of public cloud where these clouds stand with their unique identities are bound together as a one source. The advantages and features of private cloud security concern and public cloud like elasticity of on demand unlimited service and flexibility are included in hybrid cloud.

4. **Community Cloud:**
   The community cloud one which has been organized and managed by organization according to the purpose or function of cloud user. The best example of community cloud is government agencies which are connected and they have shared all credentials information in privately. This type of cloud are basically developed for fire departments, government agencies, community swimming pools etc.

Service Model

Different cloud services are associated with different cloud deployment models based on their purpose*(1)*.

1. **Infrastructure as a Service:**
   In this cloud service model, it provides virtualized computing resource over the internet, like virtual machines, virtual storage, virtual infrastructure etc. The client is responsible for different aspects of deployment like operating system, applications, user interaction with the system while all infrastructure is managed by IaaS service provider. All these service vendor offer direct access to the hardware resource. Examples of IaaS provider are Amazon web services, at&t, bluelock, ca technologies, cloudscaling etc.

2. **Platform as a Service:**
   Different applications are serviced as platform to deploy different applications on cloud infrastructure. PaaS provider enables software. PaaS is cloud computing platform that allows creation of application without maintaining them and purchasing software or applications. Amazon web services, Appistry, AppScale, ca technologies, EngineYardare few examples of the PaaS providers.

3. **Software as a Service:**
   Here application or software are being hosted over virtual infrastructure, software is licensed on the subscription basis to the user. It is software distribution model. Examples of SaaS provider are abiquo, accelops, Akamai, Appdynamics, apprenda etc.

Cloud Challenges

Dynamic workload variations
Workflow scheduling
Resource availability
Optimizing resource utilization

II. OVERVIEW OF RESOURCE ALLOCATION

Nowadays, number of users are increased for the different cloud on demand service like data processing, memory power, data storage because of cheaper and availability of cloud technology in availability of internet *(2)*. Cloud technologies create an environment which provide a platform to business due to high capability of data processing and manage work load. In last few years such technologies like grid computing, virtualization, and service oriented architecture have given huge contribution to cloud environment.
Resource allocation is an important part of cloud computing, allocated resource need to be utilized maximum. Different users have different type of requirement based on quality of service. From service provider's point of view, resources utilization should be maximum and energy consumption should be minimum. To schedule and maximize utilization of resources, proper resource allocation techniques required. If user is aware of required energy for their task it can be helpful for service provider in reducing energy consumption by resource. In resource allocation problem, an auction based resource allocation technique can be addressed best.
In the auction based allocation, user bid for the resource as per their demand (i.e. the number and type of instance) and maximum price they are willing to pay. There are many economic model proposed. Though most of algorithm focus on maximizing service provider's revenue. Researchers have done a lot for reducing energy consumption. Users are not aware of how much amount of energy consumed by their resource which is reserved for their application. User should know the energy required for their request, so it can be help to reduce energy consumed by the resource which is in ideal state.

There is mainly two type of pricing model (*8)*, first is fixed price model and another auction based approach. Here the fixed price model has several drawbacks like it is not economical efficient next is fixed price model may not give the satisfaction price based on market need. Inabilities of fixed price model resource allocation it can be remove using auction based technique. Among the different type of auction, one of the auction based technique is combinatorial auction *(7)*.
In the combinatorial action mechanism the bidder participating for the bundle off resource rather than a single instance. This technique is useful when bids are complimentary to each other bidder *(7)*. The example of this technique, service providers offer small and big resource instance. It is

beneficial that resource provision to user in the form of group of resource. It is useful for user that, it removes the process of requesting for instance separately for each.

III. RELATED WORK

In the past, many auction based mechanism has been proposed *(6-9)*. Double auction mechanism for grid computing by J. Gomoluch and M. Schroeder *(6)*. Combinatorial auction model applied for single cloud resource provideri.e. single instance of virtual machine. Based on price value combinatorial auction proposed by Das and Grosu *(10).* Greedy scheduling method using time constraints by Dong, Liu and Rojas-Cessa *(14).*
Considering the energy parameter in auction proposed in *(11)*. With the aim of minimizing energy consumption by data center framework was proposed to allocation and reallocation of resource *(10)*.

Algorithms for winner determination

For the determining the winner of auction there is three algorithm *(8)*. (I). Exhaustive Search Algorithm (ESA), (II). Linear Relaxation based Randomized Algorithm (LRRA), (III). Green Greedy Algorithm (GGA).

I. Exhaustive Search Algorithm (ESA)

This algorithm is also known as Exst-*k* search where *k* is the number of bids. This algorithm used for determining the optimal solution for each binary string that available solution is feasible or not. If the solution is feasible then the value of objective is calculated and compared with previous best optimal solution. The size binary string is n, and possible size of solution of binary string 2^n.A for loop from 1 to $2^n - 1$. The string containing zero element is not need to be consider.

The second part payment scheme, total bid value can be obtained sum of all bid value minus the bid, which is bidder has not participated. The author of *(12)* has proved for combinatorial action ESA is optimized algorithm.

II. Linear Relaxation based Randomized Algorithm (LRRA)

Linear Relaxation based solving method is widely accepted because of simplicity, to solve NP-hard problem*(13).* This algorithm is used for fractional allocation and rounding the resulting allocation randomly *(15)*. The chance of winning for user can be increase by bid increasing the bid value or requesting the less number of resource this algorithm sort the bid value in decreasing order of winning value.

III. Green Greedy Algorithm(GGA)

This algorithm work in two steps. First is ordering step and second decision step. In the ordering step bids are sorted in descending order on pre-defined criterion. This criterion is calculated by algorithm as ratio of user's bid value and total cost of energy consumption. In the decision step first bid is entered in empty set of winner and then it is verified that given solution is feasible or not. If it is feasible then next bid continue the

solution set. If it is not feasible then it removes recently added bid and continue with the next bid in ordering. The solution is called feasible if it satisfied the problem constrains.

IV. PROPOSED METHODOLOGY

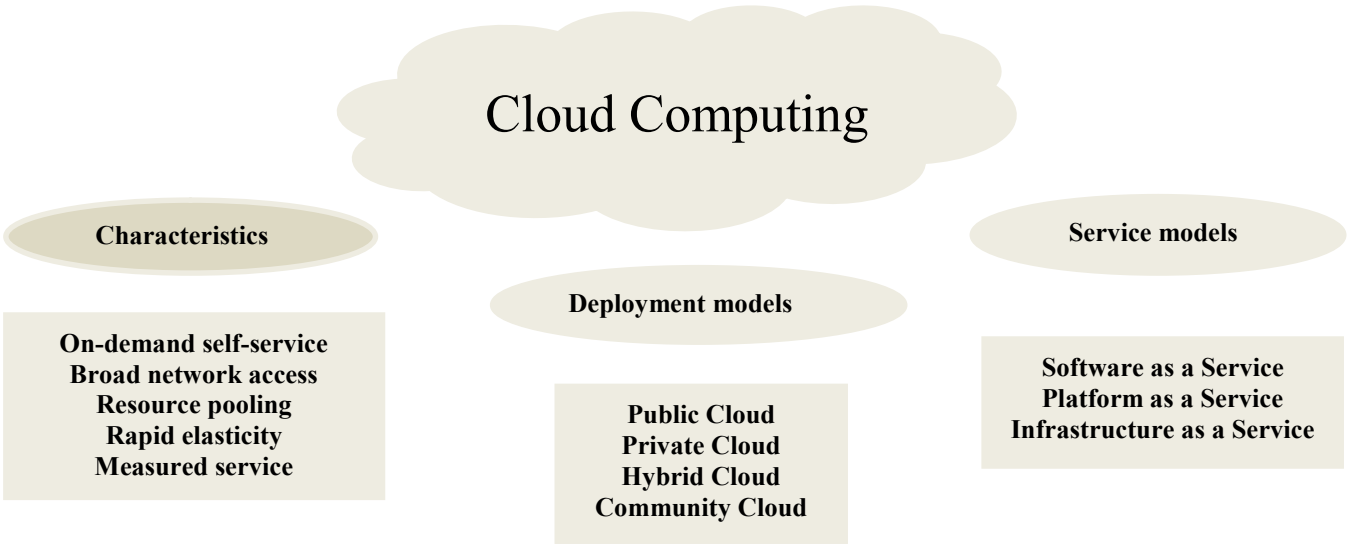
Different algorithm has different advantages based on their characteristic in combinatorial auction. Overall the performance of ESA algorithm is good but for green cloud computing energy consumption should be minimum and high performance needed. To achieve that, modification can apply in GGA so we can get high performance.

V. CONCLUSION

In this paper, three monotone and truthful algorithm are discussed to determine the bid winner and to compute payment for winner. As show in table 1, we can say that Green Greedy Algorithm has advantage over other algorithm and GGA has higher revenue of energy i.e.energy consumption is less, for the task bundle resource winner determinination. For any cloud system GGA is suggested for cloud resource allocation with arbitrary number of bidder.

VI. FIGURES

Fig. 1 shows the cloud computing characteristics, deployment model and service model.



VII. TABLES

Table 1. Comparison of algorithm on different parameter

Parameter	ESA	LRRA	GGA
Economical view	Good	Worst	Best
Gross revenue	Less	Medium	High
Net revenue	Low	Medium	High
Energy cost	High	Medium	Low

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Initial Sample Study on Effect of Grinding Process on Health of Operator using Pulmonary Function Test & Pulse Oximeter

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ABSTRACT

Grinding process accomplishes the finished surface through material removal in the form of powder, which do enter into the lungs of human during inhalation. Health problems is expected to be reported amongst the worker exposed to grinding process together with occurrence of respiratory, digestive and skin disease. The aim of this study is to report evidence of exposure to grinding process, adverse effect on blood oxygen saturation and respiratory function. The study group consisted of 3 healthy subjects aged between 29 to 50 years group from a random group as precursor to advance study. A significant decrease in Peak Expiratory Flow Rate (PEFR) is observed who have habits of tobacco chewing, smoking and drinking and their parents having similar habits in exposed than non-exposed group. This method is to set initial benchmark study in terms of using two equipment correctly and indicating understanding with outcomes.

SUMMARY

Pulmonary function tests are used as assessment tool on subjects with suspected respiratory disease and pulse oximeter helps to identify the percentage of oxygen saturation in blood of the subjects.

Keywords: Grinding Process, , Health Problem, Pulmonary Function Test, Pulse Oximeter

INTRODUCTION

Grinding is a surface generation process used to shape and finish the components made of metals and other materials in 10 mm zone with multipoint grinding wheel. The precise surface finish can be achieved by grinding process, which is far better than the turning and milling process. (*1*) Metal working fluids are used in grinding process to keep the surface of workpiece and tool cool, reduce the friction between the workpiece and tool, and advance the surface integrity of workpiece. Increase the tool life as well as productivity. Health problem are reported among the workers exposed to metalworking fluids, like incidence of respiratory, digestive, skin cancers and increase the rates of cough and phlegm. (*2*) The health issues are also associated with the cutting fluid mist for wet and dry grinding as reported by Adler, Hii, Michalek and Sutherland. (*3*) Ultimately, the fluid type and composition has a major impact on the

grinding process and therefore on the overall technological, environmental and economic impact of the product. (*4*)

Lung Capacity: Usually human takes breathe 12-20 times / minute. Human lung expand and contract supplying oxygen to the human body and removing waste product carbon dioxide from it. (*5*) The respiratory system defense system help prevent airborne particles from getting into the lungs and causing damage. When somebody inhales, the air is drawn in through the nose or mouth into the upper respiratory system, which consists of the nasal passage, trachea and conducting airways. The air becomes moist and makes various twists & turns through the nasal passages and branching airways. Particles 100 μm or larger are not typically drawn in the body by inhalation because of their size. Particles in the size of range of 10-100 μm are unable to make the turn and impact on the nasal hairs, nasal mucosa. Soluble particles simply dissolve, while insoluble particles are transported up the conducting airways by the ciliated epithelium and swallowed. Even smaller particles less than 10 μm in size are normally capable to pass through into the pulmonary part of the lungs, where gas exchange, respiration occurs. Thus, particles that get in touch with this part of the lungs are called respirable particles and if dumped, are in general eliminated by particle eating cells called macrophages. (*6*) Considering grinding process particles comes under this range of the case for report in this paper.

MATERIALS

The current study is performed on subjects belonging to different ages working in academics and Industries. This is a cross-sectional study, which is performed in machining as well as grinding department. Study is conducted in accordance with ethical concerns of the organization and the permission of all the participants are taken aforementioned to the study. Ahead of beginning with the method, the subjects were chosen on the basis of random sampling by applying inclusion criteria, elucidated the entire method in detail.

The inclusion criteria included smoker and non-smoker, tobacco users and nonusers, working exposure and no exposure of the process.

The subjects were assessed using tools as following:

1. Spirometer (Pulmonary Function Test): Spirometry test is practical examination of the lungs' function. Spiromentry help identify and supervise airways decline in samples with indications, risk factors or suspicion of airway syndrome. Spiromentry precisely determine Forced Expiratory (FE) in 1 s, Forced Vital Capacity (FVC) in 6 s, and it should be accounted both as the fixed measurement and as a fraction of normative data. The contour of the flow volume loop gives supplementary information with regard to the locality of obstruction. Pulmonary function tests are cluster of examinations that determines lungs capacity, strength in terms of air exchange.(*7*)
2. Weighing Machine: A weighing machine with 0-150 kg is used to measure the weight of the samples.
3. Measuring tape: A 5 m ± 1mm measuring tape is used for anthropometric measurement of the height of the subjects.
4. Pulse oximeter (S_PO_2): Pulse oximetry permits non insidious examining of samples during anesthesia. Blood includes hemoglobin in together it's oxygenated and deoxygenated state. Oxygen saturation is described as the proportion of oxygenated hemoglobin to the total absorption of hemoglobin present in the blood. Arterial oxygen saturation is determined non insidious using a pulse oximeter. The pulse oximeter uses two technologies pulse plethysmography to identify the pulse waveform and spectrometry to notice oxygen saturation. (*8*) This helps identify conclusively respiratory blockage if any on lung circulating & respiratory system.

The measurements are made to identifying as under:

VC: Vital capacity is the maximum volume of the air which can be exhaled or inspired during either a maximally forced (FVC) or a slow (VC) manoeuvre. VC is normally equal to FVC unless airflow obstruction is present, in which case VC is usually higher than FVC.

FEV1: Forced expired volume in 1 s is the volume expired in the first second of maximal expiration after a maximal inspiration and is a useful measure of how quickly full lungs can be emptied.

FEV1 / FVC: Expressed as the percentage of the VC or FVC (whichever volume is larger) and gives a clinically useful index of airflow limitation.
PEFR: Peak expiratory flow rate is the maximal expiratory flow rate achieved and this occurs very early in the forced expiratory manoeuvre. (*9*)

METHODS

According the subjects were called and a demonstration made to the Spirometry procedure. It requires sanitize mouthpiece to be held in the mouth and normal inhalation exhalation to be done with mouth to get used to the process of exhalation. Then they were asked to make forced blow through exhalation marking PEFR in at least three trials. Thereafter they were asked to make inhalation marking slow manoeuvre.
For the Pulmonary function test at least three technically acceptable manoeuvres are taken from which the best manoeuvre is been considered for the final result of each subjects.
All individual examination is acceptable if it meets the following acceptability criteria:
The subjects followed directions; a continuous maximal expiratory manoeuvres during the examination was attained and was established from full inspiration; No indication of uncertainty at some stage in the examination; Examination executed with rapid start; PEFR has sharp increase; No premature termination; No leaks; No cough; No obstruction of the mouthpiece of the instrument.

Description of Samples:

Table 1 records the detail of physical parameters of the 3 subjects, chosen from a set of subject in this report to bring out difference and sense the response using selected probe. According to measurement subject 1 obese, subject 2 is overweight and subject 3 is normal weight.
Table 2 summarizes the details of the habit of the three subjects giving with precedence. It also records the grinding and machining exposure of three subjects.
Table 3 summarizes the detail of pulmonary function test parameters predicted and actually achieved by the RMS Helios pulmonary function test device for the same 3 subjects. Subject 1 is an academician having no industrial exposure while subject 2 is working as laboratory instructor in academic engineering workshop, also works part time in industry in the grinding shop from last 25 years. Subject 3 is working as laboratory instructor in academic, also works part time in machine shop from last 30 years. Different background of each subjects help to identify the effect of process on health in this study.
Table 4 summarizes the detail of pulse oximetry in three samples by using S curve Fingertip Pulse Oximeter.
Fig. 3 compares the contour of flow loop of three healthy samples from which one can determine the problem in airways or lungs of the sample.

RESULTS AND DISCUSSION

During the performance of the examination following subject related problems found:
Submaximal efforts, Leak between leaps and mouthpiece of the instrument, Incomplete inspiration or exhalation, Uncertainty at initial stage of expiration during the examination, Cough, Tongue closure, Obstacle of the mouthpiece by the tongue, Poor posture.
It is also noted that all effort of measurements will be variable in samples that are not cooperative.
Fig. 3 suggests that Subject 1 having normal loop, but may have restrictive lung disease which may be seasonal or due to early exhalation. Subject 2 is having noticeable airways obstructions, as is in very severe stage as his FEV1 / FVC $< 70\%$ and $0\% \leq$ FEV1 $\leq 30\%$. It is hereby suggested medical supervision to get cure. Subject 3 is having normal loop, which suggests to have no obstructive or restrictive lung disorder. Still this result may differ for change of instrument or by the proper practice the result may improve as it is preliminary study.
This examination result one cannot indicate that the obstructive or restrictive lung disorder is the adverse effect of the grinding process because subject 2 is also having the habit of chewing tobacco as well as he was ex-smoker even his blood relation having the similar habits. On the other hand subject 1 and 3 having

no habit of tobacco chewing or smoking so their lungs working is proper. Even the value of PEF admits the same, which lying near to normal values of PEF of same group of ages. Subject 3 shows the effect of age over the lung.
From the pulse oximetry no indication of circulatory disorder process is noticed. So, to identify the exact adverse effect of the grinding process on human body requires observation of examination on more numbers of subjects. But this investigate probe helped to identify the subject susceptibility to environment and awareness about the working environment. This also helped identify development of safeguard in working practice.

CONCLUSION

This study predicts the effect of grinding process on human body with comparison of exposed and non-exposed subjects. It also helps identify that the pulmonary function test and pulse oximeter could be used as diagnostic tool for the respiratory disorder. Where in pulmonary function indicator is better indicator than pulse oximeter. Nevertheless the pulse oximeter is not suggested to be dropped as with subject having severe indication of respiratory disorder will have overlapping effect on circulatory system. The reverse of this also may also be true that subject having circulatory disorder history may have overlapping history of respiratory disorder too. But still one cannot precisely determine that adverse effect is the result of grinding process as the subject having the habit of tobacco chewing and smoking, also predicts the effect of age over lungs. So, to get the exact effect more number of subjects requires to be examined.

FIGURES

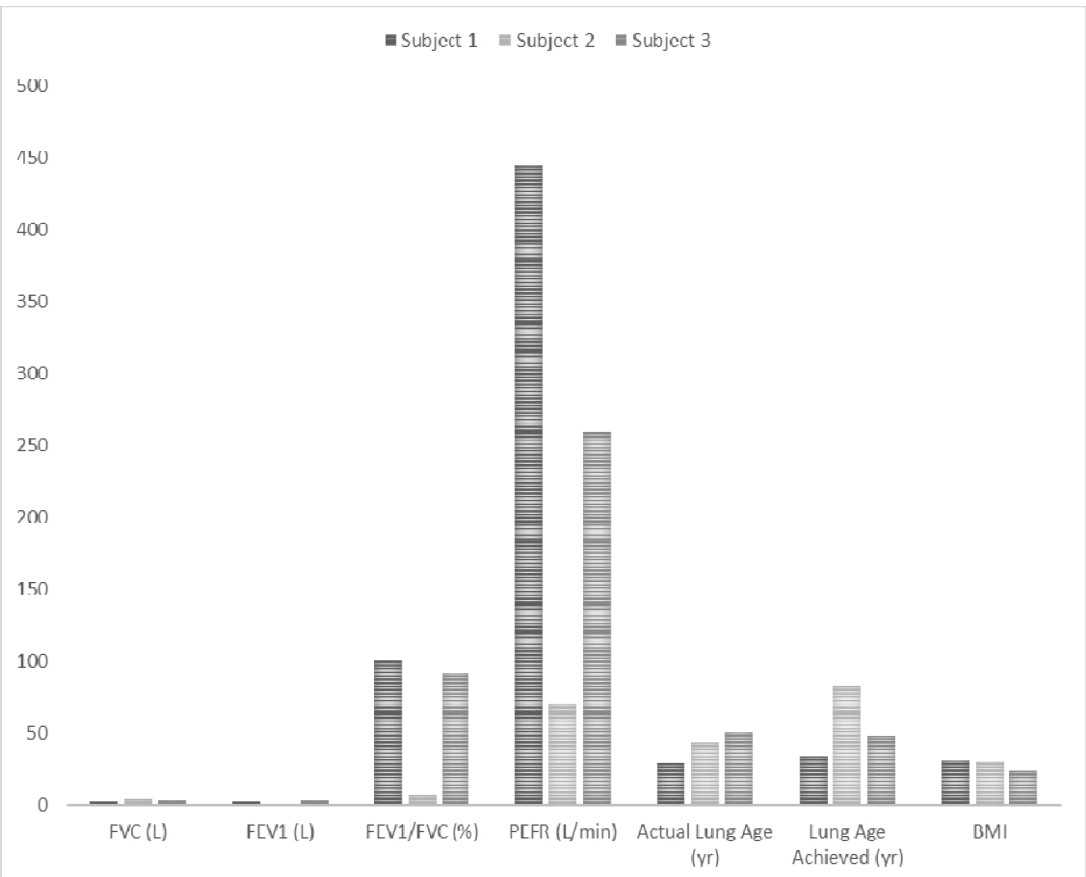


Fig. 1. Comparison of measurement and pulmonary function parameters

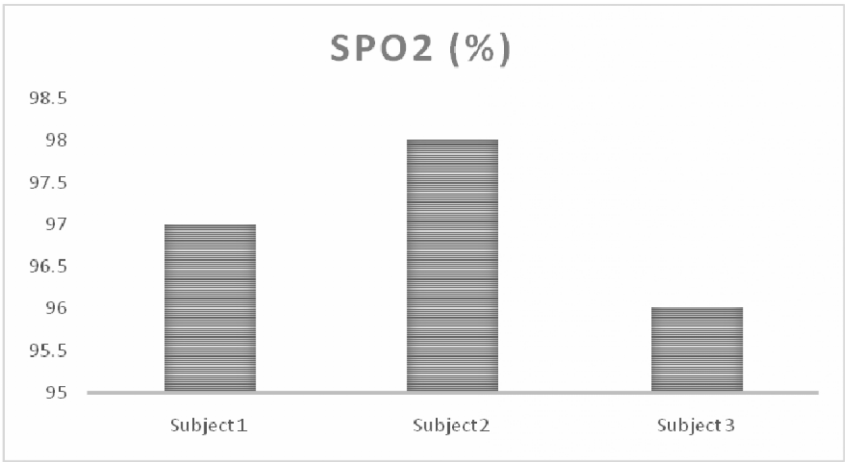
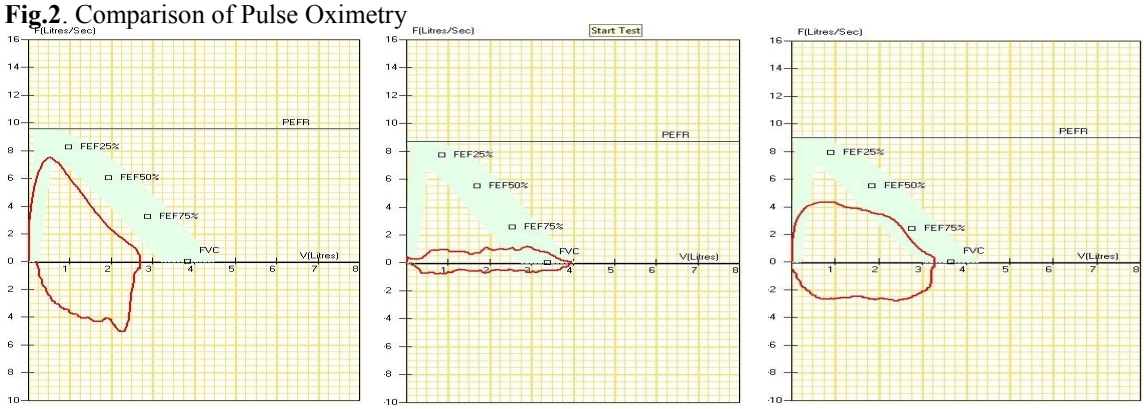


Fig.2. Comparison of Pulse Oximetry



(a) (b) (c)

Fig. 3. Comparison of Contour of Flow Volume Loop, (a) Subject 1, (b) Subject 2, (c) Subject 3

TABLES

Table 1. Physical Measurements

Sr. No.	Subject	Age (yr)	Weight (kg)	Height (cm)	BMI
1	Subject 1	29	94	175	31
2	Subject 2	43	84	169	29
3	Subject 3	50	74	177	24

Table 2. Habit and Grinding Exposure in Years

Sr. No.	Subject	Smoker		Tobacco user		Grinding Exposure (yr)	Machining Exposure (yr)
		Self	Parents	Self	Parents		
1	Subject 1	No	No	No	No	Nil	Nil
2	Subject 2	Ex-smoker	Yes	Yes	No	25	Nil
3	Subject 3	No	Yes	No	Yes	Nil	30

Table 3. Pulmonary function parameters

Sr. No.	Subject	FVC (L)		FEV1 (L)		FEV1/FVC (%)		PEFR (L/min)		Lung Age Pred. (yr)	
		Pred.	Actual	Pred.	Actual	Pred.	Actual	Pred.	Actual	Pred.	Actual
1	Subject 1	3.85	2.68	3.26	2.68	84.68	100	9.59	445	29	34
2	Subject 2	3.36	3.97	2.73	0.28	81.25	7.05	8.67	70	43	82
3	Subject 3	3.66	3.27	2.90	2.99	79.23	91.44	9.00	260	50	48

Table 4. Pulse Oximetry

Sr. No.	Subject	S_PO_2 (%)
1	Subject 1	97
2	Subject 2	98
3	Subject 3	96

ACKNOWLEDGEMENT

School of Physiotherapy for lending the instruments and help training using instrument receive during recording.

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Frequent Pattern Mining with University Library Information System Database using FPMBM

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ABSTRACT

Frequent pattern mining plays an important role in the university library data analysis. The problem of association rule mining for different books in a large library information system (LIS) database is to identify crucial frequent patterns. Also relation between different books in a pattern of DB transactions examined with few more different algorithms. Among these algorithms, Apriori which costs lots of time compare to FPMBM (Frequent Pattern Mining using Boolean Matrix). To address this issue, FPMBM is used for finding frequent itemsets. So our study focuses on frequent pattern mining using FPMBM. This study helps university students to find the frequently used books/objects of library and suggests them to choose appropriate and best among the available options from the library. In FPMBM, operations are performed using Boolean matrix for k-pattern of all frequent patterns generation process. List for a number of iterations helps to reduce the number loops for frequent patterns generation. This also reduces scans over the LIS database. And it uses the vertical data approach with LIS database. The FPMBM algorithm was tried utilizing both synthetic and genuine library information data, and the exploratory results demonstrated that FPMBM algorithm beat over Apriori algorithm.

SUMMARY

FPMBM is used to solve the problem of association rule mining for different books/resources details available in a large library information system (LIS) database and also to identify the crucial frequent patterns.

Keywords: Association rule, Frequent pattern mining, Library Information System Database, Boolean Matrix, FPMBM

INTRODUCTION

With a large amount of data available for mining due to computer based systems, a huge quantity of data is found in a university library. Important information hidden in this database can be discovered. Expectation on such data is to find useful rules to incorporate student personnel services (*1, 6*). User behaviour on library information system database can be analyzed in order to further enhance the library services which can be considered to a newer scope (*11*).

Due to advancement in information engineering, data mining found as a most useful weapon for the civilization in the last few decades. This results into availability of large amounts of data. From this data, knowledge is revolved which found to be as the most useful information. There are many areas where data mining is useful. This includes "library information system", "supermarket with attractive discounts", "web page links with combination of different keywords together found", "medical field with a group of symptoms for a syndrome", etc (*1, 11, 12, 14*).

Data mining directs to the expansion of succeeding tasks, i.e. "data collection and database creation, data management (including data storage and retrieval, and database transaction processing), and advanced data analysis (involving data warehousing and data mining)" (*1*). Few facts for data mining are : "the explosive growth of data: from terabytes to peta bytes", "data collection and data availability due to automated data collection tools, database systems, web, computerized society", "we are drowning in data, but starving for knowledge" (*1*).

Data mining can be defined as – "knowledge discovery from data" or "extraction of interesting (non-trivial, implicit, previously unknown and potentially useful) patterns or knowledge from the huge amount of data" (*1*). "The actual discovery phase of a knowledge discovery process" (*13*). Propelled advances and more up to date developments with exceedingly digitized gadget lead to heaps of information found in the present business sectors. Employment of scanner information in a college library system delivers a lot of information.

Mainly imperative strides for succeeding of knowledge discovery shown in Fig. 1 (*1*).

1. Data cleaning – "eradicates data which are noisy and not consistent".
2. Data integration – "merges data from different numeral sources".
3. Data selection – "retrieval of data from database based on selective factors".
4. Data transformation – "summarized or dashboard kind of view of the given facts".
5. Data mining – "knowledge extraction".
6. Pattern evaluation – "to recognize most fascinating patterns belonging knowledge depends upon few significant transactions".
7. Knowledge presentation – "representing or showing summarized results".

Transactional datasets or "operational datasets": As shown in Fig. 2, a transaction normally contains a TransID as well as the record of books building a transaction (like books issued from the library).

Association rules are conditional affirmations that aid rendering the relationships between distinct data in a transactional DB (*1*). An example for an association rule can be "If a student issues a book A, he is 60% expected to issue book B" (2). An association rule includes 2 divisions, "a predecessor (if) and a subsequent (then)". A predecessor – "an object originates in the data" and a subsequent – "an object that is originated in combination with the predecessor" (*1*). Researchers can concentrate on this range for mining frequent patterns. This can happen from the little and/or from the immense amount of actualities,

where the realities are either transactional or relational (*7*). **"**A set of items (such as itemsets, sub sequences, or substructures) that appear in a dataset frequently are known as frequent patterns" (*1*). There are numerous sorts of frequent patterns, association rules, and correlation relationships.

Library Users: There are many types of users who are using university library. Considering a university having different courses running in it like BCA, MCA, MScIT, B.Tech, M.Tech, B.Pharm, M.Pharm PhD, etc., students and faculties search books as per their requirements. So the library information system should help users about some recommendation on frequently used books.

MATERIALS AND METHODS

Four major frequent pattern mining approaches like Apriori (*2*), Direct Hashing and Pruning (DHP) (*3*), Frequent pattern growth (FP-Growth) (*4*), Vertical data format approach (ECLAT) (*5*). These techniques are tested to work with transactional database. Each one is having few challenges. But there is an FPMBM approach to overcome these challenges (*10*). FPMBM technique makes better use of Boolean matrix which is detailed with LIS database for frequent pattern mining (*8, 10*).

FPMBM Algorithm (Frequent Pattern Mining with Boolean Matrix) depicted in Fig. 3:

Effective frequent patterns can be generated from LIS data using FPMBM. Vertical layout for data is concerned with FPMBM (*10*). Each database transaction is converted to vertical format before mining. This shows that each row represents a particular pattern and each column for a particular transaction in a database table.

Example: 1 - We pursue a sample of a basic library information system DB table with 9 transactions and minimum support is 2 as shown in Table 1.

Let's consider some details of books like:

B1 – "Programming in ANSI C"
B2 – "C: The Complete Reference"
B3 – "Computer Fundamental and Programming in C"
B4 – "C Programming: A Modern Approach"
B5 – "C for Dummies Vol. I"

i) As shown in Table 2, convert horizontally data into vertical layout.

ii) As shown in Table 3, generation of Boolean matrix from the vertical layout. Calculate sup_count from the given matrix. Find frequent 1-pattern list.Update no._of_iterations list properly.

iii) AND operation

a. As shown in Table 4, perform AND operation and generate of Boolean matrix. Calculate sup_count from the given matrix. Find frequent 2-patterns list. Update no._of_iterations list properly. Ignore patterns whose sup_count<min_sup.

b. As shown in Table 5, perform AND operation and generate of Boolean matrix. Calculate sup_count from the given matrix. Find frequent 3-patterns list. Update no._of_iterations list properly. Ignore patterns whose sup_count<min_sup.

This method continuously followed for every k increment of factor 1. This works until there is no further scope of pattern generation. Consequent to above experiment, here following frequent patterns are found:

Frequent patterns of 1 book are: “B1, B2, B3, B4 and B5”
Frequent patterns of 2 books are: “(B1, B2), (B1, B3), (B1, B5), (B2, B3), (B2, B4), (B2, B5)”
Frequent patterns of 3 books are: “(B1, B2, B3), (B1, B2, B5)”

Mining Association Rule:

For rule B1 ⇒ B2 : Let minimum confidence threshold is, say 60%.
support = support({ B1 U B2 }) = 4/9 = 44.44%
confidence = support({ B1 U B2 })/support({ B1 })
= 4 / 6 = 66.6%
This rule is selected as a strong rule from association rule mining.

For rule B1 ⇒ B5 : Let minimum confidence threshold is, say 60%.
support = support({ B1 U B5 }) = 2/9 = 22.22%
confidence = support({ B1 U B5 })/support({ B1 })
= 2 / 6 = 33.3%
This rule is rejected as would not be considered as a strong rule from association rule mining.

Let’s elaborate this situation. A student has issued a book B1 (“Programming in ANSI C”) and he wants to issue another book. But he is in confusion about which book to be issued. By considering above two rules, he will be recommended that book B2 (“C: The Complete Reference”) is more preferable than B5 (“C for Dummies Vol. I”) which is justified and shown in comprised result discussed above by following association rules.

Fig. 4 illustrates the execution tree of the FPMBM algorithm over the transaction database mentioned in Example 1 for minimum support value 2. A frequent candidate is shown in a solid box, and an infrequent candidate is shown in a dotted box. An edge represents the ANDing relationship between size k candidate and size (k -1) frequent pattern. The Fig. 4 also illustrates the fact that a pair of frequent patterns is used to generate a candidate pattern, whereas no candidates are generated from an infrequent pattern.

Example Explanation:

We had L = {{B1}, {B2}, {B3}, {B4}, {B5}, {B1,B2}, {B1,B3}, {B1,B5}, {B2,B3}, {B2,B4}, {B2,B5}, {B1,B2,B3}, {B1,B2,B5}}.

1. Let’s take l = {B1, B2, B3}.
2. Its all nonempty subsets are {B1, B2}, {B1, B3}, {B2, B3}, {B1}, {B2}, {B3}.
3. Let minimum confidence threshold is, say 60%.
4. The resulting association rules are shown below, each listed with its confidence.
   I. R1: B1 ^ B2 → B3
      - Confidence = sc{B1,B2,B3}/sc{B1,B2} = 2/4 = 50%
      - R1 is rejected.
   II. R2: B1 ^ B3 → B2
      - Confidence = sc{B1,B2,B3}/sc{B1,B3} = 2/2 = 100%
      - R2 is selected.
   III. R3: B2 ^ B3 → B1
      - Confidence = sc{B1,B2,B3}/sc{B2,B3} = 2/2 = 100%

- R3 is selected.

IV. R4: B1 → B2 ^ B3
- Confidence = sc{B1,B2,B3}/sc{B1} = 2/6 = 33%
- R4 is rejected.

V. R5: B2 → B1 ^ B3
- Confidence = sc{B1,B2,B3}/{B2} = 2/7 = 29%
- R5 is rejected.

VI. R6: B3 → B1 ^ B2
- Confidence = sc{B1,B2,B3}/ {B3} = 2/2 = 100%
- R6 is selected.

In this way, three strong association rules have been found and help us to recommend available options to students.

RESULTS AND DISCUSSION

Comparison of performance of the FPMBM with various techniques is depicted in figures 5 to 9. Implementations of discussed techniques are performed in Java. As well as the compilation is done with Java compiler and code is written in Netbeans IDE 6.8. Testing dataset (*9*) is considered from FIMI repository. Table 6 illustrates characteristics such datasets. This study helps the student to recommend book issue based on current as well as previous book issue history. Frequently used patterns for books help many students and faculties to analyse the importance of sequence and collective use of books of same domain.

From the figures 5 to 9, we can discover that the FPMBM technique exhibits the best result comparatively for the library information system database.

CONCLUSION

Apriori is the basic technique for frequent pattern mining. But it consumes more time and less efficient than FPMBM (*10*). Also FPMBM defeats few more discussed algorithms for library information system database. Frequent pattern mining from library information system database helps a lot to university to justify the optimum utilization of library learning resources.

FIGURES

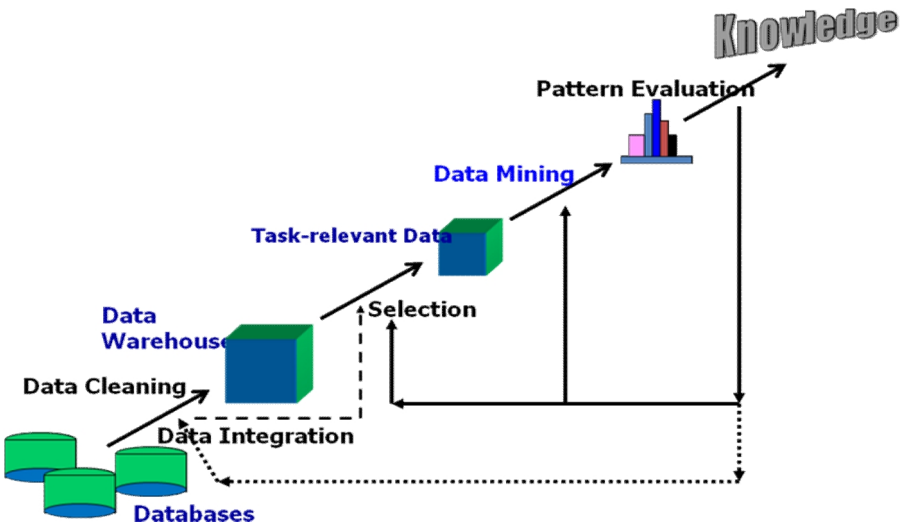


Fig. 1. Knowledge Discovery (*1*)

Trans_ID	List of book_IDs
T100	I1, I3, I8, I16
T200	I2, I8

Fig. 2. Sample Entry in a Transaction Dataset (*1, 10*)

Algorithm : FPMBM

Steps:

1. Convert list of transactions available in D into Vertical Data Layout D_Ver.
2. Generate Boolean Matrix from D_Ver. Maintain a list of NUM_ITR i.e. useful for K+1 patterns generation.
3. Find frequent 1-patterns from the given Boolean Matrix. Support count for each pattern is simply done by summing the number of 1's in each row of the itemset within Boolean Matrix.
4. Increment K by one.
5. Perform candidate patterns generation for K-patterns with consideration of Boolean Matrix. Maintain a list of NUM_ITR i.e. useful for K+1 patterns generation.
6. Find large patterns by minSupport.
7. Repeat through step-4 Until Boolean Matrix is null for all candidate patterns creation.

Fig. 3. FPMBM Algorithm (*10*)

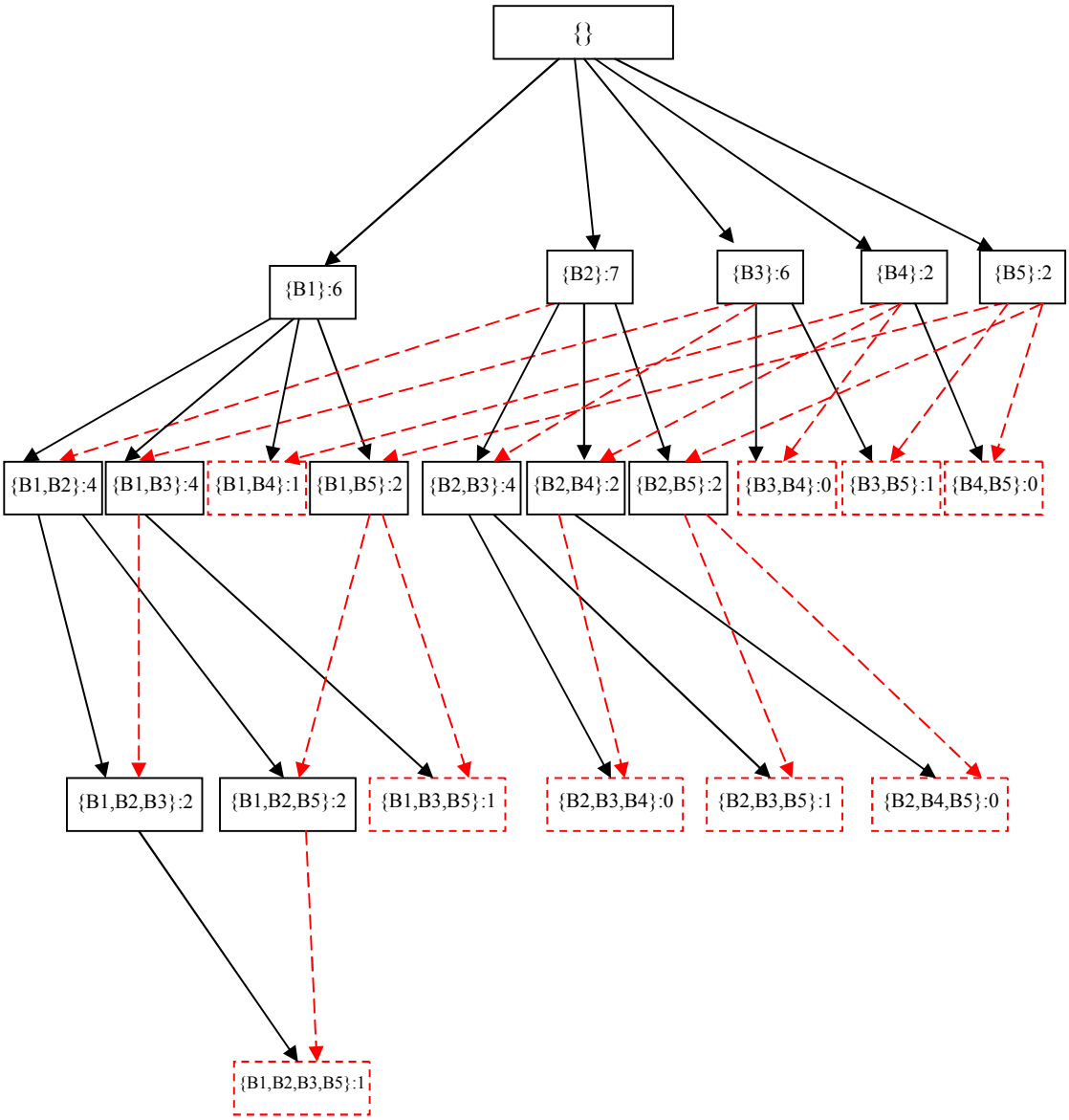


Fig. 4. FPMBM Diagram for LIS Database Example

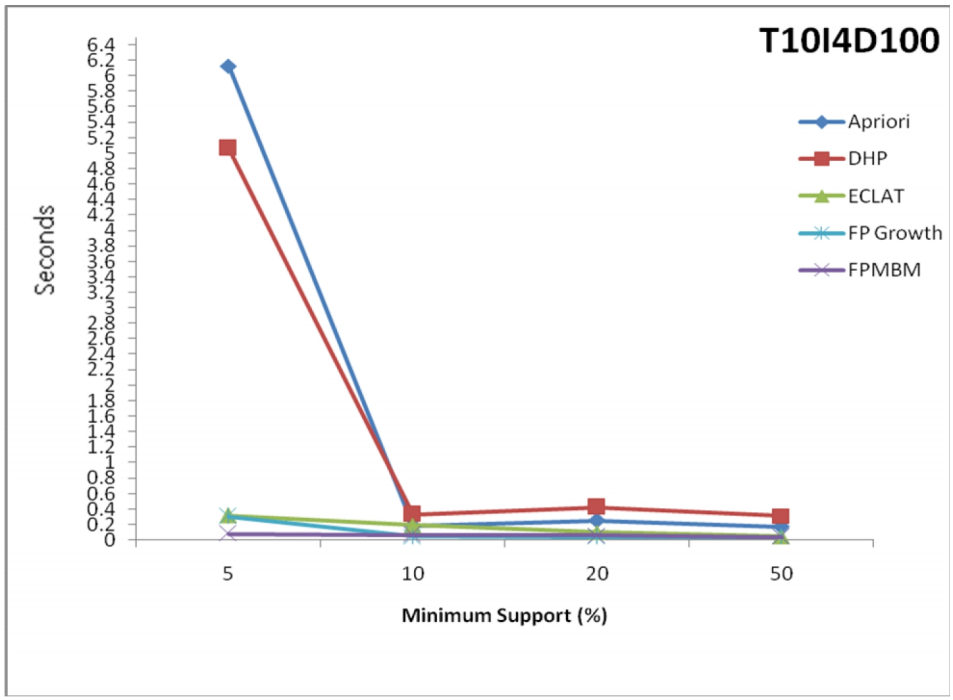


Fig. 5. Execution time (in seconds) required by five different algorithms in T10I4D100 dataset with different minimum support threshold.

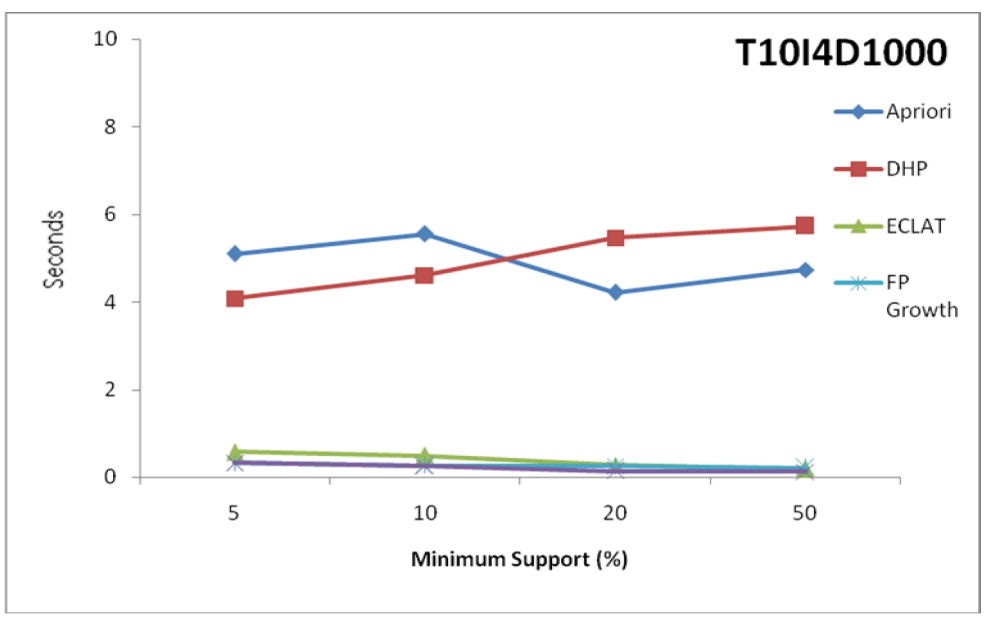


Fig. 6. Execution time (in seconds) required by five different algorithms in T10I4D1000 dataset with different minimum support threshold.

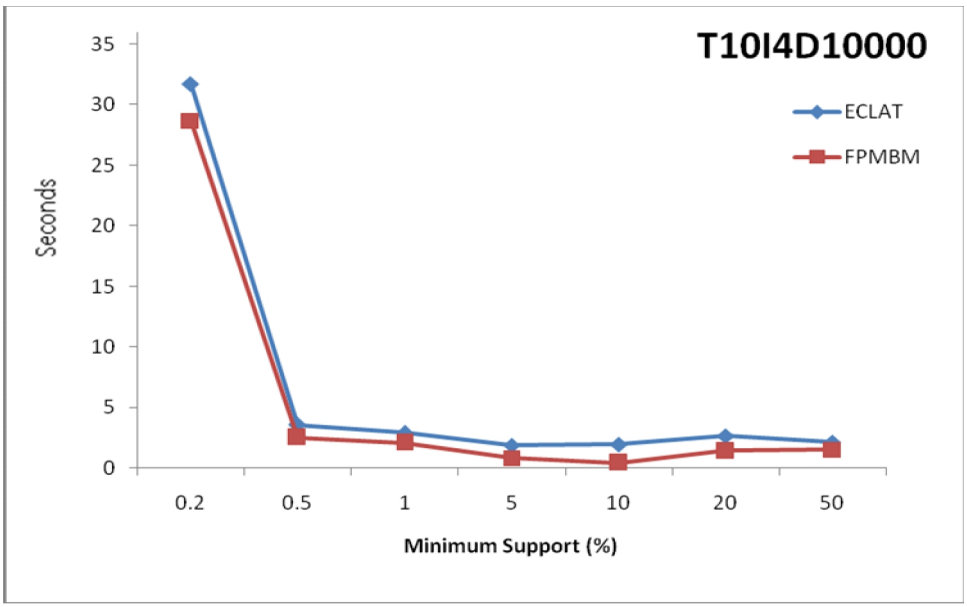


Fig. 7. Execution time (in seconds) required by two different algorithms in T10I4D10000 dataset with different minimum support threshold.

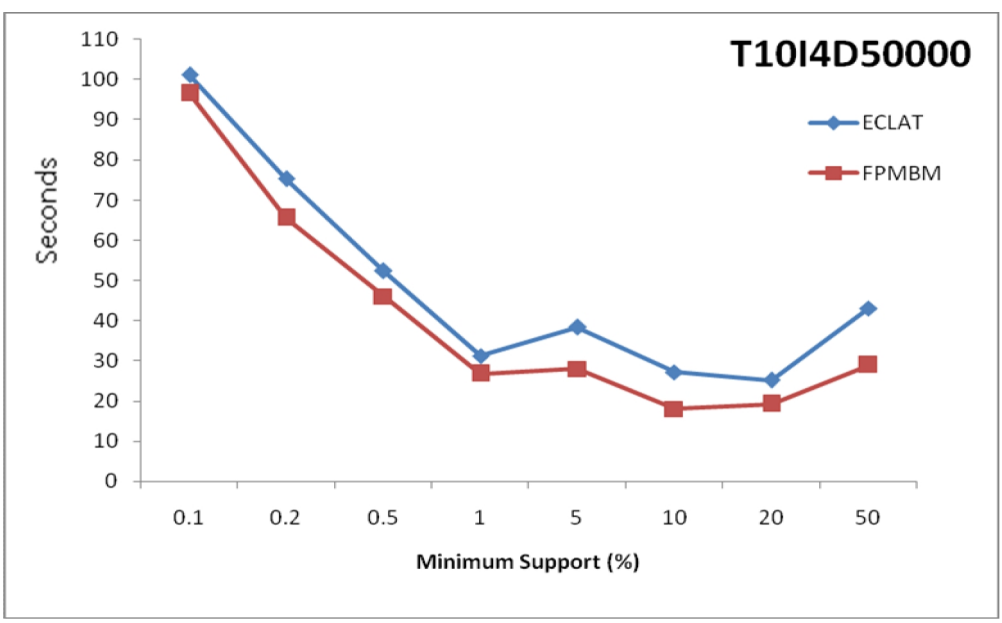


Fig. 8. Execution time (in seconds) required by two different algorithms in T10I4D50000 dataset with different minimum support threshold.

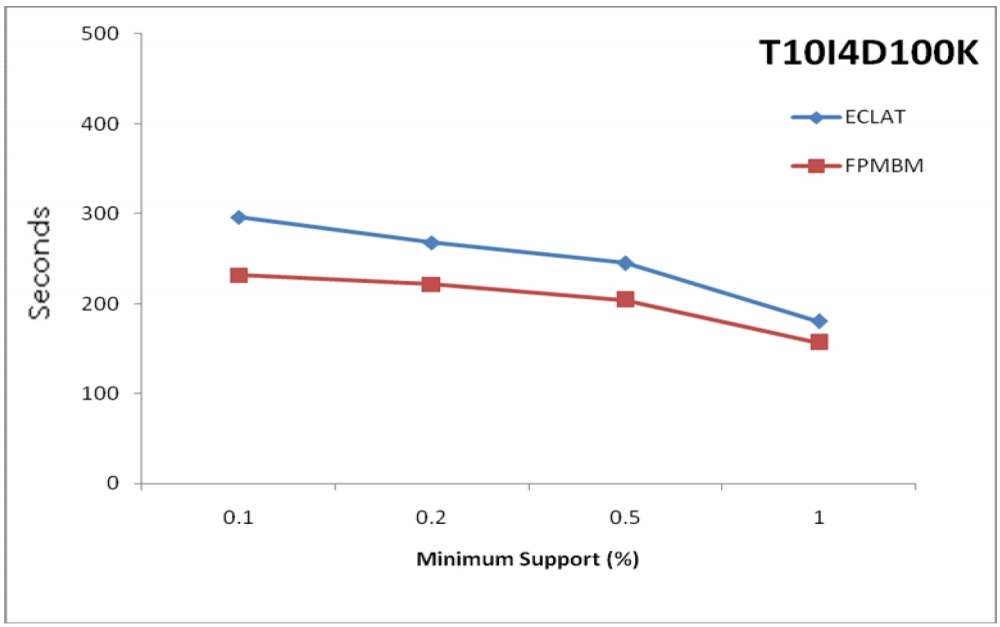


Fig. 9. Execution time (in seconds) required by two different algorithms in T10I4D100K dataset with different minimum support threshold.

TABLES

Table 1. LIS Database Table	
TID	**Record of bookIDs**
T001	B1,B2,B5
T002	B2,B4
T003	B2,B3
T004	B1,B2,B4
T005	B1,B3
T006	B2,B3
T007	B1,B3
T008	B1,B2,B3,B5
T009	B1,B2,B3

Table 2. Vertical Data Format of Transaction Table	
Itemset	**Transactions IDs**
B1	{T001, T004, T005, T007, T008, T009}
B2	{ T001, T002, T003, T004, T006, T008, T009}
B3	{ T003, T005, T006, T007, T008, T009}
B4	{ T002, T004}
B5	{ T001, T008}

Table 3. Boolean Matrix with Vertical Layout of Table 2											
TransID / Itemset	**T001**	**T002**	**T003**	**T004**	**T005**	**T006**	**T007**	**T008**	**T009**	**Sup_Count**	**No._of_Iterations**
B1	1	0	0	1	1	0	1	1	1	6	4
B2	1	1	1	1	0	1	0	1	1	7	3
B3	0	0	1	0	1	1	1	1	1	6	2
B4	0	1	0	1	0	0	0	0	0	2	1
B5	1	0	0	0	0	0	0	1	0	2	0

Table 4. AND Operations with itemsets of Table 3											
TransID / Itemset	**T001**	**T002**	**T003**	**T004**	**T005**	**T006**	**T007**	**T008**	**T009**	**Sup_Count**	**No._of_Iterations**
(B1,B2)	1	0	0	1	0	0	0	1	1	4	2
(B1,B3)	1	0	0	0	0	0	1	1	1	4	1
(B1,B5)	1	0	0	0	0	0	0	1	0	2	0
(B2,B3)	0	0	1	0	0	1	0	1	1	4	2
(B2,B4)	0	1	0	1	0	0	0	0	0	2	1
(B2,B5)	1	0	0	0	0	0	0	1	0	2	0

Table 5. AND Operations with itemsets of Table 4											
TransID / Itemset	**T001**	**T002**	**T003**	**T004**	**T005**	**T006**	**T007**	**T008**	**T009**	**Sup_Count**	**No._of_Iterations**
(B1,B2,B3)	0	0	0	0	0	0	0	1	1	2	1
(B1,B2,B5)	1	0	0	0	0	0	0	1	0	2	0

Table 6. CHARACTERISTICS OF DATASETS USED FOR EXPERIMENT EVALUATION

[1 – Apriori, 2 – DHP, 3 – ECLAT, 4 – FP Growth 5 – FPMBM]

Dataset	**Records**	**Algorithms Comparisons**	**Remarks**
T10I4D100	100	1,2,3,4,5	Top 100 records from T10I4D100K
T10I4D1000	1000	1,2,3,4,5	Top 1000 records from T10I4D100K
T10I4D10000	10000	3,5	Top 10000 records from T10I4D100K
T10I4D50000	50000	3,5	Top 50000 records from T10I4D100K
T10I4D100K	100000	3,5	-

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(Proceedings available for download at rku.ac.in/icre)

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Comparative Analysis of Different Task Scheduling Approaches in Cloud Environment

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ABSTRACT

Cloud computing is collection of services. In that third party vendor buys the infrastructure and then it delivered to the cloud as a service to the end users. So end users not worry about the managing their own infrastructure. Due to that quality of cloud environment many organizations, health cares and education departments are moving towards the cloud. It provides on demand access of dynamically scalable resources. So at time based demanding users are increased and due to that availability of resources are decreases. For that scheduling of tasks are needed. It uses maximum responses with minimum completion time. Task scheduling is one of the fundamental issue in cloud environment. To solve that issues meta-heuristic algorithms are proposed. Task scheduling maps the tasks to appropriate resources that optimize the one or more parameters or objective of Scheduling. This paper gives different task scheduling algorithms and comparative study between all that.

SUMMARY

This paper gives the brief introduction about the cloud environment and its service models. It also gives the comparative study between different task scheduling algorithms in cloud computing.

Keywords: Cloud computing, Task Scheduling, Improved cost based algorithm, PCO, GA, ABC, ACO

1. INTRODUCTION

Cloud computing is type of distributed and parallel system that consists of the inter-related and virtualized computers. In that virtual pool of resources (*e.g.*, Network, application, services, storage and servers) are there. These resources are provided to the demanding users from service provider through negotiation between end users and service provider. They build one contract between them and according to that users have to pay for the same *(1)*. Service provider deliver that services over the internet. Cloud computing has ability to quickly scale up and scale down the virtualized resources. In that customer not need to buy the resources from the third party, they uses the resources and pay for that as a services. So, it save the money and time of customer *(2)*. User can access it around the globe area.

Main aspect of the cloud computing is to manage various kind of platforms, computing power, storage, infrastructure and different services. Then after it assigned to the external users on demand. It also provides the very large number of resources. In the form of services it provides storage, network, data centre and software. For computation purpose it provides platform as a service. Virtualization is one of the main aspect of cloud computing. Using that aspect it provides the basic infrastructure to build an application.

Now a days, number of users increases and they also increase the use of cloud environment. So at a time availability of resources also decreases. So for that satisfies the customer requirement on demand, scheduling is necessary. So main goal of scheduling is to manage the all resources, balance the traffic over the network, maximum utilization of resources with minimum completion time. In that task scheduling perform very important role. It maps the task on available resources. So for that many meta heuristic approaches like improve cost based task scheduling algorithm, particle swarm optimization (PSO) algorithm, independent task scheduling based upon genetic algorithm (GA), optimize activity based costing (ABC), Genetic Simulated Annealing Algorithm for Task Scheduling and Ant Colony Optimization (ACO) algorithm are used. They are used for minimize the makespan or completion time with maximum utilization of resources. So due to that it improves the scheduling parameter like performance and throughput.

1.1 Service models

Cloud computing provides different type of resources and offered different types of services that is described below:

Infrastructure as a service (Iaas): IaaS offers the basic infrastructure to build an application. Using virtualized technology, this model shares the hardware resources for executing the service. Main purpose of using this model is to make resources accessible by application and os *(2)*. It has capability to adding new equipment or resource in a simple manner. Example of IaaS providers are CloudSigma, Amazon Elastic Cloud Computing (EC2), GoGrid and Amazon S3.

Software as a service (SaaS): In that software application is provided as a service to the end users. As per demand user can purchased the application software and also used that. SaaS is one kind of distributed model. In that applications are hosted by service provider and then customer can use that over the internet *(3)*. Example of SaaS providers are Google Apps, Salesforce.com

Platform as a service (Paas): Paas is refers for delivering associated services and operating system over the internet without downloads and installation. It is typically provide platform on which software can be developed or deployed. Example of Paas providers are Google App Engine, Heroku, Microsoft Azure, GridGain.

1.2 Deployment model

Cloud computing itself a model that provide self-managed on demand resources in availability of network connection. In cloud computing environment based upon user's requirement and usage of application and services, there are main four models are there. That are described below.

Public model: In the public cloud, cloud infrastructure is available for users in general clouds or large industry group and in that service provider, make available all cloud services (application, storage, resources, etc.) over the internet *(3)*.That is hosted on internet and user can use that on demand and pay for that only. Public cloud providers are Google App Engine, Microsoft Azure and Amazon EC2. In public cloud data is available 24/7.

Private cloud: Private cloud is one kind of organization or infrastructure that is managed by cloud service provider or third party. In that main aim is to serve the organizational data to only member of the organization or only authorized users. The main purpose behind the developing the private cloud is security concerns that the data of any private organization should accessed in that network. Example of private cloud is Eucalyptus Systems, Amazon Virtual Private Cloud (Amazon VPC), IBM smart cloud, Microsoft Private Cloud *(3)*.

Hybrid cloud: Hybrid cloud is one kind of integrated cloud. It can be an arrangement or combination of two or more cloud (private, public or community cloud). Basically it combines the features or advantages of two or more cloud *(4)*. So security concern like private cloud and elasticity of on demand unlimited service & flexibility like public cloud in one cloud is referred as Hybrid cloud. In hybrid cloud two or more cloud integrated or bounded together with standardized technology, but it remains as individual entity. An Example of hybrid cloud is Amazon Web services (AWS).

Community cloud: It mainly used for sharing purpose. For the sharing and distribution of service between group of similar organization not for anyone of local user or single organization. This type of cloud are basically developed for fire departments, community swimming pools, government agencies. The best example of community cloud is government agencies which are connected and they have shared all credentials information in privately and Facebook is also one kind of community cloud *(2)*.

2. CLOUD ARCHITECTURE

Cloud computing is general term, that delivers the resources to the end users over the internet. In that many service provider are there. In that three type of service model are there that provide the basic requirement of users on demand. In that IaaS is provide the basic infrastructure to the user. PaaS is provide the platform as a service to the users and SaaS is provide the complete application to the user on demand. So most important part or feature is internet for the cloud. That is illustrate in figure 1.

3. OVERVIEW: TASK SCHEDULING

Scheduling is maps and manages inter-dependant task on distributed resources. Main goal of scheduling algorithm is distribute the load on processors with maximum utilization of resources and minimize the total completion time. In scheduling first of all give the information of current availability of resources. Then it collects the information related this current resources. Then after resource is selected based on resource parameter and task. At last task is submitted to the resources *(4)*.

3.1 Need of scheduling in cloud computing

There are many scheduling parameters like performance, response time, processing time, makespan, cost, scalability, throughput, resource utilization are there. Initially scheduling algorithm were implemented in Grid. In grid, user were find the subset of resources for their application. Whereas in cloud, first of all find the way of allocate some VMs on them and then after it schedule the task on VMs. But in grid environment performance are reduced. Main reason to move towards the cloud is scalability (easily scale up or scale down the resources) *(6)*. Due to the scalability of resources, we can provide resources to the application as per requirement. Sometimes many cloud application requires complex execution environment and this environment is difficult to create on grid resources. There are many cloud services like storage, processing power, memory power, hosting platform are available at very lower cost.

In task scheduling, task are mapping according to the resources. Cloud users use the cloud resources through the internet. In that it needed resources at any time and use that resources at any time. In task scheduling, major activity is task. As we know cloud users use the cloud resources through internet. Due to that, users need anytime or on demand resources. For that at a time based availability of resources is checked and for that scheduling of Task is necessary.

The scheduling of task in cloud means allocate computer machine to the task in such a manner that maximize the total utilization of resources and minimize the total completion time or find best suitable resources for execution of task. Main reason for using Task scheduling algorithm is better utilization of resources, minimize the makespan and achieve better performance.

4. EXISTING TASK SCHEDULING ALGORITHMS

1> Improved cost based Task scheduling algorithm:

This algorithm schedules the task group in cloud computing platform. In cloud computing, resources are having different performance, different throughput and different computational cost. In cloud environment, for computing purpose always not a single job or task is there. In that many times grouping of job also done. So due to that grouping, communication ratio between job and resources is optimizes *(7)*. Main purpose of this algorithm is to measures the cost of resources and performance of computation. How process the available resources, according to that various tasks are combined. So doing by that it improves or increased the execution ratio, execution of tasks or data transfer rate. This algorithm is take less time while grouping of job is done compared to when grouping of job is not done *(8,9)*.

2> Particle swarm optimization algorithm (PSO):

PSO *(10)* is simple and effectiveness. In the cloud area, there is a random solution space are there. So PSO is begins the process of optimization with a random solution space and searches for the optimal solution by updating the potential solutions through the iterations, similarly to Genetic Algorithms. The particles “fly” over the searching area looking for better solutions. In that it is initialized through the random distribution of the particles in the searching space *(11)*. Main focused of this algorithm is to minimize the total cost of transferring and executing of application. For the application, resources are provided by the cloud service provider. In PSO algorithm initially position and velocity of particles are

random. By evaluating the fitness function, each particle finds or calculates the fitness value for optimization purpose. In cloud environment various tasks are there. Those are dependent on each other. So this algorithm is consider the communication cost between all the tasks. So for the cost saving purpose we are doing comparison with Best Resource Selection algorithm (BRS). So in that PSO based algorithm achieve better performance with three time saving of cost over BRS algorithm *(12)*. It also achieve good distributed workload onto resources. By increasing the particles and resource number, this algorithm is used for any number of tasks.

3> Independent task scheduling based upon Genetic Algorithm (GA):

There are many assumption related tasks are there:
-> Task are non-pre-emptive (each of them is independent)
-> Task are aperiodic (In priori, task arrival times are not known.)
-> Each task has two ways of access of process unit. Shared (in that case it can share the resource with another task) and exclusive (in that case no other task can share the resource with another task) *(14)*. Genetic algorithm is mainly based on the "Selects the Best, Discard the Rest" principle *(13)*. GA algorithm handles multiobjective problems. GA algorithms also easily interfacing with existing simulation and models *(13)*. Based on inheritance and natural selection theory that GA algorithms widely used in solving scheduling problem *(14)*. For adapt memory constraint and request of high performance in cloud environment the GA in task scheduling was introduced. In cloud computing very large volume of solution space are there. So find the optimal solution from that in polynomial time, task scheduler used GA algorithm techniques.

In basic Genetic algorithm initial population size is generated randomly. So very much less chances to generate better child from themselves. In Improved Genetic algorithm Initial population size is generated using Min-Min and Max-Min technique. In that algorithm it consider both time and resource utilization.

4> Genetic Simulated Annealing Algorithm for Task Scheduling:

This algorithm is based upon the QOS requirements. QOS requirements mean it measures the overall performance of computer network. QOS requirements include the user's satisfaction criteria towards the different services and applications, means it satisfies the user's requirement. For example, For complex task, some CPU need more time to complete that, some other need more storage to store the data, etc. QOS includes the different type of parameters like throughput, delay, jitter, cost, CPU power etc. In that, parameter's dimensions and order of magnitude are different. Using those parameters, we can implement the different type of task on cloud resources. For finding the resources, set the criteria which satisfy the task's expectation and QOS requirements, different weight of parameters are given according to the type of task. In this algorithm it considers the value of both simulated annealing algorithm and Genetic algorithm *(15)*. Simulated annealing algorithm not only accepts the optimal solution but also it accepts the limitedly lower solution with random solution space. In this algorithm, first evaluate the steps of Genetic Algorithm. After selection, crossover and mutation in GA, Simulated annealing comes into the picture. It helps to improve local search ability of GA. So this algorithm efficiently completes the finding and allocation of resources in Cloud Environment.

5> Optimized Activity Based Costing (ABC):

In cloud computing each of task is different from one another. In traditional way there is no relation between overhead application and cost of resources. In that task scheduling cannot measure the cost of cloud resources properly. In cloud computing, task scheduling is totally depends on user's requirements. ABC algorithm actually works on optimized way of resource allocation in cloud computing *(17).* This algorithm measure both cost of object and its performance. Activities will be performed on virtual os. Resources are provided over these virtual systems by the original system. Tasks are independent or dependant on the other tasks. Tasks needed the resources that might be not available on single datacentre. So, after considering all possibilities, tasks are divided into two groups: Partially available (group of tasks which will require resources from other data centre) and Available (Group of tasks which can be complete performed on a single data centre). In that according to the priority of task, allocate the resources and maximize the performance of Activity *(18).*

6> Ant colony Optimization (ACO):

Ant colony Optimization algorithm is dynamic algorithm. It is based upon the behaviour of ant. It finds the optimal or best path between their colony and source food *(20).* It is metaheuristic optimization algorithm. It discovers the path via pheromone trails. More pheromone on path increases probability of path being followed. While walking towards the colony and food source, ant leaves the pheromone on the ways they move. The pheromone intensity on the passages increases with the number of ants passing through. As the time goes, smaller path will draw more pheromone and thus the pheromone intensity helps ants to recognize smaller path to the food sources. This algorithm used in many real time application like travelling salesman problem, multidimensional knapsack problem, job shop scheduling, vehicle routing problem, data mining, connectionless routing problem and many more other applications *(21,22).*

In the task scheduling, there are many metaheuristic approaches are there for improving the scheduling parameters. Comparative study between all that approaches is describedin Table 1.

5. PROPOSED WORK

This paper highlighted six different metaheuristic approaches, they have its own merits and demerits based on the user requirements and QOS requirements. In existing task scheduling algorithms *(9, 11, 13, 14, 15, 20)* when priority is considered starvation problem is created. There is a future scope of research based on priority and improving scheduling parameters. In cloud,taskScheduling based ACO approach *(1, 20)* reduces makespan parameter for utilization of availableresources optimally. Improvement in the efficiency of ACO algorithm needed to balance the load in cloud environment. Based on it, proposed work is related to finding the precedence between tasks and balancing of load in ACO approach.

6. CONCLUSION

This paper concentrate on different cloud service models and deployment models. Service models provides the different services like infrastructure, platform and software on demand to the end users. This paper also gives the different task scheduling strategies. Task scheduling is one of the most important issues now a days. These approaches are helpful to minimize the total completion time or makespan with maximum utilization of resources. So due to that it improves the scheduling parameters like performance

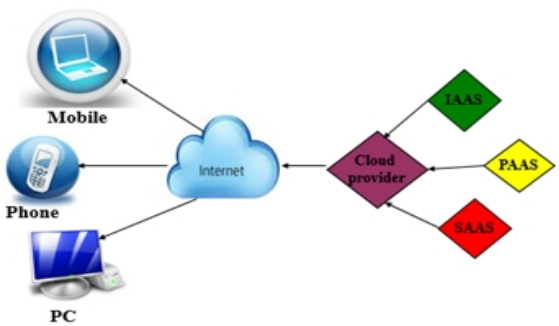
and throughput. In that different task scheduling algorithms are also compared and prepared comparative study of that algorithms.

FIGURES

Fig. 1.Cloud service Models



Fig. 2 Cloud Architecture



TABLES

Table 1. Comparative analysis of task scheduling algorithms

Task Scheduling	*Static*	*Dynamic*	*Parameters*	*Merits*	*Demerits*

Algorithm					
1. Improved cost based Task scheduling algorithm	No	Yes	Performance and cost of resources	It improves the computational ratio and computational performance. It also measures the resource cost.	In that processing cost and execution time is higher.
2. *Particle swarm optimization algorithm*	No	Yes	Processing and transferring cost and time	It used for any number of tasks by increasing the resource number and particles. It achieves better performance with three times saving of cost over BRS algorithm.	This method suffer from partial optimism. Method cannot work out of the problem scattering and non-co-ordination system. Lack of both reliability and availability criteria.
3. *Independent task scheduling based upon GA*	No	Yes	Makespan, execution time, resource utilization	Consider resource and time utilization.	Large solution space in Genetic Algorithm.
4.*Genetic Simulated Annealing Algorithm for Task Scheduling*	No	Yes	QOS requirements	This algorithm satisfies the QOS requirements of users. It also efficiently completes the searching and allocation of resources.(Helps to improve the solution space)	Hard to programme because of large number of parameters.
5. *Optimized ABC*	No	Yes	Cost and performance of object	It optimizes the resources and maximize the performance.	Resource always not available on single Site. So complexity increases.
6. Ant colony optimization	No	Yes	Makespan and cost	Used for dynamic application, Inherent parallelism, it also manage the positive feedback account for immediate discovery of good solution.	Probability distribution changes by iteration, Theoretical analysis is difficult.

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Improved Resource Allocation using IBA Algorithm for Multi Queue Job Scheduling

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ABSTRACT

Cloud Computing is large group of remote servers and networks which allows centralized data storage and online access of computer services. Cloud is scalable, flexible, and supports multi-media applications. Job scheduling is big issue in cloud computing. Scheduling is more than one process to be loaded into CPU and CPU provides shared memory allocation in each process. Different types of scheduling algorithm such as First in first out, Shortest job first, Round –Robin and Improve backfill algorithm. Using improve backfill algorithm resource utilization increase. We have proposed multi queue job scheduling method using IBA algorithm to reduce the waiting time and improve resources. Themulti queue job scheduling (MQS) method achieves better performance of virtual machine and improve throughput.

Keywords: cloud computing, Job Scheduling algorithm, IBA algorithm, virtual machine

I. INTRODUCTION

Cloud computing is large group of data centers and servers process element (*1*). It is provide scalable and flexible resources. Cloud computing are different type of cloud service like infrastructure as a service (IaaS), platform as a service (PaaS), software as a service (SaaS). Various services detail in following,

Infrastructure as a Service (IaaS) is provides physical virtual Services over internet. IaaS is also provides host application servers, storage services, and high scalable resources in our internet. User can be used to pay on per basis services. E.g. Amazon EC2. Platform as a service (PaaS) is provide the customer run time platform as a service application on cloud Computing. The customer can be used as a pay on demand services. E.g. Azure. Software as a service (SaaS) is provides distribution service model. This application is hosted on network. The service providers deploy the software and user can be directly use software as a services. User can no need to updating and no extra hardware requirements. E.g. SalseForce.Com.

II. SCHEDULING IN CLOUD

Scheduling is more than one process to be loaded into cpu and cpu provides shared memory allocation in each process. different type of scheduling algorithm and different of technique are avalable in cloud computing. The different types of jobs are different values like small,medium,and large types of job values. We can use different type of scheduling algorithm and technique to improve resource utilization and allocation for job scheduling(*1*).

Different type scheduling scalability& flexibility in following(*1)*

1 Static Scheduling: Static means all the information are fixed.The jobs are pre-schedule and resource allocation values are pre assign of process in task. The different types of jobs are different values of them.it means which job is small task process allocation and which job is large task process allocation in virtual system is fixed in scheduling.

2 Dynamic Scheduling:All the information of job scheduling is run time schedule. The different types of jobs are different values of them. It means which job is small task process allocation and which job is large task process allocation in virtual system is scheduling in run time process. It is critical to maintain in load balance problem.

3 Online Scheduling: In this process scheduling, job is schedule at a time no job is waiting for the next time of interval.

4 Offline Scheduling: In this process scheduling, job is waiting for the next task scheduling interval.

Benefits of scheduling are simple listed following:

- Job execution faster.
- Improve processing capability.
- Reduce power consumption.
- Maximum utilized services and resource.

III. SCHEDULING TECHNIQUE

Job scheduling is big challenge in maintain of cloud computing. user can be used different type of scheduling algorithm base on requriment (*10*). The different type of scheduling algorithms are following.

1. First In First Out.
2. Shortest Job First
3. Round Robin.

The different type of scheduling algorithms in detail are following:

1.First In First Out-(FIFO): It is a first come and first serve algorithm process. first come means which process come first that process start first at a time arrive time(*9,10*).Advantage:It is very simple process scheduling. Easy to coding time.Disadvantage:It is not better performance at a result.Last job is wait long time in process scheduling.

2.Shortest Job First-(SJF): Shortest job first algorithm. In this process job is execute base on completion time of job. the small job execution time will be first arrive at a time of process element (9,10).Advantage:Small job is first execution. Small job complite process is high. Disadvantage:Large job is wait a long time. priority of job is not consider.

3.Round Robin-(RR): Round Robin algoritm is base on interval task. in this process small interval time task will be execution of job and after is waiting a turn on time. when the small interval of job is complite first of virtual server(*10*).Advantage: improve FIFO. It is Simple to implement.High throughtput. Disadvantage: Task interval time may be small or large.

IV. RELATED WORK

The scheduler technique is arrival time and burst time of process. Some job is ascending order to execution in time and some job is short time execution. Because of job value use of client (*4*).The different types of scheduling algorithm are FCFS, SJF and RR in details given by above.FCFS, SJF, RR algorithms in resource utilization is less and process competition times more of them. (6)

FCFS, SJF, RR algorithms are not used in task scheduling, because sum of the region is given below: FCFS base some job is large that time job competition time is more of them and queue is waiting for a job scheduling(*7*). SJF is count small complete time of job in process. It means small job is first executed and the large job is wait process at a time.(*7*) RR algorithm is execute interval time of process and interval is small or large.

The Improve Backfill Algorithm is used various meta scheduler process. The different type of job likes small, medium and large. Type of job allocation is different at execution time on virtual machine. The scheduler process, some jobs are waiting at a time of process scheduling. IBA algorithm is schedule in cloud environment and overcome the load balance problem with maintain of physical system. (*3,8*)

The improve backfill algorithm in following step.

IBA algorithm: (*5*)

1. Sort the jobs according to the required number of PE's
2. Place the large job in the last position.
3. Place the second largest job in the right side of the pool.
4. then ,check for the next job
5. if the sum of left pool< sum of right pool, then put the job in the left pool otherwise in the right pool
6. Concatenated both the left pool.

The backfill algorithm used to random number generates value. Physical virtual machine is allocation in small, medium and large type of job in system. Job scheduling is occupied by using IBA algorithm and balance spiral method (BS) in following detail: Algorithm is implement to the server at a time;the backfill algorithm is work at first step to each process occupied job id and collects every job in FCFS base algorithm. Second step the large job is place in the last position at a time of virtual server. Third step the first priority of job is schedule in the first at execution time. Example of job scheduling in **fig1**: Suppose job sequence dynamic value given by sequence {j1, j2, j3, j4, j5}.according to the job values are given in detail {5,4,2,6,9}. J5 is last position at a time but j1 is also allocation in first position. The reaming jobs are {j4, j2, j3}. J1 in the first position left= j1, right= j2.note that j5 not included in right. Remaining jobs are {j3, j4}.Nowsum left <=sum right that time place the large job j4 from the left side. After sum left= >sum right. (*3*) Remaining job j3 in the right. New job sequence is getting by left and right with last position j5 as {j1, j4, j3, j2, j5}Now base on IBA algorithm, sum of left < sum of right that time put the job in left position. Compare job {j4,j3} base on SJF algorithm. Job scheduling in values {j1,j4} small and {j3,j2} medium and j5 will be last position at execution time. (*3*)

The IBA algorithm is high resource utilized and reduces delay. IBA algorithm is more flexible of job scheduling and reduce starvation problem, load balance problem. Advantage: Resource utilization is high. Better throughput. Disadvantage: Scheduling will be difficult. Large number of job to allocation mostly uses. (*5*)

V. PROPOSED METHODOLOGY

Scheduling is complicated issue in cloud environment. As show in table1 IBA algorithm is get better through-put and less waiting time of job scheduling. Using IBA algorithm we have proposed improved resource allocation algorithm for MQS method in job scheduling.We have modified the step in IBA algorithm and proposed new algorithm (*5*). Proposed algorithm is used to improve through-put, deadline of job completion time and less waiting time in job scheduling (2). The expected result is to reduce average waiting time, improve system utilization and get high throughput of virtual system.

VI. CONCLUSION

Scheduling is main issue in cloud environment. The various types of scheduling algorithms are FCFS, SJF, RR and IBA. The comparison of various types of scheduling algorithms as show in table 1. IBA algorithm is schedule the job in CPU process at a time and achieves the better performance of job scheduling. Using IBA algorithm reduce waiting time and increase throughput of resources.

FIGURE

("Fig. 1 Improve Backfill Algorithm.") (3)

Job id	Processor Need
J1	5
J4	4
J3	2
J2	6
J5	9



TABLE

("Table1.Comparison ofvarious scheduling algorithm.")*(9, 3);*

ALGORITHM	METHOD	COMPLEXITY	WAITING-TIME	THROUGH-PUT	IMPLEMENT-CODING
FCFS	OFFLINE	LOW	LARGE	LESS	EASY
SJF	OFFLINE	LOW	LARGE	LESS	EASY
RR	OFFLINE	HIGH	LARGE	LESS	LARGE
IBA	OFFLINE	HIGH	LESS	HIGH	LARGE

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Comprehensive research for success parameters of Maintenance Management Strategy (MMS) implementation within Indian SMEs

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ABSTRACT

In a global competition and unwind economic situation, powerful Maintenance Management Strategy (MMS) might be proven as lifeguard for SMEs. Though the numbers of large scale Indian enterprises are getting the fruit various lean concepts, SMEs still not have changed their traditional approach. As they work with limited resources, it becomes very important to address the generic issues pertaining to MMS. This paper has investigated wide literature in context of SMEs and evaluated current practices of maintenance management in SMEs where LMCS project is executed. Methodology of structured questionnaire and semi structured interviews was used to review and to reveal the various parameters determining a successful implementation of maintenance management. This paper not only offers insights into these highly diverse issues, but also suggests best practices for ensuring successful execution of various lean principles especially for Indian SMEs.

SUMMARY

The novelty of research project stems from the realisation of critical parameters for successful and sustainable implementation of maintenance management strategy to establish world class manufacturing.
Keywords: Maintenance Management Strategy (MMS), Implementation Success Parameters, Quality Management(QM), Small and Medium-sized Enterprises(SMEs)

1. INTRODUCTION

In Indian National GDP, the share of manufacturing sector is stagnated to 15-16% only. The National Manufacturing Policy of Government of India envisages share of manufacturing to reach target of 25% of the National GDP by 2022 with creation of 100 million jobs*(1)*. To achieve a sustained rate of growth, the manufacturing sector needs to build and maintain competitiveness to face the challenges posed by globalization. To overcome challenges, the Ministry of MSME, Government of India has launched Lean Manufacturing Competiveness Scheme (LMCS). Under it, a cluster of 8to10 MSMEs are formed to implemented various lean principles. The pilot phase of LMCS was executed in year 2011 for 100 Mini Clusters. National Productivity Council (NPC) of India functions as National Monitoring and Implementing Unit (NMIU) for facilitating implementation and monitoring of the scheme reported only 58 % success rate for pilot phase so far. The evaluation report has recommended the continuation of the scheme keeping in view benefits amounting to about 20% increased in productivity of units and scheme is approved for new 500 clusters*(2)*. In Surat (Gujarat), Surat Engineering Vikas Association (SEVA) which is a Special Purpose Vehicle (SPV) has facilitated implementation of LMCS in two new clusters for year 2015-16. This research work is also facilitated by SEVA for identifying issues pertaining to execution and sustention of maintenance management initiative as in Indian SME work culture the 'Sustention' success rate is much less than 'Implementation' success rate.

2. SCOPE OF STUDY

In the post liberalization and globalization business era, ease in international trade, privatization, disinvestments and deregulation have thrown several challenges to SMEs in the fast developing economies like India*(3)*. It becomes very difficult for MSMEs to compete effectively as they are still working with callous attitude of towards the maintenance management and productivity*(4)*. This has put Indian SMEs in a very annoying position. P Deshmukh*(5)* identified different challenged for Indian SMES as: Non-availability of highly skilled labor at affordable cost, Absence of adequate knowledge, technology, low production capacity, ineffective marketing strategy, constraints on modernization and expansions, increasing exports, enhanced competition from China and a few low cost centers of production. U Dombrowsk and T Mielke*(6)* observed that globally more and more enterprises have start focusing on the visible parts of lean production systems with the various methods like kanban, 5S, SMED, FIFO and many more and it is very effective to achieve short term improvements, but after a few years, the lean programs of many enterprises do not meet the expectations anymore. According to D Seth and D Tripathi*(7),* an insight into the India's manufacturing sector also reveals that maintenance and human factors have remained as neglected areas since long.

The available literatures on SMEs are focused on developed countries; as such the work on developing countries is lacking*(8)*. There is a lack of systematic empirical research to prescribe what are the issues really may prove crucial for developing, implementing and substation of Maintenance Management Strategy in context of lean principles for Indian culture. Although Lean provides benefits over prior approaches to production, it also creates new challenges like: prioritization of various elements; prevent all losses; maintain productivity; achieve zero defects; zero breakdowns; bump off variability etc. In the first phase of research, an attempt is made to explore and identify the core parameters to flourish Indian SMEs.

3. MATERIALS AND METHODS

A combination of research methods; comprises literature survey, SMEs' practices and personal interviews has been employed to explore the various parameters. The literature survey has not provided sufficient information on issues that affect the successful execution of maintenance management strategy in SMEs. The data collection process involved Engineering and Allied Ancillaries in Surat, India related with

project of LMCS. The reason behind the selection of these SMEs is that they have at-least some experience of implementation of lean principles. The research methodology is shown in figure 1.

By the combination of relevant key words, about 150 articles / papers were found from on line sources like Emerald, Scopus, Ebsco, Google Scholar and Sciencedirect; and total 50 references were used in this paper along with profound investigation of SMEs by questionnaire and discussion.

Structured questionnaires and semi-structured interviews are often used in mixed method studies to generate confirmatory results despite differences in methods of data collection, analysis, and interpretation*(9)*. In the research methods literatures, questionnaires and interviews are seen as having differing and possibly complementary strengths and weaknesses. The questionnaire tool is usually seen as a more objective research tool that can produce generalisable results but the main area that may be threatened is faulty interpretation of question may lead to wrong result. According to Bryman*(10)* questionnaire research can be seen as over-reliant on instruments and thus, disconnected from everyday life, with measurement processes creating a spurious or artificial sense of accuracy. While interviews provide contexts where participants can ask for clarification, elaborate on ideas, and explain perspectives in their own words. But at same time, the interviewer can to lead or manipulate interviewee responses. So by considering all pros and corns, it is decided to use mix method for getting response from SMEs.

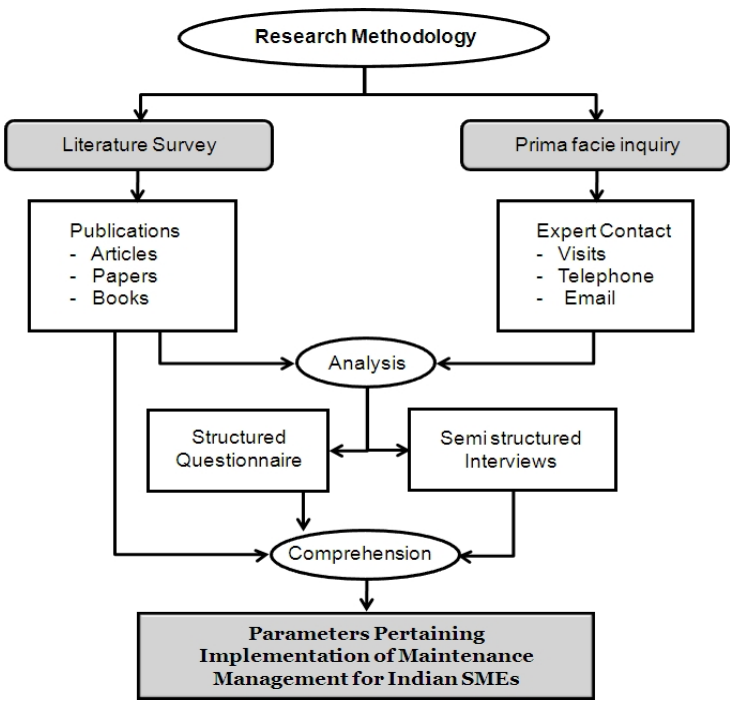


Fig. 1. Research Methodology

The data is gathered through questionnaire and interviews with multiple employees and through careful observations from 10 different SMEs. The employees have alike views on the challenges and on the solutions for challenges posed by internationalization process. The brief information of is below: Out of 10 SMEs:

- 7 SMEs were of make to order type and all were more than 10 years old.
- 5 MSMEs have 1 year of experience of Quality Management (Implementation of LMCS) and 5 are currently engaged in same

- 8 SMEs were certified for ISO 9000.
- 8 SMEs have standard manual for Maintenance related activities.
- No SMEs updates manual time to time and no SME follows Maintenance Vs Repair or Replace cost.
- In all SMEs, daily-routine maintenance was done by operators whereas maintenance people were called for any other special type of maintenance.
- 7 SMEs agreed to have entity to established and practised Maintenance Management Strategy or any other Quality Initiative.

4. COMMON PARAMETERS FROM LITERATURE REVIEW

The five common parameters identified from literature review are presented in table 1. The methodology of particular research for driving these parameters is minted second Column.

Table 1. Common parameters for successful implementation (from literature review).

Author(S) **Year** **Country**	**Quality Initiative** **Methodology**	**1.** Lack of Top management commitment Sincerity & Long term commitment	**2.** Training & Education; Literacy of worker	**3.** Mind set & Cultural aspect of workers Worker Involvement	**4.** Limited Resources and Resource Allocation	**5.** Human Resource Limitation and additional work/ multitasking
E. Aspinwall & M. Elgharib *(11)* 2013,**UK**	TPM Literature Review & Case Study		x	x	x	x
R. Singh *et al. (12)* 2013, **India**	TPM Case Study	x		x		
B. Shah, *(13)* 2012, **Malaysia**	TPM Case Study	x	x	x	x	
K. Ng *et al. (14)* 2011, **Malaysia**	TPM Literature Review	x	x	x		
K. Joshi *et al.(15)* 2008, **India**	TPM Literature Review	x	x	x		
Ahuja & Khamba *(16)* 2008, **India**	TPM Questionnaire	x	x	x		
M. Rodrigues & K. Hatakeyama, *(17)* 2006, **Brazil**	TPM: Interviewing operators & maintenance people	x	x		x	x
Seth & Tripathi *(18)* 2005, **India**	TQM & TPM Questionnaire	x	x	x	x	
F. Chan, *et al.(19)* 2005, **Hong Kong**	TPM Case Study	x	x		x	
J. Hannson & F. Backlund *(20)* 2002, **Sweden**	TQM, TPM or RCM Literature Review & Case Study	x	x			
C. Bamber, *et al. (21)* 1999, **UK**	TPM Case Studies & Literature Review	x	x	x		
K. McKone *et al. (22)* 1999, **USA + Japan and Italy**	TPM + TQM, JIT & EI Questionnaire	x	x			
L. Jorge et al. *(23)* 1997, **Caribbean**	TPM Case Study		x	x	x	
F. Chen *(24)* 1994, **USA**	PM; Questionnaires, plant visit & discussions	x	x			

Above parameters may be briefly summarised as:

- **Lack of top management commitment and sincerity for QM:** Top management act as a driver of Quality Management (QM) implementations, creating values, setting goals and systems to satisfy customer expectations and to improve an organization's performance. Establishment of quality culture is not over night process, long term commitment of top management is prerequisite.
- **Training and Education:** If employee do not receive formal, systematic training for QM (or maintenance management also), then employee involvement in framework is not at all effective. Companies need to view training cost as investment instead of cost. Particularly in country like India, literacy of worker is big issue.
- **Mind set and Cultural aspect:** In conventional work culture of Indian SMEs, to change the rigid mindset of employees is the biggest challenge and it require well defined and designed tactic.
- **Limited Resources:** SMEs work with limited capital amount and investment in a domain - up gradation in existing production and maintenance facilities require additional resources. Hence resource allocation is always critical issues.
- **Human Resource Limitation:** SMEs works with limited workforce including top management and multi tasking allotments detract the focus from QM (or maintenance management) and focused improvement process.

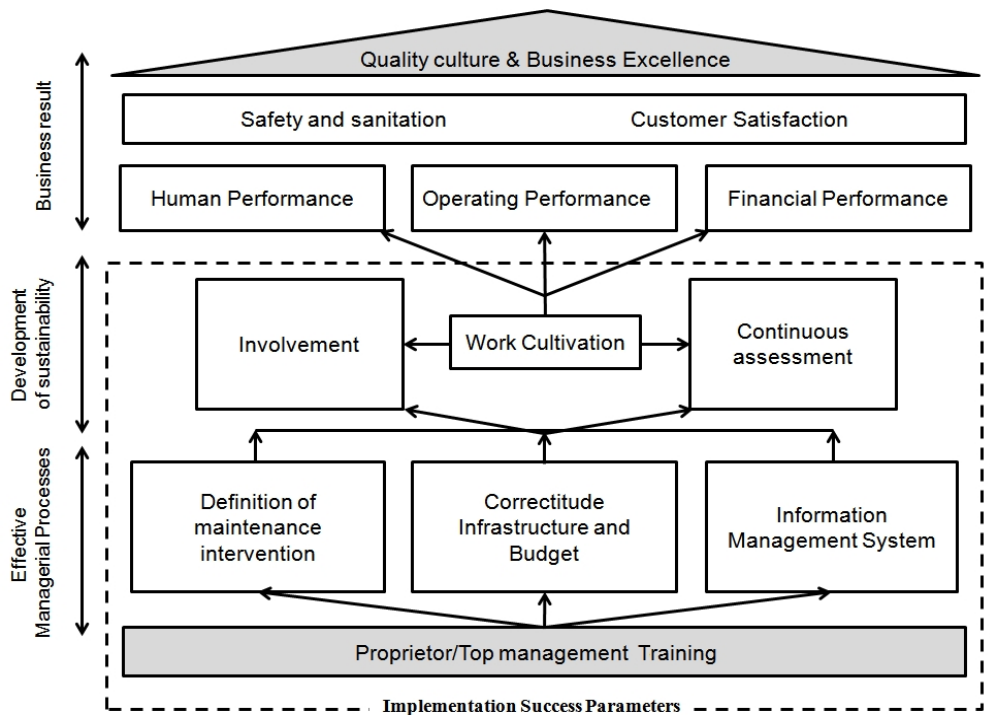


Fig. 2. Framework for Successful Implementation (Comprehensive Success Parameters)

5. COMPREHENSIVE PARAMETERS FROM SMES' CURRENT PRACTICES

This research has presented the differences between the comprehended parameters for the firm compared to the parameters given in literature. The parameters founded from current practices of SMEs along with literature review are classified in three stages: **Proprietor Training (S0), Effective Managerial Processes (S1) and Development of Sustainability (S2)**. These comprehensive (independent / input) parameters are grouped as shown in figure 2. (The performance / output parameters are not included in this paper.)

Stage 0. Proprietor/Top Management Training (S0):

B. Shah*(13)*, K. Ng *et al.(14)*, I. Ahuj and J. Khamba*(16),* M. Rodrigues and K. Hatakeyama*(17)* and many other studies over TPM-TQM have found training and education of employee as one of the most important parameter. A. Gupta and R. Garg*(25)* stated that managers need to learn to plan for higher equipment effectiveness and implement improvements aimed at achieving zero breakdowns and zero defects. In presented frame work of implementation, Proprietor / Top management's exemplary training is considered as the foundation. Especially in SMEs, proprietor or top management has to address much larger variety of functions. In such case, training and knowledge of following topics may prove crucial not only for QM implementation but also for growth of business.

- Strategic Communication
- Integrated Goal Setting
- Effective Team Building
- Conflict Management
- Implementation Policies
- Budgeting and Controlling
- Assertiveness Skills
- Value System Atribution

Parameters:

1. Training of proprietor/top management for basic MMS concepts and benefits.
2. Data recording and interpretation training
3. Vocational training and retraining frequency
4. Knowledge of philosophy and methodology of productive maintenance.
5. Knowledge of maintenance guidelines, methods and execution plan

Stage 1. Effective Managerial Process and Strategic Management (S1):

The effective integration of maintenance function with engineering and other manufacturing functions in the organization can help to save huge amounts of time, money and other useful resources in dealing with reliability, availability, maintainability and performance issues*(26)*. The maintenance manager / proprietor needs to set policy, facilitate the determination of targets and help operator/craft man to get on work. The effective managerial process includes coordination and renovations of existing facilities and establishment of new strategy to call quality culture.

Parameters:

1. Specific work-skills training (Training for standard way of utilization machines / resources)
2. Development of smooth cross-functional relationship (congenial interpersonal relationship - Including compatibility and co-ordination)
3. Scheduling lean maintenance activities.
4. Establishing standard procedure of maintenance (Maintenance framework).
5. Strategy for 5W1H for source of contamination (and thus inclusion of maintenance in production strategy)

S1.1 Definition of maintenance intervention:

The need for maintenance is predicated on actual or impending failure - ideally, maintenance is performed to keep equipment and systems running efficiently for at least design life of the components *(27)*. The design life of most equipment requires periodic maintenance and failure in doing so shorten the operating life of equipment*(28)*. Two maintenance interventions - Reliability-centred Maintenance and Total Productive Maintenance - have seen significant industrial application over the last decade*(29)*. Selection of maintenance strategies depends on the system of production, level of automation and schedule of a production system*(30)*. The various types of maintenance innervations are shown in figure 3.

Parameters:

1. Development of manual for all kind of maintenance interventions and periodic up-gradation
2. Establishment of calendar for inspection and maintenance activities

3. Use of technology in Planned Maintenance (use of vibration, oil analysis, infrared or thermal technology, ultrasonic, or optical or laser alignment for prediction etc)
4. Definition of standard MTTF, MTBF and MTTR
5. Distribution of machine into different areas/section and make category as for maintenance person (Internal and External) and as per time span (e.g. daily, weekly, fortnightly etc)
6. Definition of standards for inspection
   (1. Nuts and bolts; 2. Proximity switches; 3.Valves; 4. Gauges and dials; 5. Lubricators; 6. Level indicators; 7.Belts safety devices; 8. Motors and couplings; 9. Pipe joints etc)
7. Definition of all points for regular tightening, cleaning, lubricating etc (regular inspection)
8. Forecast for spare-parts, consumables and other inventories
9. Assignation of work order to production personnel/maintenance personnel/other.
10. Multiple task allotment (and shift wise distribution, if)

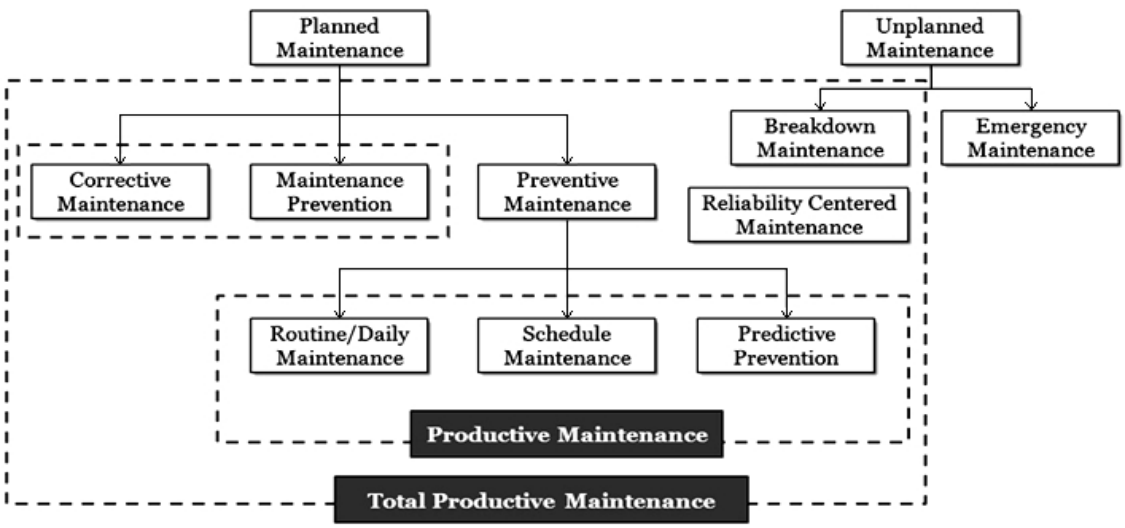


Fig. 3. Various types of maintenance

S1.1.1 Visual Management

Visual management has emerged during the past decades within manufacturing and service organizations, as a system that through visualization enables the employees to better understand their role and contribution*(31).* Visual technique helps against lack of attention during maintenance. This may applied to operating range marking on gauges direction of flow marking, direction of motor rotation, maximum operating parameter, match marking on parts, valve normally kept closed and open in operation etc*(15).*

Parameters:

1. Visual management by colour coding, stickers, matching marks, range on gauges in colour
2. Visual display of quality-maintenance information at work stations
3. Visual display of inspection route map
4. Charts showing schedule compliances on the shop floor

S1.2 Correctitude Infrastructure and Budget:

Strategic investments in the maintenance function can lead to improved performance of manufacturing system and enhance the competitive market position of the organization*(32).* The lack of proper infrastructure affects businesses at every level and ends up being a deterrent to healthy growth. Availability of basic infrastructure including Power, Water and Waste Management is a challenge for

enterprises across all cities of India*(33)*. In addition, there is a shortage of skilled human recourse and the existing human resource is also not satisfied with top management as basic amenities are unavailable.

Parameters:

1. Budget allocation for meeting minimal necessities infrastructure (like clean drinking water, meal, sanitation arrangements etc)
2. Budget for health check-up facility and Mediclaim (bottom level employee)
3. Budget for Maintenance Activity (Spares / Consumables cost, Repair costs from the preventive maintenance, Cost of External Human Resource)
4. Workforce up gradation - Training cost
5. Investment in a domain - up gradation of existing production facilities

S1.3 Information Management:

The modeling of *information* for maintenance has become important, especially with the introduction of proactive maintenance management. M. Kans and A. Ingwald suggested that common database supports the development of applications e.g. for monitoring the performance of a production process, for deciding the most cost-effective maintenance policy or for simulating possible maintenance solutions*(34)*.

S1.3.1 Information recording

D. Sanga reported that SMEs have negative attitudes in compliance to proper record keeping for their business. And suggested that the concerted effort should be made by the government and other stakeholders to set out proper guidelines for record keeping and SMEs owners must be trained on key skills for records and information management*(35)*.

Parameters:

1. Detail information of the machine and tools for maintenance activities (e.g. Brand model, Serial number, Code, Resource person etc)
2. Information classification according to capacity and priority – (Divide the machine/parts in category and give the rank)
3. Information related to maintenance intervention (for corrective, preventive and productive maintenance - tracing total maintenance expenditures and costs)
4. Work order information
5. Tracing the information of current condition and standard condition
6. Information of machine downtime and small stops as a measure of efficiency
7. Consumables and non consumable for maintenance management

S1.3.2 Information Management System

Computer Managed Maintenance System (CMMS) is integrated set of computer programs and data files designed to help in the better management of maintenance by organizing and tracing the myriad of data required for running maintenance operations effectively. The benefits of CMMS include reduced cost, better organizational methods, reduced paperwork, improved communications, equipment/asset life, reduced energy costs, improved environmental controls, and improved record keeping for meeting regulatory requirements*(36)*. Today numbers of such web based or computerized information management systems are available. The common features of such system are shown in figure 4. O. Durán*(37)* proposed fuzzy AHP approach (Analytical Hierarchy Process approach) for the selection of CMMS as there is no article has considered ambiguity and uncertainty factors when selecting effective CMMS.

Parameters:

1. Data storage in easy and in structured manner
2. Quick accesses - Information is readily available to operators.
3. Easy updation of manuals

4. Availability of the cost of quality data in the division (including data of scrap/wastage data)
5. Easy tracing - the cost of maintenance Vs Failure (and tracking of all other kind of cost)
6. Analysis/Audit of maintenance activities

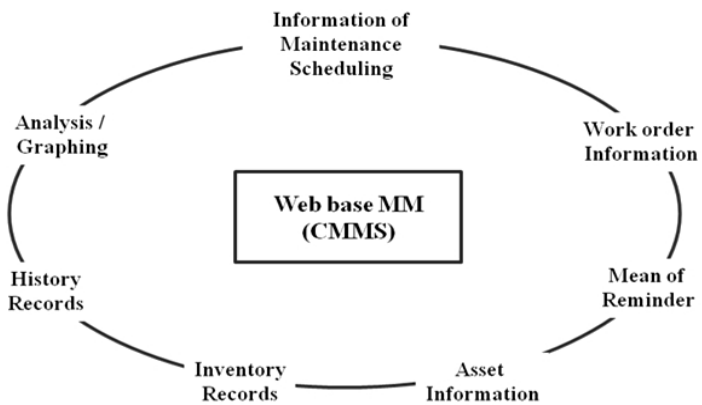


Fig. 4. Common Feature of Web based / Computerized Maintenance Management

Stage 2. Development of Sustainability (S2):
The term 'Sustainability' has become a commonly used and widely accepted expression in both an ecological sense and a business sense *(38).* The beginning of the third millennium witnessed significant changes in organizations strategy in terms of focusing on sustainability as a mean of organizational development*(39, 40).* To achieve sustainability, organizations must change their business models and undergo a process of unprecedented organizational change, prioritizing social responsibility and business ethics*(41).*

S2.1 Involvement:
In the various studies related to TPM execution and sustainability of quality initiative, '*Involvement'* of top management and employees is found one of the critical parameter for success *(13, 16, 15).* And it is most crucial in case of SMEs as the priority of proprietor/top management is always those issues which are directly related to 'Business' not related with implementation. No efforts for QM /MMS yield the desired result until and unless complete involvement. Lack of top management involvement leads to less involvement of employees and ultimately it result in failure.

Parameters:
1. Role definition of top to bottom level employee
2. Full and continued support from top management for MMS. (Long term commitment)
3. Involvement by continuous monitoring and review of progress of maintenance schedules
4. Encourage craftspeople / production operators to work together on issues

S2.2 Continuous Assessment
The performance measurement for production process is very important for sustaining firms. Managers need to make decisions from this correct assessment *(42)*. Most organisations that do not devote sufficient attention to their human capital, particularly in the measurement of quality and effectiveness, usually under-utilise the talent of their workforces. So it is wise for proprietor / managers to invest in assessment *(43)*.

Parameters:
1. Development of performance standards for assessment - Method of assessment
2. Frequency of assessment - Continuous and formative assessment

3. Evaluations of positive and negative attributes of employee
4. Rewarding / unrewarding of employee
5. Guidance after assessment.

S2.3 Work Cultivation

The organizations can assure *sustained performance gains* and develop *quality culture* by cultivating the work attributes so that withstand against continuous market challenges.

Parameters

1. Degree to which MMS is considered as quality improvement and as a way to increase productivity (Creation of quality culture)
2. Encouragement and guidance to share opinions and ideas about improvements
3. Effective integration of maintenance function with engineering and other manufacturing functions (Interdisciplinary approach of maintenance)
4. Establish relation between product/process characteristics and quality characteristics
5. Creation of benchmark for maintenance organization (including self benchmarking)

S2.3.1 Strategic collaborations

SMEs managers usually show a traditional individualistic attitude*(44)*. Unfortunately many managers have not noticed yet, that the great asset of the companies is their collaborators*(17)*. Small businesses often suffer in globalized markets from fierce competition in terms of product, labour and finance. On the contrary, being an independent but cooperative partner of network could be a real possibility to obtain advantage for the SMEs*(45)*. I. Deschamps, et al.*(46)* found that SMEs do not care about understanding and improving their capabilities about intellectual property and are not equipped with adequate tools and best practices for managing the overall collaborative mechanisms in general.

Parameters

1. Research and intellectual properties collaboration
2. Collaboration and coordination in MMS implementation (Cluster concept)
3. Collaboration in external human resource
4. Collaboration in product manufacturing between specific SMEs
5. Collaboration for research between SMEs and Universities / College

S2.3.2 Employee empowerment and satisfaction

Employee empowerment is crucial in order to create commitment in the employees' minds to this end; the management should involve all employees in defining work objectives, specifying how to achieve them and setting ambitious but realistic targets*(43)*. Shortage of skill worker is common problem found during study. In such situation, job satisfaction proves a highly salient antecedent of turnover intent *(47)*.

Parameters:

1. Worker's legal empowerment
2. Involvement of employee in target setting activity
3. Frequent interaction – employee orientation and gathering
4. Policy for family betterment (Help for child education)
5. Financial assistance in case of emergency
6. Incentive scheme and reward

S2.3.3 Safety, Sanitation and Environment Control

The objective of MMS is not limited to high rate of quality production but it is extended to creation of healthy and safe environment. After all, it calls for sustainable development which involves more than only consideration of machines but it seeks enhanced safety and health of personnel in line with improving environmental factors.

Parameters

1. Hazard awareness and safety training
2. Provision of safety gadgets like hand glows, glasses, shoes, apron etc (at work spot)
3. Use of displays and warning lamps/signals
4. Maintain the state of being clean and conducive to health

S2.3.4 Housekeeping

Housekeeping helps to improve morale by improving the physical surroundings of the worker and prevent the damage to the equipment due to extraneous particles. 5S is one most popular and widely used housekeeping technique. M. Moradi studied the effect of 5S on each pillar of TPM and concluded that the execution of 5S is an essential prerequisite of implementation of TPM. The 5S, in particular, can improve the Overall Effectiveness of Equipment (OEE).

Parameters:

1. Classify the items, fixtures, tools, papers, books etc, into different categories based on daily/ weekly/quarterly/monthly use.
2. Fix the location of all items with provision for quick and easy retrieval (less than one minute)
3. Schedule for disposal of unwanted items and wastage including files-papers etc.
4. Specify all gangways and machine areas and mark them clearly
5. Keep machines and workplace clean (daily cleaning schedule and assign clear cut duties)
6. Standards procedures for operation (machine and process)

S2.3.5 Financial Capabilities and Government Policies

SMEs are financially inept and harbour poor financing arrangements. Financial inadequacy is thus a major hindrance to the adoption and subsequent implementation of successful lean within SMEs*(49)*. They fear that the application of lean or other quality initiative within any organisation requires financial resources to hire consultant, as well as to aid the actual implementation of such ideas. Imparting greater vitality and growth impetus to the SMEs in terms of output, employment and exports and instilling a competitive culture based on heightened technology Government of India has launched many programs. Various relevant schemes like ASIDE, Credit Guarantee Fund Trust Scheme, Cluster Development Programme, Credit Linked Capital Subsidy Scheme, Technology Upgradation Scheme being operated by different Ministries will be suitably dovetailed for the benefit of Micro, Small and Medium Enterprises*(50)*.

Parameters:

1. Awareness for different schemes for SMEs
2. Regional association for cooperation with government for research and improvements
3. Rising the capability by financial assistance from possible sources

6. CONCLUSION AND SUMMARY

The driving force behind this work is the desire of Indian SMEs to improve their productivity and profitability. This paper has described the parameters that assure the successful implementation of sustainable Maintenance Management Strategy within Indian SMEs. The outcomes of questionnaires and interviews suggest that successful implementation of quality initiative is possible within short time frame as SMEs have some inherent advantage of size and leadership but current practices is not leading them to long term sustainable quality culture. The reasons are summarized below:

- Priorities of proprietor / top management is differ
- Deficiency of formal procedure and disciplined approach
- Lack of skilled human resources
- Lack of data recording and analysis system
- Lack of experience of using Consultants and Collaboration

It is concluded that proper training and education is necessary not only for changing the mind set and established quality culture but also eliminate continued scepticism about the benefits of maintenance management in SMEs. Effective Managerial Process, Correctitude Infrastructure, Proper allotment of budget, and CMMS are necessary attributes for implantation success. Involvement, Continuous assessment, Strategic collaborations, Employee empowerment and Satisfaction, Creation of safety and sanitation by housekeeping, Financial capability and Government policies are key factor for development of sustainability.

This prima facie research would provide SMEs with indicators and guidelines to develop their own sustainable Maintenance Management Strategy by emphasising critical parameters presented in paper so that one can assure the sustention of various lean principles and achieve world class manufacturing.

ACKNOWLEDGEMENT

The authors like to thank Surat Engineering Vikas Association (SEVA) for providing the platform for this research and facilitating data collection. The authors appraise SMEs' participation as they have provided all necessary information related to this study. The Authors would like to thanks exam panel of RK University for giving constructive suggestions and providing guideline to enrich the content. The authors wish to acknowledge the anonymous reviewers for accepting this paper for International Conference on Research and Entrepreneurship organized by R K University, Rajkot (India).

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Entrepreneurial Potential of a Pen-shaped Plasma Torch

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ABSTRACT

The paper describes a new concept of the non-thermal pen-shaped plasma torch. Plasmas have found applications in a variety of areas like plasma welding, melting, cutting, nitriding and treatment of polymers and textiles. It is being used extensively nowadays for a variety of industrial applications. The hydrophilic properties of plastic strip and polyethylene can be enhanced by surface treatment done by plasma torch. This device has simple construction and has been efficaciously used for the same in past. Conversely, the present status of the device is still not feasible for practical applications because of its limited effective surface treating area. In this vision, the paper present a case of development of the new design of the "pen-shaped atmospheric plasma torch", which is efficient and necessary. It is anticipated that entrepreneurial potential is huge in terms of enhancing the existing potential of surface treatment industry and new product development.

SUMMARY

Design and development of pen-shaped plasma torch.

Keywords: Plasma, Plasma-troch, Pen-shaped, Surface treatment, Hydrophilic

INTRODUCTION

Plasma is called as 4th state of matter, unlike from the gaseous states, liquid and solid which is a high temperature ionized gas. Processing at atmospheric pressure is favored on industrial scales due to the simplicity and viability (*1*).

So, Plasma is a gas wherein electrons, ions, and neutral gas species co-exist. The mixture of charged and neutral gas species is unique and responds to electromagnetic forces (*2*).

Plasmas can be classified in different ways according to its many characteristics, such as approximations of the model which defines them, density, degree of ionization and temperature.

Hot plasma (thermal plasma) is that which approaches a state of local thermodynamic equilibrium. A hot plasma is also known a thermal plasma. This plasmas can be formed by flames, electric sparks and atmospheric arcs.

Cold plasma (non-thermal plasma) is that which ignores the ions thermal motion. As a result, only the electric force is considered to act on the particles. The pressure force and the magnetic force can be ignored in case of non-thermal plasmas. Examples of cold plasmas includes the fluorescent tube flow discharge and ionosphere.

Plasma	**State**	**Example**
Thermal plasma (Quasi-equilibrium)	$T_{electron} \approx T_{ion} \approx T_{gas} \leq 2 \times 10^4 K$ $n_{elements} \geq 10^{20} m^{-3}$	Arc plasma, Plasma torches, RF inductively coupled discharges
Non thermal plasma (Non-equilibrium)	$T_{electron} >> T_{ion} \approx T_{gas} = 300$ to 10^8 K $n_{elements} \approx 10^{10}$ m^{-3}	Glow discharge, Corona, Atmospheric pressure plasma jet torch

Table 1. Classification of plasma.

Thermal plasmas are described by proximate equality between neutrals, ions and electrons or by an equilibrium. These plasma sources gives a high flux of heat and are predominantly used in areas such as waste material treatment by plasma and plasma material processing. By using microwave devices and plasma torches, the commonly used thermal plasmas can be generated. Due to high temperature thermal plasma can process even the most noncompliant wastes including nuclear elemental waste, industrial waste, medical waste, municipal solid waste, toxic waste, bio-hazard etc. in due course reducing environmental pollution caused due to them. But there are several technical applications wherein the high temperature characteristic of thermal plasmas neither anticipated nor necessary. Thermal plasmas in certain cases even becomes prohibitive. In such application, cold plasmas become more appropriate (*3*).

Non-thermal plasmas (cold plasmas) are those in which the neutral elements and plasma ions maintains approximate room temperature. Cold plasma produces active (energy full) electrons instead of heating the whole stream of gas because the most of the coupled electrical energy is predominantly controlled to the electron elements of the cold plasma. The approximate room temperature characteristics of the neutral elements and ions of the cold plasma provides the opportunity of using non-thermal plasmas for low temperature plasma application and for the treatment of bio-logical tissues and polymers that are very

heat sensitive. There are remarkable potential to employ these cold plasma sources in a wide range of applications (*3*) and this is possible due to its extraordinary characteristic features of cold plasma that includes high selectivity, presence of reactive species, a strong thermo dynamic non-equilibrium nature and of course low temperature of gas.

INDUSTRIAL NEED OF PLASMA

Plasma arc torches are being applied today as unique heating tools in industrial surface treatment processes. The many benefits of using plasma torches are being demonstrated in different production plants and prototype equipment around the world. And the successful demonstration of this versatile, low-mass, controlled high-enthalpy, selective-atmosphere, heat source is generating new applications.Industry today is benefiting from plasma heating and will in the future derive more benefits from this proven heating technology. There are many different types of plasma arc torches available. Single torch power ratings range from 2 kilowatts to 60 megawatts. The plasma heaters at the low and high ends of the power range are employed in the universities and in the national research laboratories for basic scientific studies. The industrial sector, with its emphasis on durability and economy, has adapted only the plasma torches in the range from 0.25 kW to 8 mW depending upon the application. The operating life of plasma systems is very important in industry for economic reasons. Industrial processes that use plasma torches must be capable of many hours or weeks of continuous operating life, unlike the several minutes of operating life demanded of the 60-mW plasma heater that is used for re-entry heating simulation (*2*).This paper presents the state-of-the-art of the plasma torch and discuss their industrial uses. But, the paper will place emphasis on non-thermal plasma torch only due to research scope of the author.

ENTREPRENEURIAL POTENTIAL OF NON-THERMAL PLASMA TECHNOLOGIES

Recently, there has been a marked tendency to use non-traditional technologies for the material production, surface treatment and waste utilization of different origin. Using the energy of plasma flows in industry has allowed us to modify old and develop new technologies. In particular, this concerns surface treatment and chemical industry. Operated and controlled plasma heating allows us to obtain an efficient mode of the technological process, ensuring the maximum useful output under minimum specific expenses or cost of material and energy. Besides, high-temperature heating plasma by flows creates conditions of strong disbalanced, when high energy particles participate under the moderate middle-mass temperature in the working process. New knowledge of these processes has allowed the researchers to create a new technologies in the field of surface treatment industry (*4*).

The cold plasma technique uses cold gases to disinfect the surfaces of packaging or food products. The technique has the potential to inactivate micro-organisms on the surface of products and packaging materials at low temperatures. Non-thermal plasma is drawing a lot of consideration from the industry belonging to food. Hardly a surprise, because many cleaning options are not heat-resistant, cleaning with water is expensive and chemicals are often out of the question. But gas reaches every nook and cranny. Food and bio-based research has many years of experience in cold plasma processing, including microbiology, product research, technology development and process impact.Plasma technologies have gained a new base in surface and coating industries. The main essential uses of these technologies are;

– Theoretical generalization of research results in the field of plasma chemistry, building material processes and surface treatment with the aim of processing materials with the help of low-temperature plasma or cold plasma.

– Packaging materials dis-infection: Packaging materials and vegetable microorganism can be inactivate by using non-thermal plasmas. Particularly for temperature sensitive products, this can have a clear benefit compared to heat treatments.

– Food products dis-infection: non-thermal plasmas can be used for inactivation of both bacteria and vegetative cells that are located at the surface of a food product. The appearance and quality of the food does not effected as a result of low temperature treatment. Research and development is vital for scaling up and implementation of cold plasma since non-availability of an industrial equipment at the present.

CURRENT STATUS AND APPLICATION OF PEN-SHAPED PLASMA TORCH

The atmospheric pressure plasma has various applications in surface treatment and coating technologies, including the pre coating adsorbates removal (*5, 6*), anti-bacteria (*7*) and the hydrophilic property improvement of the polymer materials (*8*).The hydrophilic properties of plastic strip and polyethylene can be enhanced by surface treatment done by pen-shaped plasma torch. This device has simple construction and has been efficaciously used for it in past. Conversely, the present status of the device is still not feasible for practical applications because of its limited effective surface treating area (*9*).

The reported uses of pen-shaped plasma torch are as follows:

– Surface treatment of polymers like polyethylene and plastics to increase hydrophilic properties.
– Improve printing ability of polyethylene film (*10*).
– De-coloration of dye solution of textile industries.

MOTIVATION BEHIND RESEARCH ON PEN-SHAPED PLASMA TORCH

Literature review itself shows that the non-thermal plasma technology has various industrial applications as mentioned above. These technologies to become gradually commercially worthwhile in the upcoming days. But, it is also reported that the present status of the plasma torch is still not feasible for practical applications, hence in this vision it is necessary to develop novel design of the pen shaped plasma torch.

MATERIAL AND METHOD FOR NEW CONCEPT OF PEN-SHAPED PLASMA TORCH

It is expected that some modifications in the existing design parameters of the pen-shaped atmospheric plasma torch system would enhance the effective treating area and thus would fulfill the prerequisite of practical industrial applications. The subsequent discussion shows new research methodology and materials for efficient design of the pen-shaped atmospheric plasma troch. The major focus are on feed stoke gas from monatomic to diatomic because the diatomic gases have higher specific heats than monatomic gases; the cathode material from SS to tungsten because of its low thermionic work function and higher free electron emission capacity which leads to perfect ionization process the goal of any plasma generation method (*11*) and the anode material from SS to copper because of its higher thermal conductivity. The major change in construction of the pen shaped plasma torch is introduction of arc stabilization method. The existing design has straight flow of driving gas but in new design the swirling effect to the flow pattern of the driving gas shall be introduced for stabilization of the plasma arc. The erosion of the anode can significantly reduce by application of swirl flow of the gas because it keeps the arc plum in the center of the nozzle and rotates the arc and anode attachment point (*12*). This shall lead to prolonged torch operation for better surface treatment of the component.

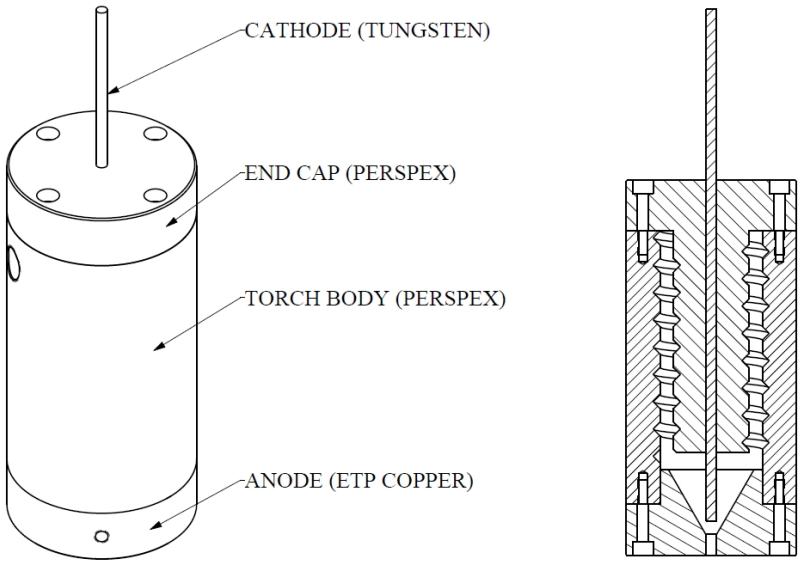


Fig. 1. New concept of pen-shaped plasma torch.

RESULTS AND DISCUSSION

Based on state of the art report, it can be concluded that the non-thermal plasma technology has various industrial applications and this technology to become gradually commercially worthwhile in the upcoming days for surface treatment and coating industries. Due to the simple configuration and respectable treating performance of the pen shaped plasma troch the entrepreneurial potential of the device to become powerful equipment for surface treatment in future is highly expected. Nonetheless, the development of the pen shaped plasma torch at the present stage is not feasible for practical applications because of its limited effective surface treating area. Therefore, further investigation to improve the effective treating area of the pen shaped atmospheric plasma torch system is very crucial. Through the study of influence by new design parameters and materials the effective treating area could be increased. Nowadays industries requires cost effective and prolonged torch operation in this competitive market. It is anticipated that newer design will be able to satisfy this industrial requirements. The literature also reports that entrepreneurial potential is huge in terms of enhancing the existing potential of surface treatment industry and new product development.

CONCLUSION

The literature supports that entrepreneurial potential is huge in terms of enhancing the existing potential of pen-shaped plasma torch device in surface treatment industry and as a new product development. For that reason it is worthwhile to do research on pen-shaped plasma torch.

ACKNOWLEDGEMENT

The author would like to acknowledge Dr. Ajitkumar N. Shukla (Director), RK University, Rajkot, Gujarat, India, for providing the valuable support and guidance during the research.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

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RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

Another look at the foundry sand quality for the Furan No-bake mould system

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ABSTRACT

In current global market quality of product places a very vital role for customer satisfaction. It is a challenge for foundry industry to remain competitive in terms of cost & quality. Nowadays Furan No-Bake binder system is increasingly used in foundry applications for achieving good dimensional accuracy. The quality of the cast products produced using this system mainly depends on the properties of the mould. Though there is a good dimensional accuracy, one major defect repetitively observed in FNB casting is sand inclusion. Here the attempt has been made to avoid this defect by considering various parameters affecting this defect. Sand inclusion is mould related defect and quality of mould basically depends on type and property of sand. In this work the various sand tests are performed to get a quality mould. It has been observed that Loss on Ignition (LOI), Grain fineness number (GFN), and Scanning Electron microscopy (SEM) tests give better prediction about sand inclusion defect.

SUMMARY

Sand tests are performed to evaluate major casting defect.

Keywords: SEM, Resin, Furan, Foundry, Catalyst, LOI

INTRODUCTION

There are three main classes of binder systems heat activated, no bake and cold box. This paper is mainly dealing with no-bake furan binder. Furan No-bake processes is binder system for producing cores, core moulds or plain moulds. Furan resin is made of furfuryl alcohol and its most important application is foundry. Since this binder system does not require a baking cycle to generate practical mechanical strength the furan binder has been acknowledged as the first true no-bake binder.

The no-bake furan resin bonded sand, with which self setting mould and core can be achieved at room temperature, is characterized by high strength, high dimensional accuracy, fast hardening rate, high production efficiency *(1)*. Therefore, furan resin is widely used in casting practice since in different countries. Nowadays due to better quality and environmental aspect our Indian foundries are also moving towards such sustainable solution to remain competitive.

The sand in a no-bake mould is mixed with a liquid resin and hardens at room temperature *(2)*. To produce a quality mould, foundry sand should be of uniformly sized, high quality silica sand which will combined with a binder and used to form moulds. The physical and chemical characteristics of foundry sand will depend in great part on the type of casting process and the industry sector from which it originates. In modern foundry practice, sand is typically recycled and reused through many production cycles. Industry estimates that approximately 100 million tons of sand is used in production annually of that 6 - 10 million tons are discarded annually and are available to be recycled into other products and in industry. It is important to purchase high quality size-specific silica sands for use in their molding and casting operations.

Objective

In our country No-bake furan binder system is adopted by some limited industries. One of such industry, I have visited is Krislur Castomech Pvt. Ltd. Founded in 1980, it is one of the most promising and reputed industry in terms of quality of sand casting. It is situated at GIDC Bhavnagar. The Industry has been certified by ISO 9001:2008. The main emphasis of the industry is production of motor body and its aligned parts.

To analyze defect rate in Furan No-Bake Binder (FNB) casting, past one year defect data was collected and analyzed through Pareto chart. Here Fig.1 shows a Pareto chart for one January month's rejection data. Same way other data were analyzed using Pareto chart and the same results were drawn.
It has been observed from above graph that the major defect which is faced by industry is sand inclusion. It was necessary to find out actual reasons behind the defects with use of Cause and Effect Diagram for analysis purpose. Fig.2 shows the actual photograph of motor body casing in which sand inclusion defect is present.

The defects need to diagnose correctly hence Cause and Effect Diagram has been prepared as shown in Fig.3 to identify and classify the reasons that are responsible for defective casting production and lower productivity of organization *(3)*. It is also important to note that the mechanical strength of resin bonded core depends on the adhesive force between the resin binder to sand grains and the cohesive force of the resin film itself *(4)*. A fish bone diagram is prepared to find out reasons behind this defect.

This figure determines the potential cause which causes defects. From causes and effect diagram we can identify remedies behind defect. It has been identified that sand inclusion is a mould related defect. Major parameters affecting this defect are sand properties and strength of the mould. Hence it is important to consider physical sand test to improve casting quality.

SAND PROPERTIES THAT AFFECT QUALITY OF THE CASTING

The FNB molding/core sand mixture used consists of three ingredients, namely washed silica sand, resin and hardener. Silica sands possess almost all the characteristics but it has some draw back like higher thermal expansion, lower chilling effect and lower refractoriness compared to chromites sands or Zircon sands *(5)*. Along with thermal expansion density, melting temperature, shape of the sand grain, pH values are also important properties looking to the following aspects. Density is important because the heat content will be higher if the density is higher. The melting temperature is important because the sand will be "wetted" more easy if the temperature is coming close to the liquid metal temperature. This will lead to penetrating and burn in of sand – metal. The shape of the sand grains, from round to angular, is important because it has an influence on the necessary amount of binder to get an equal strength. The pH is important because it will indicate the necessity for catalyst or even indicate that an acidic system is not applicable.

Regeneration, or reclaiming, is necessary to keep the chemical bounded sand system economic. The importance of reclaiming increases due to increasing difficulty and cost for dumping or re-using the "used sand" *(6)*. But, after using the sand once, a lot of reacted products (binder and catalyst) are present in the sand. By re-using the sand, various consequences occur as mentioned in Table.1.

Sand testing is essential to understand the properties of the resin bonded sand mould. Some of the most important sand testing is moisture, permeability, strength, compactibility and loss of ignition test *(7)*. In this study to avoid sand inclusion defect in furan no-bake sand GFN, LOI, SEM and compressive strength test are performed. These four tests are performed for one particular lot of sand and its effect has been observed on casting.

TEST PERFORMED TO AVOID SAND INCLUSION DEFECT

Sieve Analysis test

To produce high quality sand, we need a good grain distribution. This grain distribution together with the grain surface (specific surface) determines the necessary amount of resin and catalyst. Therefore the "AFS-number" or "GFN-number" and the "Specific Surface" must be evaluated *(8)*. Grain size and distribution of the base sand influence many properties of a molding mixture.

The grain size of the base sand influences the strength properties of bonded mixtures, an inverse relationship existing between compression strength and grain size in clay bonded sands. Table.2 shows the sieve analysis test data which was performed in Krislur Castomech Pvt. Ltd. on reclaimed sand.

Loss on Ignition

Loss on ignition is performed to test to measure amount of moisture or impurities loss when the sample was ignited. It also determines the presence of organic or other gas forming materials in the sand mixture. As per Fatta e.t. al. LOI gives an objective idea of the gas development that can happen and of the possible decrease in hardness / strength of the sand. LOI of the reclaimed sand is a useful check to determine the amount of combustible materials remaining on the sand after reclamation. These overall indicates the potential danger for gas defects in castings and the level of surface quality. The best value for the LOI of reclaimed sand is zero, but less than 3% is generally acceptable *(9)*.

The loss-on-ignition is the difference in weight before and after ignition of the sand sample. LOI is performed at an AFS-defined temperature as shown in Fig.4. The main method for determining LOI involves heating samples to a temperature at which organic materials volatilize and decompose. LOI measurement indicates the amount of combustibles in raw sand. In chemically bonded sand, they absorb binder and reduce its effectiveness. Thus, LOI measurements can provide essential information about the overall quality of a foundry's sand system.

SEM for Sand grain size

There are mainly 3 types of grain shape: “rounded”, “angular” and “sub-angular”. All three have different properties and behaviour. A well sorted sediments (e.g. particle size, shape, roundness and sorting) is one in which the grain are all about the same size. Particle roundness referred to the smoothness of a grain, regardless of its shape *(10)*.The angularity of sand is estimated by visual examination with a low power microscope and comparing with published chart shown in Fig.5.

A setup was prepared at M S University Baroda and SEM was performed wit help of electron microscope as shown in Fig.6 Rounded sand offer excellent permeability but lack any interlocking strength, in an unbounded state. In a bounded state require less binder though with a reduced permeability, but good flow characteristics.

Angular sand exhibit lower permeability though with superior interlocking strength when unbounded. In a bounded state require more binder to cover the higher surface area, and are prone to produce significant sand fines due to sharp corners breaking from mechanical handling.

Predictably sub-angular sands are a median between rounded and angular. Better permeability than angular, but not as good as rounded when unbounded, better interlocking strength than rounded, but not as well as angular. More binder is required than rounded, but not as much as angular, with less fines generated than angular, but more than rounded, and a similar mid-point of flow ability *(6, 11)*. It is common practice for foundries to use a mixture of sand shapes to achieve a balance of the above properties for their specific molding, handling and casting needs.

Compression test

The way in which the mould box is filled, does have a large influence on the final strength of the sand. When the molten metal is poured in the mould, the sand in contact with the hot metal starts losing its strength. At this stage the molding sand must possess the sufficient strength to retain the exact shape of the mold cavity and at the same time it must be able to withstand the metallostatic pressure of the liquid material. The principal special feature in the testing of furan sands is the significance of the time factor in the development of properties, and thus of the point in the interval from mixing to full hardening is considered as 1hr, 4^{th} hr and 24^{th} hr *(12)*.

Compressive strength test was performed using a universal testing machine (UTM) as shown in Fig.7 at Krislur Catomech Pvt. Ltd as per IS 1918:1966 *(6)*. This test was performed by considering resin percentages are considered as 0.80 % of sand and catalyst as 30% of resin, which is common practice of foundry industry.

RESULTS AND DISCUSSION

Sieve analysis test

AFS grain fineness number – is the average grain size, and it corresponds to the sieve number whose opening would just pass all the sand grains if all were of the same size. As per American Foundry Society

(AFS) two graphs can be plot, one is percentage retained and other is cumulative shown in Fig.8 and Fig.9.

LOI Results

As per AFS standard LOI test is performed at 980 ^{0}C with help of reclaimed furan no-bake sand and data was collected as shown in Table.3

Fig.10 shows time versus loss of percentage graph for above data. From the graph we can say initially with respect to time percentage loss will increase. When time reaches 140 min the loss in weight remain constant. Even when sand kept in oven for more than 2hr the loss on ignition remains constant. So the maximum percentage loss is 2.3 for respective sand which is within AFS limit *(12).*

This results show good LOI values because according to Brown., LOI must be kept. below 3%. The experiment and graph shows the higher the time of burning, the more organic material will burn and the the lower the loss of ignition will be.

SEM Results

Grain shape has a considerable influence on binder adhesion and quantity, due to the variance in grain surface area. Grain shape also influences mold strength and permeability. The finer the sand and the higher the specific surface and the more angular the grain, the more resin is needed.

Predictably sub-angular sands are a median between rounded and sub-angular. It offers better permeability than angular, but not as good as rounded when unbounded, better interlocking strength than rounded, but not as good as angular. More binder is required than rounded, but not as much as angular, with less fines generated than angular, but more than rounded, and a similar mid-point of flow ability *(12).*
So it is common practice for foundries to use a mixture of sand shapes to achieve a balance of the above properties for their specific molding, handling and casting needs.

In practice, sand grains contain mixed grain shapes, depending on origins of sub-angular to rounded grain mixture would be the best combination *(13).*
SEM Fig.11 shows the sand topology of FNB Mould. It reveals combination of Rounded and semi angular shape which give excellent permeability and flowbility.

Compression test results

Compressive test performed using universal testing machine at Krislur Castomech Pvt. Ltd. and data are collected at 1st hr, 4th hr and 24th hr as shown in Table.4. The factors that could compromise the strength values are mixing speed, time and surrounding temperature.

As per industries review this results are within a requirement.

CONCLUSION

Four controllable factors chosen for the experiments are LOI, GFN, Grain size and shape and Compressive strength of sand. The obtained results for this test shows that sand type, its quality and size are ineffective parameters to inclusion defect.

In Furan resin bonded sand moulds, two different ratios of resin and hardener were employed, and the maximum compression strength was obtained. As per Foseco Ferrous Foundryman's Handbook, Strength

of mold depends on resin type and addition. So we can say the factors that could compromise the strength values are mixing speed, time and surrounding temperature. As all test results are as per requirement, by considering above parameters again the test can be performed to find out reason behind defect.

After this research we can conclude that many parameters are affecting the performance of Furan No Bake binding system. For defect free casting especially sand inclusion defect, all the process parameters must be set according to the standard.

FIGURES

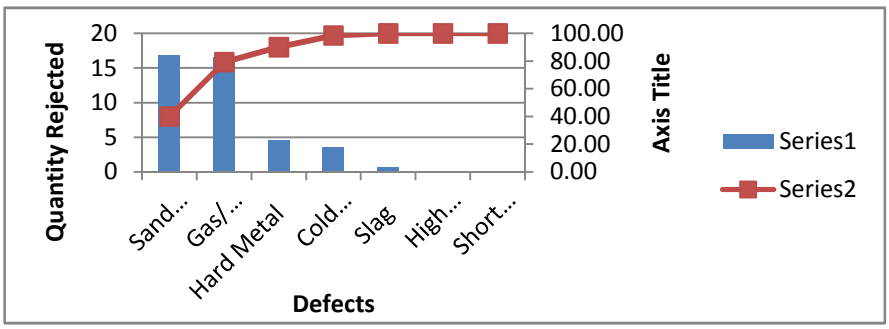


Fig.1 Pareto chart prepared for one month's rejection data



Fig.2 Sand inclusion defect in actual Motor Body Casting

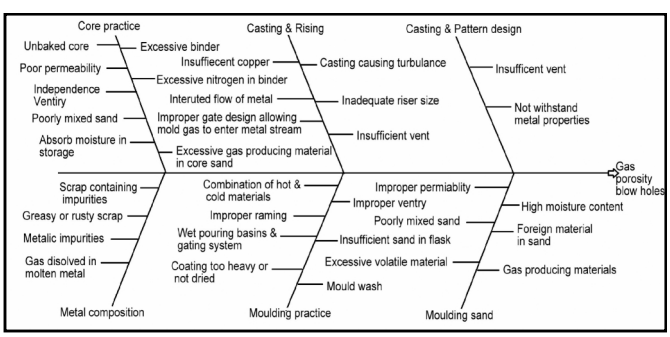


Fig.3 Causes and Effect diagram for Sand Inclusion defect



Fig.4 LOI test performed at Industry

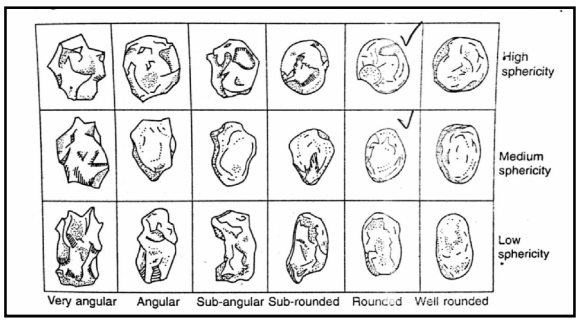


Fig.5 Sand Shape (Brown 1999)



Fig.6 Set up of Scanning electron microscope at MSU



Fig.7 Compressive strength test with help of UTM

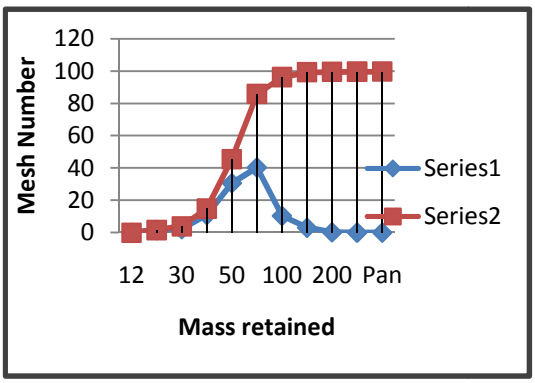


Fig 8 Mass retained on different Mesh No

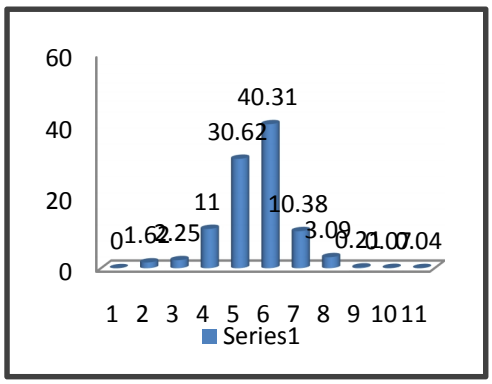


Fig.9 Column chart for Mass retained on different Mesh No.

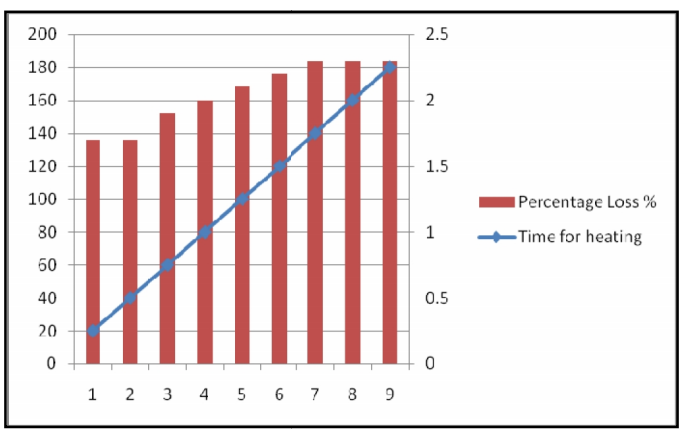


Fig.10 Loss on Ignition graph

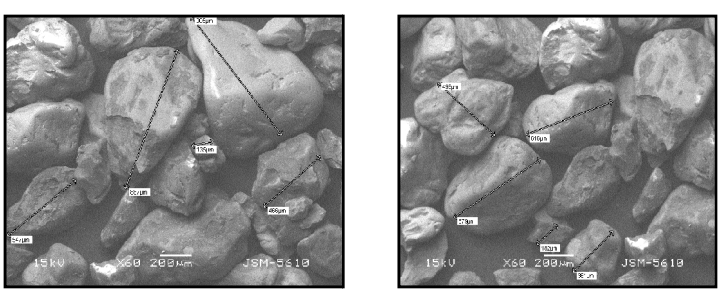


Fig. 11 SEM image of Sand

TABLE

Table.1 Consequences and its effect due to reclaimed sand

Consequences	**Its Effect**
Build up of resin particles	LOI increases
Build up of catalyst	pH decreases (needs less catalyst)
Sand grain gets smaller	AFS number increases
Requires less binder	Because specific surface increases and the roundness of the grains gets better
Build up of nitrogen	Problems with pinholes

Table.2 Sieve analysis test

Sr. No.	**ASTM mesh No.**	**Mass Retained (gm.)**	**Cumulative**	**% Retained**	**Multiplication factor**	**Product**
1	12	0	0	0	5	0
2	20	1.62	1.62	1.63	10	16.2
3	30	2.25	3.87	2.26	20	45
4	40	11	14.87	11.04	30	330
5	50	30.62	45.49	30.75	40	1224.8
6	70	40.31	85.8	40.48	50	2015.5
7	100	10.38	96.18	10.42	70	726.6
8	140	3.09	99.27	3.1	100	309
9	200	0.21	99.48	0.21	140	29.4
10	270	0.07	99.55	0.07	200	14
11	Pan	0.04	99.59	0.04	300	12
	Total ™ =	99.59		100	Total (Tp) =	4722.5
Fineness No. = Tp/Tmm =						47.42

Table.3 LOI test data

Sr. No	Time for heating (min)	Weight before (gm)	Weight after (gm)	LOI	Percentage Loss %
1	20	10.04	9.84	0.017	1.7
2	40	10.02	9.86	0.017	1.7
3	60	10.02	9.83	0.019	1.9
4	80	10.03	9.83	0.020	2.0
5	100	10.03	9.82	0.021	2.1
7	140	10.01	9.79	0.022	2.3
6	120	10.01	9.78	0.023	2.3
8	160	10.03	9.80	0.024	2.3
9	180	10.02	9.79	0.023	2.3

Table.4 Compressive test results

Resin		Catalyst		Sand Temp. ^{o}C	Compressive strength (gm/cm^2)		
Resin (%)	Resin (gm)	Catalyst (%)	Catalyst (gm)		1^{st} hr	4^{th} hr	24^{th} hr
0.80	18	30	10	45	7.48	16.10	20.71
0.80	18	30	10	42	7.23	16.25	20.24
0.80	18	30	10	42	7.51	16.22	21.60

ACKNOWLEDGEMENT

The authors acknowledge the help and support of Mr. Mahesh Madhnani, CEO Krislur Castomech Pvt Ltd.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th& Jan. 6th, 2016)

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RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

Performance Analysis of Equal Area Distribution Algorithm Implemented for Augmentation of LEACH – A Wireless Sensor Network Protocol

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ABSTRACT

Today's era is the era of energy efficient MEMS (micro-electro-mechanical systems). The mammoth amount of growth in the fields like Embedded Systems and Wireless Communication has opened the doors of the development of cost and power effective, multifunctional sensor nodes which are having capability to communicate untethered in short distance. In this paper we have implemented an Equal Area Distribution Algorithm to augment the performance of LEACH – A WSN Protocol. In the LEACH protocol the selection of CH is a randomized process. The implementation for Equal Area Distribution approach provides the fix number of CHs per round and which depends upon the remaining number of alive nodes. So it provides the required randomization for effective WSN and also maintains the advantage of Maximum Number of CHs per round. The proposed algorithm augments the working duration of the network, communicated data bytes in the network and required amount of energy.

SUMMARY

Equal Area Distribution approach is implemented for LEACH – A WSN Protocol.

Keywords: WSN Sensor 1, LEACH 2,Cluster Head Selection Algorythm 3,Equal Area Distributed Approch 4

INTRODUCTION

We are living in the era in which the development of Physics, Microelectronics, Control Systems and Material Science has led us to development of the MEMS – which is Micro, Electro, Mechanical Systems. Just like the livings, a variety of modern devices and systems totally depends upon the data captured from the real world around it. This sensory data is the outcome of the Wireless Sensor Networks and which indirectly depends upon the advances in the field of MEMS and Embedded Systems. The sensed data represents the physical or environmental conditions measured by the tiny sensor nodes which are the part of the Wireless Sensor Network. These nodes are capable enough to monitor physical entities like temperature, motion or vibration, pressure, or sound and send the collective information to the central computing system called the Base Monitoring Station.

The advancement in the field of MEMS has truly augmented the concepts of SoC, i.e. System on a Chip, and enabled us to design and manufacture the chips which are initially assumed to do only functions with logic now can sense and also can respond. MEMS has also played a vital role in the designing of the low power and high frequency transreceivers on chip, which are the stepping stones of Wireless Sensor Networks. The devices which are having capability to sense, process, communicate and actuate is the basic building block requires to form the sensing networks and are commonly called "mote". These motes are something with characteristics like cost effective, power efficient, sensor nodes, with multifunctional abilities, which are tiny in dimension and can also communicate without losses for little distances. These little nodes or motes, which has capability to sense, process the data and communicate the same, force the idea of network of sensors founded on cooperative work of a thousand number of nodes. Such network characterizes a noteworthy advance over old-fashioned sensors, which are organized in the following two ways:

- Sensors can be situated far from the definite phenomenon, i.e., roughly known by sense discernment. In this tactic, large sensors that use some multifaceted techniques to differentiate the targets from ecological noise are required.
- Numerous sensors that accomplish only sensing can be arranged. The locations of the sensors and communications topology are prudently engineered. They convey time series of the sensed sensation to the central nodes where calculations are accomplished and data are merged.

WSN faces its limitations like battery life, limited computing capabilities with less memory and power consumptions when we try to implement it for remote places. These life threatening parameters demand a multi sectional architecture to transmit data and self-organized architecture to control the energy consumption in sensors. As far as the sensing part is concerned, if sensors are largely deployed in remote areas and if each one of them starts to communicate and data transmission, a huge amount of data congestion, collisions and draining of limited energy will be experienced in the network. To address this issue clustering of Nodes will be prime option. The clustering approach works in a hierarchical manner where nodes are segregated into smaller clusters and CH is been elected for each & every cluster. The fellow nodes convey their own sensed data to particular CHs and then CHs groups data and again transmit to the chief base monitoring station. This type of Clustering in WSN facilitates the efficient energy utilization and results in extended network lifespan. Low Energy Adaptive Clustering Hierarchy, i.e. LEACH, is cluster based routing protocol for WSN with a random cluster head selection algorithm.

In (1), authors have discussed the following issues related to Wireless Sensor Networks…
What is sensing and its applications
Key design factors for WSN

- Communication architecture for Sensor Network
- Algorithms and Protocols for each layers of WSN
- Research areas for the realization of sensor network

In (2), the authors have characterized the present application zones and related applications for those ranges of WSNs. This Paper provides a thought regarding the examination work and their long haul application objective in the era of Wireless Sensor Networks. They have likewise specified the ebb and flow examination ventures in the diverse parts of the world. Toward the end of the Paper they have likewise said the Open Research Issues for the understudies and analysts also.

In (3) the authors have exhibited Low Energy Adaptive Clustering Hierarchy convention which was the vitality proficient convention contrast with Minimum Transmission Energy convention and static grouping system.

In (4) the authors have actualized another convention called PEZAC - Power-Efficient Zoning Clustering Algorithm to get the benefit of PEGASIS and LEACH. The calculation takes a shot at standard closer hubs with sink make littler bunch and more distant hubs make ale group. The bunches are made into fan-molded locale. The CHs correspond with one another with multi-bounce directing strategy. In any case, Complexity is expanded on the grounds that every hub will have hub ID so BS will need to keep record of this. It will help in enhancing the adaptability of the remote sensor system.

In (5) the authors have given the Two-Levels Hierarchy for LEACH to enhance LEACH as far as vitality utilization and lifetime. TL-LEACH utilizes arbitrary turn of nearby group base stations. This grants to better dispersion of the vitality load among the sensors in the network particularly when the thickness of system is higher. What's more, weakness of this calculation is it requires higher set up time and system unpredictability is higher as Clustering is done at two levels

In (6) the authors have introduced Variable Round LEACH calculation to enhance the system life time. VR-LEACH creates variable round time is ascertained which is subject to number of CHs and Energy of hubs and so forth. In VR-LEACH, First, at set-up eliminate sort the hubs whose vitality is not exactly the normal vitality; different hubs take an interest in selecting groups.

In remote sensor system vitality gathering is fundamental in a few applications, particularly when sensor hubs are set in non-reachable zones like fight territory (7). Some of utilizations sun based product LEACH (sLEACH) has been anticipated by creators (7) in which lifespan of the remote sensor system has been enhanced through sun based force. A few hubs are encouraged by sunlight based force and these hubs will go about as bunch heads essentially relying on their sun based status in sLEACH. In sLEACH both of LEACH and LEACH - C are amplified.

Drain imagines all hubs are homogeneous regarding vitality which is not practical methodology. Specifically, round uneven hubs are appended to different CH, for this situation CH with expansive number of part tribute will deplete its vitality as contrast with bunch head with littler number of related part hubs. Besides, portability backing is another issue with LEACH steering convention, to moderate these issues, M-LEACH is proposed in (8). M-LEACH permits portability of non-bunch head hubs and

group head amid the setup and consistent state stage. MLEACH additionally considers remaining vitality of the hub in choice of bunch head.

MATERIALS AND METHODS

Low-Energy Adaptive Clustering Hierarchy, it is a protocol which is application-specific. This protocol is a clustering oriented one that comprises the subsequent features…

- randomization, adaptiveness, self-designed cluster creation,
- confined control to transfer data,
- media access with very low energy, and
- Data Aggregation like data processing is Application specific

In LEACH protocol, the nodes found themselves hooked on native clusters, through one node temporary as the CH. All non-CH nodes must convey their sensed data to the CH, whereas the cluster head node must accept data from all the cluster fellows, accomplish signal processing jobs on the data e.g., data accumulation, and communicate data to the isolated base station. Consequently, being a cluster-head node is much more energy severe than being a non-cluster-head node. In the situation where all nodes are inadequate of energy, if the CH were designated a static during the classification of epoch, as in a stationary algorithm for clustering, the CH sensor nodes would quickly use up their partial energy. Once the CH runs out of energy, it is no longer active.

Process of LEACH protocol is separated into parts. Each part initiates when the clusters are organized with a setup phase, tailed by a steady-state phase. Setup phase includes three section in it.

Section A: Determining Cluster-Head Nodes

In Determining CH nodes, we need to project the way such that there are a definite no of clusters, k, during each round. Second, we need to try to regularly allocate the energy absolution when all the nodes are in the network so that there are no exaggeratedly employed nodes that will turn out of energy afore the others. This will undertake signal processing tasks on the data, and transfer the data to a last user who may be faraway; equally distributing the energy capacity between all the nodes in the network requires that each node takes its turn as CH. Thus, the cluster making technique should be proposed such that nodes are CH nearly the same time, presumptuous all the nodes begin with the equal amount of energy. The Cluster Head selection threshold is intended to assurance that a planned portion of nodes, P is elected CHs at each round. Supplementary, the threshold guarantees that nodes which helped as Cluster Head in the past 1/P rounds are not designated in the present round. To meet these necessities, the threshold T(n) of a competing node n is expressed as,

$$Pi(t) = \frac{k}{N - k * \left(r \, mod \frac{N}{k}\right)} : Ci(t) = 1$$

Or (1)

$$Pi(t) = 0 : Ci(t) = 0$$

Where If Ci (t) is the sign of the task key whether or not node has been a Cluster Head in the most recent(r mode(N/K)) rounds (i.e., Ci (t) = 0 if node has been a CH & 1 else), then apiece node should choice to vote a cluster head at round with probability Pi(t). This can be comprehended by setting the probability of receiving designated as a Cluster-head as a role of a nodes energy level relative to the

combined energy long-lasting in the network, somewhat than virtuously as a drive of the amount of times the node has been CH:

$$Pi(t) = \frac{Einstant\ (t)}{Ecumulative(t)} * k \qquad (2)$$

Where Einstant is the recent energy of node I, Ecumulative is entire energy given by.

Section B: Set-up Phase

When nodes have voted themselves as CHs using probabilities in Equ. (1) or (2) the CHs nodes should let all supplementary nodes in network tells that they have designated this part for the prevailing round. To make this happen separately CH node broadcasts a message of advertisement (ADV) utilizing a non-determined carrier sense multiple access (CSMA) MAC protocol. In short altogether NCH eavesdropping to publicizing message will have hand-picked one node whose signal power is supreme as its Cluster Head and send its unique ID, Cluster Head ID and an entreaty message to be fellow of its cluster. Underneath figure displays the graph of the procedure for distributed cluster creation in LEACH (3). Begins subsequently CHs obtain all demands from Non-CHs. After this the CHs broadcast their unique ID, validation signals to their group fellows and the Time Division Multiple Access schedule to be cast-off throughout the steady state phase which begins following.

Section C: Steady-state Phase

The Steady State phase contains of following stages. In first part NCHs utilizes the Time Division Multiple Access list to communicate their own sensor data with their own unique ID number and unique Cluster Head ID to particular Cluster Head. Roster averts crashes amongst data messages and permits Non-CH to turn off their radio devices until its assigned time section. In second part upon getting data packets from its cluster nodes, the Cluster Head wholes the data and directs them to the BS alongside with its unique Cluster Head ID and Base Station ID. Communication among a Cluster Head and a Base Station is attained using static spreading code as well as CSMA. Figure 2 displays the steady state process of LEACH (3).

RESULTS AND DISCUSSION

Change proposed here is constraining the quantity of group heads in each round or as such isolating the range into limited number of matrices. By implementing this we are governing the vitality dissemination between hubs and making it level.

As depicted in the above areas in LEACH the Cluster Head choice is irregular and bunch arrangement is additionally erratic. This irregularity effects in a bumpy vitality circulation among hubs. In this projected strategy we have isolated the entire zone into the limited fix number of segment. At start of each round proposed change partitions the entire region into limited areas and after that chooses the Cluster Head from every segment. The division of region is an arbitrary process so by doing this we are just as dispersing the vitality load in the middle of the considerable number of hubs similarly. Likewise, every hub will get an opportunity to end up a Cluster Head and this will bring about an all the more even appropriation of vitality. The reenactment examination of this methodology demonstrates a huge change in the system life time and throughput of the system.

CONCLUSION

The Paper incorporates diagram of remote sensor system and issues of asset obliges like vitality, constrained accessible transmission capacity and so forth. Additionally, it incorporates LEACH and its impediments like bunch head is chosen in view of edge worth produced by seeing arbitrary variable. After the examination of LEACH convention, we have closed the principle disadvantage of LEACH convention which is uneven appropriation of bunch heads. We have attempted to enhance this con by actualizing even territory dissemination criteria. By this we have enhanced the Life Span of system and throughput of

system. The execution of LEACH has been enhanced as far as Life Duration of Network and information exchanged from group hubs to group head and toward the end of the BS.

FIGURES

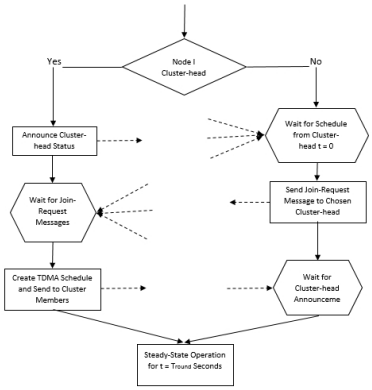


Fig. 1 Algorithm for Cluster Formation

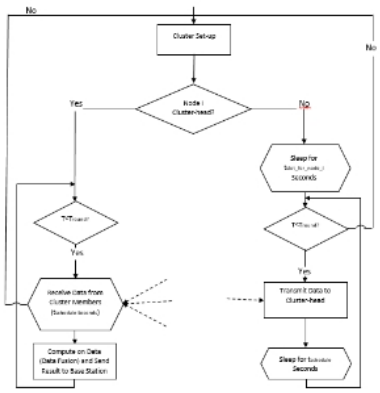


Fig. 2 Steady State

Simulation Results & Analysis for LEACH & LEACH-C

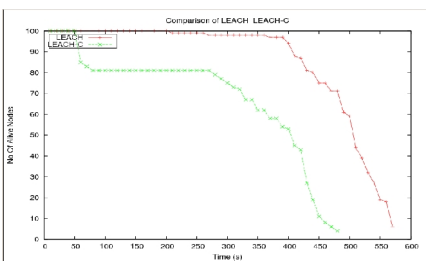


Fig. 3 Time against Alive Nodes per Round

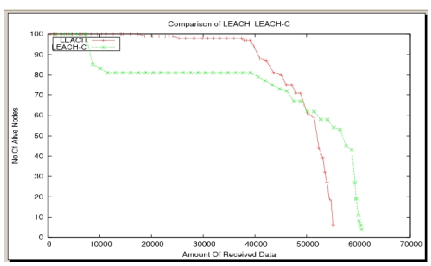


Fig. 4 Data Bytes Received at BS against Alive Nodes

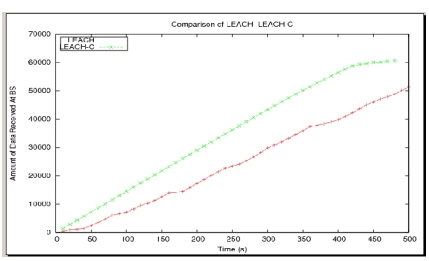


Fig. 5 Time against Data Bytes Received at BS

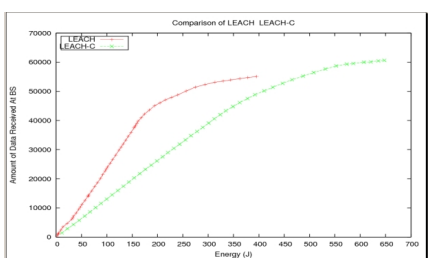


Fig. 6 Energy against Data Bytes Received at BS

Proposed Augmentation &Simulation Results

A. Number of Cluster Heads/Round = 4

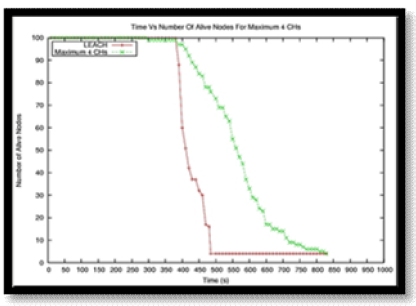


Fig. 7 Time against Alive Nodes

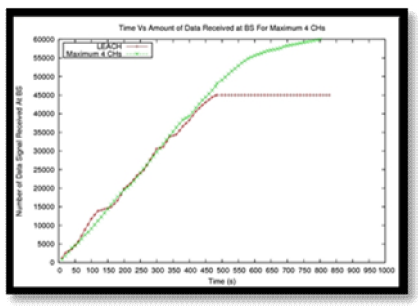


Fig. 8 Time against Data Bytes Received at BS

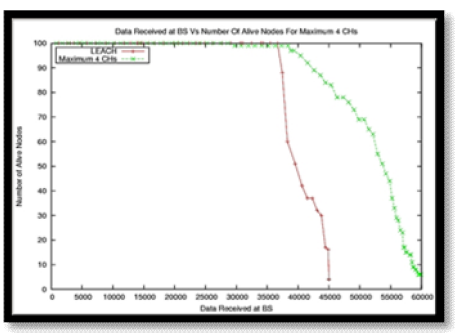


Fig. 9 Data Bytes Received at BS against Alive Nodes

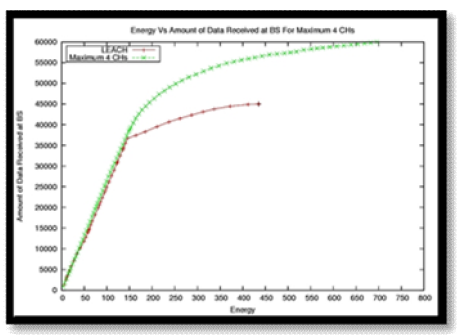


Fig. 10 Energy against Data Bytes Received at BS

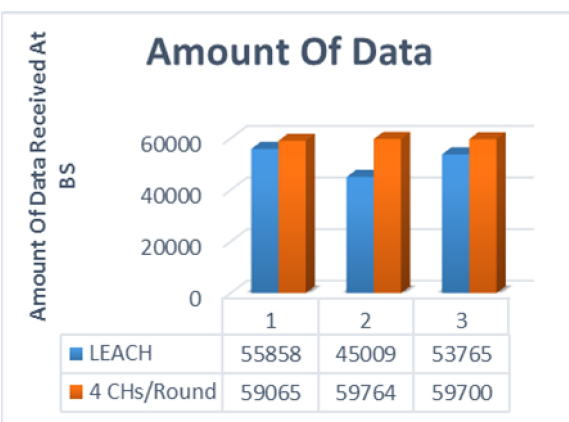


Fig. 11 Total Data Bytes Received at BS

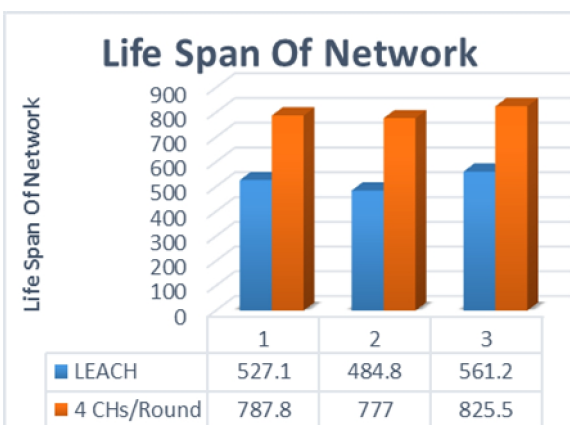


Fig. 12 Life Span of Network

B. Number of CHs/Round = 5

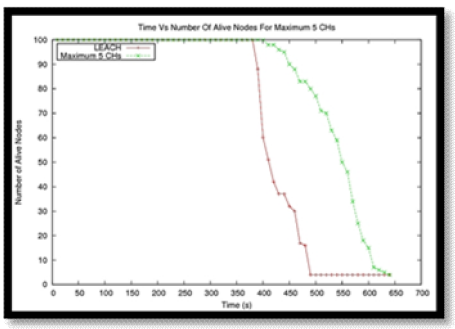


Fig. 13 Time against Alive Nodes

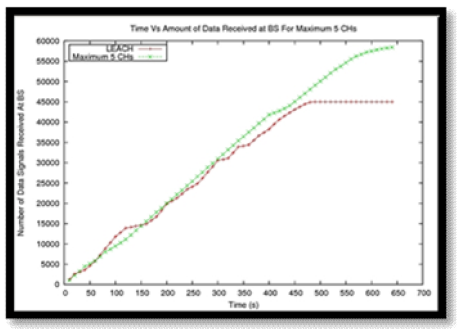


Fig. 14 Time against Data Bytes Received at BS

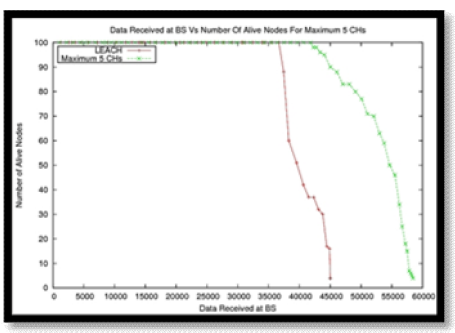


Fig. 15 Data Bytes Received at BS against Alive Nodes

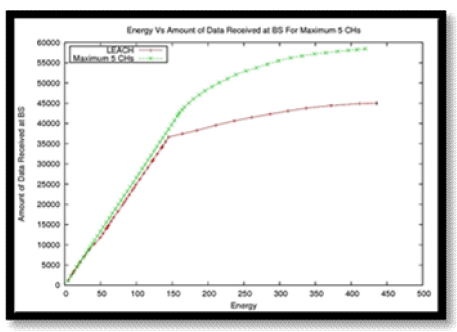


Fig. 16 Energy against Data Bytes Received at BS

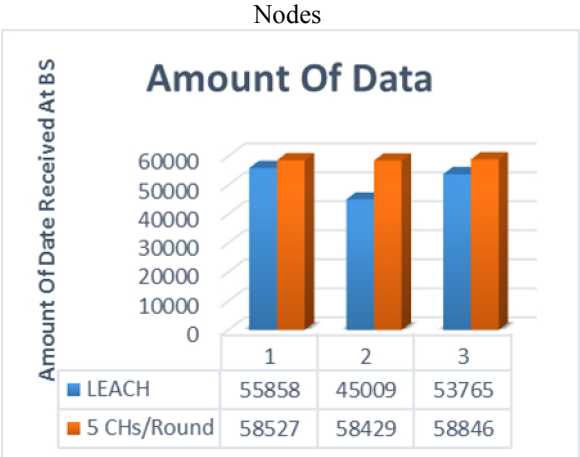


Fig. 17 Total Data Bytes Received at BS

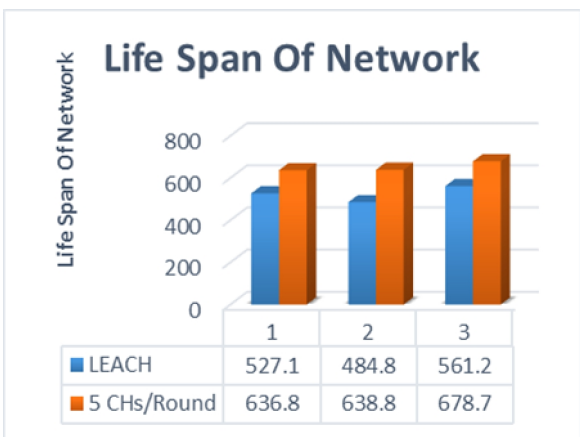


Fig. 18 Life Span of Network

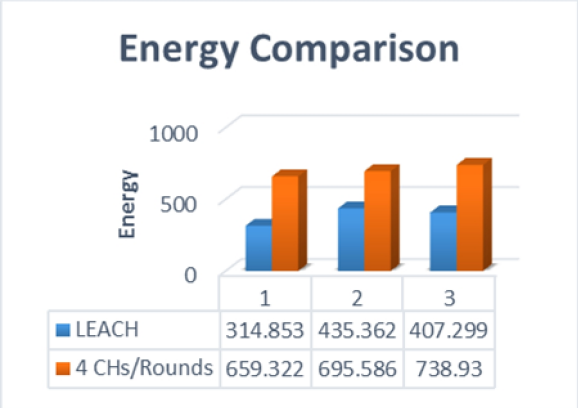


Fig. 19 Consumption of Energy

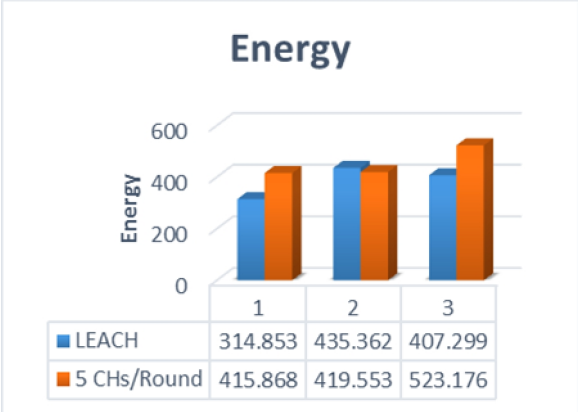


Fig. 20 Consumption of Energy

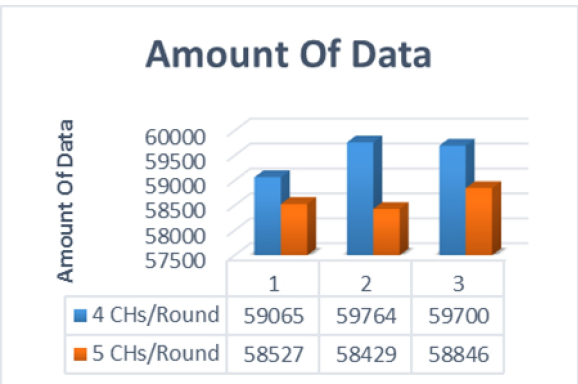


Fig. 21 Total Data Bytes Received at BS

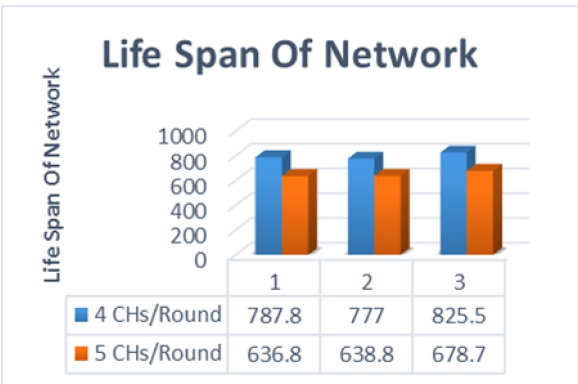


Fig. 22 Life Span of Network

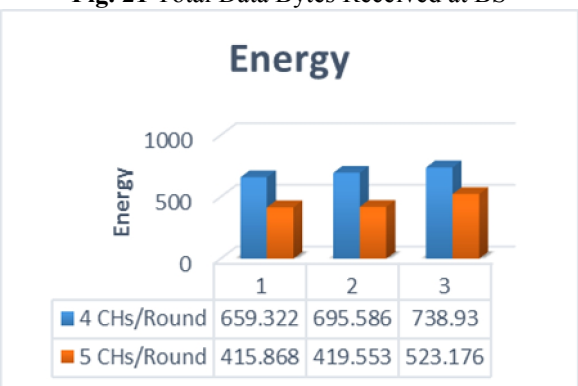


Fig. 23 Consumption of Energy

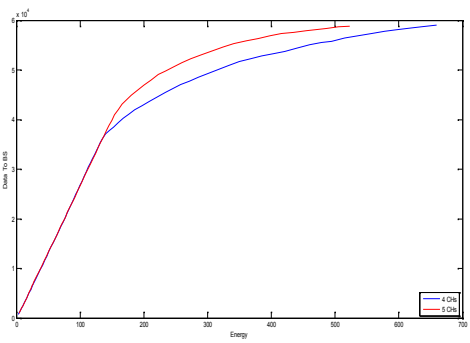


Fig. 24 Energy against Data Bytes Received at BS

As shown in Figures, the projected technique advances the prevailing LEACH protocol in terms on total life span of the network by making more number of alive nodes & throughput of the network by quantity of data. The most important developments can be observed in Figure 24. Figure 24 shows the data acknowledged at Base Station for a specific rate of Energy intake is too much extraordinary in upgraded protocol related to the conservative one.

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Using Big Data Analytics for developing a Crime Predictive Model

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ABSTRACT

In this growing field of technology, rate of cyber-crimes is increasing and are challenging the capabilities of investigation people. The data generation regarding crime is also increased nowadays which is mostly digital in nature. Nowadays generated data cannot be handled efficiently with the use of traditional analysis techniques. Instead of using traditional data analysis techniques it would be beneficial to use Big Data Analytics for that huge data. Primarily collected data will be distributed over geographic location and based on that clusters will be created. In second phase the created clusters are analyzed using Big Data Analytics. Finally that analyzed clusters are given to the Artificial Neural Network which will results in production of prediction pattern. That prediction pattern can be used by security authorities for allocating resources that helps in reducing crime.

SUMMARY

Using Big data Analytics for analyzing crime related data to develop crime predictive model.

Keywords: Crime Mapping, R tool, HDFS,

INTRODUCTION

Increasing crime day by day is the main issue in front of human society. Crime occurs when the personnel space or work space of offender and target intersects at a single point *(1)*. Target may be single person or group of people or say a territory. Crime might accidental or it might be planned. Accidental crime is

unfortunate and unexpectedly occurs. Accidental crime occurs at any places. The group of people fight with others for a small matter which may harms the people which are not having any relation with that matter. Planned crime is the crime which is implemented intentionally. The person whose intention is to do crime, primarily research the target or target area and study it accordingly to implement crime. Secluded places have the higher chances for crime to occur where police patrolling is less *(1)*.

Crime mapping is used to analyze, map and visualize crime incidents or crime pattern to have an idea for predicting the crime occurrence. Crime mapping thus helps the security as well as police to accommodate their resources accordingly for preventing crime *(2)*. In earlier time crime mapping could be done by few peoples who were having special tools. Nowadays both scholars as well as practitioners have the capacity to map the crime using available criminal spatial data and with the help of developed advance technology. Thus crime mapping is mainly implemented to reduce the crime from the society by identifying the Hotspots (the places where crime can occur at higher rate) *(3)*.

In earlier time the data regarding crime are mostly the police complaints, news paper's report and articles which are available in hand written format or printed but as the technological development advances the data regarding crime are available in hard copy as well as soft copy format. Past scenarios are different as the lower crime rate was there, the data generated regarding criminal activities was also low. On that less amount of data traditional data analysis techniques are efficient to analyze and predict the crime. The past data related to criminal activities plays a vital role in mapping crime and prediction of places where crime can occur*(3)*. Analyzing that data available in earlier time was very tedious and time consuming task by traditional data mining techniques even though the data was very less. Data generation nowadays is vast due to increased crime rate which cannot be handled by traditional data analysis techniques. This vast generated data is Big Data which can be easily treated with the help of Big Data Analytics *(4)*. Digital data may be structured, semi-structured or unstructured. Mostly the digital data which are analyzed till now was a structured kind of data for predicting crime *(4)*. Structured data can be considered as the data arranged in tabular format with the help of suitable rows and columns. Previous data are helpful to predict the volatile places or say hotspots. After applying some data mining techniques like clustering, classification and other techniques the places having higher chances of crime to be occur were identified and police capabilities can be allocated there. Nowadays the use of internet is increasing rapidly. The use of internet is also responsible to provide communication between criminals for completing their targeted mission. So the data generation is in huge amount which is mostly in semi structured or unstructured data format and can be analyzed using clustering for Big Data *(5)*. To analyze such huge amount of data either in semi-structured or unstructured format traditional data mining techniques are not that much capable. For that purpose data Big Data Analytics is used.

Generation of data increases exponentially and traditional infrastructure is somewhat incapable of handling such a vast data. Using Big Data Analytics these vast data which includes unstructured or semi-structured data can be handled *(6)*. As the input given to Hadoop might be in semi-structured or unstructured but the output generated from Hadoop will results in structured data*(9)*. The mapper and reducer will contain the prediction algorithm and map reduce is used to handle such data and produce the results in half the time that is taken by traditional data mining methodology.

R tool is used to distribute the data geographically. This tool is capable of generating geospatial representation of data geographically distributed data. Different packages are available with this tool which needs to be installed in order to perform the data distribution. Data analysis as well as different visualization patterns of distributed data can be obtained from this tool.

Artificial Neural Network is the collection of different processing neurons or nodes (processing elements) which gives the prediction based on available data or clustered data. The prediction accuracy of Artificial Neural Network is normally very high as compared to other systems like Fuzzy Logic Series or Bayesian Network *(11)*. The main disadvantage of Artificial Neural Network is that it takes time to learn Artificial Neural Network implementation.

RELATED STUDY

Crime can be considered as an "act against the law which harms the innocent peoples and results in acquiring punishments from the legal authorities like law enforcement or judiciary authority of government". Different types of crime are mainly traffic violations, fraud, sex crime, arson, drug offenses, violent crimes, murders, robbery, damage, theft and cyber-crime *(1)*. It can be observed that the past data which were relevant to criminal activities are helpful for predicting the crime hotspots.

Crime data analysis can be done using data mining techniques with the tools like weka tool, rapid minor tool, R tool, KNIME, ORANGE and Tanagra etc. Mostly the crime data analysis is done using k-means clustering technique of data mining. Due to development in technology the criminals are using their technological equipment for doing crime. That digital data is being used to analyze the crime *(3)*. The analyzed crime will be useful in predicting the hotspots. Again the data used for analyzing and for prediction purpose using data mining is structured data, when there is unstructured or semi-structured data, data mining techniques are somewhat time consuming at that moment. *(4)*Obtained criminal data was taken, preparing that data for rapid minor tool and perform k-means clustering on that data to obtain the clusters. After obtaining clusters, analyzing that clusters to predict the crime.

Other data mining techniques can also be applicable to analyze the crime data and prediction can be done to identify the hotspots. Other technique includes mainly classification, aK-means clustering algorithm, Expectation maximizing algorithm etc. After applying aK-means clustering algorithm it might provide improved results then what we obtain after only applying k-means clustering*(4)*. K-Means algorithm can be implemented in Big Data Analytics *(5) (7)*. These are some traditional and time consuming techniques to map the crime as it requires more over the structured data.

To distribute the data relevant to crime geographically is also a tedious task but now it can be implemented using the tools like R tool. With some geospatial packages that needs to be installed and running with the R tool will greatly influence the data to be distributed over geographic areas. Clustering of that criminal data can be done using appropriate technology. *(8)* In Big Data Analytics GA (Genetic Algorithm) based clustering can also be implemented for analyzing or implementing clustering.

The apache foundation provided the powerful tool for storing and analyzing the data in different clusters separately. Different cluster's data are processed separately where map reduce is used to produce some fruitful results and also uses the HDFS (Hadoop File System). As the different clustered data are processed simultaneously the processing is very fast as compared to traditional storage and processing scenarios and tools. HDFS is a component or say file system of Hadoop which is distributed in nature *(9)*. In HDFS meta-data deals NameNode servers and data regarding applications deals with the DataNode servers *(10)*. With the use of map reducing it is possible to process the semi-structured as well as unstructured data so this Hadoop platform is used nowadays for large data that contains the data of all data model format. Using Big Data Analytics is to overcome the problems like increment in data size which should be stored and analyzed, varied data recording methods and infrastructure, due to its complex nature and time consumption.

Artificial Neural Networks are used to provide the prediction pattern based on analyzed data or given data. Other soft computing techniques like fuzzy time series, Bayesian networks are also used for prediction purpose but Artificial Neural Networks are more powerful as they provide more accuracy as compared to other techniques. Bayesian networks are totally depends upon the selection of parameters and in Fuzzy Time Series results are effected by various factors. Drawback of using Artificial Neural Networks is to learn how to implement that *(11) (12)*.

Nowadays the field of digital forensics is also approaching to analyze the crime in order to predict the crime which helps in crime mapping ultimately results in identifying the places where crime can occur. The field of forensics uses some data mining technique as well as trying to use the concept of big data analytics for crime mapping. Digital forensics is the branch of computer science and engineering which deals mainly with collecting evidences which are digital in nature and can be obtained from digital device such as smart phones, computers, laptops, tablets, palmtops. The major problem over here is also the generated data which is in huge amount. That couldn't be handled with the existing infrastructure and that is the reason for approaching big analytics *(13)*.

RESEARCH OBJECTIVE

Due to increased crime rate, vastly generated criminal data cannot be efficiently analyzed by traditional data analysis techniques. Objective of this research is to analyze such vastly generated data using Big Data Analytics for providing the analyzed clusters to Artificial Neural Network which in turn produces the crime prediction pattern. Produced prediction pattern can be utilized by police department for allocating their resources in order to reduce crime rate.

PROPOSED WORK

Using R tool, Big Data Analytics and Artificial Neural Network we are going to perform the crime mapping. It contains mainly three phase – Distribution of data geographically and creating clusters, Cluster analysis of created clusters and prediction of crime.

Distribution of data geographically is the first phase where the available data is distributed over geographical areas. Here the available data is related to crime. This can be implemented using the R tool with the geospatial packages. With that the clusters are created after allocation of centroids. The KDE (Kernel Density Estimation) technique will be used for estimating or creating clusters on the basis of mapped data. As this created clusters are being utilized by cluster analysis phase.

Hadoop platform is used for cluster analysis purpose which is second phase. Clusters created in primary phase are used as input this phase and suitable clustering algorithm is to apply over here for the analysis purpose. Hadoop can perform parallel processing on different clusters the processing will be fast as compared to traditional processing capabilities. This will result in less time consumption and gives the output earlier than the normal data mining cluster analysis process. The GAMMA Test is used for cluster analysis over this phase.

As shown in fig.1, Analyzed data of cluster analysis i.e., identified cluster from the Hadoop is utilized by the Artificial Neural Network as an input for crime forecasting purpose is third and final phase. It also uses the regression tree prediction specification and classification. The output of Artificial Neural Network is the pattern that predicts the crime rate at different places or the places where the chances of crime occurrence are high. Artificial neural network is selected to predict the pattern because it's quite

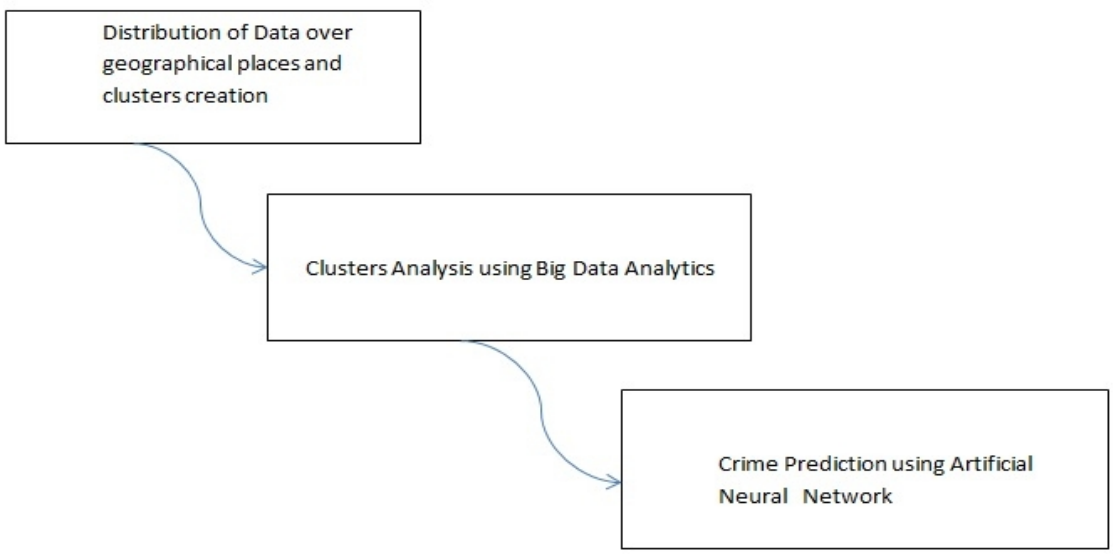
good than other network in terms of producing pattern as well as it takes less time for producing the pattern.

CONCLUSION

The proposed work focuses on crime prediction by crime mapping with recorded data using the latest technology. The model helps in reducing crime for the security authorities. The model also helps the authorities in investigation of crimes. Using Big Data Analytics with clustering approach reduces the investigation time and helps in retrieving the hidden information through correlation and categorization.

FIGURES

Fig. 1 Crime Prediction process Model



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Interference effect Measurement of GPS and LTE by simulation

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ABSTRACT

This paper presents a measurement study of interference effect GPS and LTE signal using Simulation in MATLAB2014. We here stated basic physical characteristic of GPS signal as BPSK modulation and LTE signal as QAM. We quantify the impact of LTE and GPS signals on the BER checking. In our controlled testing, we characterize the interference properties of both technologies.

SUMMARY

On the BER simulation of radio signal one can easily judge the susceptibility of interference.

*Keywords:**GPS, LTE, MATLAB, SIMULATION***

INTRODUCTION

Today, GPS is essential and core technology of our day to day navigation and many other time precision technology for accurate timing. 4g technology which helps us to provide data much faster to feel like Wi-Fi on the go up to 10Mbps and the most promising for the Data hungry tech savvy. Potential interference of 4g LTE and GPS receiver is an essential problem to attend. Research and testing of this possible interference source are necessary because GPS has a chief role in so many important systems that the public depends upon for its safety and welfare. We are here using MATLAB base desktop programming environment, which grants you to do simulation interactively with our test data. In MATLAB one can

easily modify the code on the go. The Graphical output can be visualized. Many benefits we can count of MATLAB, so we selected best simulation environment, MATLAB for our Simulation tool. If we have basic knowledge of programming than it's easy to put our logic in MATLAB.

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Physical-Layer Characteristics

2.1 GPS Signal Structure

L1 and L2 two carrier frequencies which are used in GPS signal transmission **(1, 2)**. GPS signal uses BPSK (Binary phase shift key) phase modulation, in which signal sifted with a half circular phase shift over successive intervals. Each SV (satellite vehicle) have distinctive PRN (Pseudo Random Noise) sequence related with each SV and by a common navigation data. The fundamental frequency: of L1 is 10.23MHz, which is consist of two PRN Code the C/A code and P code where L2 consist of only one C/A code. Military GPS communication uses the P code. **(3)**Let's recall the basic equation of BPSK from **(4).**

$$S\ (t) = \frac{\sqrt{2E_b}}{T_b}\ c\ \ (2\pi f_c t + \pi(1-n)),\ \text{n=0, 1} \qquad (1)$$

This produces two phases, 0 and π. In the specific procedure, binary data is often taken with the following signals:

$$\boldsymbol{S}_{\mathbf{0}(t)=\frac{\sqrt{2E_b}}{T_b}c\ \ (2\ f_c t+\pi)} = -\frac{\sqrt{2E_b}}{T_b}\boldsymbol{c}\ \ (2\ f_c t+\pi) \quad \text{For binary 0(2)}$$

$$\boldsymbol{S}_{\mathbf{0}(t)=\frac{\sqrt{2E_b}}{T_b}\boldsymbol{c}\ \ (2\ f_c t)} \quad \text{For binary 1} \qquad (3)$$

Where carrier-wave's frequency is the fc. Hence, the signal–space can be represented the signal by single basis function.

$$\emptyset(t) = \frac{\sqrt{2}}{T_b}\ \boldsymbol{c}\ \ (\mathbf{2}\ \ \boldsymbol{f_c t}) \ (4)$$

[Fig.1. GPS signal generated in MATLAB]

From the above eq. (4) we generated the GPS's BPSK signal modulation in MATLAB simulation. In the above figure first signal is carrier signal. Second signal as message signal. And the third signal is Phase shifted key signal is combination of message and carrier signal.

2.2 Signal structure in LTE

The LTE is advancement of the requirement of the high speed rate of transmission. Multiple channel bandwidths provided by LTE (1.25-20 MHz). For achieving the speed and quality of service orthogonal frequency division multiplex (OFDM) for PHY layer, MIMO technology in LTE.

More than one narrow band sub-carriers used in wide channel bandwidth by OFDM technology. Inter symbol interference mitigate in the frequency domain which are mutually orthogonal in all sub-carriers shown in Figure (2).

[Fig.2: mitigate the internal symbol interference]

OFDMA has many advantages with and some disadvantages like high peak-to-average power ratio (PAPR) and high compassion to frequency offset and. PAPR is a result of the random constructive addition of sub-carriers and outcomes in spectral spreading of the signal which result at least as adjacent channel interference. To resolve the problem of PAPR add cyclic prefix on the uplink of Single Carrier FDMA (SC-FDMA). **(6)**

Let's, recall QAM signal equation from **(5).**

$$\begin{aligned} s(t) &= \Re\{[I(t) + i\iota\ (t)]e^{i2\pi f_0 t}\} \\ &= I(t)\cos(2\pi f_0 t) - Q(t)\sin(2\pi f_0 t) \end{aligned}$$

Where, i^2=-1, *I*(t) and Q(t) are the modulating signals, f0 is the carrier frequency and $\Re\{\}$ is the real part.At the receiver end cosine and sine signal received estimates of *I*(t) and Q(t) by demodulated using coherent demodulator.

It is possible to detect the modulation signals because of the orthogonality property. In most situations, one can demodulate I (t) just reproducing transmitted signal with a cosine signal:

$$\begin{aligned} r(t) &= s(t)\cos(2\pi f_0 t) \\ &= I(t)\cos(2\pi f_0 t)\cos(2\pi f_0 t) - Q(t)\sin(2\pi f_0 t)\cos(2\pi f_0 t) \end{aligned} \quad (6)$$

Using standard trigonometric identities, we can write it as:

$$\begin{aligned} r(t) &= \frac{1}{2}I(t)[1 + \cos(4\pi f_0 t)] - \frac{1}{2}Q(t)\sin(4\pi f_0 t) \\ &= \frac{1}{2}I(t) + \frac{1}{2}[I(t)\cos(4\pi f_0 t) - Q(t)\sin(4\pi f_0 t)] \end{aligned} \quad (7)$$

High frequency terms removes by Low-pass filtering. This filtered signal is natural by, the in-phase element can be received individually by the quadrature component. Likewise, one can reproduce with a sine wave and then low-pass filter to extract.

[Fig.3. Signal spectrum of signal for coding 8-QAM with SQRC]

From the above equation (7) we have created the 8QAM with square-root-cosine (SQRC) signal in MATLAB simulation.

BER Measurement Results

The bit error-rate expression of phase-shift keying (PSK) and 8-quadrature amplitude modulation (QAM) are gained in the presences of the phase error. One can examine the performance penalty by averaging over the date of the phase error, as a function of the phase error variance.

For BPSK, where erfc(x) = 2/√π • x+∞ exp (−t2)dt and γb is defined as the signal-to-noise ratio (SNR) per bit; **(7)**

$$P_b(e|\Delta\theta) = \frac{1}{4}\left[e \quad \left(\sqrt{\gamma_S}\sin\left(\frac{\pi}{4}-\Delta\theta\right)\right) + e \quad \left(\sqrt{\gamma_S}\sin\left(\frac{\pi}{4}-\Delta\theta\right)\right)\right] \quad (9)$$

[Fig.4: Bit Error Rate for BPSK simulation in MATLAB]

From the figure (4) of SNR we can deduce the bit error rate for BPSK modulation signal simulation and theoretical difference if the difference more, than modulationtechnique more of suspicious to the interference.

[Table 1: Bit rate error for BPSK]

The error probability of 8QAM symbol is obtained by the error probability of each branch (4-PAM) and is given by: **(8)**

$$P_S = 1 - \left(1 - \frac{2(S_{\cdot} \quad 4-1)}{S_{\cdot} \quad 4} Q\left(\sqrt{\frac{3\gamma_S}{3}}\right)\right)^2 \quad (10)$$

[Fig.5: Bit Error Rate of 8QAM modulation]

From the above Graph of SNR (Eb/No) we can deduce the bit error rate for 8QAM modulation signal simulation and theoretical coded and theoretical encoded difference if the difference more, than the modulating technique of suspicion to the interference.

CONCLUSION

We have a GPS BPSK signal expression and LTE 8QAM signal expression and figure generated by MATLAB. We have a BER expression of 8QAM and BPSK with the phase error$\Delta\theta$. As we can easily conclude GPS BPSK more differ in simulation as we assumed in theory and 8QAM have not muchdifference. On the above simulation and data of SNR, the BPSK as GPS signal and 8QAM as LTE signal we can find some fact that 8QAM is more secure than BPSK so GPS is easily victim of interference.

FIGURES

Fig.1 GPS BPSK signal generated in MATLAB.

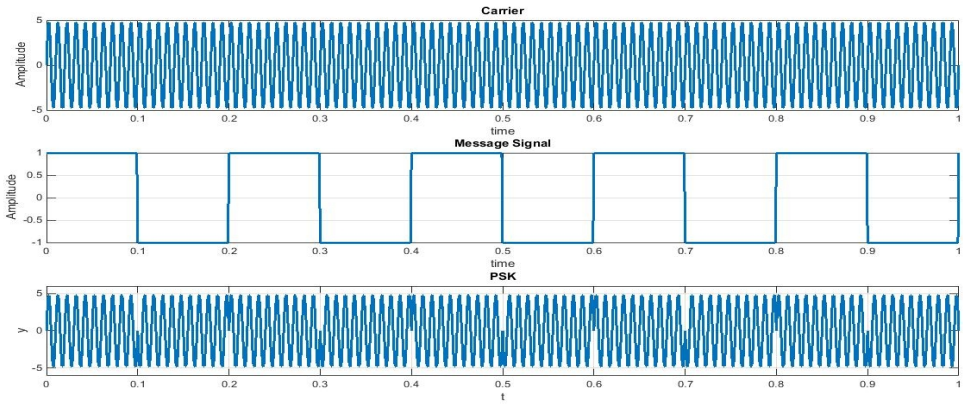


Fig.2 Mitigate the internal symbol interference.

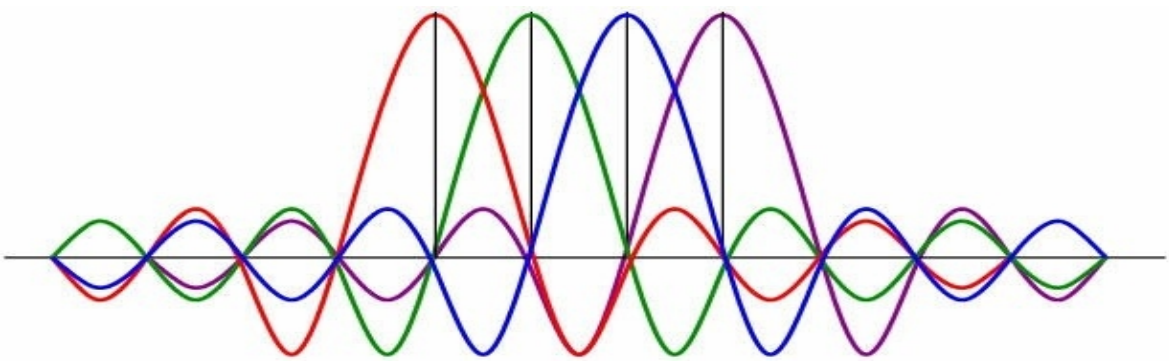


Fig.3 LTE Signal spectrum of signal for 8-QAM

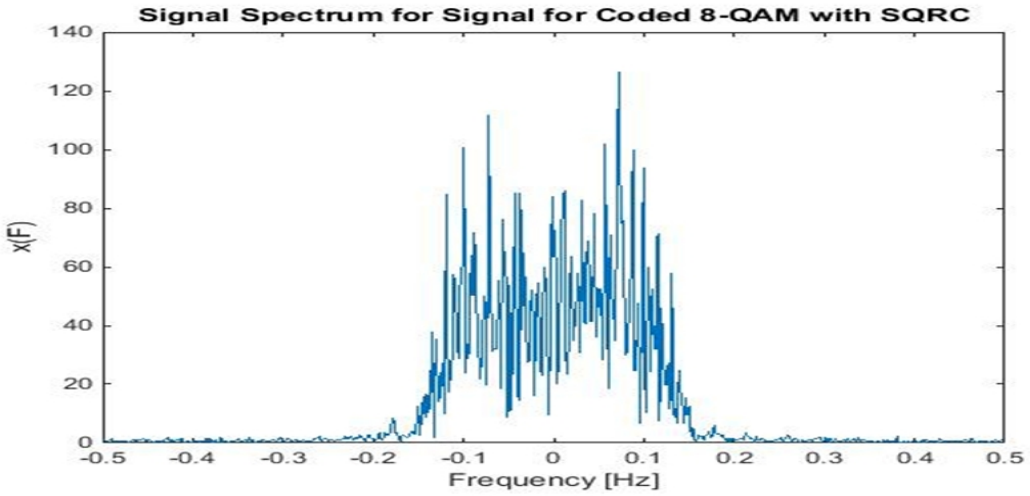


Fig 4 Bit Error Rate for BPSK simulation in MATLAB

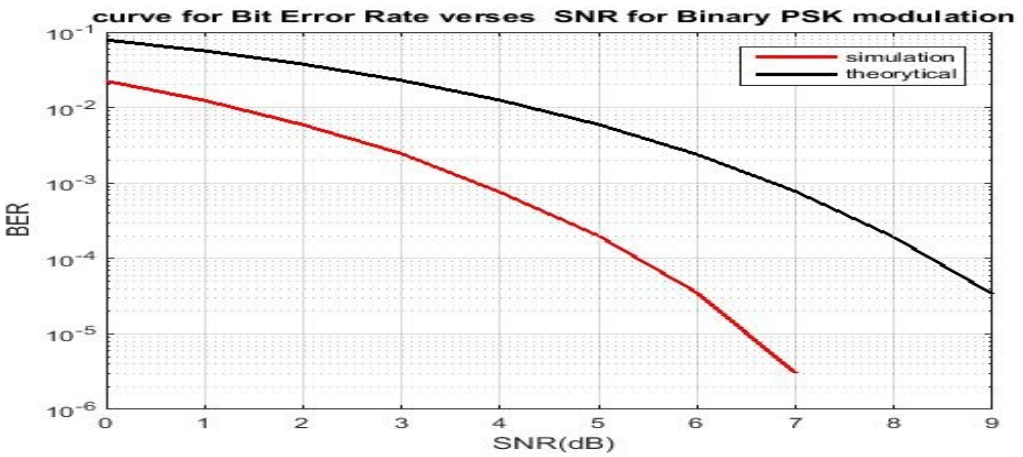
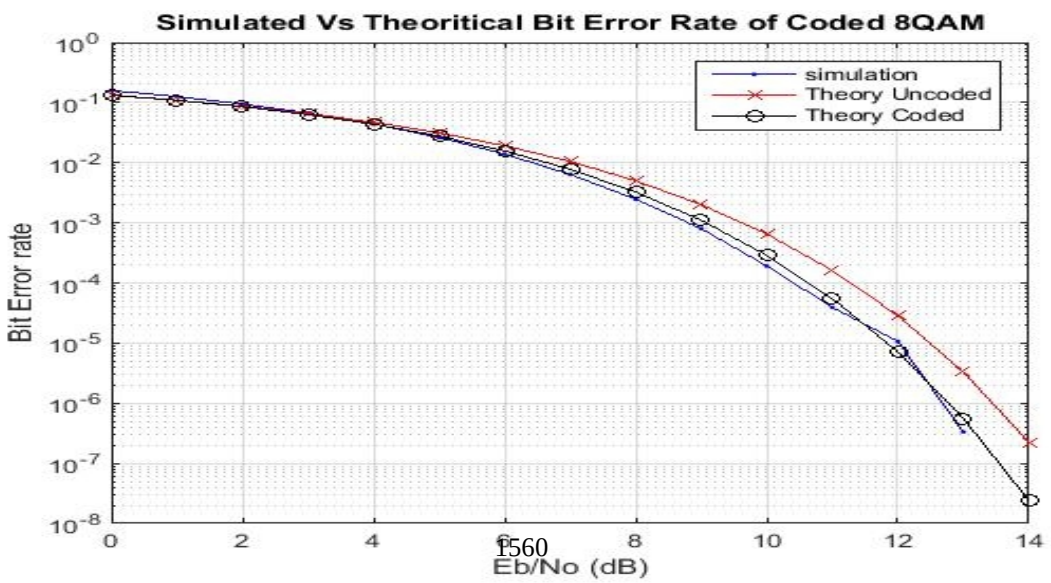


Fig 5 Bit Error Rate for of 8QAM modulation



TABLES

Table 1. Quantitative estimation of the Bit rate error for BPSK

	Bit Error Ratio for BPSK	
SNR(dB)	Simulation	Theoretical
0	20^-2	80^-2
1	12^-2	60^-2
2	50^-3	30^-2
3	25^-3	20^-2
4	80^-4	12^-2
5	20^-4	60^-3
6	30^-5	25^-3
7	20^-6	80^-4
8		20^-4
9		32^-5

Table 2. Quantitative estimation of the Bit rate error for 8QAM

	Bit Error Ratio For 8QAM		
SNR(dB)	Simulation	Theory Uncoded	Theory Coded
0	14^-1	14^-1	12^-1
1	12^-1	12^-1	10^-1
2	80^-2	80^-2	80^-2
3	60^-2	60^-2	60^-2
4	40^-2	40^-2	40^-2
5	30^-2	30^-2	30^-2
6	15^-2	20^-2	17^-2
7	60^-3	10^-2	80^-3
8	25^-3	50^-3	30^-3
9	80^-4	20^-3	12^-3
10	20^-4	65^-4	30^-4
11	40^-5	17^-4	45^-5
12	10^-6	30^-5	60^-6
13	30^-7	32^-6	45^-7

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RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

Comparison of Various Improved Optimized Link state Routing Protocols in Mobile Adhoc Networks

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ABSTRACT

Mobile Adhoc Network is a wireless network, which is self-configuring of mobile nodes communicated by wireless link. Now a day's MANETs is a wider area for the research in which energy efficiency is the key feature to research where minimum power consumption is required in the network. Optimized Link state Routing (OLSR) protocol is a table driven, proactive protocol, which provides an immediate route between nodes when needed. In this paper, different improved techniques of OLSR, which used to improve energy efficiency in the network, are described. At the end of the paper all techniques are compared and founded that the best technique which will improve energy efficiency in the network. In future for energy efficiency the routing table can be modified by saving multiple shortest path for immediately next shortest link available in case of first link goes down without running route discovery method.

SUMMARY

Energy efficiency by OLSR protocol in mobile Adhoc networks.

Keywords: *OLSR, Multipoint relaying, Routing protocol, energy efficiency.*

1. INTRODUCTION

1.1 MANETs

Mobile Adhoc Networks (*1*) are the types of wireless networks, which is infrastructure less. It is a decentralized wireless network. In MANETs every node itself router. In MANETs nodes are in mobile nature, because of that the network topology is changed frequently .

1.1.1 Characteristics of MANETs (*2*):

- No any mediator network device is required for communications.
- Each node is working as intelligent node.
-Ease of deployment, Speed of deployment, Multi-hop network.

1.1.2 Advantages of MANETs (*2*):

- No need of geographic position for access to inform and services.
-Infrastructure less and no need to administration, also nodes are itself routers.
-Provides scalability- able to add more nodes.
- Improved flexibility than wire network.
- Provides Robustness due to decentralize administration.
- Free to set up The network at any place and time.

1.1.3 Applications of MANETs (*2*):

- Military battlefield.
- Collaborative work.
- Local level.
- Personal area network and Bluetooth.
- Commercial sector.

1.1.4 Limitation of MANETs (*2*):

In refuge system Vulnerability is a Limitation. Without verifying user's identity, the system does not allow data access (*2*), so if unauthorized data manipulation will done on a system, it may be unsecured to the system. There are more vulnerabilities in MANET than wired networks.

1.2 OPTIMIZED LINK STATE ROUTING PROTOCOL (OLSR)

Optimized Link State Routing protocol (OLSR) (*3*) is the link state protocol, so it provides the link immediately when required because of its proactive nature. OLSR floods link or control messages to keep updated of the every node in the network and maintains table up-to-date. Instead of transmitting the control messages to each node, OLSR will transmit it to particular selected nodes who are Known as multipoint relays. The multipoint relays task is to disseminate the messages in the network.

1.2.1 MULTIPOINT RELAYS (MPRs)

The main perception of the MPRs is to decrease the flood of the broadcast packets in the network. It reduces replica of retransmission, in same region. Multipoint relays are a subset of nodes, which are selected by its neighbors. Each MPR node in the network retransmits its packets to the two hop neighbor. The multipoint relays (MPRs) of the node are following in figure 1.

1.2.2 PROTOCOL FUNCTIONING

The OLSR protocol contain various functions those are responsible to perform the task of routing. these functions of the protocol are discussed follow :

1) **Containing Neighbor node Information (neighbor discovery)** (*4*)

In this function, the HELLO messages is broadcasts by Each node periodically with containing its neighbors information and its link position. These control messages does not transmitted in unicast manner but are transmitted in the broadcast manner. These are sent to one hop neighbors. The HELLO message contains:

- Neighbor addresses list to which there available a valid bi-directional link.
- List of addresses corresponding to nodes, i.e. the nodes that are heard by this node by which HELLO message received. The structure of a HELLO message is displayed in figure 2.

2) Link state declaration (*4*)

Link state routing protocols are based on nodes flooding the network with information about their local links. In OLSR link state emitted describes link to neighbor nodes. This is done using TOPOLOGY CONTROL(TC) message. The format of a TC message is shown in figure 3.

MPR optimization is done by TC messages flooding . This is done at a regular time period. but when changes are identified in the MPR set, TC messages are generated immediately.

1.2.3 OLSR TABLE CALCULATION

OLSR contain two tables:

1) Topology table:

Every node contains a topology table from TC messages. Routing table store data from topological table the format of the topology table is shown below in figure 4:

2) Routing table:

A Routing table contains final result of the OLSR protocol. Each node creates its information about routes to the each node of the network topology, and using the Dijkstra shortest path algorithm, it calculates the shortest path to any destination. A routing table stores the shortest path which is calculated from Topological table. The classic OLSR routing calculation algorithm is triggered, Once a node identifies the change in network topology, OLSR routing calculation algorithm finds the routes with less cost/distance from the source node to every destination. the format of the routing algorithm is shown in figure 5.

If there any modification in table of neighbor node or in topology table, a routing table is recalculated after every change.

2. IMPROVEMENT IN OLSR

2.1 IMPROOVED OLSR IN MANETs:

In this paper author has modified the TC packet, and HELLO packet , he added a field named threshold energy and residual energy, these fields increase the lifetime of the node that improves the performance of the network*(5).* The proposed technique evaluates optimal route according to the number of hops and available energy. The load will be mainly assigned to the main route, but The number of nodes in MPR subset can also be reduced if the energy of the intermediate nodes is going to reach to threshold (given by the, user), then another path to be considered. This will provide a shortest hop route as well as optimal node energy consideration for longer life span of the network.

2.2 IMPROVED OLSR BY DESCARDING LOOPS:

This paper contains the purpose to provide a better quality of the package delivery ratio and the throughput*(6),* that is required powerful routing protocol standards, which can give assurance delivering of the packages to destinations, and the throughput on a network. For achieving this purpose author improved the OLSR routing protocol by technique eliminating the unnecessary loops.The technique is following: When a node transmits a package to other nodes within its own network area, packets will be transmitted by nodes known MPR to the other nodes. Consequently, if the package enters into a loop, then two possibilities occur; if the package is IP Header and the package takes less than 255 steps to reach the destination, then author set its number of steps to zero dynamically to give the package the second chance to reach the destination, and if the package used more than 255 steps, and the package is not IP Header, then author discard the package, otherwise, many packages will not get chance to deliver to the destination in the network and this will create network traffic, increase high level of delay in the package delivery, bandwidth occupation, and the package delivery ratio and network will be reduced.
In the above method, author proved that, by discarding the unnecessary loops, the package delivery ratio (PDR) and the throughput is improved by about 20 percent. In this method, by discarding the less required, the package delivery rate and throughput in the network is improved.

2.3 MOBILITY ENHANCEMENT IN OLSR:

The work done in this paper*(7)* concerns the improvement of the formula used in the routing protocol Mob-OLSR for measuring mobility of nodes by automating the setting. Herein, the improved protocol is called a Mob-2-OLSR protocol. In OLSR every node in the network can be found within a set of other neighbor nodes. The main perception of the Mob-OLSR protocol is to find a metric which measures node mobility of neighbor node, which are entering and leaving in the network. In this paper By Mob-OLSR PDR, Average Delay and Average throughput are calculated and proved that this enhanced OLSR gives better energy efficiency than basic OLSR.

2.4 IMPROVED MPR SELECTION ALGORITHM:

In this paper(*8*), the node localization is proposed to combine with node localization technology for the purpose to an improved algorithm. In this algorithm the information about node localization is used, the typical method is to select MPR is reduced. It can make full use of the network area and resources. It reduces more routing packets required to deliver in the network, therefore it can improve the network transmission ability.

In this algorithm author introduced new technique, that is calculate the angle between two adjacent nodes, and forward the packet which have less angle or degree from MPR node. This improved algorithm is implemented in the network simulator, the results are studied, it shows that the improved algorithm ,is feasible and applicable, and the location-based selection method is proper and exact.

2.5 REDUCING ROUTING OVERHEAD IN OLSR:

The main purpose of this paper(*9*) is to provide a improved quality of the package delivery ratio and the throughput, that is required of influential routing protocol standards, which assures of the packages delivery to destinations, and the increase throughput on a network. Author focus on the imprecision of state information, more specifically the residual energy level of nodes that is collected by the control messages of the OLSR protocol.

Imprecise information affects the efficiency of the OLSR protocol. Author analyze some parameters of OLSR protocol that forces the inaccuracies in the neighbor nodes energy level information and compared between ideal parameters result and realistic parameters result of OLSR protocol. This analysis concluded that the tuning of OLSR improves the residual energy information of nodes and simulation results demonstrated a significant improvement in the package delivery ratio and throughput.

3. COMPARISON

We have compared all different techniques which are used in different paper which are referenced here and we also found the advantages and disadvantages of each technique. And found the best technique among them due to its less disadvantages with compared to another techniques.

Here a comparison table of all the techniques is following Table 1:

From above comparison table last technique is a most suitable technique with less limitations and less disadvantages.

5. PROPOSED WORK

Proposed work will modify OLSR for the purpose of less energy consumption by altering the hello message by adding residual energy field. Prior to send hello message, each node adds its own residual energy within hello message and adds residual energy field within TC message.

Each node sends Hello message for finding hop count and each node creates neighbor table of one hop count and 2 hop count. Based on this table, each node selects MPR. MPRs are selected nodes that have more than one hop as well as high residual energy. After selecting MPR, only MPR nodes broadcast route_request. For sending route_request MPR nodes send TC (topology control) message. TC message contains MPR list, therefore each node will get an idea about complete topology. Over here, we will modify TC message and also verify residual energy of MPR.

After receiving TC message, each node maintains topology table and routing table and will store multiple shortest path in routing table from topology table with 1st path contains high energy MPR and 2nd path contains second highest energy MPR. If any link will goes down then router immediately get 2nd path with another MPR. So in case of failure of link, no need to calculate residual energy and to run algorithm. Therefore here time and energy will consume less.

4. CONCLUSION

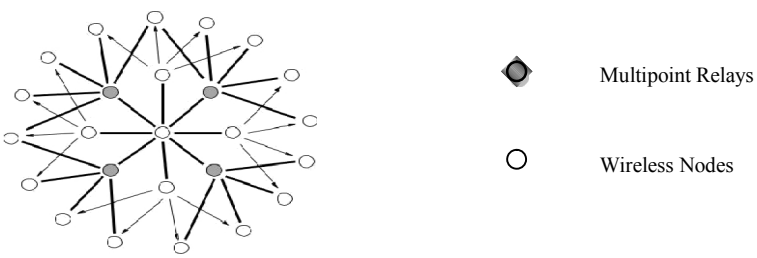
MANET is a very large field to research and growing very fast in the world of technology, that's why demand of efficiency in MANET goes higher day to day. OLSR is the most suitable protocol to find the shortest path in MANETs.

Also, many Improved OLSR technologies are presented to consume the least energy while finding the shortest best path in the network. this paper presents the multiple energy efficiency modified techniques in OLSR protocol. Also comparison among all techniques and here presented their advantage and disadvantage of each.

Also found the better one from them and from that found the future conclusion for energy efficiency in MANETS.

5. FIGURES

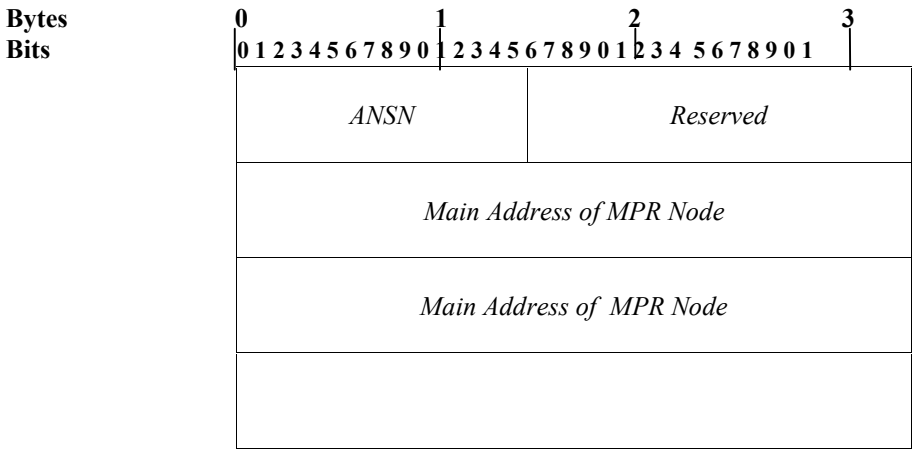
" Figure 1: Multipoint relays (*3*)"



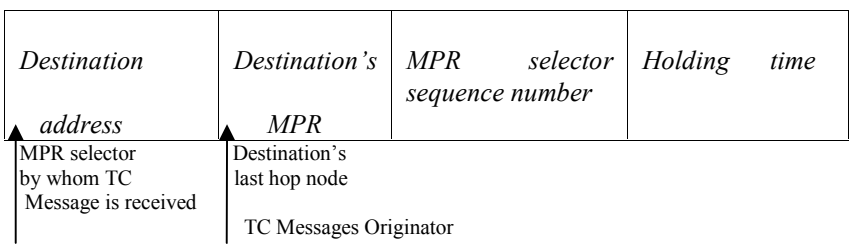
" Figure 2: HELLO message (*4)"*

Bytes	**0**			**1**				**2**				**3**		
Bits	**0**	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**0**	**1**	**2**	**3**
	Reserved								*Htime*			*Willingness*		
	Link Code				*Reserved*				*Size of Control Message*					
	address of one hop Neighbor													
	address of one hop Neighbor													

“ Figure 3: Topology Control message (*4*)”



“Figure: 4 Topology table (*4)”*



“ Figure: 5 Routing table (*4*) **“**

Destination address	*Next hop address*	*Distance*

6. TABLES

"Table : 1 Comparison between techniques :"

IMPROVED OLSR	*METHOD*	*ADVANTAGE*	*DIS – ADVANTAGE*
2.1)Improved OLSR in MANETs	*Modified the HELLO and TC packet by threshold energy parameter*	*Provides shortest hop path as well as optimal node energy for longer life span of the network topology.*	*Still overhead to find the shortest path if a link goes down.*
2.2)Improved OLSR by discarding loops	*Eliminating the unnecessary loop*	*Decrease delay in the network.*	*IP header retransmission is there, so also again bandwidth and energy consumption is there.*
2.3)Mobility Enhancement In OLSR	*Improved protocol Mob-2-OLSR*	*Improves the weakness of the Mob-OLSR protocol.*	*In MANET difficult to measure the degree of mobility because of the continuous changing topology of the network*
2.4) Improved MPR selection algorithm	*The algorithm for improvements of MPR selection.*	*It reduces TC message transmission in the OLSR protocol because of nodes in MPR set can also be reduced.*	*Difficult to calculate an MPR set when nodes are moved out of network range*
2.5)Reducing routing overhead in OLSR	*OLSR Improvement by calculating the residual energy level of the nodes*	*Improves PDR and throughput ratio.*	*Less limitation with comparing with above four techniques.*

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

(Proceedings available for download at rku.ac.in/icre)

RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

Enhancing the TCP NewReno Protocol in Wireless Ad-Hoc Networks

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ABSTRACT

Nowadays, wireless network in support of access the internet is increasing. There are two types of wireless network such as infrastructure and ad hoc networks (infrastructure-less). Transmission Control Protocol (TCP) is a connection-oriented, byte-stream, reliable, end-to-end, full duplex communication protocol. There are many variants of TCP, such as TCP Tahoe, TCP Reno, TCP New Reno, TCP Vegas, TCP Westwood, etc. On a wireless network, congestion is mainly detected through bit error; duplicate acknowledgement, RTT (Round Trip Time) and retransmission timer expire. We analyzed a major congestion control mechanism in TCP NewReno. TCP NewReno does differentiate issues of packet losses due to congestion or bit-error. But it always decreases congestion window in issues of packet loss and its decrease the performance. In this paper, modification introduced in TCP NewReno over a wireless network using calculate the duplicate acknowledgement and retransmission timeout, improve the performance related to congestion control.

SUMMARY

In this paper, modification introduced in TCP NewReno over a wireless network using calculate the duplicate acknowledgement and retransmission timeout, improve the performance related to congestion control.

Keywords: TCP NewReno, Congestion control, Congestion window, Duplicate Acknowledgement, Retransmission timer.

INTRODUCTION

Fixed Wireless Networks was provided with incorporated connections. Wireless Adhoc Networks are a set of nodes that provided identical or similar quality of present at the same time functions that work as station and router. MANET (Mobile Adhoc Network) defines that Wireless Adhoc network is a separate system and its nodes are also allowed to move a given network. The characteristics of Wireless Adhoc network provide the function that is flexibility, stability, and effortlessness of utilize and operation (2). The major problem faced by Adhoc Wireless Network such as hidden terminal, exposed terminal, irregularity, network separation, routing problems and power constraints (1).

The primary aims of this protocol that include the set up of an along a connection, along the length release of data packages, stream control, error control, congestion control, dependable, a series of bytes, and connection-oriented transport layer protocol such as TCP for Wired and Wireless Network. There are many variations of TCP, such as TCP Tahoe, TCP Reno, TCP New Reno, TCP Vegas, TCP Westwood, etc. In a wireless network, congestion is mainly detected through bit error; duplicate acknowledgement, RTT (Round Trip Time) and retransmission timer expire. There are many matters in designing a TCP for Adhoc Wireless Networks that are induced traffic, induced throughput unfairness, division of congestion control, reliability and flow control, power constraints and bandwidth, active topology, etc.. For the duration of TCP communication, the TCP is to provide functionality to always inform to sender about the current capacity of the buffer (5). The distribution factor moves a congestion window that is basically to provide successful transmissions and send the information about the congestion window limit. The spot of the congestion window also limited may be outcome in below operation of web resources. An outsized congestion window might in excess of deliver the network that provides an outcome that segments are dropping at the overfilled node (5).

Nowadays TCP NewReno is designed for a Wireless Adhoc Network (6) and also Wired Network. In TCP NewReno, timeouts and reception of three duplicate acknowledgements permit the Fast Recovery (7). In TCP NewReno frequent retransmission timeouts, which is the primary problem of the performance degradation over Wireless Adhoc Network (6).

Congestion Avoidance in TCP:

Congestion is providing simple to overfill or overcrowd network. Congestion control is required for both to exhaustion of network and to improve the utilization of a network (5). Congestion is mainly occurring when the bandwidth of the network is limited or traffic record may go beyond the capacity of the network bandwidth. In an overcrowded network that reduces the network throughput as well as performance.

Congestion Avoidance Algorithms for TCP NewReno:

There are a variety of algorithms that are used for better performance and also to avoid congestion in TCP NewReno. These algorithms make sure that our packages are not misplaced (3). There are the primary phases of algorithms included Slow Start, Congestion Avoidance, Fast Retransmission and Fast Recovery.

TCP NewReno is modified version of TCP Reno. In TCP NewReno and TCP Reno that only difference between algorithms, phase of fast recovery. In TCP NewReno, several packet losses are more stable than TCP Reno. In TCP Reno, when duplicate packets are pick up, then it's enter into fast retransmit phase, but it will not get away into the fast recovery phase. In TCP NewReno, several packets are lost that is provide a signal that sequence of packets are being lost and it should be retransmit the packets. When

more packets are being lost from a series of data that it is sending data without retransmission time out (9).
The TCP New Reno is a very capable to handling multiple packets lost. If one packet lost that is considered, it is having a restriction to identify the lost packet and retransmit the packet per RTT.

The steps for congestion control in TCP New Reno are detailed below:

(1) Slow-Start Phase:-

In slow-start phase, to begin with cwnd (congestion window) that is equal to 1. In an each packet acknowledges the cwnd is increment until the congestion window (cwnd) is greater or equal ss_threshold.

(2) Congestion Avoidance Phase:

In Congestion Avoidance Phase, the congestion window is linearly increased by one segment of every RTT (Round Trip Time) while the network congestion is not detected. Their process is working until the pick up by timeouts or three duplicate acknowledgments.

(3) Fast Retransmit Phase:

After receiving the three duplicate acknowledgements that TCP dispatchers enter the Fast Retransmit Phase in Fast Retransmit Phase. The transmission rate has slowed down by the sender. If congestion is identified by timeouts, then ssthresh is position in the direction of half of the present congestion window. The congestion window is put, that is equal to 1 and insert into the Slow Start Phase. If the congestion is identified by three duplicate acknowledgements, then sender inserts into the Fast Recovery Phase without retransmit packet lost waiting for the retransmission timer to be expired. Then ssthresh is position in the direction of half of the present congestion window and the new congestion window to add the ssthresh to the pick up by duplicating acknowledgements that insert into Fast Recovery Phase.

(4) Fast Recovery Phase:

There are two acknowledgements in Fast Recovery Phase. [1] Full ACK: all segments are brilliant next to the start of the phase. In Full ACK, the congestion window is equal to the ssthresh and end of the Fast Recovery Phase and insert into the Congestion Avoidance Phase. [2] Partial ACK: some segments are brilliant. In Partial ACK, the congestion window is reduced by one less than pick up acknowledgement.

MATERIALS AND METHODS

In a wireless network, congestion is mainly detected through bit error; duplicate acknowledgement, RTT (Round Trip Time) and retransmission timer expire. Congestion control is very important role in computer networks. We broke down a major congestion control mechanism in TCP NewReno.

A. Adaptive Back off Response Approach (ABRA)

Here TCP NewReno, we analyzed that to calculate new RTO (Retransmission Timeout), namely Adaptive Back off Response Approach (ABRA) (8). ABRA is the result of a retransmit. This procedure is frequently, pending an ACK in support of the retransmitted packet have been arriving. The retransmission stimeout period that possibly will be extremely extensive and the path may be re-established a few instance back. It is a complete waste of time. So the ABRA is used in smoothing Round Trip Time (SRTT) (8), that is depends on pulling through the morals of slow start threshold, congestion window, and smoothed Round Trip Time, while retransmission timer will be expires. There are used to build up RTO intervals by two each time and build up with an assessment that is called back-up between single

and double, depending on the last_srtt, which is a weighted norm of last measured retransmitted timeout (8). Min_srtt is the smoothed round trip time with value 0.1 seconds. Max_srtt is the maximum smoothed round trip time with value 0.6 seconds.

In this result, to improve the performance metrics that were measured, including data packet pick up packet drop, packet retransmitted, and throughput.

Additional modification of the Fast Recovery algorithm to pick up the functioning of TCP NewReno, it has been set up with the intention of the TCP NewReno is useless in term of consumption of connection capability and unreasonable in throughput (5). There are some problems behind this process like [1]. Congestion window is split in two irrespectively of the deceased tactful of the network because long as a packet failure is observed. [2]. The crisis emerges with NewReno is with the aim of once at hand are negative response packet losses, except packets are reversed by additional than 3 duplicate acknowledgments; NewReno inaccurately introduce Fast Recovery, and split in two its congestion window. The basic thought is to change the congestion window (cwnd) of the TCP transmits depend on the stage of congestion on the set of connections as a result seeing that to permit assign additional packets in the direction of the address.

B. Modified Fast Recovery Algorithm:

Designed for the period of Congestion Avoidance, when the TCP sender picks 3 duplicate ACKs, it takes up the Fast Retransmit phase to retransmit come again present to be lost packet with no for the future retransmission timer to end (3,5). After that it calculates the new congestion window (cwnd), sets the ss_threshold to the higher of two segments and cwnd, and sets the cwnd to the ss_threshold value plus the amount of pick up photocopy acknowledgements and remains by way of the Fast Recovery phase (5). The TCP sender raises the cwnd by individual used in favour of all pick up photocopy acknowledgment, and forwards a fresh segment if authorized. With one-sided ACK, it retransmits the acknowledged segment and returns. With complete ACK that is producing the cwnd to the ssthresh value and enters into the Congestion Avoidance phase. If the TCP sender observed dead via timeout expires, it sets the ssthresh to the highest of two segments and cwnd, and sets cwnd to one segment, and insert on to the Slow Start algorithm.

Step: 1
With 3 DUPACKs: congestion window that is situate to 2(cwnd − Avgnum);
Ssthresh that is identical to utmost 2 congestion window;
Cwnd = ssthresh + 3;
Each Duplicate ACK pick up that congestion window is incremental;
Post fresh packet if permit;
Following one-sided Ack;
Wait in fast recovery;
Retransmit after that missing packet (per RTT);
Following complete Ack:
The Congestion window is identical to the ssthresh;
Way out Fast Recovery;
Call up Congestion Avoidance Algorithm;

Step: 2
When Timeout: window that is situate to 2(cwnd − Avgnum);
Ssthresh that is identical to utmost 2 congestion window;

That time the Congestion window is equal to the one;
Call up Slow Start Algorithm;

In this result, improve the packet delay and throughput transferring extra packets to the target.

C. Comparison of TCP NewReno and Routing protocol (DSDV and AODV):

We analyzed that improvement of TCP NewReno over MANET, comparison with TCP. We also include that important role of routing protocols in MANET, involve the study of DSDV and AODV routing protocol. In TCP New Reno, together timeouts and response of 3 DACK allow faster recovery (2, 3). t presumes the packet with the aim of without delay result the one-sided ACK pick up at fast recovery is missing, and retransmits the packet. Once there is not accurate and marks in lower functioning. In TCP, the packet failure, whichever outstanding to congestion or node motion results in a raise in the RTT (Round Trip Time) that raises the timeout and the adjustment congestion window (7).

There are two categories in routing protocols: proactive and reactive routing protocols. In proactive routing protocols, every link failure result in table update. In reactive routing protocols, updating arises only when node is applied. We analyzed that combination of TCP NewReno and DSDV (Destination Sequence Distance Vector) for the Wireless Network(7).

DSDV is the proactive routing protocol, all node work as a router. AODV (Ad hoc On-demand Distance Vector), is a reactive protocol that is considered for MANET. On demand and a distance vector routing protocol that is implying a path is well-known by AODV stating a target simply scheduled demand. As soon as the dimension of the network raises, a node-to-node routing protocol like AODV is additional popular (7).

On the TCP layer, congestion window of the TCP sender adapts the form on the stage of overcrowding in the arrangement therefore that additional amounts of packets can be reassigned. We remark to facilitate in TCP, all the spread packets create a round trip (RTT) reverses starting the receiver to the pickup. The RTT switches throughout the overcrowding as the web service load changes. On top of the foundation of receiving RTTs, we know how to able to manage our congestion window. These are RTT measure guidance in formative timeout and in TCP's retransmission scheme.

We make use of the most recent little facts of RTTs of conveyed packets and acknowledgements at pick up and dispatcher side, in that order and calculate a normal of its RTTs. The dissimilarity in common RTT and RTT of newest acknowledgement pick up is second-hand to decide the fresh standards of cwnd and ssthresh at the fast recovery phase of TCP New Reno (5,7) If dissimilarity in RTT's is unfixed, cwnd require that can not to be real changed linearly, whereas for outsized dissimilarity that we can modify the congestion window exponentially. It assists in achieving aim to agreement among the position of congestion, take full advantage of throughput and decrease timeouts.

D. Improve TCP NewReno in Multi-hop Wireless Networks (MWNs):

The presentation of NewReno reduces particularly that act as a Multi-hop Wireless Networks (MWNs). Persistent retransmission break that is a notable crisis of the routine reduction of NewReno over MWNs. Sender identifies the loss of a packet by the Retransmission Timeout that time the receiver retransmit the first unacknowledged data and reboot the congestion window that is equal to the 1 mss (maximum segment size) and the slow start phase that is increase according to this process. In MWNs, when multiple packets are missing beginning one window of segment that time retransmission timeout are

obligatory (6). In TCP NewReno, it cannot knob the persistent drop of congestion windows volume outstanding to retransmission timeouts and several missing packets of the refresh move packet from one window of data segment for the duration of the fast recovery (6).

We analyzed that new TCP variant called SRA (Sudden Recovery Algorithm), retransmission timeouts (RTO) that perform several missing packets transmitted fresh packet at the time fast recovery and comparison with TCP. The Fundamental arrangement of the algorithm that is to amass the uppermost series of quantity transmitted to a next volatile by the side of the time that sender enter into existing Fast Recovery action and measure up to the an unacknowledged packet that is used by the time of retransmission timeouts (7). It can also restore the several losses of packets on the outside of the retransmission timeout. Merely it suffers a crisis of the large RTT modification of MWNs and that time retransmission time-outs is frequently working with this mechanism. TCP NewReno that is compared with TCP SACK and Westwood that is originally a modification of TCP Reno.

Immediate Retransmission (IR) method is newly introduced process for particular managing retransmission timeouts if the dispatcher close timeouts for the defeat of fresh transmitted packets at the time of fast recovery. In Fast Recovery Phase, R-RTO store the uppermost series integer that variable is known as 'NewRecover' whenever receiver has a complete absence and then location the dimension of cwnd to slow start threshold (ssthresh) by the expire of the Fast Recovery and enter into the congestion avoidance state. When the dispatcher pick up three dupacks that time duplicate ACKs are a smaller amount or identical value that stored variable into the 'NewRecover'. There are two conditions (1) if yes ,retransmits the losses of a packet without delay, and (2) if no,the losses of a packet that retransmit is not enough duplicate ACKs, or several packet failures of R-RTO requirements to wait for timeouts.

Inside the Fast Recovery Phase, 'seq_before_timeout' variable that is store the value of the uppermost series number the sender-side. For the duration of fast recovery, the size of the congestion window is increased by the new sent packet by the sender and also renew the cost of 'seq_before_timeout'. Retransmission timeout (RTO) arises that time the sender investigate the packet to exist retransmitted (Unack) or not by RTO that value is smaller or equivalent to the cost of stocked variable that is a 'seq_before_timeout'. While a outcome, the dispatcher straight away retransmits the packet with no dropping the volume of cwnd. Show in Figure.1.

E. Improve TCP NewReno in Heterogeneous Wireless Network:

The Heterogeneous Wireless Network system has been designed with planned advantages of wireless characteristics like flexibility, transportability and effortlessness of utilizing and employment. Heterogeneous network situation associates mobile terminals among several web integrates which are able to selecting the best expected system or connection for records (2).

We analyzed that TCP crises arise outstanding to set of connections handovers and come up with a cross-layer assisted handoff TCP (TCP- CLAH) to upgrade on the solid achievement of diminishing client supposed glitches. TCP-CLAH scheme compared with TCP NerReno and outcomes are calculated. TCP-CLAH exceeds TCP NewReno in terms of jitter, round- trip time, and queue size, generating a lot added powerful, authentic, and handoff challenging and resourceful information transfers (2).

This is the main contribution of this work:
(1) TCP frameworks to be an improved service condition throughout handovers.
(2) Dropping congestion window of the period of handoff is important for guaranteeing service link and diminishing distraction and glitches.

(3) To progress service link for the duration of handovers from end to end BDP-based congestion window modification.

TCP's drawbacks during Handoff: aborts the older links previous to handoff completes, and aborts the older link relationship merely as the fresh connection is available. There are two types of issues like high-delay network to low-delay and vice versa.

BDP (Bandwidth Delay Product) and Handoff:
We analyzed that in instructing to decrease link glitches and preserve sufficient service link for the duration of a handoff the bandwidth delay product (BDP) of the connection have got to be quantified. The BDP store highest sum of information that can be transmitted in a set of connections link at a few applied time. It presents the total of information is transferred earlier than the rest ACK is arriving (2). The BDP is serving as the result of a link's power and its round-trip time.

F. Modification of TCP NewReno:
The modification of TCP NewReno that is used three mechanisms to identify the congestion control to get better performance in Wireless Network. We broke down a major congestion control mechanism in TCP NewReno. TCP NewReno does differentiate issues of packet losses outstanding to congestion or bit error. Only it always decreases cwnd (congestion window) in issues of packet loss and its decrease the execution.

1. **LT-TCP (Loss Tolerant TCP)** that is easily performed in a wireless network. In this mechanism, basically work with ECN (Explicit Congestion Notification) starting a set of connections routers. LT-TCP is used for FEC (Forward Error Correction) means to get back fragment errors and damages. It is basically a definite an error correcting segment is pre-generated and then reserved (5). PFEC (Proactive Forward Error Correction) segments that transferred beside with the original record segments. RFEC (Reactive Forward Error Correction) segments that transferred throughout the retransmission of before transferred segments.

2. **Indirect-TCP (I-TCP)** employs a TCP separating come up with dissimilar flow control and congestion control method for the infrastructure and infrastructure-less network [5]. There are two connection points' Base station (fixed host) and Mobile host (wireless connection). A base station clearly relocation, position in sequence to some other base station for the duration of handoffs. I- TCP brings quicker version to nomadic and wireless association cracks.

3. **TCP probing** mechanism that record segment is postponed or misplaced; the sender arrives interested in a probe cycle as a proxy for retransmitting and dropping the congestion window size. In a probe cycle that query data are changed connecting the dispatcher and pick up and also check the system and the usual transmission is set aside. There are two errors are generated in probe cycle such as constant error that TCP shrinkage its congestion window (cwnd) and ssthresh and the temporary accidental error that the sender continue transmission next to the identical window volume before inserting into the probe cycle.

TCP NewReno compared with TCP Westwood and TCP Reno. It also checked the performance of TCP Westwood and TCP Reno with TCP NewReno. TCP NewReno simply identifies the actual congestion in the set of connections or not. If data slice are misplaced outstanding to bit error, after that there is not a reduced transmission rate. We also preserve a number of timeouts and the number of 3-dupacks knowledgeable for the duration of an end. We work out the proportion of the number of timeouts to the number of 3-dupacks. We note that a very small percentage (in between 0.01 to 0.1) of the amount of timeouts and amount of 3-dupacks is to identify the bit error event, not a congestion event and a far above

the ground percentage (greater than 0.25) of the amount of timeouts and the amount of 3-dupacks that is identified as a congestion event.

We analyzed that TCP NewReno is improving the performance over wired and wireless environment. It also improves good part, throughput, and solve the bit error problem.

PROPOSED WORK:

We are proposing the differentiate issues of packet losses due to congestion or bit error. But it always decrease cwnd (congestion window) in all issues of packet loss and decrease the performance of throughput. The traditional TCP-NEWRENO cannot differentiate reasons of segment loss through either congestion or bit on wireless link. Therefore we proposed new approach for finding reason of loss with the help of flag (F) of TCP Header. We use 1 and 0 bit flag TCP Header for strong indication of congestion or bit error. We use F=0 for congestion and F=1 for bit error. This algorithm states if any segment loss due to any reasons during communication, receiver has been received out of order sequence numbers. When receiver receives out of order sequence number, it will start observing timing of next successive segments and continuous retransmit DUPACK. If next successive segment suffer from delay, then receiver assumes that congestion occurs in the medium and segment loss due to congestion. So set FLAG=0 in the third DUPACK. If next successive segment does not suffer from delay and received continuously without delay, then receiver assumes that congestion not occurs in the medium. So set FLAG=1 in the third DUPACK. In the TCP-NEWRENO, when the sender receives 3 DUPACKs, it immediately reduces congestion window (cwnd) to half without knowledge of reason of loss through either congestion or bit error. So it degrades the performance in real time communication. We modified TCP-NEWRENO protocol to improve the performance of TCP-NEWRENO. In our case when sender receives 3 DUPACKs, it does not immediately reduce congestion window to half. But sender observes the FLAG status of third DUPACK. if F=0 in third DUPACK, the sender assumes that packet loss due to congestion and set cwnd to half and enter into Fast Retransmit phase of TCP. if F=1 in third DUPACK, the sender assumes that packet loss due to bit error and not reduce cwnd to half and enter into Congestion Avoidance phase of TCP. So in this way we improve the performance and results show difference performance of TCP-NEWRENO and Modified TCP-NEWRENO.

CONCLUSION

TCP is association oriented and consistent transport protocol. TCP congestion is assuming packet losses generally outstanding to timeouts and three duplicate acknowledgement of transmission. Each TCP variant has a dissimilar mechanism for congestion control and loss recovery. TCP NewReno congestion window grows a great deal more rapidly. So they unwontedly reduce the congestion window due to bit error and packet loss. We will identify causes of packet loss and improve congestion of TCP New Reno over Wireless Network.

FIGURES

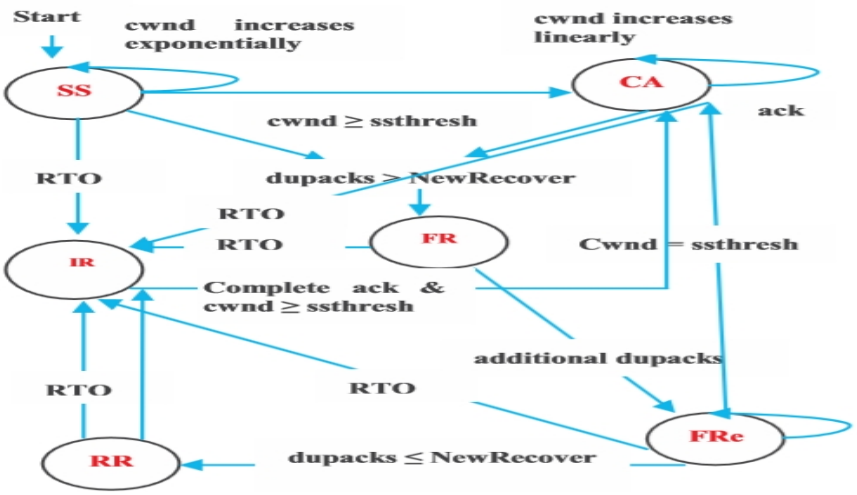


Figure: 1 State Transition Diagram of SRA (6)

TABLES

Comparison of TCP-NewReno Methods:

Sr. No.	Methods	Parameter	Limitations
1.	TCP Congestion Control Algorithms	Threshold, count of timeouts, Count of 3-dupacks, ratio of TC:DC, Ratio of TI:TD	Unwontedly reduce congestion window
2.	Immediate retransmission	Throughput, Fairness	If multiple losses of newly sent packets from one window of data occur, R-RTO cannot avoid RTOs.
3.	Round Trip Time	Packet delivery fraction for AODV, DSDV. Throughput	Routing link breaks, we need to find a new route message toward the destination
4.	Bit rate adaption	Throughput, Packet Delivery Ratio, Packet Drop Ratio, End-to-End Delay	End-to-End delay is lacking in this performance parameter
5.	Round Trip Time	Packet Received, Packet Drop, Throughput, Packet Delivery Ratio, No. of Nodes, Pause Time, Packet Retransmitted	It is limited to detecting and resending only one packet loss per Round Trip Time.
6.	Fast Recovery Algorithm	Throughput, Packet Delay	(1)It halves its congestion window irrespective of the network as long as packet loss is detected, (2)when there are no packet losses, but packet are reordered by more than 3-dupacks,mistakenly enters Fast Recovery, and halves its cwnd.

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An experimental investigation on the natural frequency of free un-damped, damped, and forced damped vibrations

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ABSTRACT

There are three well known analytical methods to calculate the natural frequency of a simple vibrating system. Namely are equilibrium method (D'almbert's principle), maximum energy method, and Rayleigh's method. In this paper, experimental investigations on the frequency and amplitude of different vibratory system are carried out by using universal vibration analyzer machine for each of the case, (i.e. free un-damped, damped, and forced damped) and harmonic response of dynamic vibratory system is also discussed. It is found that the results of experiments are quite similar with those of the analytical methods. The results also validate that damping effect reduces the amplitude of vibrating body and it leads towards concept of vibration isolation.

SUMMARY

The frequency of free-un-damped, damped, and forced damped is calculated experimentally and discussion is carried out.

Keywords: Harmonic exciter, magnification factor, speed ratio, phase angle, Universal vibration analyzer machine.

INTRODUCTION

Vibration is commonly known as an oscillatory motion. It is either simple or random. Any simple vibration is determined by means of three factors (i) Amplitude (ii) Frequency, and (iii) Timing of the oscillations relative to some fixed time. Natural frequency is a free frequency of any vibrating system and does not depend onto the intensity of the stress, but only on the physical characteristics of the system. In the free un-damped vibratory system, there is no element that causes dissipation of energy during the motion of mass, the amplitude of motion remains constant with time (Ignore resistance due to air).In

actual practice there is decrement in the amplitude of vibration with time due to resistance offered by surrounding medium. Such type of vibratory system is known as damped vibratory system. In forced vibratory system external excitation is applied either harmonic or non harmonic but periodic, or non-periodic, or random in nature. Harmonic response and transient response are the consequent effect due to harmonic excitation and transient excitation respectively. Here, investigation is carried out with vibratory system surrounded by air.

MATERIALS AND METHODS

Principle of each of three analytical methods and its procedure are described subsequently. Equilibrium method states that "the summation of forces acting onto the body is equals to zero." Maximum energy method states that "the summation of kinetic energy of body and potential energy of body is equals to zero." Rayleigh states that "the maximum kinetic energy of body is equals to maximum potential energy of body." Procedure of analytical methods is as follows: Free body diagram for different case of vibration is shown (Fig. 1) respectively. It is also shown that the initial displacement is in downward direction, hence the inertia force and damping force is in upward direction. At instant, the procedure of each method is given for the free un-damped vibration. See (Fig.1.–(a))

Equilibrium method [1]: The equilibrium method is also known as D'Alembert's Principle. When mass 'M_e' is attached to a free spring, its static deflection would be say 'δ_{st}', due to the self weight.

$$k = \frac{W}{\delta_s} = \frac{M_e g}{\delta_s} \quad \text{(Eq.1)}$$

Where, W = self weight,

δ_s = static deflection

As per D'Alembert's principle, a dynamic body can be converted into a static equilibrium by considering that an inertia force F=$M_e\ddot{x}$ acting at C.G of the body in the opposite direction of the acceleration. Magnitude of inertia force is equal to the product of the mass and the acceleration.

$$\sum \text{Forces acting onto body} = 0 \quad \text{(Eq.2)}$$

$$-M_e\ddot{x} + M_e g - k(x + \delta_s) = 0 \quad \text{(Eq.3)}$$

$$M_e\ddot{x} = M_e g - k - k\delta_s \quad \text{(Eq.4)}$$

Where, x = Displacement

From Eq. (1), we can write Eq. (4) as,

$$M_e\ddot{x} = -k \quad \text{(Eq.5)}$$

$$\ddot{x} + \frac{k}{M_e}x = 0 \quad \text{(Eq.6)}$$

The General Equation for Simple Harmonic Motion is given by,

$$\ddot{x} + \omega^2 x = 0 \quad \text{(Eq.7)}$$

Now Comparing Eq. (6) with Eq. (7);

$$\omega^2 = \frac{k}{M_e} \quad \text{(Eq.8)}$$

$$\omega_n = \sqrt{\frac{k}{M_e}} \quad \text{(Eq.9)}$$

$$T_p = \frac{2\pi}{\omega_n} = 2\pi\sqrt{\frac{M_e}{k}} \quad \text{(Eq.10)}$$

The frequency in hertz will be,

$$f_n = \frac{1}{T} = \frac{1}{2\pi}\sqrt{\frac{k}{M_e}} \quad \text{(Eq.11)}$$

Where,

ω_n = Angular natural frequency of the sring mass system

T_p = Time period

f_n=Natural frequency of system

Maximum Energy method [1]: The energy conservation law states that "The energy can neither be created nor destroyed, but it can only convert from one form to another." According to the statement,

$U = P + K = C$ (Eq.12)

Where,

U = Total Energy

PE = Potential Energy

KE = Kinetic Energy

$K = \frac{1}{2} M_e \dot{x}^2$ (Eq.13)

Where, $\dot{x} = v$ o the m .

$P = \frac{1}{2} k x^2$ (Eq.14)

According to Eq. (12),

$U = K + P$

$U = \frac{1}{2} M_e \dot{x}^2 + \frac{1}{2} k x^2 = c$ (Eq.15)

Differentiating Eq. (15) with respect to time,

$\frac{d}{d} = \frac{2}{2} M_e \dot{x}\ddot{x} + \frac{2}{2} k \dot{x} = 0$ (Eq.16)

$\dot{x} \neq 0$ Because the body has a deinte velocity,

$M_e \ddot{x} + k = 0$ (Eq.17)

From Eq. (7), we get,

$\omega_n = \sqrt{\frac{k}{M_e}}$ (Eq.18)

Rayleigh's method [1]: Principle of this method states that the maximum kinetic energy of a system is equal to its maximum potential energy. The maximum kinetic energy occurs when the system has maximum velocity, and the maximum potential energy occurs when the system is at maximum displacement from its mean position.

Let,

PE_{max} = Maximum Potential Energy

KE_{max} = Maximum Kinetic Energy

$X = A$

$P_m = KE_m$ (Eq.19)

Considering SHM,

$x = X \sin \omega$ (Eq.20)

When, $\sin \omega = 1$ means that the displacement is maximum,

$x_m = X$ (Eq.21)

Differentiating Eq. (20),

$\dot{x} = X \cos \omega$ (Eq.22)

When, $\cos \omega = 1$ means that the velocity is maximum,

$\dot{x}_m = X$ (Eq.23)

From, Eq. (21);

$P_m = \frac{1}{2} k x^2{}_m = \frac{1}{2} k X^2$ (Eq.24)

Similarly from equations Eq. (23);

$K_m = \frac{1}{2} M_e \dot{x}^2{}_m = \frac{1}{2} M_e X^2 \omega^2$ (Eq.25)

From, Eq. (19), Eq. (24) and Eq. (25)

$\frac{1}{2} k X^2 = \frac{1}{2} M_e X^2 \omega^2$ (Eq.26)

From Eq. (7), we get,

$$\omega_n = \sqrt{\frac{k}{M_e}} \quad \text{(Eq.27)}$$

According to equilibrium method the equation of motion for free un-damped vibratory system can be written as (See Fig.1.–(a)),

$$M_e\ddot{x} + k \ = 0 \quad \text{(Eq.28)}$$

According to equilibrium method the equation of motion for free damped vibratory system can be written as (See Fig.1.–(b)),

$$M_e\ddot{x} + c\dot{x} + k \ = 0 \quad \text{(Eq.29)}$$

According to equilibrium method the equation of motion for forced damped vibratory system can be written as (See Fig.1.–(c)),

$$M_e\ddot{x} + c\dot{x} + k \ = F_0 s\imath \ w \quad \text{(Eq.30)}$$

Universal vibration analyzer machine is used to investigate all the experiment regarding simple as well as harmonic vibratory system. Universal vibration analyzer machine with experimental set up is as shown in (Fig. 2.) Speed regulator is also used to investigate the effect of vibration for various speeds in case of forced vibratory system. This machine is not able to perform experiment of forced un-damped vibration system at very high speed. Plotter rotates at a speed of 29 mm/s to record the oscillatory motion for various cases of vibration. From the oscillatory motion time period is calculated by means of the ratio of the length of unit cycle to the plotter's speed and its inverse gives the frequency for respective case. Plotter diagram for each case and its results are discussed in subsequent sections.

In free un-damped vibratory system the body is displaced by means of external force and then, it is allowed to vibrate up-to its equilibrium position without any disturbance. Figure –3 is a plotter diagram for the free un-damped vibratory system.

Theoretically, we can calculate the value of natural frequency of free un-damped vibratory system as given below:

Where,

Mass of beam= 1.5 kg.

Length of beam= 0.78 m.

Let the exciter be at 'l' meter apart from the trunion.

k = Equivalent stiffness of the system = 4*stiffness of spring = 4 *3270 = 13080 N/mm.

Equivalent weight at beam centre, producing same moment about trunion so we can write,

Me = m * l / 0.39 = 7.5 * 0.39/0.39 = 7.5 kg.

Where, m = mass of exciter = 7.5 kg.

Me = Equivalent weight at centre

Therefore total mass acting at beam centre, $m_t = m_e + 1.5$ kg = 7.5 + 1.5 = 9Kg

Natural frequency for free un-damped vibratory system is equals to,

F= $(k/m)^{1/2}/2\pi$ = $(13080/9)^{1/2}$ /2*3.14 = 6.07 Hz.

In free damped vibratory system the body is displaced by means of external force then, it is allowed to vibrate up-to its equilibrium position without any disturbance. Figure –4 shows the diagram for the free damped vibratory system.

In forced damped vibratory system the body is continuously displaced by means of harmonic excitation. Figure –5 shows the graph of forced damped vibratory system. Figure 6 –8, is plotter diagram for forced damped vibration for various speeds at N_1, N_2, and N_3 respectively. ($N_1 < N_2 < N_3$)

DISCUSSION

The plotter diagram of free un-damped vibratory system validates that the amplitude of vibration remains constant (See Fig. 4.). It takes too much time to reach at its equilibrium position. The difference between theoretical and practical value of natural frequency for free un-damped vibratory system is 15.4%. Equation-(28) states that the motion of a given system is depend onto the two parameters that are

equivalent mass and equivalent stiffness. As the mass is increases the value of frequency will decreases but the value of frequency is linearly vary with stiffness of spring.
The plotter diagram of free damped vibratory system (Fig. 4.) validates that the around half of amplitude is decreased. The reverse amplitude is just little below of mean line implies that the damping system is under damped vibratory system. From the table-1, it is clearly seen that there is a reduction in the value of frequency of the free damped vibratory system than that of the free un-damped vibratory system. It clearly shows that the vibration amplitude decreased by means of damper.
Figure–5 shows the plotter diagram for the forced damped vibratory system having harmonic response. For better vision graph is divided into three sections. (See Fig. (6 – 8)) .Equation-(29) states that the motion of a given system is depend onto the three parameters that are equivalent mass, equivalent stiffness and damping co-efficient. As the mass is increases the value of frequency will decreases and the value of frequency is linearly vary with stiffness of spring. The effect of damping co-efficient is shown in terms of damping factor ($\boldsymbol{\xi = c/c_c}$,$\boldsymbol{c_c = c}$ [illegible]) [4]. According to the value of $\boldsymbol{\xi}$, There are three cases of vibratory system namely, over damped (*4*) ($\boldsymbol{\xi > 1}$), critical damped (*4*) ($\boldsymbol{\xi = 1}$), under damped (*4*) ($\boldsymbol{\xi < 1}$) vibratory system. The relation between amplitude and time for each of the given cases are as shown in (Fig. 9,). Investigation is carried out for various speed of exciter and results are recorded (see Table 1.).The graph shows that as the speed of the exciter is increases, the amplitude of vibratory system as well as frequency are also increases. Whenever the natural frequency of vibrating body is coincides with the frequency of external excitation, the phenomenon known as *resonance*, which leads to excessive deflection or failure of a body. (See Fig. 10.)
It is always essential to calculate the amplitude of given vibratory system to ensure that the system will not find resonance condition up to some extent. The amplitude of forced damped vibratory system is given by,

$$X = \frac{F}{\sqrt{[(k-mw^2)^2+c^2w^2]}} \qquad \text{(Eq.31) (2)}$$

$$\emptyset = \tan^{-1}\left(\frac{c}{k-mw^2}\right) \qquad \text{(Eq.32) (2)}$$

Where,
X =Amplitude of the system c =Damping co-efficient
m =Mass of the system k =Stiffness of the system
F =Harmonic external force w =Speed of system
$\emptyset$ =phase angle
Magnification factor is a ratio of given amplitude to the static deflection of system. It means that the effect onto the amplitude of vibration system having dynamic excitation with respect to the amplitude of same vibratory system as in case of static excitation.

$$\delta_s = \frac{F}{k} = D \quad u \quad s \quad f \quad F \ , \qquad \text{(Eq.33) (2)}$$

$$r = w / w_n = \text{frequency ratio,} \qquad \text{(Eq.34)}$$

The (Eq.31) and (Eq.32) becomes,

$$X = \frac{1}{\sqrt{[(1-r^2)^2+(2\xi r)^2]}} \qquad \text{(Eq.35)}$$

And

$$\emptyset = \tan^{-1}\left(\frac{2\xi}{1-r^2}\right) \qquad \text{(Eq.36)}$$

Relation between magnification factor and speed ratio, and phase angle and frequency are given.
(See Fig. 11.)

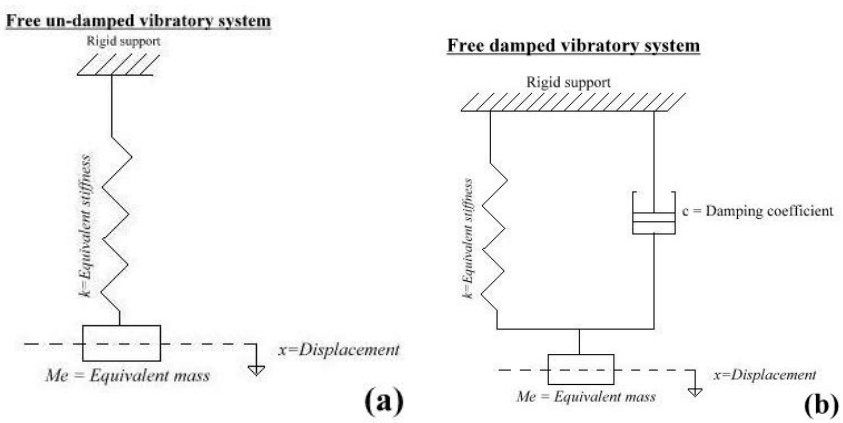
Characteristics of magnification factor and phase angle (*2*) :- (See Fig.–11)
In the case of a constant force (when r =0), the value of M=1.It is seen from the graph that for any specified value of *r,* a higher value of damping reduces the value of magnification factor. Also it is seen

that reduction in magnification factor in the presence of damping is very significant at or near resonance because damped natural frequency of the system and it is always less than the natural frequency of a system. The amplitude of vibration is maximum at the resonance condition. The amplitude of forced vibration becomes smaller with increasing values of the forcing frequency M tends to zero as r tends to infinity. (Ex. Rotor blades in the aero-plane are rotating at higher speed to reduce the amplitude of vibration. The variation in the value of phase angle is due to damping. Without it is either 0° or 180°. The phase angle increases as the value of damping decreases above resonance. For $\xi > 0$and $0 < r < 1$ the phase angle is lags the excitation. For $\xi > 0$ and $r > 1$ the phase angle is leads the excitation. For $\xi > 0$ and $r = 1$ the phase difference between the excitation and the response is 90°.For and large values of *r*, the phase angle approaches 180°, implying that the response and the excitation are out of phase.

CONCLUSION

Based on experimental results of given vibratory systems and literature, we concluded that the motion of a free un-damped vibrator system is depends on equivalent mass and equivalent stiffness. The motion of a free damped vibratory system is depend onto the three parameters that are equivalent mass, equivalent stiffness and damping co-efficient. For forced damped vibratory system, as the speed of external exciter increases the amplitude of vibration is increases and after resonance, we find one higher speed at or beyond which vibration of system tends to zero. From the results shown in table we can say that the Use of damper will reduce the cycle time. It is also seen that there is a difference between the values of frequency for the free un-damped vibratory system; the difference is subjected to mechanical error (Ex. Lubrication and loose joints etc.) as well as its surrounding (Not created vacuum).We also conclude that the amplitude of any vibratory system should either less or higher than that of at resonance condition to prevent it from extreme deflection.

FIGURES



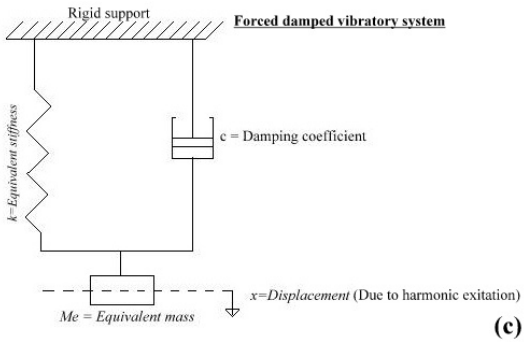


Fig. 1. FBD of free un-damped, damped, and forced damped vibration system



Fig. 2. Universal vibration analyzer machine

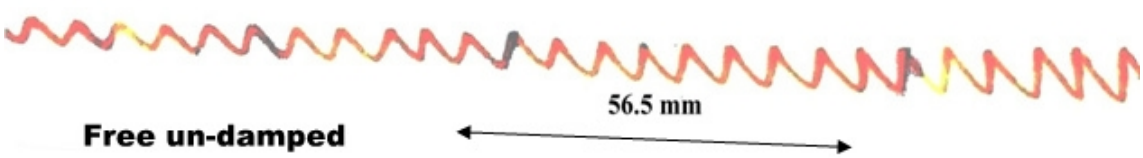


Fig.3. Free un-damped vibration

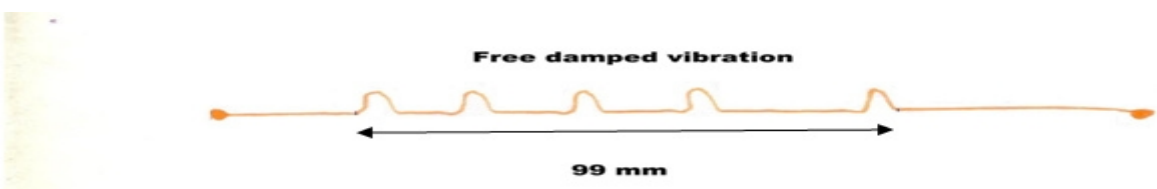


Fig.4. Free damped vibration

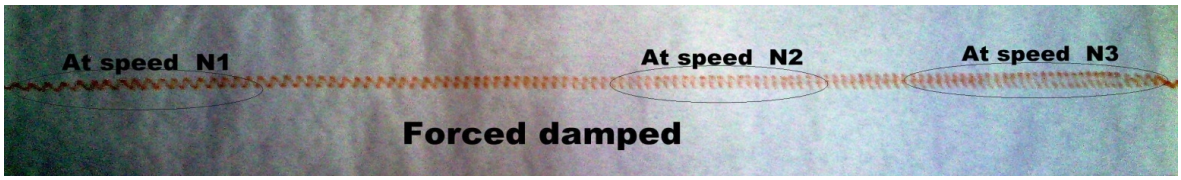


Fig.5. Forced damped vibration

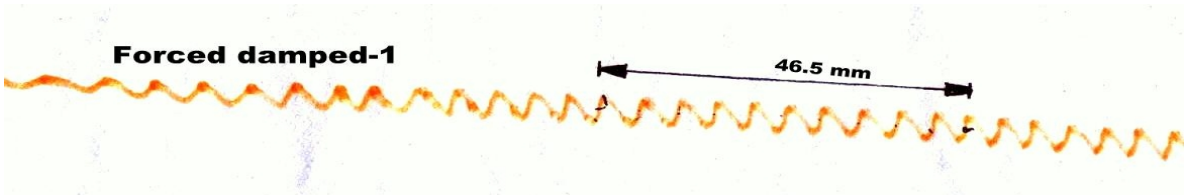


Fig.6. Forced damped vibration for speed N_1



Fig.7. Forced damped vibration for speed N_2

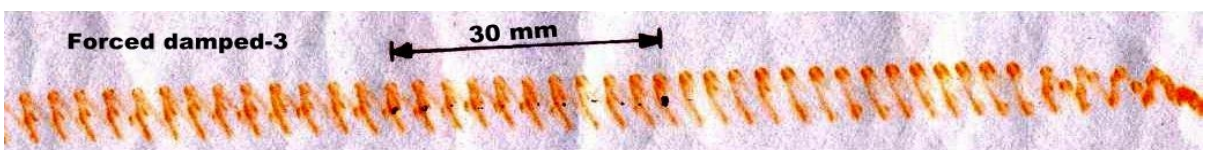


Fig.8. Forced damped vibration for speed N_3

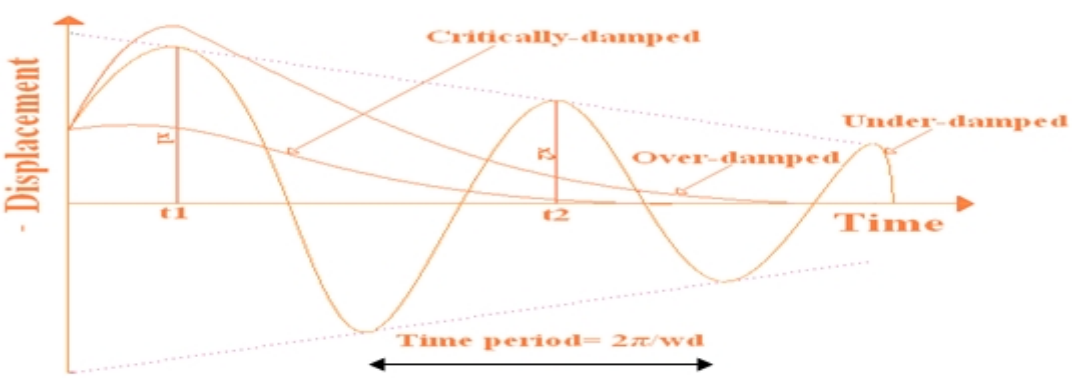
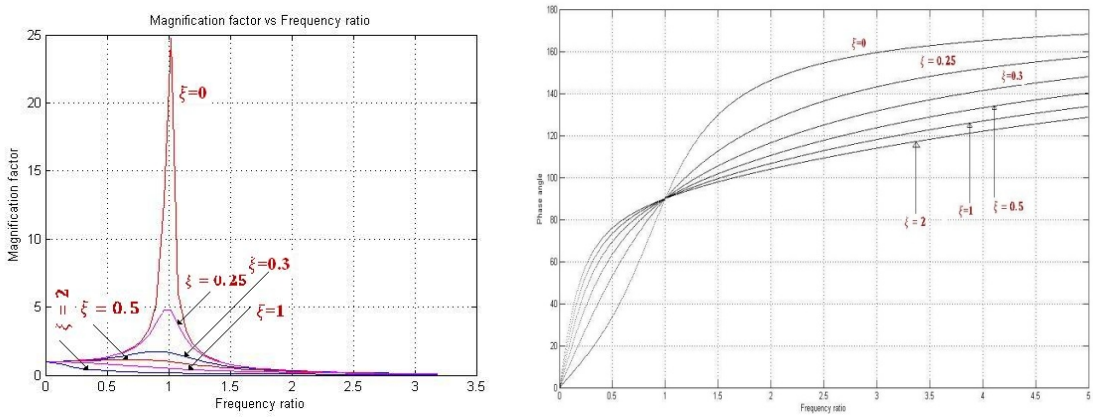


Fig.9. Over damped, under damped and critical damped vibration



Fig.10. Tacoma Narrows Bridge [3]



(a) (b)

Fig.11. (a) Magnification factor vs. frequency ratio
(b) Phase angle vs. frequency ratio

TABLES

Table.1. Experimental values of frequency:

Free un-damped		Free damped		Forced damped	
Time period per unit cycle	Frequency	Time period per unit cycle	Frequency	Time period per unit cycle	Frequency
0.194 seconds	5.13 hertz	0.341 seconds	2.93 hertz	0.16 seconds	At speed N_1 6.23 hertz
				0.11 seconds	At speed N_2 9.09 hertz
				0.10 seconds	At speed N_1 9.71 hertz

ACKNOWLEDGEMENT

We are thankful to the Department of Mechanical Engineering of RK-University, tramba- Rajkot (Gujarat), India and colleagues for providing very good facilities as well as very good support for the successful completion of this work.

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(3)Tacoma bride, Farquharson's photo, Historical Photography Collection, University of Washington Libraries.

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Appendix:-

Matlab coding:-

```
% magnification factor vs frequency ratio
clc;
clear all;
r=0:0.4:3.2;
z=0;
e=0.02;
t=0.3;
u=0.5;
d=1;
i=3;
x1=1./((1-r.^2).^2+(2*z*r).^2);
x2=1./((1-r.^2).^2+(2*e*r).^2);
x3=1./((1-r.^2).^2+(2*t*r).^2);
x4=1./((1-r.^2).^2+(2*u*r).^2);
x5=1./((1-r.^2).^2+(2*d*r).^2);
x6=1./((1-r.^2).^2+(2*i*r).^2);
plot(r,x1,'r')
hold on
plot(r,x2,'m')
hold on
plot(r,x3,'b')
hold on
plot(r,x4,'r')
hold on
plot(r,x5,'m')
hold on
plot(r,x6,'r')
hold on
grid on
xlabel('magnification factor')
```

```
ylabel('frequency ratio')
title('magnification factor vs frequency')

% Phase angle VS Frequency ratio
clc;
clear all;
for j=0:0.5:3
   for r=0:0.005:5;
      F=atand(2*j*r/(1-r^2));
      if F<0
         F=F+180;
      end
      plot(r,F,'k--');
      xlable(Frequency ratio);
      ylable(Phase angle);
      grid on;
      hold on;
   end
end
```

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(Proceedings available for download at rku.ac.in/icre)

RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

The security perusal of big data in cloud computing environment

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ABSTRACT

In modern era no one can deny the fact that cloud computing has emerged as one of the most significant technology which is consisting a demeanour to leverage computing as a utility. This has become possible because virtualization has provided the robust base for the implementation of the cloud computing. Enormous data is going to get produced depending upon the usage behaviour and the heterogeneity among the users, further it is fact the generated data holds cardinal importance when it is altered, modified or shaped into a predefined format (information), which later can be batter utilized to identify certain patterns which results into batter business decisions for any organizations. At the same time one need to take into serious consideration the scale and rate at which the data is getting generated and the ways and means in which it could be made secured with the help of security algorithms.

SUMMARY

The paper highlights probable usage of various security algorithms for the security of big data on cloud.

Keywords: Cloud Computing, Big Data, virtualization, security

INTRODUCTION

The providers of cloud utilities consist of mainly three models namely software as a service, platform as a service and infrastructure as a service, here we are about to emphasize on infrastructure as a service model which is capable of providing utility services like storage, computation power, etc. our area of concern is about storage because the enormous amount of generated row data has to be made persistent somewhere, so that it can be further processed and utilized. As it has been mentioned that the data which

is to be stored on the cloud is not physically occupied by the owner, the biggest concern from the end user's perspective is about integrity of data.

When we talk about the scale and rate of the data which is getting produced and stored on the cloud it becomes quiet cumbersome to manage it effectively and efficiently as when we talk about big data it could be either in structured format, un-structured format or semi-structured format.to handle these much variety of data at this huge scale it is prime necessity to develop some robust security mechanism and tools.

Big Data

A model for enabling ubiquitous, convenient, on-demand network access to a shared pool of configurable computing resources, like Networks, Servers, Storage, Applications and Services, that can be rapidly provisioned and released with minimal management effort or service provider interaction.

The heterogeneous data (structured, semi-structured and unstructured) which is enormously huge in scale processed , computed and analyzed to capture certain patterns which could be utilized to make appropriate decisions. In short we can brief big data as anything which is beyond human and technical infrastructure needed to support storage, Processing and analysis.

Cloud computing

It is a technology which is offers services virtually on demand basis to the customers which is the best feature of the cloud that it offers 'pay as you go' that is based upon the amount of resources consumed by the clients for the specific duration. It is responsibility of cloud providers to keep log of the same and provisioning or de-provisioning of the resources based upon the needs. The charges to be conducted per usage and the resources to be provided are to be mentioned in the service level agreement which takes place between the consumers and the providers of the cloud.

How big data works on cloud computing

All of us are aware with the fact that in digital world the amount and the type of data which is generated by various sources like web application, portals, web sites and other domains. It might not be feasible for the small scale organization or industries to handle these data, they can easily opt for the cloud services and virtually store the data at other locations. Cloud service providers like amazon, Microsoft azure and Google are having the facility to store the data of the clients and provide the infrastructure for the same.

Data security

The biggest concern of the cloud computing is the security of the data which is stored on the cloud, data owners are mainly worried about where the data is stored exactly and the security preventions and the measure the service providers of the cloud are practicing for the same. If the security of the data is not maintained in that case it may Couse several unfavorable circumstances like denial of services, un-availability of the data, compromise in the confidentiality of the data.

Denial of services.

Denial of service is a kind of attack on any system in which the system is exploited in a way that it is no more capable to handle the requests from the clients and process it effectively. This will lead to the congestion in the system and results in to denial of the services to the clients. The person with the malicious intent may flood the system with n number of requests or provide the processing requests beyond the capabilities of the system so that it becomes bottleneck.

Integrity

Integrity of the data is nothing but making sure that the credential data which is stored on the cloud is not tempered by any intruder or the person with malicious intent. The objective behind doing this is to either make the data loss or change the data in a format that it is no more of use for the concern persons. There are tools available in the market such as vulnerability scanner which performs monitoring of the cloud system and perform scanning in the cloud system for known vulnerabilities.one can use the option to schedule the scan periodically or on specific date and time so that it can be assured that the data remains untouched. Further if the data is going to be updated by multiple users then also it has to effectively synchronize such that any user can access the same updated copy of it.

Confidentiality.

Confidentiality mainly refers to keeping the data private by allowing access to legitimate users. This includes privacy of not only sensitive data but also Meta data or usage logs. Access control mechanism and protocols plays the vital role in this.

Availability.

Availability of the data refers to make the data available as per the anticipation and expectation of the end user. When so ever legitimate user requests for the data it has to be provided with the same.in order to achieve this it is required to have well defined architecture and concise service level agreement.

Cryptography

A great deal of quality work have been carried out till now in order to enhance the security of big data on cloud, despite of it there is a fissure which could be exploit in order to make the system vulnerable, which is the reason that researchers have been trying to make the system as robust as possible by implementing various encryption algorithms and technique. There are several algorithms capable of making the data theft prone system and also equipped to maintain the integrity and confidentiality of the data. In this paper we are going to walk through such encryption algorithms (*1-2*) categorized as symmetric key algorithms, asymmetric key algorithms and hash algorithms. All mentioned techniques works on the basis of encrypting the data at one and decrypting at other. Which is nothing but altering and tempering of the content of the data with intent of making it not understandable to the intruders, hence making the data meaning less for them. The exact reverse process is to be carried out in order to get the data in the normal format so that legitimate receivers of the data can have meaningful access.

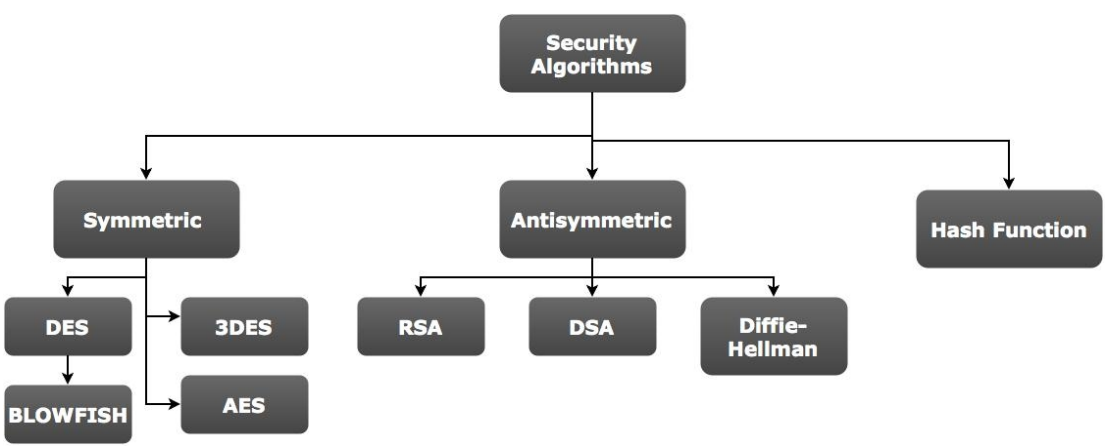


Fig. 1: Types of security algorithms.

Symmetric Algorithm

In symmetric encryption algorithm, encryption and decoding requires that the same algorithm and key are utilized to both encipher and disentangle the message. There is a private key that is utilized to encode and decode the message at both finishes. Symmetric encryption key technique is greatly quick and proficient for preparing scrambles and decode message. Symmetric encryption calculation gives secrecy, trustworthiness and accessibility however it neglects to give genuineness and non-revocation.

Advanced Encryption Standard (AES)

Advanced Encryption Standard (AES)(6)(7) symmetric encryption algorithm with key length of 128-bits for this purpose

- The client chooses to utilize cloud benefits and move his information on the cloud.
- User presents his administration necessities with CSP's and picks supplier offering best services.
- When movement of information to the picked CSP happens and in future at whatever point an application transfers any information on the cloud, the information is encrypted and after that sent.
- The encryption procedure is done utilizing AES calculation.
- Once encrypted, information is transferred on the cloud.
- Any solicitations to peruse the information will happen after it is decrypted on the clients end and after that plain content information can be perused by the asking for.

The plain content information is never composed anyplace on cloud. This incorporates a wide range of information. This encryption arrangement is straightforward to the application and can be coordinated rapidly and effectively with no application changes by any stretch of the imagination. The key is never put away by the encoded information, since it may bargain the key moreover. To store the keys, a physical key administration server can be introduced in the client's premises.
This encryption arrangement secures information and encryption keys and ensures they stay under client's control, and are never uncovered away or in travel.

Data Encryption Standard (DES)

The Data Encryption Standard (DES)(3) is a symmetric-key block cipher. At the encryption site, DES takes a 64-bit plaintext and creates a 64-bit cipher text, at the decryption site, it takes a 64-bit cipher text and creates a 64-bit plaintext, and same 56 bit cipher key is used for both encryption and decryption. The encryption process is made of two permutations (P-boxes), which we call initial and final permutation, and sixteen Feistelrounds (4). Each round uses a different 48-bit round key generated from the cipher key according to a predefined algorithm.

DES performs a beginning stage on the whole 64 bit piece of information. It is then split into two, 32 bit sub-squares, which are then gone into what is known as Feistel rounds. Each of the rounds are indistinguishable and the impacts of expanding their number is twofold - the calculations security is expanded and its transient proficiency diminished. Toward the end of the sixteenth round, the 32 bit L15 and R15 yield amounts are swapped to make what is known as the pre-yield. This [R15, L15] connection

is permuted utilizing a capacity which is the precise reverse of the beginning change. The yield of this last change is the 64 bit figure content.

3DES

The algorithm was proposed in late 1990s which is nothing but enhancement of DES. In this the technique of the encryption is pretty much same as the previous version but connection is performed three times to reach to the next level.it occupies 64 bit square size with 192 bits of key size. The performance level is quite low and the consumption of the resources is more comparatively.

Blowfish

The algorithm was developed in early 1990s became the widely used one. The best thing about the algorithm is it works with variable key and the size of block cypher used for the same is 64-bit with 132 bits of key size.it is a very robust algorithm.in general the algorithm occupy very less time complexity as it is capable of providing adequate processing at very low consumption of resources.

Asymmetric Encryption

Asymmetric encryption algorithm utilizes two keys rather than one. One is a private key just known not beneficiary of the message and the other is an open key known not and can be uninhibitedly disseminated. Either key can be utilized to encode and unscramble the message. However in the event that just key A is utilized to encode the message then just key B can be utilized to unscramble it. Alternately, if key B is utilized to scramble the message then just key A can be utilized to decode it.
Asymmetric algorithms are slower than symmetric calculations. Yet, it has preferable key dissemination over symmetric calculation. It has better adaptability furthermore gives credibility and non-renouncement

RSA

The RSA algorithm named after Ron Rivest, Adi Shamir, and Leonard Adleman. It depends on a property of positive whole numbers. RSA utilizes secluded exponential for encryption and decoding. RSA is a calculation for open key cryptography, includes an open key and a private key. The general population key can be known not and is utilized for encoding messages. Messages scrambled with general society key must be unscrambled utilizing the private key.

Diffie-Hellman (DH) encryption

DH algorithm (9) utilizes a half breed model from symmetric and asymmetric. In this system, asymmetric encryption is utilized to trade private key safely over an open system for private key dispersion. General society key is shared among all. At that point both celebrated can utilize their private keys to encode and decode messages utilizing symmetric cryptography.

Digital Signature Algorithm (DSA)
In DSA algorithm the random signature value K has to be distinctive, undisclosed, and in chunk. It has to be in this manner otherwise the private key would be disclosed to attacker.one simply cannot use the same value of k multiple times in that case if few bits of k is compromised then easily DSA would be cracked by any attacker.

Hash Function

Hash function (10) helps to maintain integrity of the data. It provides a seal/ shield to the data before transmission. If the seal is found to be broken at the receiving end, it will state that something has been changed in the file. Hash function can be a checksum, index data in hash table, etc. They are used in Message Authentication Codes (MAC), Digital Signatures and many information security applications. There are many hash functions available such as Message Digest (MD) and Secure Hash Algorithms (SHA). Various versions of hash functions have been published like MD-2, MD-4, MD-5, SHA-1, SHA-224, SHA-256, SHA-384 and SHA-512.

Performance assessment of Security algorithm on cloud environment

For the performance measurement we have elected main parameter mean processing time for various inputs. The mean processing time is defined as the total time required to encrypt data. The cloud environment implemented on eclipse SDK and Google App engine. For the implementation purpose we have gone through different algorithms like AES, DES, Blowfish, MD5 and the data input sizes: 15 KB, 30KB, 45KB, and 60KB.

Table-1: Comparison of Mean processing time of the algorithms on cloud environment

Input	AES	DES	Blowfish	MD5
15KB	2.2	4.9	2	1
30KB	2.9	6.1	2.6	1
45KB	3.3	8.25	3	1
60KB	4	9.75	3.2	0.5

Mean processing time is calculated in milliseconds

CONCLUSION

It can be observed from the experiments that as we increase data input size the time required to encrypt data is also increases. Further among the all security algorithms MD5 provides the consistent performance even after increasing the data input size, while other security algorithms consumed more time.

In future we will be implementing security algorithms with increased data input size in simulation environment like cloudsim with integration of eclipse framework to analyze the results, we are also planning to implement hash function to enhance the security level of big data in simulated environment.

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A Shot Boundary Detection utilizing Abrupt Transition

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ABSTRACT

Video processing gives a comprehension of the scene that it depict. It is a fundamental part of various innovations including video observation, mechanical autonomy and mixed media. It speaks to a range of exploration with immense development in the later past. Video shot limit location is one of the exploration works in the field of video preparing. Numerous scientists are attempting to advance distinctive calculation in this appreciation. Here we introduce a brief writing overview that portrays the work done till date.

SUMMARY

A shot boundary detection using adaptive histogram method for different types of videos.

Keywords: Video shot boundary detection, Cut detection, Threshold, Histogram Difference

INTRODUCTION

The preeminent necessity of any sight and sound industry is video. Throughout the years businesses has created far reaching and finish measures and procedures to file, store, alter, recover, succession and present video material. Shot limit discovery is generally the opening stride toward programmed video indexing and skimming (1). It depends on the reorganization of visual discontinuities brought on by the moves, to section a video stream into basic continuous substance units for resulting abnormal state semantic examination. The discontinuities typically found amid scene change or shot change. These

discontinuities happen in type of moves of various types which are classified into two gatherings: unexpected (as hard cut) and progressive (disintegrate, blur in, become dull, wipe) (2, 3). Routinely, if there exist outlines that are converged by the nearby shots yet have a place with neither of them, the move is known as a continuous one; else, it is known as a cut.

There are two diverse shot move classes of video altering in particular unexpected and progressive moves. The procedure of recognizing the diverse shot move inside of a video is known as video shot location or shot based video division or video apportioning (4, 9). This location assumes a basic part in video examination, video indexing, video rundown, systematizing the Digital Video Library as an organized Internet application for putting away, sorting, recovering the video arrangements.

An unexpected shot move is a sudden change starting with one video shot then onto the next and is just alluded as hard cuts or cuts (5). A delicate cut speaks to a progressive move between two shots which implies an arrangement of video edges that fits in with the first and the second video shots. The shots are gathered amid the altering stages utilizing assortments of strategies like blur in, become dull, disintegrate and wipe and these are considered as steady move impacts in the video successions (6).

The key issues of the shot limit discovery strategies are (i) decision of the element for representation of video edges ii) decision of likeness/separation measurements (iii) calculation for distinguishing unexpected and continuous transitions.The visual substance contrast between sequential casings inside of the same shot is basically brought on by two components: movement impact and lighting changes. Pixel based, Histogram based, Edge based, Motion based routines can be utilized for video shot limit identification. According to pixel based technique, the force estimations of the pixels of the same areas of the sequential edges don't change extensively unless there is a shot limit. The beginning pixel based calculations forms the aggregate of outright pixel power contrasts and if the distinction is more prominent than certain worth then video shot limit is dissected (10,11).

Additional strategy called as the histograms which don't change with the spatial alterations inside of an edge. Histogram contrasts are heartier against the article movement with a consistent foundation. Additionally, histogram contrasts are delicate to camera movement, for example, tilting or zooming, panning. Histograms speaks to the worldwide force of hues into an edge, once in a while two edges may have fundamentally same histogram. Further the edges additionally demonstrated valuable in shot limit (7).

MATERIALS AND METHODS

Using adaptive histogram based on the value of threshold different cuts will be find out using find out adaptive threshold based value Unexpected moves are moderately simple to identify, as there're regularly a major contrast between the two move outlines. The issue is proportional to identify this huge difference. This distinction can be measured on a pixel by pixel premise, in square based way, or taking into account some worldwide qualities of the edges, for instance, shading histogram and force histogram.
One of the compelling way is power histogram.

In this paper first converted videos into number of frames and compute the color histogram. According to the histogram we get the intensity value of different frames and calculate the histogram difference. After Calculate the mean and variance of the frame-to-frame difference, calculate the threshold value and get the cut transition frame number.

1. First converted videos into numbers of frames.
```
imwrite(currFrame, opFullFileName, 'jpeg');
```
2. Compute the color histogram
```
Co_Int = 256/numOfBins;
HG = zeros(numOfFrames, numOfBins);
```
3. Calculate the histogram difference
```
HD = [zeros(1, numOfFrames-1)];
fori=1:1:numOfFrames-1
HG(i) = sum(sum(abs(HGr(i, :) - HGr(i+1, :))));
end
```
4. Ccalculate the threshold value and get the cut transition frame number

In a persistent video casing progression, the histogram distinction is little, while for sudden move identification, the force histogram distinction spikes. Indeed, even there is an outstanding development or brightening changes between neighboring edges, the force histogram distinction is generally little contrasted and those tops brought about by unexpected changes. Accordingly, the distinction of power histogram with a legitimate edge is successful indistinguishing unexpected moves.

The threshold value to determine whether the intensity histogram difference indicates an abrupt transition.

RESULTS AND DISCUSSION

The presented shot limit discovery calculation has been tried on diverse video information sets. We have performed numerous tests on a different types of videos and got attractive outcomes. As appeared in figure. is the first casing which is shot change frame at frame 168, frame 321 and frame 459 from 1000 frames from movie scene and their histogram in which highest value represented the different frame will be started.

As same with the news video shot change start at frame 528 and frame 630 from 1000 frames from news scene and their histogram in which highest value represented the different frame will be started also same for cartoon and cricket. In addition with I have compared all this results with selective histogram.



Frame 167 Frame 168

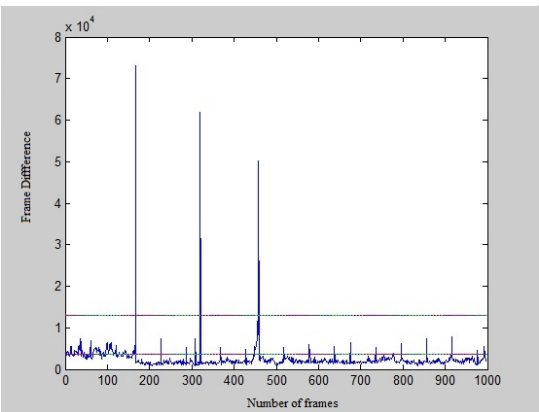


Frame 320 Frame 321

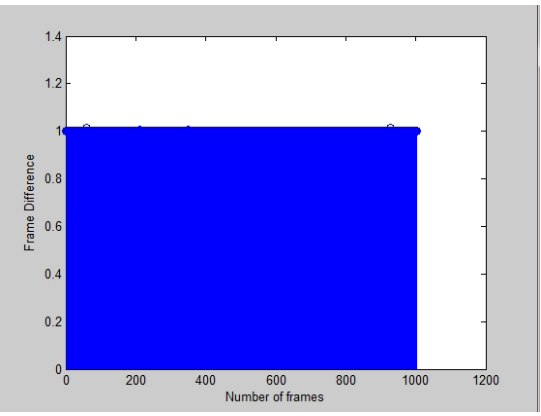


Frame 458 Frame 459

"**Fig 1**. Frame no. 167 to 459, where shot move can be seen between casings"



"**Fig.2** Video Shot Boundary detection for movie using additive histogram"



"**Fig 3**Video Shot Boundary detection for movie using selective histogram"





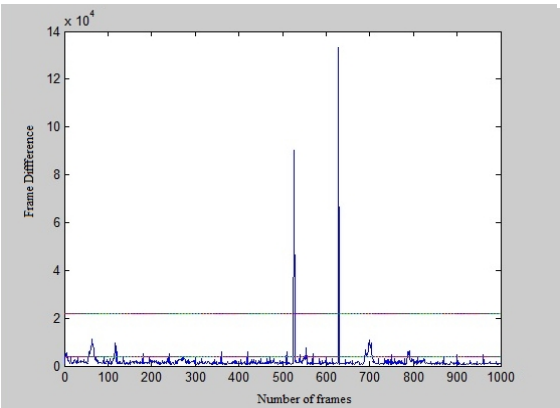
Frame 526 Frame 527



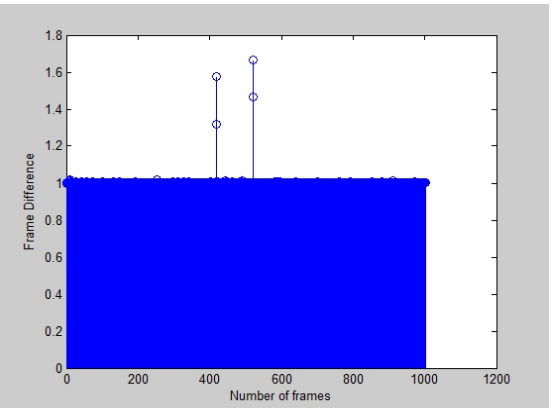


Frame 629 Frame 630

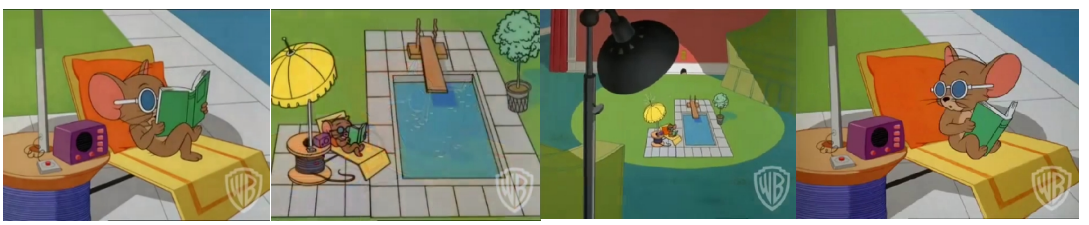
"**Fig 4**. Frame no. 526 to 630, where shot move can be seen between casings"



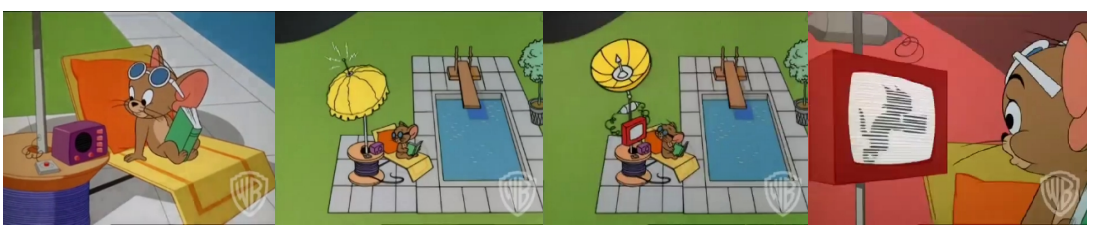
"**Fig.5** Video Shot Boundary detection for news using additive histogram"



"**Fig 6** Video Shot Boundary detection for news using selective histogram"



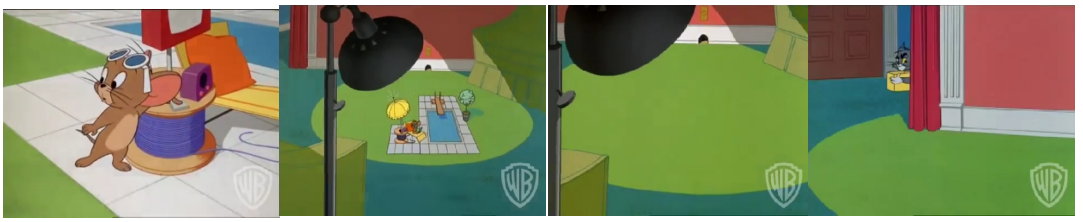
Frame 78 Frame 79Frame 228 Frame 229



Frame 268 Frame 269 Frame 361 Frame 362



Frame 410 Frame 411 Frame 522 Frame 523

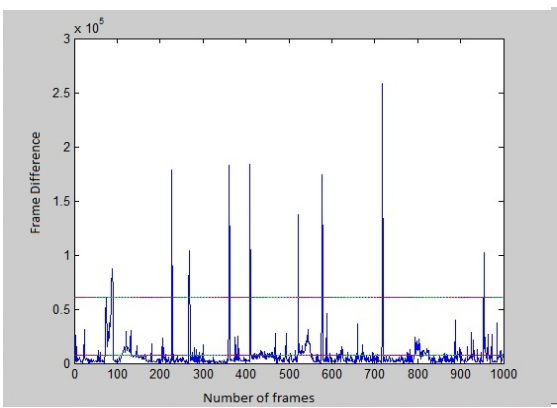


Frame 578 Frame 579 Frame 718 Frame 719

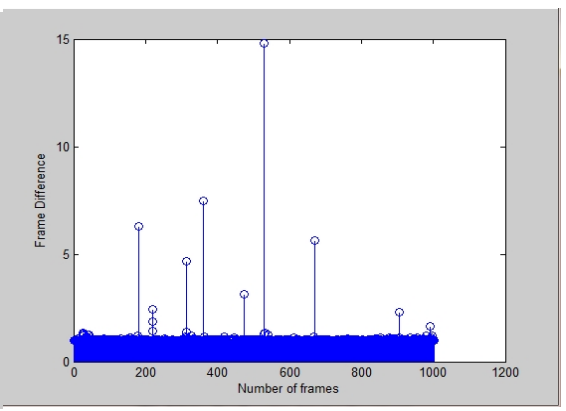


Frame 954 Frame 955

"**Fig 7**. Frame no. 78 to 955, where shot move can be seen between casings"



"**Fig.8** Video Shot Boundary detection for cartoon using additive histogram"



"**Fig 9** Video Shot Boundary detection for cartoon using selective histogram"



Frame 213 Frame 214

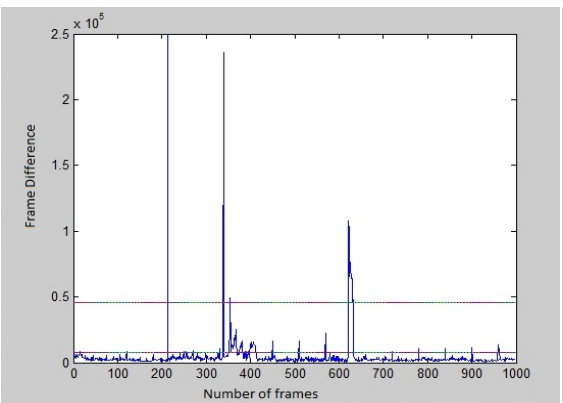


Frame 339Frame 340

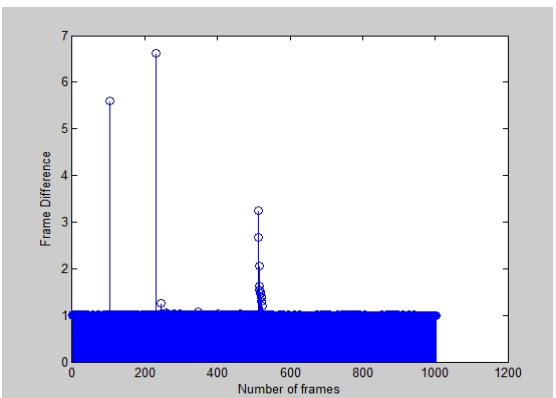


Frame 624 Frame 625

"**Fig 10**. Frame no. 213 to 625, where shot move can be seen between casings"



"**Fig.11** Video Shot Boundary detection for cricket using additive histogram"



"**Fig 12** Video Shot Boundary detection for cricket using selective histogram"

CONCLUSION

Exact shot change recognition is vital for sorting out video substance into significant sections for video section investigation. In this paper we have exhibited novel way to deal with the location of shot limits. We have initially uprooted the impact of enlightenment change, as regularly light impedance is mixed up as shot limits and its end is the real test to the shot limit recognition calculations. A shot limit identified if

the distinction in the edges is higher than the edge esteem. In addition with I have compared all this results with selective histogram. Using adaptive method results is better than selective for different types of videos.

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Comparative Study on Digital Image Watermarking based upon combined frequency domain transformation

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ABSTRACT

Millions of internet operators are everyday share multimedia information everywhere. Security is particularly important concern for the internet technology purpose behind is repetition of data, manipulation of existing information and unauthorized access. Watermark is technology which hide the some essential information in existing data with protection of illegal access of it. In this paper represent comparative study of different watermarking techniques and their result analysis as well as describe an invisible& combination of different frequency domain procedure for watermarking areDiscrete Cosine Transform (DCT), Discrete Wavelet Transform (DWT)&Discrete Fourier transform (DFT) which produce more robust output as compare to individual procedure. Performance measurement based on Peak-Signal Noise Ratio (PSNR), Mean-Square Error (MSE) and Signal-Noise Ratio (SNR) value. Proposed technique is evaluated against different kind of geometric, security, compression and cropping attacks. Based on performance measurement factors found combine technique get more robust result as compare to individual.

SUMMARY

Efficient Digital Image Watermarking with use of combine Frequency domain transformation techniques.

Keywords: Spatial Domain, Frequency Domain, DCT, DWT, DFT, PSNR, MSE, SNR

INTRODUCTION

In digital watermarking procedure insert a watermark in variety of data field like colourful 2D/3D images, audio files, video files and text documents etc. The complete embedding procedure are describe in fig.1 where watermark inserted in specific location desired by specific authentication key. Whenever

watermark is embedded it's paced as watermarked image. Every multimedia object which is numerically processed and attacks can be intently or perhaps unintentionally (7). So, placed watermark is should be more robust against all the different kind of attacks which is possible for present application. Any authorized person can recover the original watermark through insert the private key and extract the original watermark without any distortion of existing image. Watermarking technique based on arability either its visible and or invisible mode (3). Visible watermark are less secure as compare to invisible. Third party can easily modified or destroy embedded watermark due to its specific localization. In other side for invisible domain its make very difficult for attackers to detect exact location where watermark is actually placed. Without any authority no one can destroyed the embedded watermark.

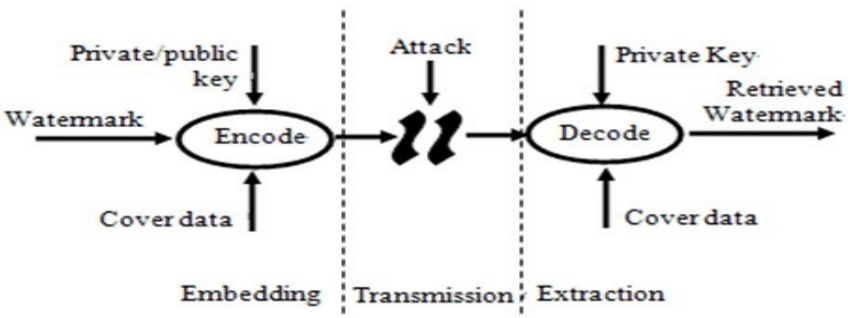


Fig.1 Process of Watermark embedding and extracting**.**

LITERATURE SURVEY

Digital watermarking works performed on new technique and basic survey for different method of execution of it. These works show how different basic techniques of secret writing is used to send secrete text and different watermarking technique is used to develop the new hybrid technique with better performance and parameters, like Mean square error, Pick signal to Noise Ration and Reduced elapsed time and also robustness.

Year	Author	Title	Methodology	Performances
2010	Naderahmadian. Y.; Hosseini-Khayat. S.	Fast Watermarking Based on QR Decomposition in Wavelet Domain	QR code image with use of means of wavelet transform	Robustness to Attacks, Visible Copyright Protection and Authentication Tool
2011	Nan Lin; Jianjing Shen; XiaofengGuo; Jun Zhou	A robust image watermarking based on DWT-QR decomposition	DWT, QR decomposition	Robustness, Invisibility and Higher Embedding Capacity
2012	Qing Liu,Jun Ying	Grayscale Image Digital watermarking Technology	DWT and Spread Spectrum	Increased Complexity of

		Based on Wavelet Analysis	Technology	Method, Extraction Is Not Good Enough
2012	Zhaoshan Wang, ShanxiangLv,Yan Shna	A Digital Image Watermarking Algorithm Based on Chaos and Fresnel Transform	Fresnel diffraction plane and Chaotic scrambling	Provide Both Good Robustness and Security
2013	Jithin V M, K K Gupta	Robust invisible QR code image watermarking in DWT domain	QR Codes and It can be scanned by QR code scanner	More Robust Than Previous Techniques
2013	Md. Maklachur Rahman	DWT,DCT and SVD based Watermarking technique to protect the image piracy	DWT+DCT+SVD apply Hybrid Watermarking	Increased NCC values than the existing DWT-SVD. Give More Robustness against different kind of NOISE. Good PSNR values that ensures more imperceptibility
2014	Mayank Agrawal , Ratnakar	Image Resolution Enhancement using Lifting Wavelet and Stationary Wavelet Transform	LWT+SWT+ ILWT Hybrid watermarking procedure	Increased Smoothness of picture quality + high resolution with ILWT
2014	AfrojaAkter , Ahsan Ullah	Digital Watermarking with a New Algorithm	2Level-DWT,DST, and Cox's Modified Algo with Salt and Pepper attack	Decreased PSNR Value with part of all the attacks proved better performance.
2015	Ms. MahejabiKhan,AjayKushwaha	A new Digital Image Watermarking algorithm based on Image interlacing , DWT,DCT	Fresnel bending plane with Chaotic climbing	Secure and Robust Watermarking based on the Combination of image interlacing.
2015	A. Amsaveni, C. Arunkumar	An Efficient data hiding scheme using Firefly Algorithm in Spatial Domain	Reversible data hiding and Firefly algo + Histogram Shifting	With Optimal location to hide the secret data with FA and histogram shifting with better quality of image.
2015	Dr. H.B.Kekre, Dr.TanujaSarode	Robust Watermarking by SVD of Watermark Embedded in DKT-DCT and DCT Wavelet Column Transform of Host Image	Adaptive Scaling Factor and DCT Wavelet + DKT+DCT Wavelet domain	DKT-DCT wavelet is more robust than DCT wavelet but histogram and noise attack robustness of DCT is better than DKT-DCT.

CLASSIFICATION OF WATERMARK

Digital Image Watermarking can be classified with two major parts. First one is Spatial domain and other one is Frequency domain which also called as Transform domain. As per the shown in fig.2 host image either proceed by spatial domain or frequency domain for embedding the watermark. In spatial model has less computation cost as compare to transform domain. Spatial domain get more robust output. But frequency strength of pixels are very sensitive to appearance any geometric attack (5, 8).

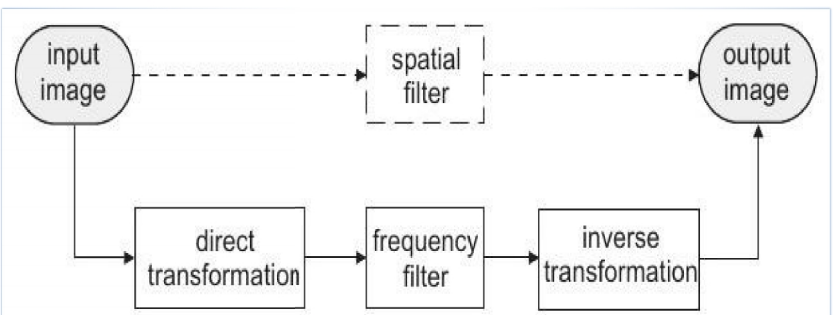


Fig.2 Classification of Watermark

Spatial domain: In Spatial domain watermark placed directly on randomly selected pixels on host image. As time of any geometric attack and compression attack watermark easily modified or may destroy form the location. Adaptive watermarking, Least significant bit, spread spectrum technique are example of spatial domain.

Additive Watermarking:
Known spatial domain method where image directly inserted on arbitrary selection of object pixels based on their intensity value (5). Disturbed noisy signal mostly follow the pattern like -1, 0, 1 or may it very from decimal value also. Through this technique watermark can easily track by any other user.

Least Significant Bit: Oldest method very popular for less computational cost and time consuming. In host image bunch of pixel value are randomly selected and their least significant bit replaced with selected watermark image. Through this procedure may image blurring change is increasing as well as due to any compression or geometrical attack it can to stable with its original intensity(2, 9) . Less robust as compare to other watermarking technique.
Frequency domain: Most of the watermark technique as based on Transform domain through its robustness and imperceptibility. Frequency domain proposed many procedure like cosine Transform, Fourier Transform and wavelet Transform. First modification apply on host image and after the inserting watermark image invers transform technique apply to recover the original image.
Discrete cosine transforms (DCT):which work based on frequency space instead of magnitude space in terms of cosine frequency domain procedure. Under the observation of different attack this transform technique is fragile specially change in position and size of the object image (4). DCT prove more robust technique as compare with spatial domain. DCT work on block based distribution of frequency value (11).

Discrete wavelet transforms (DWT): Wavelet Transform which work based on magnitude space instead of frequency space. Magnitude space work on three part like horizontal, vertical or based on diagonal (10). Host image can paced based on frequency range of the pixel in four sub bands where watermark placed on low frequency band due to high coefficient range and energy bit in low sub-band as compare with other LH, HL and HH sub-bands.

Discrete Fourier Transform (DFT): Fourier Transform which directly proceed on frequency part instead of magnitude with modification or reconfiguring cosine and sign part of the watermarked image (1). This kind of frequency transform which inserted watermark in images through two different way first with direct embedding method and other is template depended embedding(6). DFT which offers more toughness against geometrical attacks in terms of scaling, translating and rotation. In direct inserting watermarking technique is embedded by changing magnitude of Fourier transform & Phase coefficients and in template based inserting watermark image which introduced by different templates(9). Template like structure which inserted in DFT part to get judgement of transformation factor. The Main characteristic of DFT is central component where hold low frequency.

PROPOSED WORK

Basis on comparative study watermarked image must be robust against various attacks. As per the application of watermarking technique it should be copyright protected, Fingerprinting, Broadcasting Monitoring and Data Authentication. Quality of any watermark image is depend on imperceptibility and robustness. Performance of any embedded image is measure by PSNR, MSE, SNR and BER. Based on different valuation of it find the appropriate technique for the watermark. With approach of individual system its contain less computational time even less cost effective but based on the quality measurement hybrid technique are more powerful as compare to the individual. Based on the literature survey up to 2^{nd} level DWT transform level is on execution but if we try to apply 3^{rd} level of DWT is more effective with use of DCT and DFT frequency domain combination. MSE means mean square error ratio which decrease with use of DCT and DFT instead of single execution. Through my proposed work its will be proved by use of 2D+3D coloured images. Which provide more noiselessness and more robust watermarking procedure.

COMPARISON DIFFERENT WATERMARKING ALGORITHM

Algorithm	Advantage	Disadvantage
LSB	Easy to implement and understand	It lacks basic robustness
	Low degradation of image quality	Vulnerable to noise Vulnerable to cropping, scaling.
	High perceptual transparency.	
	Gain factor can be increased resulting in increased robustness	Image quality gets decreased due to very high increase in gain factor.
Patchwork	High level of robustness against most type of attacks	It can hide only a very small amount of information.
DCT	The watermark is embedded into the coefficients of the middle frequency, so the visibility of image will not get affected and the watermark will not be removed by any kind of attack.	Block wise DCT destroys the invariance properties of the system.

Algorithm	Advantage	Disadvantage
DWT	Allows good localization both in time and spatial frequency domain	Cost of computing may be higher.
	Higher compression ratio which is relevant to human perception.	Longer compression time.
		Noise/blur near edges of images or video frames.
DFT	DFT is rotation, scaling and translation (RST) invariant. Hence it can be used to recover from geometric distortions	Complex implementation
	Strongest Component of the DFT is central component which contain low frequency	Cost of computing may be higher.
	More Robust as compare to other Transform technique.	DFT also resist cropping because effect of cropping leads to blurring of spectrum .
	Good for JPEG Compression Attack and Geometric Attack.	

CONCLUSION

Digital image watermarking method is very dominant tool for copyright defence, content validation, temper recognition etc. This paper contributes a detailed study on different digital watermarking procedure and their application. It's provide relative analysis of various watermarking techniques and

fundamental procedure for inserting and extracting watermark from embedded image. Quality of any watermark image is depend on imperceptibility and robustness. Performance of any embedded image is measure by PSNR, MSE, SNR and BER. Based on different valuation of it find the appropriate technique for the watermark. So, as part of this analysis based on measurement valuation we can say that instead of applying single technique hybrid technique get more robust results for digital watermark.

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Effect of variable conductance on Ougassing rate in Vacuum chamber

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ABSTRACT

In vacuum chamber as the pressure is lower, the outgassing rate is more important i.e. the evolution of gases from the surfaces exposed to the vacuum. Conductance modulation method is a technique to measure outgassing rate by varying the orifice conductance between pumping chamber and sample chamber. At 10^{-7} mbar pressure drop and 10 m^2 surface area, Outgassing rate is calculated for different values of Conductance (C_1& C_2). where C_1 & C_2 are functions of cosine. Graphical representation of Outgassing rate and modulated conductance shows that outgassing rate varies in cyclic manner. This paper describes Conductance modulation technique, materials and methods. It also reports results of outgassing rate versus variable conductance at 10^{-5} and 10^{-9} mbar pressure drop.

SUMMARY

Outgassing rate is measured by conductance modulation method by varying conductance in vacuum chamber.

Keywords: Outgassing rate, Conductance modulation, Orifice

INTRODUCTION

Vacuum region is different in all the vacuum pumps and gauges. So it is very difficult and costly to achieved lover pressure. Also outgassing is very important in the lower pressure. So relatively outgassing is important in the rough vacuum region and more important in the ultrahigh vacuum region.

The designer must select structural material with low outgassing properties during constructing a vacuum vessel. However, other properties like strength ease of fabrication, thermal conductivity, elasticity, optical properties, resistance to high temperature, and cost are important according to the particular application. Structural materials which are commonly used in vacuum system include stainless steel, aluminium, copper, titanium etc.

Generally high vacuum systems are constructed of steel or aluminium because of their good outgassing properties. Since ultra-high vacuum systems are usually made up with prebaked the materials, hence need to withstand high temperature. So stainless steel is widely used for the vacuum vessel, connecting tubes and valve bodies.

In any vacuum system that has reached equilibrium and in which leaks have been eliminated the pressure depends on the total outgassing of the system and pumping speed of pumps.

$$p = \frac{Q}{S} \text{ mbar} \qquad \text{Eq. (1)}$$

Above formula is true only for the separate part of the system molecular flow succeed and where one can consider a volume into which gas is evolved from surfaces, and out of which gas flows due to pumping. But this formula is not applicable where large temperature differences are exist, e.g. near cryo surfaces of a cryopump.

In general the pressure in a pumped vacuum system will slowly decrease with time due to the outgassing rate of the materials, reducing as gas is removed. This is because the rate depends on the surface coverage or on the concentration of gas dissolved in the material. Fig. 1 shows a typical plot of log outgassing rate against log time.

CONDUCTANCE MODULATION METHOD

The conductance modulation method is a variant of the throughput method where the conductance of the pumping orifice is modulated (2, 3). Here the known conductance of the orifice is modulated by the following ways,

(a) changing the opening of an iris diaphragm,

(b) exposing orifices of different size in a rotary disk, or

(c) by changing the separation of a circular plunger from a large circular opening in an annular disk

The latter method is outlined schematically in Fig. 4. The outgassing rate can be found from the test chamber by measuring the pressure p_1 & p_2 when the conductance is changed from C_1 to C_2 by moving the plunger from position 1 to 2. Outgassing rate of the sample is calculated by subtracting the blank system outgassing rate.

The outgassing rate per unit area is given by this method is,

$$q_A = \frac{(p_1 - p_p)S_1}{A} = \frac{(p_2 - p_p)S_2}{A} \text{ (mbar l s}^{-1}\text{ m}^{-2}\text{)} \quad \text{Eq. (2)}$$

Now,

$$\frac{1}{S_1} = \frac{1}{C_1} + \frac{1}{S_p} \;\&\; \frac{1}{S_2} = \frac{1}{C_2} + \frac{1}{S_p} \quad \text{Eq. (3)}$$

Then, provided that P_p remains unchanged on moving the plunger, the outgassing rate per unit area is given by

$$Q_A = \frac{p_1 - p_2}{A\left(\frac{1}{C_1} - \frac{1}{C_2}\right)} \text{ (mbar l s}^{-1}\text{ m}^{-2}\text{)} \quad \text{Eq. (4)}$$

The outgassing rate per unit mass is given by,

$$Q_M = \frac{p_1 - p_2}{M\left(\frac{1}{C_1} - \frac{1}{C_2}\right)} \text{ (mbar l s}^{-1}\text{ kg}^{-1}\text{)} \quad \text{Eq. (5)}$$

MATERIALS AND METHODS

In the conductance modulation method, Outgassing rate can be obtained by varying conductance of orifice in the vacuum chamber.

Here Outgassing rate is calculated with excel spread sheet method for two specific position of crank. In this procedure one diaphragm is placed at orifice in vacuum chamber. By connecting it with crank mechanism, different value of conductance at different speed of crank can obtain.

By considering Conductance as a function of cosine,

$C \alpha f(\cos\Theta)$; $\Theta = \omega t \therefore\ C \alpha f(\cos\omega t)$; $C = K \cos\omega t$ Eq. (6)

Where, C= Modulated Conductance (l s^{-1}); K= modulation constant; ω= angular speed (rps);t=time (s)

In ideal condition, K=1, $\therefore C = \cos\omega t$ Eq. (7)

Now at initial position 1, $C_1 = \cos\omega_1 t_1$ similarly at different speed of plunger also as per Eq. (4)

$C_2 > C_1$,

Assume that C_2 is 10% higher than C_1 as in schematic diagram of conductance modulation method; at position 1 orifice area is less than at position 2.

So C_2=1.1 C_1

So below given values of C_1 and C_2 are obtained.

Also now putting values of C_1 and C_2 in eq. (1) and by assuming 10^{-7} mbar pressure drop in vacuum chamber with $10m^2$ surface area and mass of sample i.e. 50 gm, outgassing rate Q_a and Q_M calculated as shown in below table.

RESULTS AND DISCUSSION

As shown in Fig. 3 & 4 calculated outgassing rate at 10^{-7} pressure drop graphical representation between outgassing rate and modulated conductance can be compared.

CONCLUSION

Outgassing rate for any sample is calculated by Conductance modulation method in vacuum chamber with different modulated conductance. Here with the help of assumptions i.e. C_1 is a function of cosine and C_2 is taken 10% greater than C_1 as realistic value of C_2 is not satisfying condition $C_2 > C_1$, calculation is done theoretically with the help of excel spread sheet. Also Outgassing rate at same conductance is neglected due to limitation of field of graphical representation. This paper signifies that Outgassing rate is changing in decreasing manner with inverse difference of conductance and minimum outgassing rate is achieved at nearly equal conductance at orifice.

FIGURES

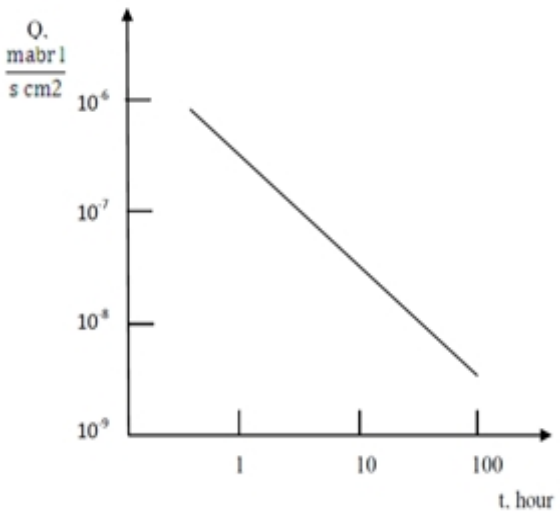


Fig. 1.Typical outgassing rate plot (1)

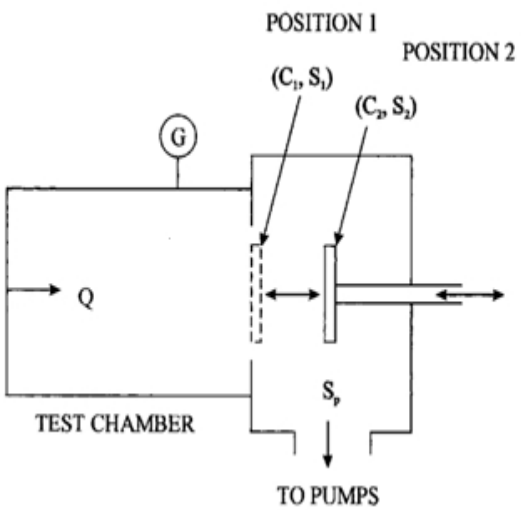


Fig. 2.Schematic diagram of the system used to measure outgassing rate by Conductance modulation method (3)

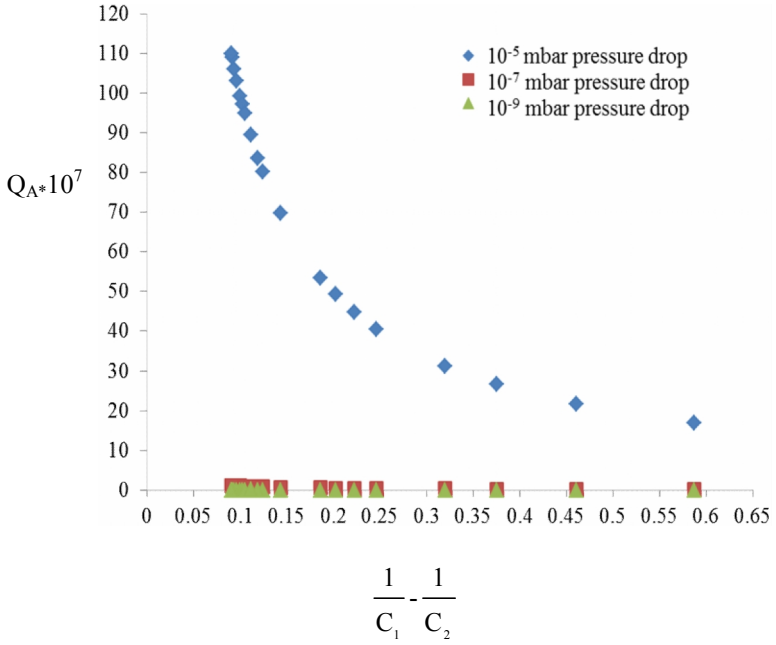


Fig. 3 Measured outgassing rate per area for different values of modulated conductance

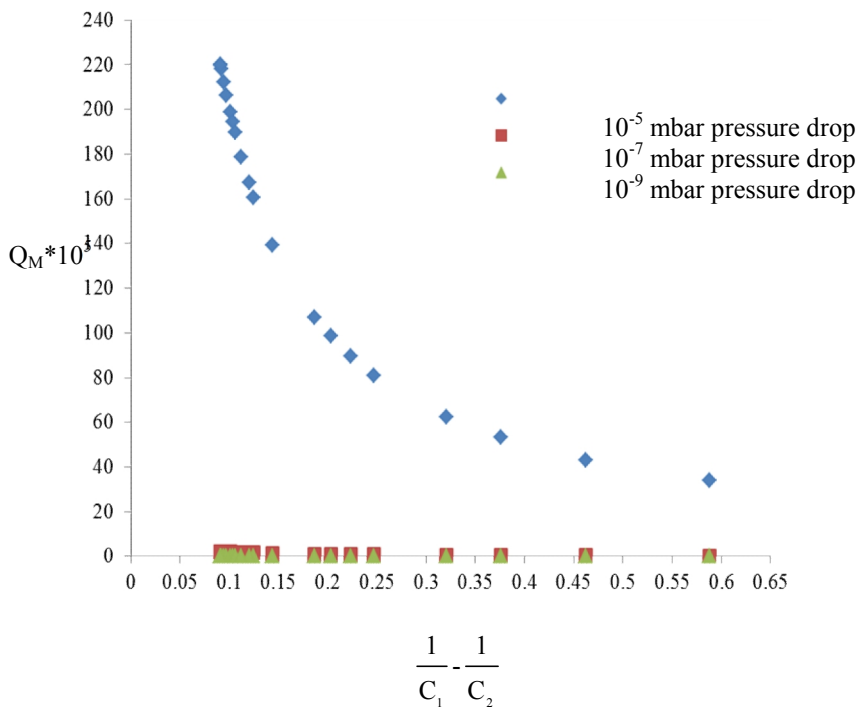


Fig. 4 Measured outgassing rate per mass for different values of modulated conductance

TABLES

Table 1. Outgassing rate at different values of C_1 and C_2

				at p_2-p_1=10^{-7} mbar	
ω_1	t_1	C_1= cos$\omega_1 t_1$	C_2=1.1C_1	Q_A	Q_M
0	0	1.00	1.10	1.1E-07	2.20E-05
50	1	0.96	1.06	1.061E-07	2.12E-05
100	2	0.49	0.54	5.359E-08	1.07E-05
150	3	-0.73	-0.80	8.032E-08	1.61E-05
200	4	-0.45	-0.49	4.929E-08	9.86E-06
250	5	0.94	1.03	1.032E-07	2.06E-05
300	6	-0.99	-1.09	1.09E-07	2.18E-05
350	7	0.90	0.99	9.942E-08	1.99E-05
400	8	-0.28	-0.31	3.123E-08	6.25E-06
450	9	-0.88	-0.97	9.721E-08	1.94E-05
500	10	0.15	0.17	1.701E-08	3.4E-06
550	11	0.76	0.84	8.36E-08	1.67E-05
600	12	0.86	0.95	9.489E-08	1.9E-05
650	13	0.63	0.70	6.973E-08	1.39E-05
700	14	-0.20	-0.22	2.167E-08	4.33E-06
750	15	-1.00	-1.10	1.099E-07	2.2E-05
800	16	0.41	0.45	4.478E-08	8.96E-06
850	17	0.24	0.27	2.664E-08	5.33E-06
900	18	-0.37	-0.41	4.054E-08	8.11E-06
950	**19**	**-0.02**	**-0.02**	**2.265E-09**	**4.53E-07**
1000	20	0.81	0.89	8.945E-08	1.79E-05

ACKNOWLEDGEMENT

The author wishes to acknowledge the assistance given by the RK University in the preparation of this article.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th& Jan. 6th, 2016)

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RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

Design & Finite Element Analysis of the Nozzles of Air-Surged Vessels

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ABSTRACT

Pressure vessels are extensively functional in numerous industrial sectors. Nozzles are essential for different processes carried out in pressure vessels. The geometrical incoherence of the vessel-wall caused by the nozzles lead to concentration of stress about the opening which causes failure of the system. Hence a detailed analysis is carried out to analyse the effect of stress on a system. This article focuses on design & finite-element analysis of these types of nozzles of air-surged vessel to know the stress distribution of the horizontal type pressure vessel attachments. Meanwhile, present study is carried out for the changing load and relative effects on stress from different geometric parameters.

SUMMARY

Design & Finite Element Analysis of various nozzles as per ASME codes.

Keywords: Air-surged Vessel, Nozzles, ASME Codes, FEA, Stress Distribution.

INTRODUCTION

Pressure vessels have extensive applications in many industrial segments such as machine-building, aerospace techniques, chemical and petroleum, oil and oil-refining industries, gas, nuclear & power engineering, etc. *(1)*. A surge tank is a supplement to a pressurized system designed to accumulate pressure fluctuations. Its purpose is to counteract ups and downs in pressure to avoid system failures, blowouts and many other complications. The surge tank is connected at the uppermost position of the system. As the pressure increases it forces the air to move up towards the vessel and it will store the air when the pressure decreases the air will reimburse the abnormality of pressure and hence pressure will be sustained. If the pressure drops rapidly, the surge tank can fill in with a standby fluid until the pressure is stabilized again. Hence, the purpose of a surge control tank is to deliver a pre-determined quantity of gas (often air) and liquid (water) at the instant a surge incident occurs.To fulfill desired necessities,inlet, outlet, vents, drains and manholes etc. are only possible with the help of nozzles in the air-surged pressure vessels *(4)*.

In the structural analysis, internal pressure is key focus for the determination of main vessel & nozzle contacts. In addition to the stresses produced by the internal pressure, the significance of external forces and moments applied to nozzle should be considered into attention. External loading is generally enforced by a piping system connected to the nozzle. A structural analysis of piping system provides the values of loads and moments*(6)*.For the stress analysis of nozzle connections in pressure vessels subjected to various external loadings, several articles including numerical, analytical, experimental research have been published. The codes propose a technique to design the junction, but do not offer any approach to compute the degree of these stresses. So, there is a prerequisite to take-out a comprehensive finite element analysis of the junction to analyze stresses at the junction and also in the vessel as well as nozzle *(3)*.

MATERIALS AND METHODS

- *Stress distribution due to Internal Pressure*

  There are two ways in which a thin cylindrical shell fails when subjected to an internal pressure*(2)*:

  * Circumferential or hoop stress: Tensile stress acting in a direction tangential to the circumference is called circumferential or hoop stress*(11)*. In thefabrication of large pressure vessels like steam boilers, riveted joints or welded joints are used*(10)*.

$$t = \frac{p \times d}{2s_{t1} \times \eta}$$

  Where, η= joint efficiency

  * Longitudinal stress: Tensile stress acting in the direction of the axis is called longitudinal stress *(11)*.

  For welded or riveted joint

$$t = \frac{\pi \times d \times p}{4s_{t2} \times \eta}$$

- *Designing of Nozzles*

  ∗ *Design Data*

The vessel is designed as per ASME section VIII DIVISION 1. It is a horizontal vessel with elliptical head used to store compressed air. The vessel has an internal diameter of 701 mm, length 1150mm & thickness 11.5 mm with an operating temperature and pressure of 8^0C to 52^0C and 1.363 MPa.

Table 1. Design data for Nozzles

Sr.no.	Name	Location	Description	Opening	Length
1	N2	Head	Air outlet	DN 50	135
2	N3	Shell	Pressure indicator	DN 50	237
3	N4	Shell	Drain	DN 50	172
4	N5A	Shell	Hand hole	DN100	253
5	N5B	Head	Hand hole	DN100	135
6	N6	Shell	Safety valve	DN 50	237

∗ *ASME codes for designing (7)*

The design of shell and nozzles can be calculated by ASME codes. Separate codes define in ASME books for designing and addenda. The standard codes use for designing are as following:

Table 2. ASME codes for Components

Component	Codes	Consideration
Shell	UG-27	Shell under internal pressure
Head	UG-37	Head under internal pressure
Nozzles	UG-45	Nominal thickness of nozzle neck

∗ *Material Selection (9)*

For different components of the air-surged vessel material selection needs to be done on the basis of requirement of functioning. The table below gives the details of material selection.

Table 3. Selection of Materials for components

Component	Requirement	Standard Material selected	Allowable stress
Shell	Withstand high stresses at low and moderate temperature services	SA-516 M Grade 485	s_t=485-620 MPa s_v= 206 MPa
Head	Withstand high stresses at low and moderate temperature services	SA-516 M Grade 485	s_t=485-620 MPa s_v= 206 MPa
Nozzle neck	Withstand low stresses at high temperature service	SA-106 M Grade B	s_t=415 MPa s_v=240MPa
Flange	Withstand low stresses at low temperature service	SA-105 M Grade 2	s_t=485 MPa s_v=250 MPa

∗ *Analytical Calculations for Nozzles (8)*

By analytical calculationsaccording to different ASME codes, the following results are obtained:

Table 4. Analytical Calculations for different nozzles

Sr. No.	Nozzle	Nominal thickness (mm)	Thickness provided (mm)	Standards Codes
1	N2	6.45	11.07	DN50.XXS
2	N3	6.05	11.07	DN50.XXS
3	N4	6.05	11.07	DN50.XXS
4	N5A	6.05	11.13	DN 100.SCH.120
5	N5B	6.04	11.13	DN 100.SCH.120
6	N6	6.05	11.07	DN50.XXS

- *Modeling*

As per the analytical design so obtained, models were created using commercial modelling software having academic license. The following figures shows the 3D models of different nozzles.

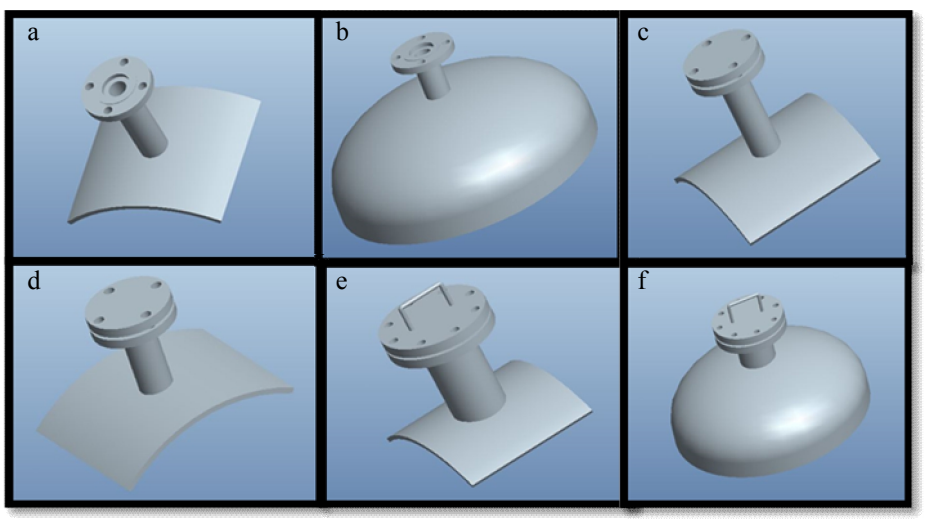


Fig.1. 3D models of (a) N2 (b) N3 (c) N4 (d) N5A (e) N5B & (f) N6

- *Structural Analysis of Nozzles*

Steady State Finite Element Analysis is carried out for each nozzle to check whether the stress developed in each of the above designed nozzles in extreme working conditions is within permissible limits.

∗ *Assigning Material Properties*

The materials selected for different components should possess definite properties as mentioned in the table no 5.

Table 5. Properties of Materials assigned

Sr. No.	Component	Material	Density (ρ) kg/m^3	Young's Modulus (E) Psi	Poisson's Ratio (V)	Tensile Yield Strength (S_t)MPa	Ultimate Tensile Strength (S_y)MPa
1.	Shell	SA 516 Gr.485	7.8	2.91x10^7	0.295	260	485
2.	Nozzle Pipe	SA 106 Gr.B				240	415

3.	Nozzle Flange	SA 105M				250	485

∗ *Meshing*

Manual meshing is a lengthy & tiresome process, but with valuable tools developing in pre-processors, the task becomes simpler. The mesh engine automatically creates themesh when the mesh density is defined along the model's edges*(10)*.

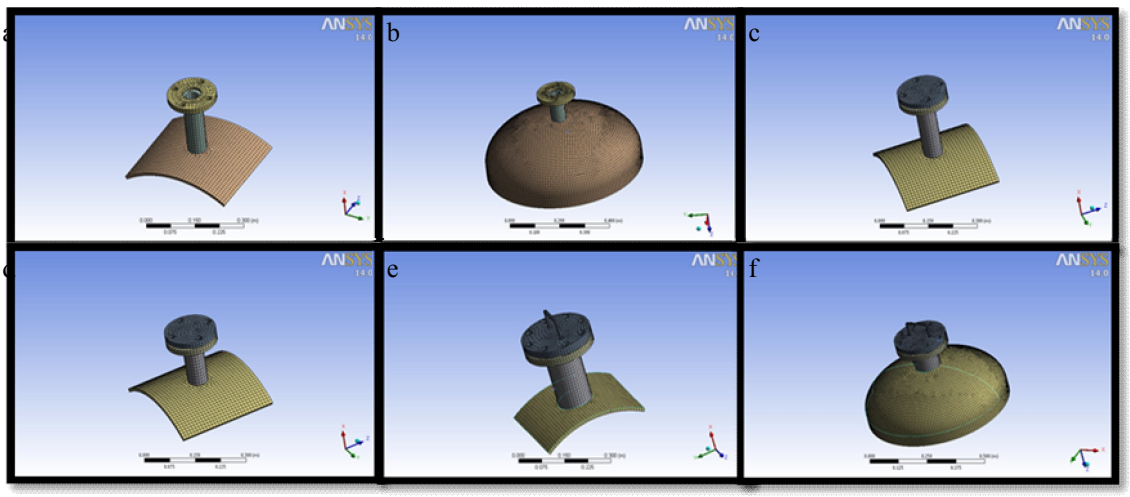


Fig.2. Meshed models of (a) N2 (b) N3 (c) N4 (d) N5A (e) N5B & (f) N6

∗ *Boundary Conditions*

For steady state structural analysis of the nozzles, the boundary conditions are taken as shown in the following figure. At the internal wall surfaces of vessel & nozzle, working pressure is applied as loading condition & at wall surface of vessel, fixed boundary conditions is applied in structural analysis. The boundary conditions shown in the figure are considered to be the same for each of the nozzles*(5)*.

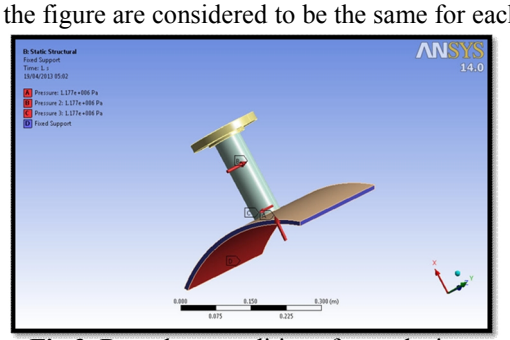


Fig.3. Boundary conditions for analysis

RESULTS AND DISCUSSION

The figures given below show the stress distribution for various nozzles discussed so far.

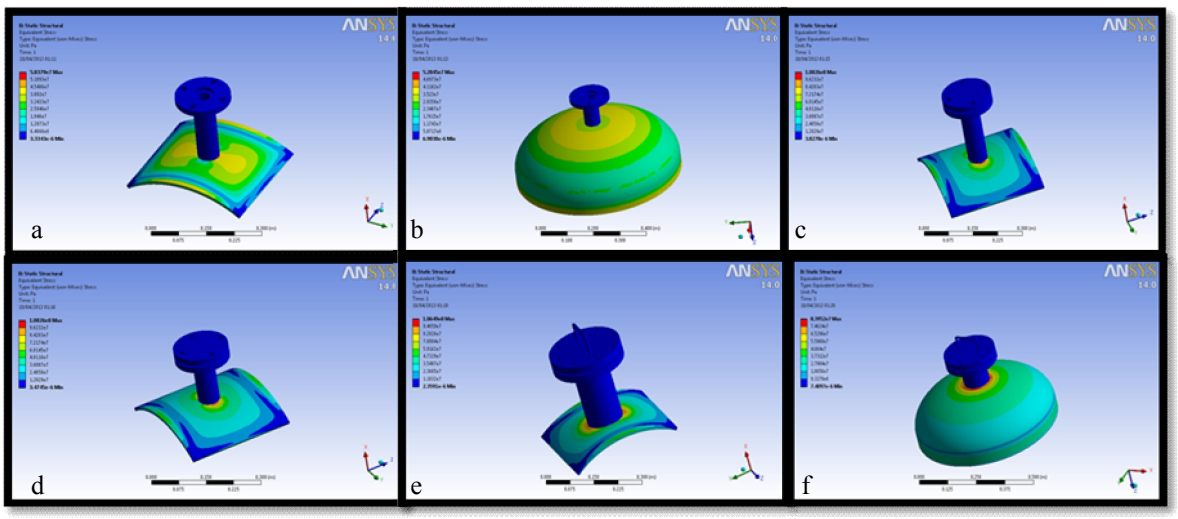


Fig.4. Stress Distribution in (a) N2 (b) N3 (c) N4 (d) N5A (e) N5B & (f) N6

From the analysis of the results, the maximum stress is found at the junction of pressure vessel-nozzle. An abrupt change in the geometry develops high stress concentration is at this junction consequently leading to a significant change in stress flow.

The following simulation results are obtained for the different nozzles each having a maximum allowable stress of 136 MPa.

Table 6. Simulation Results

Nozzle	Max. Allowable Stress for Nozzles	Simulation Results
N1and N6	136 MPa	58.91 MPa
N2		53.29 MPa
N3		109.18 MPa
N4		109.18 MPa
N5A		107.4 MPa
N5B		84.66 MPa

CONCLUSION

For all the nozzles, maximum stress valuesare found as shown in the table 6. Hence, maximum allowable stress for neck material is 136 MPa and according to the proposed design and the simulation results, maximum induced stress is 109.18MPa, which is less than the allowable stress. Hence, it is concluded that the proposed designs of all the nozzles are safe and within permissible limits.

ACKNOWLEDGEMENT

The authors are heartily thankful to Vijay Tanks & Vessels (P) Ltd, Vadodara for providing the opportunity to work with their firm and provide the resources necessary to carry out the design and analysis. The authors are also thankful to Mechanical Engineering Department, ADIT for their kind support and providing the software facilities to carry out the structural analysis.

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Classifying Challenges of E-Governance implementation in India

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ABSTRACT

India is 2nd largest country after China based on its population. Even whole country is divided based on different language, region, rituals and lots many differences. Efficient governance of such large country with such diversity is tough task. Government of India is aggressively using Information and Communication Technologies in their daily work to provide SMART (Simple, Moral, Accountable, Responsive and Transparent) governance to every citizen of India. The tools, technology and methods used in E-Governance application provide a roadmap for effective and timely delivery of services at the door step of citizen. Purpose of this paper is to examine various challenges encountered in E-Governance implementation in India. Scope of classification of challenges in this paper is limited to India only as other countries may face same or different challenges depending on their local factors.

SUMMARY

Finding and classifying different challenges in implementation of E-Governance with context of India.

Keywords: E-Governance, Challenges for E-Governance, Political Challenges, Social Challenges, Economic Challeges, Technical Challenges

I. Introduction

Every governments are aggressively using information and communication technologies in their everyday work. As a consequence, the study of e-government has increased in last two decades and many approaches to describe e-government and develop digital government research have been evolved.[1][2]. One of the way to e-government understanding describes the evolution of e-government initiatives in terms of their degree of technological and organizational sophistication.[1][2][3]. The major demand on government is that it should be more receptive to fulfil every requirement of citizens instantly, without wasting time. Citizens are expecting 365 X 24 access for many government work. This can be achieved through E-Governance only.

The terms “E-Governance” and “E Government” is sometimes used interchangeably, but E Government is just as subset of E-Governance. We can say that E-Government is an application of E-Governance. This uses the latest technologies of information and communication technology (ICT) and to make government more efficient, effective and to provide transparent services[4]. To achieve this goal, the government is focusing more to avail government services to citizens through Internet. So, E-Governance is a broader thing that deals with the whole range of the relationship and networks within governments regarding the usage and application of ICT. E-Government is a small discipline dealing with the development of online government services to the citizen and businesses such as e-tax, e-transportation, e-procurement, e-participation etc. The “E” part of both e-government and e-governance stands for the electronic platform or infrastructure that enables and supports the networking of public policy development and deployment.

II. Definition of E-Governance :

E-Governance is define by Word Bank as[5]"*E-Government refers to the use by government agencies of information technologies (such as Wide Area Networks, the Internet, and mobile computing) that have the ability to transform relations with citizens, businesses, and other arms of government. These technologies can serve a variety of different ends: better delivery of government services to citizens, improved interactions with business and industry, citizen empowerment through access to information, or more efficient government management. The resulting benefits can be less corruption, increased transparency, greater convenience, revenue growth, and/ or cost reductions.* "

As per UNESCO'[6] E-Governance is "*Governance refers to the exercise of political, economic and administrative authority in the management of a country's affairs, including citizens' articulation of their interests and exercise of their legal rights and obligations. E-Governance may be understood as the performance of this governance via the electronic medium in order to facilitate an efficient, speedy and transparent process of disseminating information to the public, and other agencies, and for performing government administration activities.* "

Advantage of E-Governance:

- Increase citizen participation in government process
- Interaction between industry and Government can be improved.
- Delivery of Government services in batter manner.
- Citizen empowerment by providing access to all information.
- Service reaches to citizen rather citizen reach for service.
- Citizen can avail all information of Government through a single window at any time and any location with a device having Internet connection.
- Decrease corruption.
- More efficient and convenience way of utilizing government services.
- Eliminate human errors in the manual process.

III. Evolution of E-Governance in India

Evolution of E-Governance in India can be considered in three phases[7]

- Phase I: 1947-1984 Information based E-Governance
- Phase II: 1984-1995 Personal Computer based E-Governance
- Phase III: 1995-onwards Internet based E-Governance

Major milestones are in this evolution process are listed here. One of the bigger milestone was the establishment of the National Informatics centre (NIC) in 1977 under Department of Electronics. Establishment of Computer Maintenance Corporation (CMC) in 1975 was another milestone for E-Governance in India. A new IT policy was introduced in India in 1984 which impacted by 100% growth in number of computers in India at 50% reduced cost[8]. In 1987, National Informatics Center Network (NICNET) was launched. After NICNET, District Information System of the National Informatics Centre (DISNIC) was introduced, which is to computerize all district offices with free hardware and software so all can take part in E-Governance initiative.A National Task Force on Information Technology and Software Development was constituted in May 1998[9]. By 2000, Indian Government had identified 12 point minimum agenda for implementing E-Governance in all union Ministries / Departments.[10]. The National E-Governance Plan (NeGP) was defined by the Department of Electronics and Information Technology (DEITY) and Department of Administrative Reforms & Public Grievances (DAR&PG). The Union Government approved the National E-Governance Plan (NeGP), comprising of 27 Mission Mode Projects (MMPs) and 10 components on May 18, 2006.

IV. Classification of Challenges in E-Governance implementation

Although the Government of India has come up with several initiatives to facilitate citizen to provide public services using E-Governance, the desired outcomes are yet to be fully realised. There are a large number of obstacles in implementation of e-Governance in India. These can be categorized under the following titles: 1) Technical Challenges. 2) Economic Challenges. 3) Political Challenges and 4) Social Challenges.

Figure 1 Challenges on E Governance Implementation in India

1) Technical Challenges:

Figure 2Technical Challenges for E-Governance

a. Scope of application: In every E-Governance Application, scope of application must be exactly predefined. After knowing exact scope of application only any E-Governance application can be designed.
b. Scale of application: E-Governance Application must be scalable. If one application is developed for one region/area than it must be extended to serve other all region.
c. Interoperability: In Government, different departments may use different tools and technology for implementing E-Governance application. But these applications must be design such that one department can use details gathered by other department/Agency.
d. Tried and Tested Technology: Technology is changing very fast but Government cannot change all technological aspect like computer servers and other hardware frequently. So only tired and tested for longer time technology must be used.
e. Permanency: Various companies are using data of last few years. Banks are using data of longer duration. But data like land records required to access like permanently. We are using land record of almost last 100 years. So E-Governance application data is stored and required to retrieve almost permanently.

f. Local Language: All E-Governance application must be developed in multilingual mode. Because every citizen is not comfortable with English language and every local language is used by specific region only.
g. Security: In E-Governance application, data of all citizen are stored. Security of these data is critical.
h. User Interface : Day by day more number of citizen are using Smart Phone. User interface of E-Government application must be such that it can be easily accessible from smart phone.

2) Economic Challenges

Figure 3Economic Challenges of E-Governance Application

a. Cost of Infrastructure: To provide E-Governance, Internet connectivity is required at all government offices. For developing country like India infrastructure and implementation cost is most important factor in implementation of e-Governance where major percentage of the population is living below poverty line. So government has to identify low cost solution for providing E-Governance.
b. Low per head Income: India is having low per head income means it is not possible that every citizen will be having personal Internet facility for accessing online E-Governance applications.
c. Portability of E-Governance Application: E-Governance application developed for one State/Region/Agency must be portable such that same application can be used for another State/Region/Agency with minor required modification if any. This will save design and development cost of same application for another state/region/agency.
d. Maintenance of Hardware: ICT field is changing very fast. It is not possible to replace whole hardware frequently. All components of

infrastructure must be identified in such a way that it should be used for enough longer period.

3) Political Challenges

Figure 4Political Challenges for E-Governance

a. Budget: Government is required to spend huge amount of money to implement required hardware and software for E-Governance application across the country. It is very difficult for any government to spare this huge amount from overall budget of country.
b. Slow decision making: In private organization, responsible team is taking decision and after than no one is going to challenge. So these organization can start working on new product very fast. But in government organization, decision making process is very lengthy. In ever changing of ICT field, this slow decision making process is a big challenge.
c. Short term approach: Government is elected for 5 years and then after election if other party gains power, it might possible that whatever plan /action proposed by previous government is revoked or changed. Due to this, any government may propose plan for short term only.
d. Integration: Government is bouquet of huge number departments at Ministry/State/District/Taluka/Village level. Integration of such versatility is a very big task and number of reforms need to establish for doing integration of all department.
e. Cyber Laws: E-Governance require every government transaction must be done online through Internet. But before implementing E-Governance completely, government must come up with precise Cyber Laws.

4) Social Challenges:

Figure 5 Social Challenges for E-Governance

a. Versatility in Language is India: According to census report, India is having 22 official languages and 1,652 languages are used as a mother tongue[11]. Prepare an E-Governance application for such huge variation is a big challenge.
b. Low Literacy rate: As per census report, average literacy rate of India is 74.04%. Again there is vast difference in rural and urban area. Literacy rate difference is even exist between male and female citizen. Usage of E-Governance application from illiterate person is very difficult.
c. Low IT Literacy: For independently accessing E-Government application every citizen must be able to use Internet either in PC, Laptop or in Mobile. But India is having low IT literacy which is challenge for implementing fully E-Governance system for India.
d. Resistance to change: Basic human nature is to resist any changes. It is very difficult to convince all citizen, government Employee and all stack holders to transfer from paper based governance system to Internet based system.
e. Finding proper application: To access various E-Governance application, citizen need to remember proper application and need to remember it.
f. Awareness: It is big challenge to let all citizen aware about various E-Governance application. Government need to run different type of awareness campaigns so people can know such applications.

Conclusion

Government of India had taken so many initiative to implement E-Governance in India like establishment of State Wide Area Network (SWAN) for Internet connectivity, State Data Center (SDC) for data management, Common Service Centre (CSC) for providing access point for E-Governance application usage to citizen and Service Delivery Gateway (SDG) for providing interoperability between various E-Governance applications. Even many Mission Mode Projects had been implemented to provide E-

Governance to National level, State level and Local Level.In this paper we have made an attempt to summarize and categorize various challenges faced by all stack holders in implementation of E-Governance application across India. Other countries of world may have some different challenges depends on their technical competency, economic feasibility, political will power and other local factors. After providing solutions of these challenges only, successful implementation of E-Governance is possible.

FIGURES

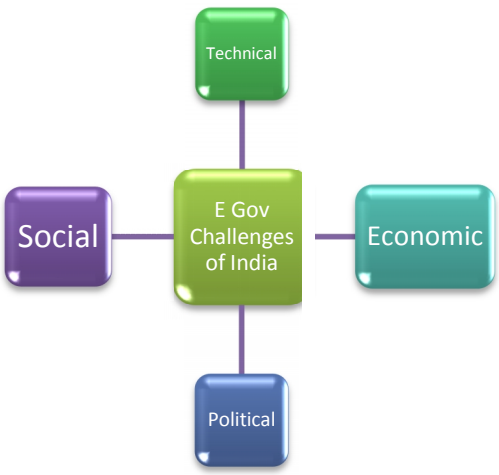


Figure 6 Challenges on E Governance Implementation in India

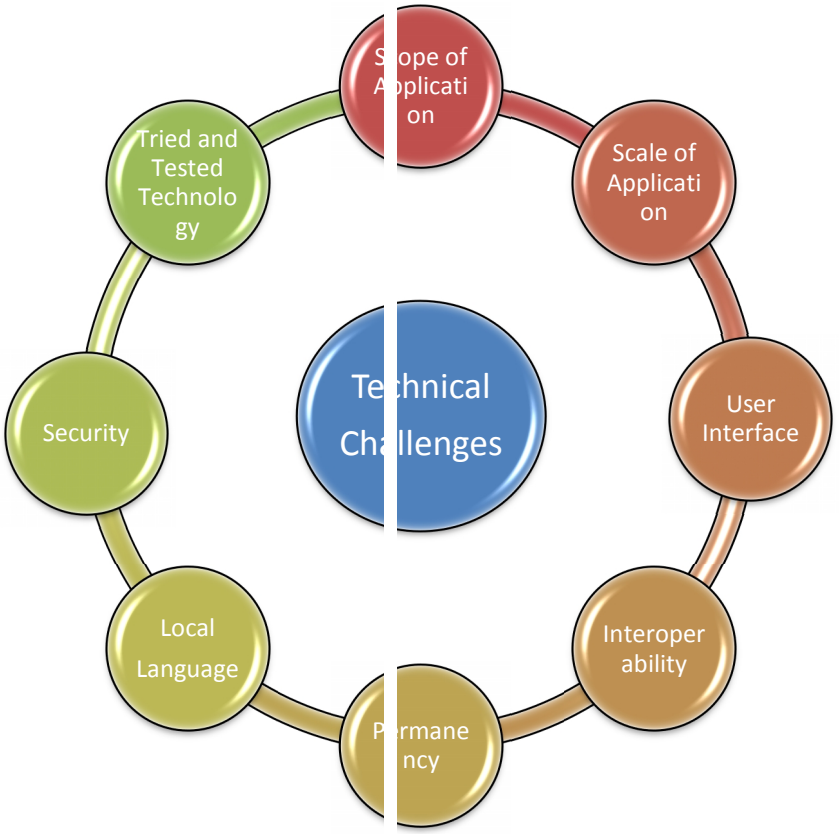


Figure 7Technical Challenges for E-Governance

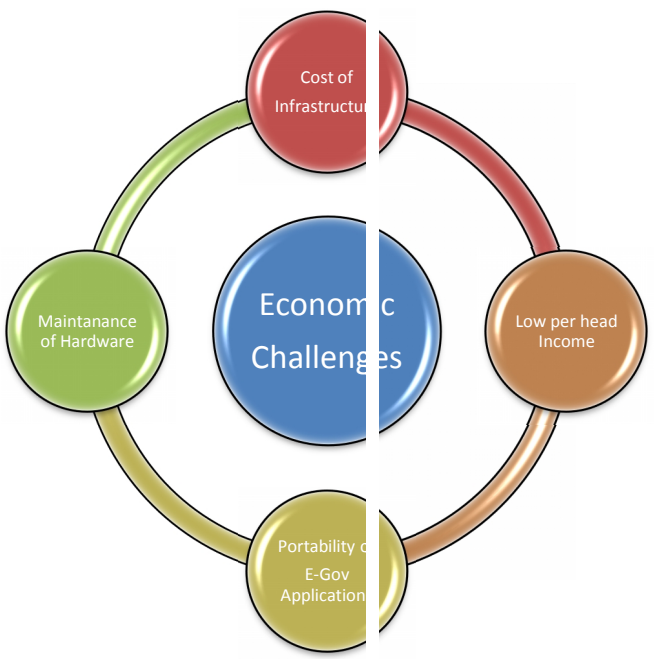


Figure 8Economic Challenges of E-Governance Application

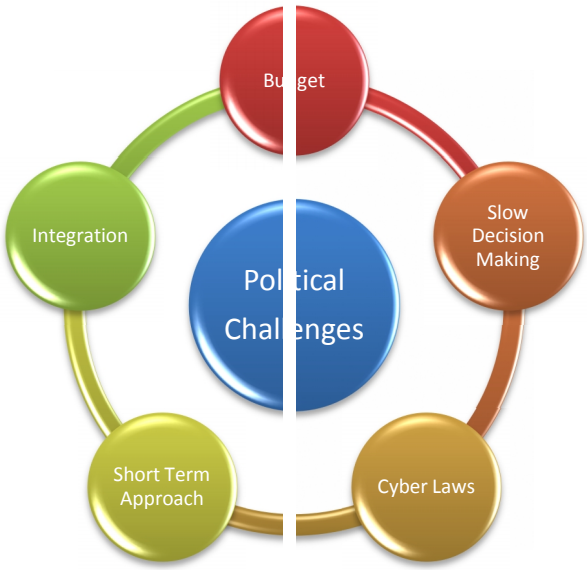


Figure 9Political Challenges for E-Governance



Figure 10 Social Challenges for E-Governance

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th& Jan. 6th, 2016)

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RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

Survey of Multicast Reactive Routing Protocols

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ABSTRACT

A wireless Ad Hoc network is a collection of mobile nodes that form a dynamic, autonomous network. Nodes communicate with each other without depending on any infrastructure. Hence, in these networks, each node acts as a host as well as a router. Multicast routing plays significant role in MANETs. These protocols face various challenges like bandwidth constraint, Quality of Service, Security, Scalability etc. In this paper we have studied various Multicast reactive routing protocols and compared them on different parameters. This will be helpful for further research and will result as a guiding document.

SUMMARY

This survey studies various multicast reactive routing protocols.

Keywords: Mobile Ad hoc Network 1,Multicast Routing 2, Security 3,Quality of Service5

INTRODUCTION

Mobile ad-hoc network (MANET) is set in an infrastructure less environment, where mobile nodes communicate with each other via wireless links. Mobile devices move as per their wish which results into a non-stationary topology. Nodes which are in vicinity communicate directly while the far away nodes require help of intermediate nodes for communication. Thus a co-operative approach is required for communication among far away nodes.

MANET has varied applications because of wide spread of portable devices and advances in wireless

communications. It can be employed at places where there is no or very little infrastructure available or it is expensive or uneconomic to build the same. Ad hoc networking allows various devices ranging from large scale, mobile, hand held, power constrained etc. [3].

Military Battlefield: More and more military equipment now equipped with some kind of computer equipment. MANET helps in building information network among soldiers, vehicles, base camp and headquarters.

Commercial Sector: MANET plays a very important role in disaster management situations like fire, flood, or earthquake. Due to natural calamity, the existing communication infrastructure would not be operational. MANET helps in building a rapid communication system and help in rescue operation.Private networks or personal area networks for the purpose of teleconferencing, video conferencing, peer-to-peer communications, ad hoc meetings, or, more generally, collaborative applications of all kinds are also the applications of ad hoc networks. *Vehicular ad hoc networks* allow vehicles traveling along a highway to exchange data for traffic congestion monitoring, intervehicle communications, and early warning of potential dangers ahead such as an accident, road obstruction, or stationary vehicle.

In this paper we have specifically selected only reactive multicast routing protocols. We believe that reactive protocols are better suited compared to the proactive counterpart. As MANET nodes are supposed to be mobile and topology changes are frequent, reactive protocols have an edge. Future research will be more focused on such protocols. This paper gives a comprehensive survey of such reactive protocols and we hope will be useful to all researchers who want to go further in this field.

MULTICAST ROUTING PROTOCOLS

Routing protocols play an important part in establishing communication among mobile nodes. In MANET as node are moving and free to enter or exit the network, the topology changes dynamically and hence routing protocols are one of the most studied / researched topic.The routing protocols can be classified as unicast – one node to one node route establishment, multicast – one node to many node route establishment and broad cast – one node all node communication. Also routing protocols can be divided in to proactive routing protocols - table driven - constant route maintenance to all the nodes in the network and reactive routing – on demand routing – establish route to the destination only when it is required. Here we are studying reactive multicast routing protocols.

In simple terms multicast can be understood as under. In a scenario when a sender wants to send data to more than one receiver i.e. a group of receivers, there can be two ways of doing this. First, a source sends data each receiver individually or second a source sends data which is addressed to a group of receivers. The former is known as multiple unicast while the latter is known as multicast. In MANETs where bandwidth is always a limiting parameter, it is always better to use multicast instead of multiple unicast. MANET applications involving data streaming like Video Conferencing, civilian operations, e-education, classroom meetings and emergency search-and-rescue are very much suitable for using multicast operations [7].

Parameters of multicast routing protocol

While designing a multicast routing protocol one must take care of basic property of MANET like limited bandwidth, frequent changes in topology, energy constraint, movement of nodes, instable paths, security and scalability [8]. We have enlisted some issues here based on this discussion.

Reliability: It means all the transmitted data must reach the destination and there should not be any loss of transmitted data packets. Military application requires such data transmission.

Robustness: Even in the presence of unfavourable environment, the protocol needs to maintain minimal data loss and maximize packet delivery ratio. There must be alternate arrangements available which come in to picture when communication through the primary links is not possible. In presence of high mobility, to sustain good quality of PDR, the multicast protocol should be robust enough.
Scalability: It means the protocol's capacity to incorporate and manage more numbers of nodes such as senders, receivers and other supporting intermediate node in the network. No of nodes in the network has direct impact on the control traffic. Multicast routing protocol needs to keep a closer look on generated control packets to accommodate more and more nodes in the network.

Efficiency: It is the ratio of packets received to the packets sent. Control packets contain no data, so to increase efficiency that must be kept in closed consideration. In case of bandwidth constraint applications, efficiency plays very important role.

Battery power control: MANET nodes are battery operated and so power constrained. Periodic messages consume power and reduces lifetime of the nodes also created partition in the network. So the selected Multicast routing protocol should contain an algorithm which specifically concentrates on enhancement of power utilization and also uniform power consumption.

Control overhead: As stated earlier, control packets does not contain data but required to maintain and tracking path. In this way it is very much important to keep a tab on generated control packets. The selected multicast protocol needs to incorporate this requirement.

Security: Due to its open environment, MANETs are comparatively more vulnerable to security attacks compared to other wired networks. So security is the key in operation of Multicast routing protocol. A well-defined security policy should be built in covering all the stages starting from network establishment to normal functioning, nodes joining and leaving, key management, intrusion detection etc.

Quality of service: A guarantee that a certain level of quality will be maintained throughout the specified operation is known as QoS. Such a guarantee is more difficult in MANET environment. A proper mechanism needs to be built in the protocol which observes and employ proper management mechanism for total traffic which can be routed through the network and use the available resources optimally.

Dependency on the unicast routing protocol: Due to varied application many different operating system and protocols exist in real world. Multicast routing protocol may needs to work with such heterogeneous networks. Such integration should be possible in the selected Multicast routing protocol.

2.2 Types of Multicast Routing

Depending the route discovery mechanism, there are three types of Multicast Routing Protocols.

1. Tree Based

In such protocols, single path is established between two nodes of the group in multicast is proposed. This will help in reducing the copies of packets that need to ne sent to other receiving nodes. Hence it provides for very efficient use of available bandwidth. For a single source least nodes are involved in the routing so they are power efficient too. But on the other hand as mobility increases and path breakages found, the established tree need to be configured once again. To counter this problem, various mechanisms like a

shared tree, losing path optimality or maintenance of multiple trees are available but they in turn requires for more storage space and increased control overhead.

2. Mesh Based

In a mesh-based multicast routing protocol, multiple paths are created. A mesh of interconnected nodes is created. In case of high mobility environment, multicast mesh based protocols are more robust compared to their counterpart i.e. tree based protocol. But this robustness comes with a cost of increased control messages being generated. This will reduce power efficiency and increase control overheads.

3. Hybrid

A useful combination of both the above kind of protocols i.e. tree based and mesh based is known as Hybrid-based multicast routing protocols. Hybrid protocols are designed to address both the issues of effective bandwidth utilization and simultaneously focusing on providing robustness. Multiple routing paths are maintained to send data packets which can result in to duplication of received data. This also results in to the created trees to be not optimal all the times.

SELECTED ROUTING PROTOCOLS

In this section we have selected various important reactive multicast routing protocols. We have studied their salient features, accessed the performance details and overall operation of these protocols.

The Shared Tree Ad-hoc Multicast Protocol (STAMP)

The Shared Tree Ad-hoc Multicast Protocol (STAMP) [9] is a multicast routing protocol which is reactive in nature and employs core-routing. More efficient and adaptive multicast communication is designed not only inside the cluster but also among the clusters. It is designed independent of the underlying unicast routing protocol. In STAMP, a core node is elected using distributed mechanism from the receiver nodes. Here the source node does not need to join a multicast group to send datagram. A shortest path is selected for datagram to transmit from sources to core node. A tree member forwards the data packet to further on the tree. Here, there is no pre assigned core node which makes this protocol different from CAMP. By a perfect blend of advantages of boot tree based and mesh based protocol, STAMP provides for efficient delivery ratio even in the situations of high nodes mobility and also for heavy traffic.

Pros: Independent of underlying uncast routing protocol, higher delivery ratio in high mobility and heavy traffic.

Cons: It is not energy efficient and QoS is not guaranteed.

The Adaptive Core-Based Multicast Protocol (ACMP)

In Adaptive Core-based Multicast routing Protocol (ACMP) [10] a tree structure is shared in the multicast group. A tree is maintained on-demand i.e. only when it is required. Whenever a multicast communication is required, core is formed and members join the multicast group. In absence of a core, tree is not initiated or constructed. All receivers would be silent. ACMP tries to balance between control overhead and efficiency of data transmission. Whenever there is any requirement for a multicast structure, ACMP selects the core. Core keeps a tab on the generated control traffic for group members to take an entry in to a multicast group. A small part of traffic is consumed by routing traffic. This results into efficient data transmission. Local route recovery and M Tree refresh on regular interval is used at the time of link failure which improve the performance of tree structure. ACMP achieves better usage of bandwidth and efficient power consumption by sending less number of for sending the required data packet to receivers.

Pros: Efficient use of available bandwidth, balance between control overhead and data transmission, efficient use of power

Cons: Selected routes are not always optimal.

The Mesh-Based Multicast Routing Protocol With Consolidated Query packets (CQMP)

In Mesh-based multicast routing Protocol with Consolidated Query packets (CQMP) [11] Query packet consolidation – means instead of each source sending advertising packets to the network, each core disseminates to the network the mappings of multicast addresses to one or more core addresses. It successfully addresses the problem of scalability. The scheme has advantages like high packet delivery ratio under high mobility, high throughput and also reduced control overhead. CQMP requires routing information from the unicast routing protocol. Also apart from routing information, unicast will also provide correct distances from source to destinations within a reasonable amount of time. Also a beaconing protocol may be embedded wit the existing unicast protocol is required for operation of CQMP. In situations of failures of router and partitions in network, CQMP will be dependent on associated routing protocols.

Pros: High packet delivery even under high mobility, reduced control overhead

Cons: Dependent on underlying unicast routing protocol,

The Enhanced On Demand Multicast Routing Protocol (EODMRP)

The Enhanced On-Demand Multicast Routing Protocol (EODMRP) [12] is a reactive mesh-based multicast routing protocol. With the help of adaptive refresh, it is proved to be a batter version of ODMRP. Based on receiver's report, refresh frequency is decided and thus adapted to current environment. When the refresh frequency is low and time between two refreshes increase, it results into loss of data for a newly joined node or for a temporarily detached existing node. It employs unified local recovery in which a node on joining or detecting a broken link performs expanding ring search and attached itself with forwarding mesh. It considers loss of packets as a link breakage and results into a reduced ratio of packet delivery. The major advantage is efficiency in lower control overhead, which results into a higher delivery rate at even increased loads. On the same hand it keeps the same ratio of packet delivery as ODMRP.

Pros: Adaptive to current environment, lower control overhead

Cons: Not so scalable, more processing overheads

The Bandwidth Optimized and Delay Sensitive protocol (BODS)

The Bandwidth Optimized and Delay Sensitive (BODS) [13] is a source-rooted mesh multicast routing protocol. The operation is accomplished in a distributed manner. To save on bandwidth this protocol selects nearest participant heuristics but in the other hand it also takes care for delay performance. A combination of BODS and ODMRP results into more effective algorithm. Under the high traffic load, BODS has better packet delivery ratio and reduced delay. It will be very useful for bandwidth sensitive and delay sensitive application like multimedia streaming. BODS can be integrated in to any existing multicast routing protocol.

Pros: effective bandwidth usage, less control overheads, good packet delivery ratio

Cons: Not suitable for high mobility scenario

The RObust Multicasting in Ad-Hoc Network Using Tree (ROMANT)

The RObust Multicasting in Ad-hoc Network using Tree (ROMANT) [14] is a reactive tree-based multicast routing protocol. It uses a group hello packet, an existing control packet which is used to fix link breakage in MAODV. ROMANT has low control overhead, high packet delivery ratio even in case of heavy traffic or in highly mobile environment. ROMANT merges partitions very efficiently. ROMANT is not dependent on the underlying unicast routing protocol.

Pros: Robust, low control overhead, high packet delivery

Cons: requires frequent announcement from core else partition may be created

The Mobile Agents Aided Multicast Routing Protocol (MAMR)

The Mobile Agents aided Multicast Routing protocol (MAMR) [15] is a reactive QoS-based hybrid multicast routing protocol. Intelligent mobile agents help in integration of any existing multicast protocol with MAMR. In MAMR, MAs are very simple packets, and travel in the network providing information on current topology and QoS information such as link delay, congestion etc. This information is used by nodes in deciding on efficient routing decisions. MAs help in finding route to the given destination when it is not present. Thus the protocol solves problem of additional delay might be required for searching a new route to the destination. It also keeps tab on generated control traffic. This information in turn will avoid broadcast route discovery. It has advantages of reduced end-to-end latency of the network and better packet delivery ratio. But MAMR requires an extra cost of processing MAs.
Pros: For the sender the time required to obtain multicast tree and routing information is less
Cons:Increased processing and control overheads

Table 1 Summary of Reactive Multicast Routing Protocols.

	STAMP	**ACMP**	**CQMP**	**EODMRP**	**BODS**	**ROMANT**	**MAMR**
Energy Aware	No	No	No	No	No	No	No
Flat	Yes	Yes	Yes	Yes	Yes	Yes	Yes
Hierarchical	No	No	No	No	No	No	No
Hybrid	No	No	No	No	No	No	No
Location aware	No	No	No	No	No	No	No
Mesh Based	No	No	Yes	Yes	Yes	No	No
QoS aware	No	No	No	No	No	No	No
Stability	Yes	Yes	Yes	No	Yes	Yes	No
Tree	Yes	Yes	Yes	Yes	Yes	Yes	Yes

CONCLUSION

In this study we presented a comprehensive study of major reactive multicast routing protocols. Multicasting can be proved very useful for a wide range of applications such as video conferencing, e class rooms, tutorials where one to many transmission is required. Basis its application, there are various characteristics which is desirable from the underlying multicast routing protocol. We have discussed some of the very important features like scalability, traffic control, QoS, Power constraint, Security, packet delivery etc. in this paper. Various protocols are designed considering one or the many features as listed. The enhancement on aspect may lead to reduction of the other aspect. So a perfect blend of the selected parameters is very much important in the final design of the protocol. The study can be a useful stepping stone who ever id willing to go ahead for research in this field.

Future Trends:
On studying various survey on multicast routing, more focus is being placed on improve QoS and hence packet delivery ratio. This obviously requires reduced control traffic and efficient routing techniques. Location details of nodes, distances between nodes, capability in terms of computation power and stored energy are taken in to consideration in designing routing protocols. The most important and highly researched area to make these protocols secure. Secured protocol versions of the existing protocols are being introduced. As more and more attacks are being surfaced, the counter measures are also introduced quickly.

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Comparison of non-co-operative spectrum sensing techniques in cognitive radio networks

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ABSTRACT

The increasing need of wireless devices and applications has formed very much constraint on the usage of existing radio spectrum. Although it is a limited and lavish resource, major portion of allocated spectrum is inefficiently used. Cognitive Radio is showing possibility of achievement for a novel approach to increase usage of present electromagnetic spectrum. Spectrum sensing is an elementary and crucial function of Cognitive Radio (CR) to detect the unused spectrum. This paper is about non-cooperative spectrum sensing techniques, its advantages and shortcomings.

SUMMARY

By comparing these techniques it is concluded that Match Filter Detection technique accurately senses the presents of Primary Users but has most complexity for implementation.

Keywords: Cognitive Radio, spectrum sensing technique, Energy detection, Matched filter detection, Cyclostationary feature detection.

Introduction

1.1 Motive for this study

The existing radio spectrum is a restricted and regulated natural resource which is being congested gradually because of increasing demands. The traditional method of spectrum allocation is considered rigid because each operator is allocated a private access to perform in a distinct frequency band and operator cannot deregulate this method. It is noticed that the allotted spectrum is under employed, as a result of the permanent distribution of the spectrum. As most of the spectrum is distributed at present, it is difficult to detect vacant bands. Thus it is abstruse either to enhance existing ones or to deploy new services. For that reason it is necessary that, multiple networks will have to share large parts of the spectrum. CR principle is an auspicious way to open the doors for such sharing. (1)- (2)

1.2 Cognitive Radios Unveiling

To provide extremely reliable communication required anywhere and anytime and to aim efficient use of the spectrum Cognitive radios are deliberately designed. On the basis of interaction with environment, CR can re-tune its transmitter parameters. This interaction is done in four separate parts which are known as main Function blocks of Cognitive radios. This function blocks are as following:

i. Sensing spectrum,
ii. Managing spectrum,
iii. Sharing spectrum,
iv. Mobility of spectrum.

1.3 A brief view of function blocks

SPECTRUM SENSING: To scan the wireless radio spectrum this technique is employed by Cognitive Radio which executes specific task of finding out availability of spectrum in other words it looks for absence of primary users which are also known as licensed users.

MANAGING SPECTRUM: Through this method superintendence of various radio frequencies are persuaded to promote efficient use in an ample manner. Prime intention of this method is to estimate that for how long the spectrum holes (absence of Primary User is anticipated as spectrum holes) are available to be accessed by Cognitive Users.

SHARING SPECTRUM: Approach of this method is to distribute estimated spectrum holes equally to each unlicensed users.

MOBILITY Of SPECTRUM: This method carries out function of maintaining uninterrupted communication during the transition from one to another frequency.

In the direction of developing cognitive radio networks spectrum sensing is the foot step amongst all four functions listed above

1.4 Spectrum sensing methods:(3)

Succeeding list is some of spectrum sensing approaches are as follows:

i. Non-cooperative or primary transmitter detection,
ii. Cooperative detection and
iii. Interference detection.

Only Non-cooperative techniques is considered and compared in detail in upcoming part or report.

SPECTRUM SENSING

Task of detecting vacant spectrum and subsequently sharing it to Primary Users without interference is an essential prerequisite of the cognitive-radio network. Identifying Primary Users is most efficient way to perceive empty spectrum which is accomplished by cognitive users who detects absence of PU in the traditionally allocated licensed radio spectrum. Once corresponding PU appears then it is responsibility of Cognitive User to make empty the frequency band. This is a precautionary measure warding off impending any kind of interference between Primary Users & Cognitive Users. **(5)**

Classification of spectrum sensing techniques:

2. NON-COOPERATIVE SPECTRUM SENSING TECHNIQUES OR TRANSMITTER DETECTION[1].

Process that identifies the presence of Primary Users based on the sensed signal energy at transmission end is a non-coherent detection.

2.1 Energy Detection

A Technique which is most favourable and easily applied to all kind of received frequency band is Energy Detection. Out of two reason for this belief one is its simplest design to implement and another is no prerequisite of any hypothetical information of PU **(8)-(9).** In the beginning of this process energy of the desired signal (on which CR may transmit) is detected and then it is compared with a threshold value. This threshold value is equal to the value of energy present in absence of all kind of PU which is only because of noise present in the environment. It is expected that the primary user is not present and the spectrum is free only if the detected signal value is lower than pre-determine threshold value. On the contrary it is assumed that spectrum is not free if the detected signal value is higher than pre-determine threshold value. In spite of the fact that it is easy to implement Energy Detector is having several limitation which leads to find other ways of Spectrum sensing.

Advantage:
Simplicity for putting into practice.
Limitations:
i) Detection is influenced by uncertainty of noise power.
ii) For given probability of detection sensing process is time-consuming.
iii) ED does not work with spread spectrum signals**.**
iv) ED fails to discriminate PU and CR user which are already present in signals. **(10)**
v) The reckoning of the threshold value used for sensing is extremely susceptible for the unpredictable noise levels which leads to formulate an environment of low SNR **(13).**

2.2 Matched Filter

In the field of signal processing Matched Filter (MF) is one of the optimal techniques and for that reason this method is acknowledged as optimal spectrum detection. Only when the transmitted signal from the source end is known matched filter is used to sense the spectrum. To be more precise it is carried out by **matching** an unknown signal with the known signal. Then it assumed that the PU is present in the spectrum if the unknown signal pattern are similar to the known signal pattern else it is consider that spectrum is free and the Cognitive User can use the spectrum of course If the pattern of the unknown signal and the known signal are unlike. Thus a condition arises that this technique is only useful when Cognitive User has an awareness of available PU signal **(9).** This process is similar to correlation process in which the unknown signal is convolved with the filter. It is important to note that the filter is so chosen that its impulse response is the echo and time shifted form of a reference signal.

Advantages*:*
i) Higher accuracy for probability of detection.
ii) Less detection time as it requires only one sample to detect.

Limitations*:*
i) MFD performance decrease because of transmitted signal characteristics are usually unknown which further leads to unwanted signal detection.
ii) MFD requires a prior knowledge of every primary signal.
iii) If the information is inaccurate, Matched Filter performs below par.
iv) CR would need a separate receiver for every sort of primary user.

2.3 Cyclostationary Feature Detection

CFD is the superior technique with compare to ED and the MFD techniques. (11) To distinguish the existence of primary users (PU) periodicity is used. Periodicity of received signal in the form of pulse trains or spreading code or sinusoidal carriers or hopping sequences or cyclic prefaces of the primary

signals. Unlike noise and interference especially stationary these cyclostationary signals have attributes of periodic statistics and spectral correlation.

Advantages:

i) CFD does better than energy detection in low SNR zones.
ii) It is robust to noise uncertainties.
iii) Existing cognitive users will improve the overall CR throughput as they may not mandatorily keep silence throughout sensing.

Limitations: (12)
i) High computational complication and
ii) Extensive sensing time.

As it is easily perceived by the above figure, that Energy based detection is the simplest methods to implement and least accurate compared to other methods. Likewise MFD is the most complex to implement in Cognitive Radio Networks, but has highest accurateness. And other methods are in the middle of these two.

CONCLUSION

From last few decades, it has been observed that spectrum has got an extreme importance particularly in research areas and turned to be the most valuable resource in wireless communication systems. Cognitive radio is considered to be the most promising technology through which spectrum sensing is permitted for fortune hunters by offering a means for the use of white spaces for spectrum utilization. Facing the problems generated because of CR, using spectrum sensing technique finds vital necessity to achieve reasonable outcomes for effective utilization of availability of spectrum and partial interference. Tread off between accuracy in probability of detection & complexity for implementation plays crucial role for selecting spectrum sensing technique. Future work is to implement algorithms for these techniques in MATLAB.

FIGURES

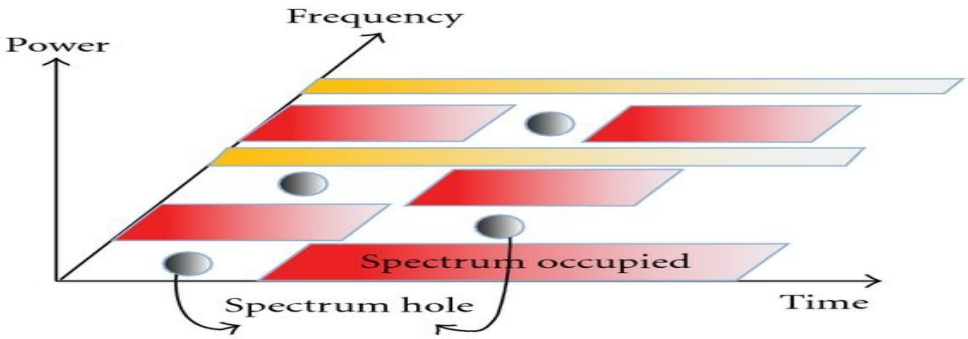


Fig. 1. Illustration of spectrum white space (4).

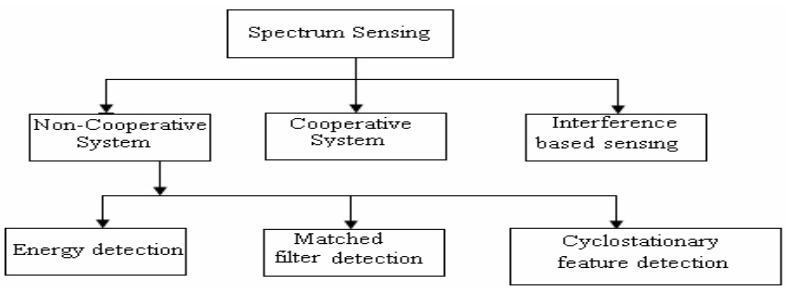


Fig 2. Classification of spectrum sensing techniques (6).

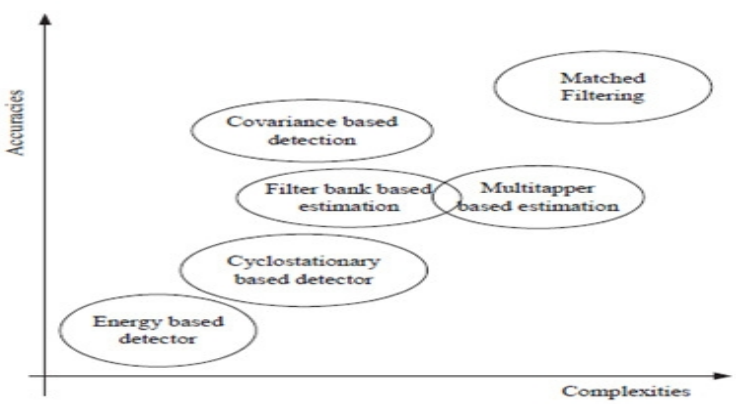


Fig. 3: Sensing accuracy and complexity of various sensing methods (14).

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Reduction of Transformer Magnetizing Inrush Current using Asymmetrical winding Technique

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ABSTRACT

Transformer is the most important apparatus in electrical power system. When a transformer is initially connected to supply mains, a fleeting current many times enormous than the full load current can flow for a short duration. The magnetizing inrush current is of very high magnitude when transformer magnetic circuits are saturated at the time of feeding transformer. The large inrush current flows when primary winding is supplied when voltage waveform is passing through zero. By using Asymmetrical winding method in the design of magnetic circuit of transformer, this high amplitude magnetizing inrush current is reduced to a considerable extent. In this asymmetrical winding technique, main focus is paid on increasing inrush analogous inductance in the design stage so as to lessen magnetizing inrush current to a considerable extent. Along with this, the equal emphasize is given to voltage drop i.e. with design of inrush analogous inductance, the inductance on account of leakage flux ought to be provided equal stress to achieve the goal with low voltage drop.

SUMMARY

Inrush current is reduced usingasymmetrical winding technique while switching on transformer initially.
Residual flux, Core saturation, Asymmetrical winding arrangement, Inrush current, Inrush analogous inductance.

INTRODUCTION

Type here. References should be cited in parentheses with an (*1*). Multiple references are separated by commas (*2, 3*);an en dash is used for a series of references (*4–8*).

Transformer is the most important apparatus of electrical power system and knowhow of its performance is first and foremost in to know how much system can be depended upon. Though care is normallytaken for overload and fault circuit current calculations, a harmfulfleeting condition may seem when a transformer on no load is fed from supply mains. Afleeting in-rush currentmanytimes the full load value *(1)* may result in the mal-operation of overload/fault relays. With the result, there takes place disconnection of the transformer from the mains. When transformer is first fed from mains, a fleeting current upto 12 times the specified current flows for a short duration. The mechanical auxiliaries of the transformer may be deteriorated on account of increased magnetic forces by this enormous current. The largest amount of inrush current is extracted when primary winding is connected at the point when no emf value is applied. Withthis start, the magnetic circuit will be saturated. The magnetic lines of force curve is originated from the same point as emf curve, the magnitude of flux is double the highest flux.The transformer magnetic circuit isnot following the linear region beyond the maximum webber in steady state. After steady state highest value of webbers, the magnetic path leaving linear portion, the current needed to generate remaining flux is going to be very high.So transformer primary will extract very high current from the source which is known as magnetizing inrush current in transformer (or inrush current) in transformer. The amplitude of inrush current is so high but it generally does not result into any permanent fault in transformer as it lasts for quite shortduration. Yet inrush current in power transformer is a problem, as it interferes with the operation of circuits as they have been designed to function. Over-sized fuse wires or circuit interrupting devices have been needed due to this large magnetizing inrush current.Noise is generated due to this current. There are present some other methods to mitigate this inrush current such as switching transformer as per our requirement in control, Phase Sequence Energisation and so on. The first requires an extra logical path but that also suffers from unexpected happenings so far as spring auxiliary is concerned as well as residual flux present in the magnetic path of transformer. To limit the inrush current by latest method of using winding with not symmetrical arrangement that is not similar to the commonly employed winding with symmetry construction in transformer design. The objective is brought about byusing winding not symmetric i.e. by varying the low voltage winding turns. So, high inrush analogous inductance is obtained by this method as well as good leakage inductance for a transformer. We can increase the inrush analogous inductance by movingthe turns assemblyof windings. But, large value of inrush analogousinductance must be designed cautiously taking into account voltage drop, the rating rupturing capacity of circuit interrupting device. When transformer design is carried out, a large no of parameters are taken into account like weight of transformer which includes weight of active parts (copper and iron), power required at the terminals and an efficiency of converting energy as well as the cost of each and every component *(3-5)*. Of course, with all this, low value of voltage drop, rupturing capacity of breaker and also control of inrush current are all taken into consideration with the same degree at a time. The magnetizing inrush current can be reduced to an appreciable extent by designing larger inrush analogous inductance and that is possible either by increasing the cross sectional area of core or by some other methodology. With increasing cross sectional area of core, the volume and hence weight of core and so cost gets affected adversely. So, designing larger inrush analogous inductance when transformer core has been saturated is by using winding not as normally symmetrical but to some degree, an asymmetry in winding is provided i.e. the asymmetric winding method can obtain this effect.

Transformer magnetizing Inrush current reduction problem: Normally, a transformer primary winding is energized by feeding it directly to the supply voltage. This direct switching may sometimes give rise to fleeting phenomenon full load which includes a current impact above specified current of transformer. The protective system will consider this current as the fault current and trip circuit though it is not a fault but this is a happening on switching. Transformer magnetizing inrush amperes are

of large amplitude, harmonic-healthy currents created when transformer magnetic cores are leaving he linear region of B-H curvet when transformer is fed from mains. These high amperes have unwanted effects like adequate damage or deterioration of life to the transformer, its auxiliaries, malfunctioning of the existing protection and what not. So, exact instant transformer feeding will give rise to high asymmetries in lines of force and saturation of one or more magnetic paths of the transformer. This saturation gives rise to large magnitude currents which have healthy harmonic content and have high direct current line. This inrush ampere is able to cause wrong operation of protecting relays as well as fuses, mechanical deterioration to the transformer windings from magnetic pulls etc. If we are in a position to make relay which will not sense this Inrush amperes initially and also by using oversized wires for fuses, these ill results on transformer as a whole can be reduced to a reasonable extent. The inrush amperes reduces to a fractional value of its starting amplitude after a few tenths of a second, and its full decay takes place only after several seconds. The most unfavorable situations are: the lines of force gets started from the residual flux, ought to vary so as to make its components vary as the supplied voltage variation, a function of time. To make it sure, Webber along with current fed, increase in first half cycle. The maximum possible value $Ø_{ee}$ of the flux is the addition of the change of flux $2Ø_m$ produced in steady state condition and of the residual flux $Ø_r$.i.e.$Ø_{ee} = 2Ø_{max} + Ø_{res} = A_{icore} (B_{rem} + 2B_m)$
where A_{icore} is the cross sectional area of the magnetic path, B_{rem} is the residual induction pertaining to flux density B, and B_m is the highest value of flux density in the magnetic circuit in the steady-state condition. Transformer designers usually work with values of 1.45 T to 1.74 wb/m^2 selected for flux density and the residual flux tesla with regards to this creation may reach values as high as 1.29 to 1.69 T. The highest amplitude of the magnetizing inrush amperes may cross full load amperes of the winding and may provide adequate electrodynamics stresses on the transformer, and cause the transformer protection to provide tripping signal. The transformer insulation get affected adversely on account of interruption of inrush current of such huge magnitudes resulting into over voltages i.e. the insulation used at various places in transformer - between LV and core, between LV and HV, inter turn insulation, insulation between two coils etc get deteriorated. The reason for this mishap is only large amplitude of magnetizing inrush current though it persists for a very short duration.

MATERIALS AND METHODS

Transformer design by using asymmetrical winding method:

When a transformer is connected to supply mains,thefleeting inrush amperes of large amplitude occurs in an electric circuit.Thisfleeting current usually causes the unintentionalbehavior of protection system which uses over current.However, the mechanical assemblies of the transformer get deterioratedon account of the magnetic stresses produced by inrushamperes. The magnetic path of transformer leaves the linear region at the time of fleeting inrush current (*6*). Now as the magnetic circuit crosses the knee point of B-H curve, the permeability of magnetic circuit becomes absolute permeability. So, the amplitude of (NΦ/I) is lessened.With decrease in this value, the inrush amperes enriches suddenly.Normally, this ampere is quite high as ten to twelve times the full load specified amperes of transformer whereas the normal primary winding current of a transformer is even less than 2% of the full load current. This study tries tocontrol the inrush amperes by making use of asymmetrical winding method. Different from usualwinding with symmetryin manufacturing of transformer design, the aim is brought about by varying the low voltage winding coil no of turns. Large inrush analogous inductance is achieved in the design of transformer by this method to limit inrush current as well as an low value of leakage inductance is also provided for small voltage drop. In latest Asymmetrical winding technique, the same attention has been paid on the appropriate voltage drop in the design of transformer. From the constructional components of transformer, the leakage inductance and inrush analogous inductance are to be studied which takes care of magnetizing inrush amperes in transformer (*6*). The major source in the

design of transformer to reduce this inrush current is the inrush analogous impedance during this period. We can increase this inrush analogous inductance by proper design using asymmetrical winding technique. So, inrush current gets reduced to a considerable extent. The distribution transformers of L-H-L and L-H-L-H construction can be used for demonstration purposes. Calculated magnitudes of leakage inductance will vary with the value of x in L-H-L construction and the values of x and y in the L-H-L-H structures. As we know the transformer is in need of a suitable leakage inductance to balance the rating rupturing capacity of the circuit breaker and less voltage drop, the asymmetrical winding technique affects the value of Inrush analogous inductance and leakage inductance. Increase in the value of inrush analogous inductance will control inrush current. In this method, the area of primary winding is increased by splitting the winding. So, this study provides a new thought in the design for a transformer to increase inrush analogous inductance so as to get the requisite. This method satisfies the controlled inrush ampere the low voltage drop simultaneously. As the magnetic circuit of transformer is crossing the linear region and knee point, there is no place for the flux lines to pass through. In the design of transformer, the lines of force take their path through the asymmetrical winding and increase inrush analogous inductance. Thereby reducing the magnitude of even initially occurring fleeting inrush amperes.

RESULTS AND DISCUSSION

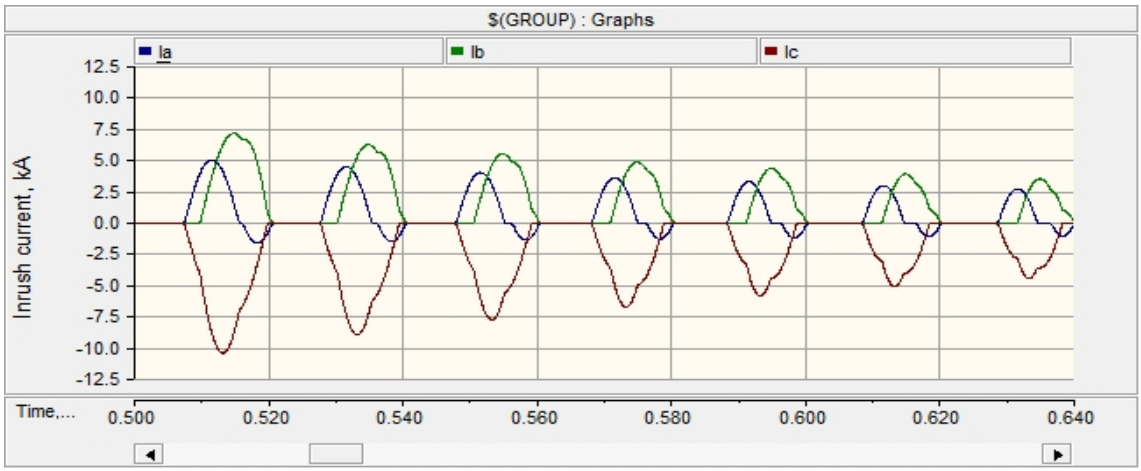
Simulation results:

3- phase Transformer specifications:
kVA=250
Voltage ratio=11000 /433volts
Connection: Delta/ Star
No load loss=550 watts
Copper loss=4800 watts, %Z=4%
Figure (1) shows Inrush current in transformer without using asymmetrical winding configuration i.e. with normal symmetrical winding as usually done. We find that the inrush current magnitude is quite high +6 kA to -8 kA while energizing transformer. As observed from figure (2), the transformer's magnetizing inrush current gets reduced to a considerable extent using asymmetrical winding configuration i.e. +2.5 kA to -3.5 kA during energizing transformer which is quite low i.e reduction in the magnetizing inrush current is 58 %.So, we can observe from figure (3) and figure (4) that the remenance affects the magnetizing inrush current to large extent. In figure 3, Remenance Effect using no load losses = 0.05 pu, the magnetizing inrush current is 1.1 pu whereas in figure 4, Remenance Effect using no load losses = 0.5 pu, we get the magnetizing inrush current as 2.1 pu. which is quite larger as compared to 1.1 pu. with low no load losses. Thus it is seen that the waveform of Transformer magnetizing inrush current with the aid of asymmetrical winding technique in the design of transformer, we can reduce the magnitude of magnetizing inrush current in transformer.

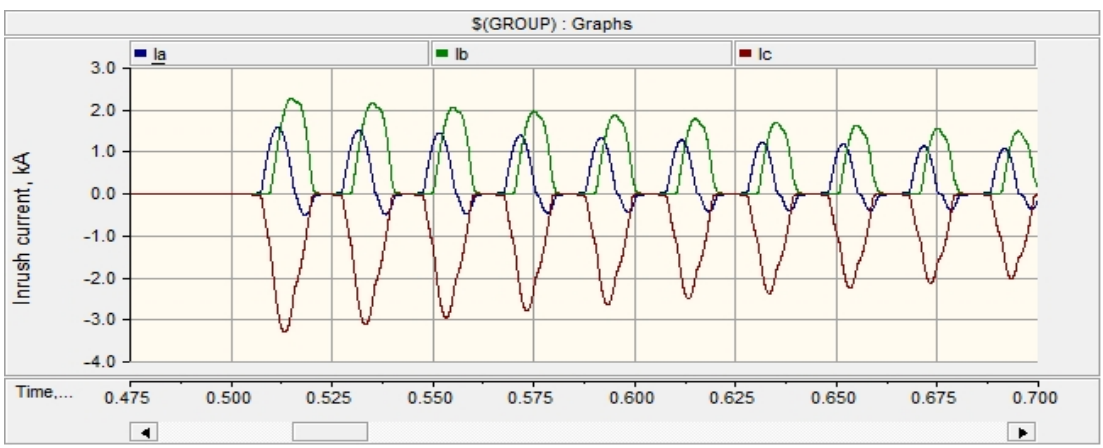
CONCLUSION

Transformer designed with asymmetrical winding technique is used to reduce inrush current up to about 58% to 60% taking into account equally a suitable voltage drop. So, this method is more practical as compared to other methods as controlled switching, sequential phase energization methods. The asymmetrical winding configuration is free from all the disadvantages of other methods. The inductance on account of leakage flux and inrush analogous inductance are analyzed from the constructional parameters of transformer. The optimum design is carried out which enables inrush current mitigation and less voltage drop.

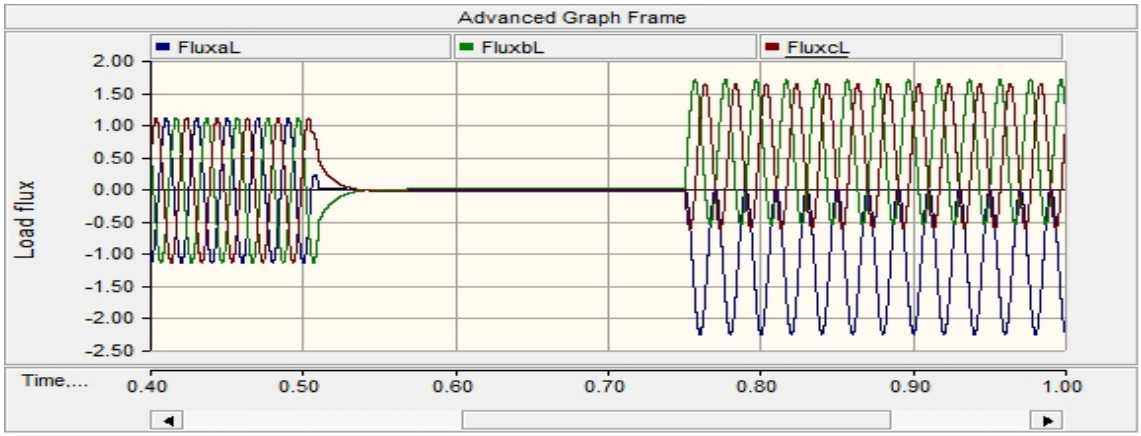
FIGURES



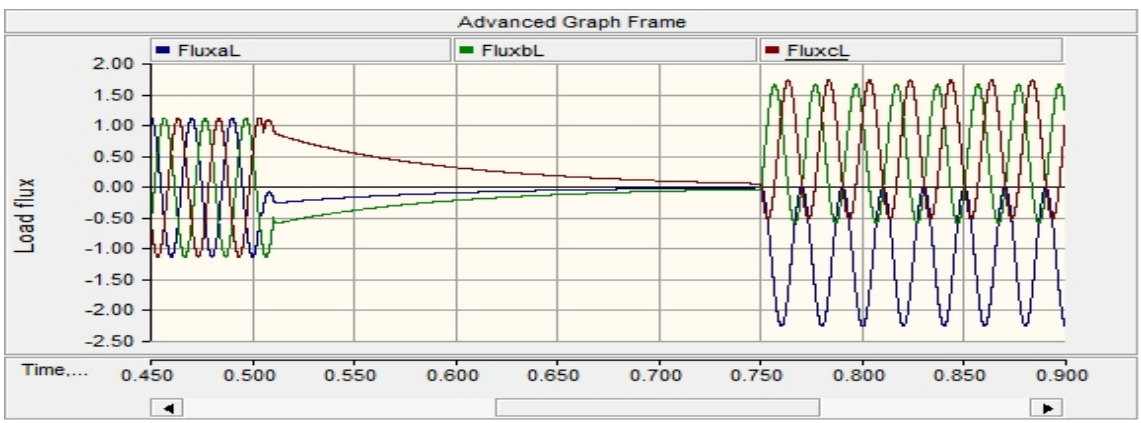
"**Fig.1**. Transformer magnetizing inrush current with usual symmetrical winding."



"**Fig. 2**. Transformer magnetizing inrush current using asymmetrical winding configuration."



"Fig. 3. Remenance Effect using no load losses = 0.05 pu"



"Fig. 4.Remenance Effect using no load losses = 0.5 pu"

Remenance	No load loss, pu	Inrush current
Present (low)	0.05	1.1
Present (large)	0.5	2.1

"**Table 1.** Magnetizing Inrush current on account of remenance."

ACKNOWLEDGEMENT

I am very much thankful to Dr. R. B. Kelkar sir and his whole hearted support every now and then. I thank my guide Dr. Heena Chandwani for providing me right direction at the right time. I am also thankful to Mr. Urmil Parikh for his kind and valuable suggestions all the times. I also thank R. K. University to provide us such a wonderful ICRE 2016 conference.

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Application of the Fenton Process for the Treatment of Pharmaceutical Wastewater

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ABSTRACT

Pharmaceutical industries are bulk consumer of water and generating toxic and recalcitrant compounds. Removal of these compounds is possible by advance oxidation processes. In Fenton process generation of hydroxyl radicals is takes place with the reaction of hydrogen peroxide (H_2O_2) and iron (Fe^{+2}) as catalyst at acidic pH. Various doses of ferrous sulphate was used from 2 g/L to 8 g/L and achieved COD removal from 20% to 60%, BOD removal 8.33 % to 41.67% and increase biodegradability index from 0.23 to 0.30. Study investigates that maximum COD reduction of 60% was achieved at 8 g/L dose of $FeSO_4.7H_2O$. So, it is investigated that Fenton process alone was not so much sufficient to efficiently remove the recalcitrant compounds and reduce COD concentration. Further, it is matter of research work, to consider various combinations of Fenton process with other advance oxidation process to achieve 100% removal of COD from pharmaceutical effluent.

SUMMARY

Fenton process can be used to remove recalcitrant compounds as pretreatment step to biological degradation for the treatment of wastewater from pharmaceutical industries which led to an increase the biodegradability index of pharmaceutical wastewater.

Keywords: Fenton processes, Pharmaceutical wastewater, Chemical oxygen demand, Recalcitrant organic compounds, Zero liquid discharge, Waste minimization

INTRODUCTION

Water is the key elements for the survival of the life on the earth. Growth of the society depends on the availability of clean and safe water. Industries use water for their manufacturing processes and other purposes. Due to industrialization, globalization, overexploitation, scarce rainfall and population explosion, water is now considered as scarce resources. For the sustainable development of the society, people have to use water wisely as per their needs and industrialists have to adopt various water conservation strategies in their industryto conserve water for future generation by means of 4R's concept i.e. Reuse, Recycle, Recover and Reduce. The concept of 4R's will help them to achieve zero liquid discharge phenomena in their manufacturing plant. The theoretical concept regarding to achieve zero liquid discharge is the separation of waste impurities in the form of solids component from industrial wastewater, treated wastewater is reused and generated solids containing some moisture content are disposed as a waste or by product. By using zero liquid discharge concepts, industries can keep their surroundings environment sustainable and eco-friendly. [1] Pharmaceutical industry are using great amount of water and it is alwaysproblematic due to the ample types of chemicals used in pharmaceutical manufacturing.Drugs are complex organic compounds which are resistant to biological degradation. Pharmaceutical industries are generating toxic and recalcitrant organic compounds. These compounds are non biodegradable, highly stable and cannot be removed by simple means of conventional treatment methods.Removal of these compounds is possible by converting their nature from non biodegradable to biodegradable organic compounds. So use of advance oxidation process is the viable solution of it. [2] These effluents must be removed as per the prescribed standards given by statutory bodies. Removal of these effluents by chemical and biological means is often difficult. Now a day, advance oxidation processes are the shows potential for the treatment of these effluents. Advance oxidation processes are the process whichformfree hydroxyl radicals in an aqueous media to promote the oxidation of pollutants to their high oxidizing power (E^o = 2.8 eV). [3] Now, treated clean water from combination of conventional biological treatment methods with AOPs can be reuse in plant boundaries i.e. zero liquid discharge or discharge into streams for the disposal of treated water. Thus, it is stated that zero liquid discharge can be achieved by combining different advance oxidation processes with conventional treatment methodsin pharmaceutical wastewater.

Among the AOPs, the Fenton reaction with all its variants and modifications shows potential for practical industrial applications. There are several divisions of Fenton's reagent including: photo-assisted Fenton, electro-Fenton, sono-Fenton and solar Fenton. [4]

In 1884, Fenton reaction was discovered by H.J.H Fenton. In Fenton process, generation of hydroxyl radicals is takes place with the reaction of hydrogen peroxide (H_2O_2) and iron (Fe^{+2}) as catalyst at acidic pH and ambient conditions. [4] Optimum pH for the Fenton process is 2.8-3.0. The activity of Fenton process decreased with an increase or decreases of the pH from 2.8 to 3.0. At pH above than 3, Fe^{3+} starts precipitating as ferric hydroxide and dissociates the H_2O_2 into O_2 and H_2O. [5] Reaction temperature is also a critical factor. Reaction rate can be increased by increasing the temperature of the reaction, but decomposition of hydrogen peroxide into oxygen and water can be takes place. [6] Hydrogen peroxide dose and iron ion concentration are two important factors which affect the Fenton process. [7] Activity of the Fenton process can be increased by the combination of with other treatment processes. [4] Fenton process has been widely used for the removal of COD, TOC and reduction of recalcitrant and toxic compounds with conventional biological treatment. [2]

Fenton process has some advantages over the other AOPs. Fenton process is takes place with short reaction time at atmospheric pressure and room temperature so; no energy is required to activate the hydrogen peroxide.
Sebasti et.al, 2003 was investigated that Fenton process can be use as pretreatment method for a polluted pharmaceutical effluents COD 362000 mg/L due to recalcitrant compounds, which is indicated by biodegradability index as low as 0.008. Tekin et.al, 2006 studied the Fenton process as pretreatment method at one of the pharmaceutical industry in Turkey for the improvement over biodegradability index and reduction of toxicity. He reported that optimum pH for the Fenton process was 3.5 and coagulation stage was 7.0.Fenton process in combination with sequencing batch reactors (SBR) was reported 98% COD and BOD removal efficiency and complies with the discharge standard prescribed by regional authority. The COD removal efficiency achieve in the Fenton process was in the range between 45% and 50%.

MATERIALS AND METHODS

A. Sampling

For the study, analytical grade chemicals were purchased. The untreated wastewater samples were collected from one of the pharmaceutical intermediate products manufacturing industry located at Vapi, (Gujarat) India.

B. Materials

In the study, hydrogen peroxidewas used as oxidant reagent, ferrous sulfate heptahydrate ($FeSO_4.7H_2O$) as source of Fe^{2+} catalyst. To adjust the pH of the sample, sulfuric acid and sodium hydroxide were used. Distilled water was used for all solution preparation.

C. Mechanism

In Fenton process, the formation of hydroxyl radicals takes place through a catalytic process in which the iron ions play a very important role. The usually acknowledged mechanism of the Fentonprocess proposes that hydroxyl radicals are formed in accordance with Equation (1), while the catalyst is regenerated through Equation (2-4). Nevertheless, a number of competitive reactions can also occur.

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + HO^- + HO^{\cdot} \quad (1)$$
$$Fe^{3+} + H_2O_2 \leftrightarrow H^+ + [Fe(OOH)]^{2+} \quad (2)$$
$$[Fe\ (OOH)]^{2+} \rightarrow Fe^{2+} + HO_2^{\cdot} \quad (3)$$
$$HO_2^{\cdot} + Fe^{3+} \rightarrow Fe^{2+} + H^+ + O_2 \quad (4)$$

D. Experimental Setup

A schematic representation of the laboratory reactor for Fenton process is shown in Fig. 2. The experiments were performed in a beaker made of borosilicate glass of 500 ml. The reactor is placed on magnetic stirrer was stirred with a magnetic stirrer for 60 minutes using a constant speed to maintain a homogenous concentration during the experiments. The simplest Fenton process requires an acidic medium, because low pH favours Fenton oxidation. Typically, the pH is first adjusted to prevent formation of iron oxyhydroxides. There are two ways to control acidity. One is to adjust the initial pH of the solution. The other is to control pH throughout the process by adding sulphuric acid or sodium hydroxide. Then iron salt, commonly ferrous sulphate (Fe^{2+}), is added and finally, (30%)H_2O_2 is slowly introduced. The pH was monitored and controlled at 3±0.2.

RESULTS AND DISCUSSION

Fenton Treatment (Fe_{2+}/H_2O_2)

In Fentonprocess, influence of Fe^{2+} concentration on percentage removal of COD, BOD_3 and BOD_3/COD from the pharmaceutical wastewater has been considered as shown in figures 3, 4, and 5 respectively withCOD 14097 mg\L, BOD_3 3000 mg\L for the contact time of one hour. It can be concluded from the figure, the dose of Fe^{2+} increases from 2 to 8 g/L, COD and BOD removal are increased from 20.0 to to 60.0 % and 8.33 to 41.67 %, respectively. Minor variation in pH was observed afterFenton treatment.Improvement in BOD_3/COD of 0.30 from 0.23 was found after treatment at 8g/L dose of $FeSO_4.7H_2O$.

CONCLUSION

It is pointed out from study that Fenton process was not so much efficient. When, these individual processes will combine with other AOPs processes, it will be more efficient than the individual process. In the study, Fenton process which is based on the generation of Fe^{2+} in the presence of H_2O_2 was used to treat pharmaceutical effluent. Various doses of ferrous sulphate was used from 2 g/L to 8 g/L and achieved COD removal from 20% to 60%, BOD removal 8.33 % to 41.67% and increase biodegradability index from 0.23 to 0.30. Study investigates that maximum COD reduction of 60% was achieved at 8 g/L dose of $FeSO_4.7H_2O$. So, it is investigated that Fenton process alone was not so much sufficient to efficiently remove the recalcitrant compounds and reduce COD concentration. Further, it is matter of research work, to consider various combinations of Fenton process with other advance oxidation process to achieve 100% removal of COD from pharmaceutical effluent.

FIGURES

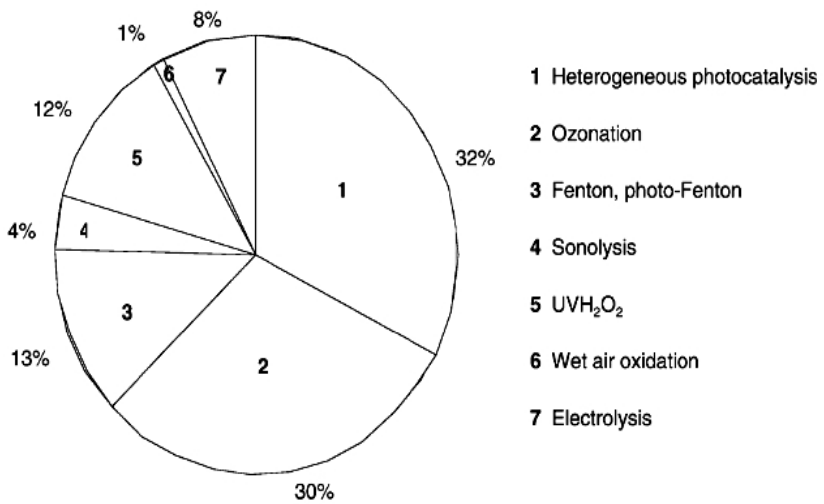


Figure 1 AOPs Tested on Pharmaceutical Effluent [3]

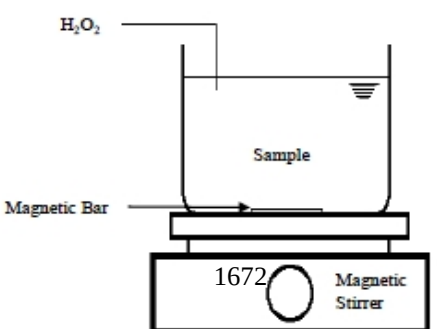


Figure 2 Experimental Setup

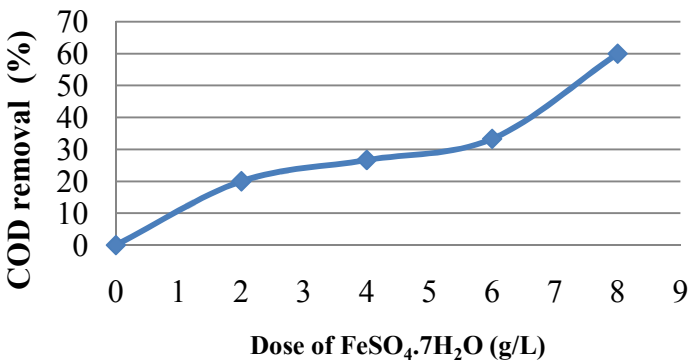


Figure3 Different doses of $FeSO_4.7H_2O$ v/s COD removal

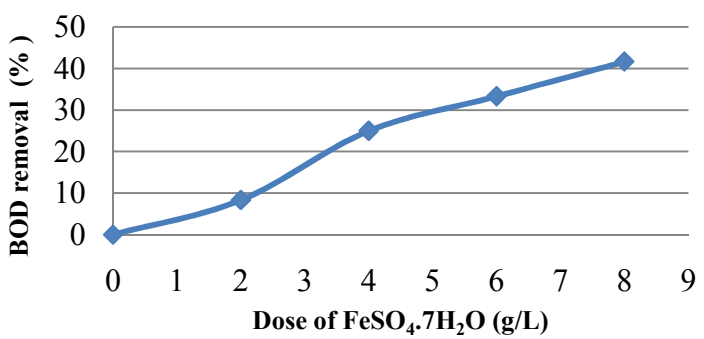


Figure4Different doses of $FeSO_4.7H_2O$ v/s BOD_3 removal

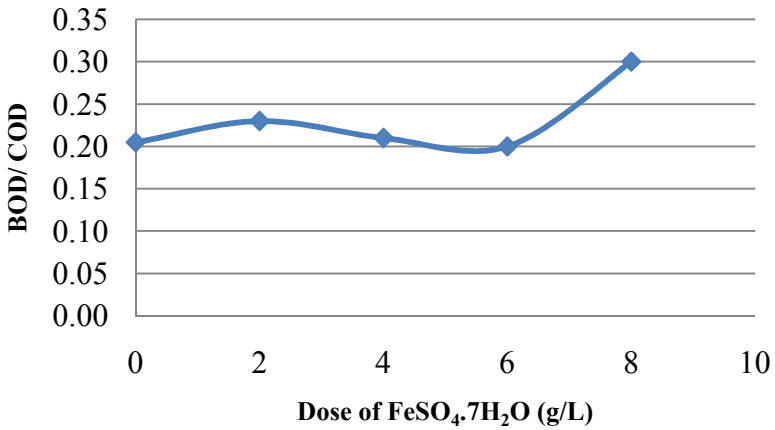


Figure5 BOD_3/COD after Fe^{2+}/H_2O_2 Treatment

TABLES

Table-1: Characterization of Wastewater

S.N	Parameters	Results (ppm)	Desirable Limit (ppm) (Inland Surface)
1	pH	12.38	5.5-9.0
2	BOD	3220	30
3	COD	14190.4	250
4	TDS	2160	2100
5	TSS	868	100

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Exergy Analysis on double effect, parallel flow 250 TR LiBr/H_2O Vapor Absorption Refrigeration System

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ABSTRACT

This paper deals with exergy analysis of the double effect, parallel flow 250 TR Libr/H_2Ovapour absorption refrigeration system. The objective of the paper is to find out losses and destructions in the system. The Property of Libr/H_2O at every state point is determined and thermodynamic model is prepared. Exergy at inlet and outlet of each component are determined. The performance parameters like exergy destruction, exergy loss, exergetic efficiency, exergy destruction ratio, exergy loss ratio and C.O.P. of the given system are evaluated. Results indicate that the coefficient of performance for double effect is 0.84- 0.94. The exergy destruction is observed in each component of VARS. This analysis provides an opportunity to optimize the given system.

SUMMARY

In this paper exergy destruction is evaluated and it is to be optimized.

Keywords: Exergy,Energy,Vapor

INTRODUCTION

The Energy optimization and utilization of waste heat has become a most critical issue in the industry. Also, CFC-based refrigerant are ozone depleting. So, today eco-friendly refrigerant is required in the chiller plant and air dryer.

Many researchers and investigators have studied and presented such system in the systematic manner.Absorption cycle is one of solution for utilizing waste heat.Development of a packed bed type solution heat exchanger has designed for better performance and to reduce the crystallizationfor single effect lithium bromide water vapor absorption cycle [1].The exhaust gas energy is utilized as a heat source for an ammonia–water absorption refrigeration system [2]. The exhaust gas energy availabilityand the impact of the absorption refrigeration system on engine performance, exhaust emissions, andpower economy are evaluated. The investigation for multi effect cycle has been carried out [3], which only considers the unavoidable exergy destruction for single,double, triple and half effectWater–Lithium bromide absorption cycles.For utilization of absorber heat [4] , GAX cycle has been introduced in which the heat exchange from generator to absorber is applied and exergy destruction is analyzed. The thermo economic optimization concept is applied for single and double effect cycle [5, 6], cost of inflows and out flows are evaluated and total cost effective model is developed. The performance of major component of series flow machine is obtained by computerized program for further investigation [7] Simulation of LiBr–H2O single stage absorption water chiller carried out for best economic performance [8] . In this paper we have included both heat reclaimer and drain heat exchanger for exergy analysis for parallel flow VARS for capacity of 250 TR

THERMODYNAMIC ANALYSIS

For the thermodynamic analysis of the VARS, the principles of mass conservation, first and second laws of thermodynamics are applied to each component of the system[10].Each component can be treated as a control volume with inlet and outlet streams, heat transfer and work interactions. In the system, mass conservation includes the mass balance of total mass and material of the solution. The governing equations of mass and type of material conservation for a steady state and steady flow system are:

$$\sum_{i=1}^{i=n} m \; - \sum_{o=1}^{o=n} m \; = 0 \qquad (1)$$

$$\sum_{i=1}^{i=n} (m \;)i - \sum_{o=1}^{o=n} (m \;)o = 0 \qquad (2)$$

Where m is the flow rate and x is mass concentration of Libr in the solution. The first law of thermodynamics yields the energy balance of each of the component of the absorption system as follows:

$$\sum_{i=1}^{i=n} (mh)i - \sum_{o=1}^{o=n} (mh)o \; + [\sum Q \; - \; \sum Q \;] + W = 0 \qquad (3)$$

For the purpose analyses following assumptions are made (a) Pressure losses in all heat exchanger and pipelines are negligible. (b)Cooling water enters at temp.32˚Cand leaves the water at 37.4˚C. (c) Inlet cooling water temp. Range is 29˚C to 33˚C to avoid crystallization. (d) The concentration of Libr must not reach above 65%. (e) Vacuum should be maintained constant and not below 6mm of Hg. (f) Absorbent pump, refrigerant pump and vacuum pump power are neglected. (g) Refrigerant enters in evaporator at 12˚C and leaves at 7 ˚C

For absorber,

Mass balance equation is:

$$m_6 + m_{16} = m_7 \quad (4)$$

Mass Concentration balance is:

$$m_6 x_6 = m_7 x_7 \quad (5)$$

Heat balance for component

For absorber

$$Q_a = m_6 x_6 + m_{16} x_{16} - m_7 x_7$$

$$= m_{23}\,(h_{24} - h_{23}) \quad (6)$$

For generator

$$Q_g = m_1 h_1 + m_1\ h_1\ - m_1\ h_1\ = m_1\ (h_1\ - h_1\ ) \quad (7)$$

For condenser

$$Q_c = m_3(h_4 - h_5) = m_2\ (h_2\ - h_2\ ) \quad (8)$$

For evaporator

$$Q_e\ = m_6(\ h_7 - h_6) = m_2\ (h_2\ - h_2\ ) \quad (9)$$

For expansion valve

$$h_5 = h_6 \quad (10)$$

$$C\ = Q_e\ /Q_g \quad (11)$$

If we neglect the environmental heat losses and pump work then we can write energy balance for the absorption system

$$Q_a + Q_c = Q_g + Q_e \quad (12)$$

EXERGY ANALYSIS

The exergy analysis gives an idea about the thermodynamic inefficiencies produced in a particular process quantitatively as well as qualitatively. This inefficiency increases the cost of final product. When the system interacts with another system and allowed to come to equilibrium, gives work as output. Exergy can be defined as the maximum amount of theoretical useful work obtainable when the system comes to the state of environment condition

In absence of magnetic, electric, nuclear, and surface tension effect, the total exergy of the system considered of four components: Physical exergy, Kinetic exergy, Potential exergy and Chemical exergy.

$$\dot{E} = \dot{E}^P + \dot{E}^K + \dot{E}^P + \dot{E}^C \quad (13)$$

Considering the system at rest and no chemical reaction takes place,

$$\dot{E}^P = m[(h - h_o) - T_o(S - S_o)] \quad (14)$$

The exergy analysis requires a proper 'Fuel-Product-Loss' (F-P-L) definition of the system to show the real production purpose of its subsystems by attributing a well defined role, i.e. fuel, product or loss, to each physical flow entering or leaving them. For HTG, LTG,SHX-1, SHX-2, DHE, Condensor, Evaporator, Absorber, Expansion valve F – P – L is calculated[11]. A detailed exergy analysis includes calculation of exergy destruction, exergy loss, exergetic efficiency, two exergy destruction ratios, and exergy loss ratio in each component of the system along with the overall system. Mathematically, all these are expressed for the component as

Exergy destruction is

$$\dot{E}_D = \dot{E}_F - \dot{E}_P - \dot{E}_L \quad (15)$$

Exergetic efficiency

$$\mathcal{E} = \frac{\dot{E}_P}{\dot{E}_F} \quad (16)$$

Exergy ratio

$$Y_d = \dot{E}_d \Big/ \dot{E}_F \quad (17)$$

$$Y_d^* = \dot{E}_d \Big/ \dot{E}_{d,T} \quad (18)$$

RESULTS AND DISCUSSION

The 250 TR VARS which is analyzed in this paper shown in Fig.1. Table.1 represents the calculated thermodynamic properties at every state points of double effect parallel flow for VARS. The calculation is done for base operating condition by using the. The result shows that the C.O.P of the VARS is 0.84 which is low. Heat removal at absorber and condenser is 1502.68 kW and heat addition at generator and

evaporator is 1524.43 kW. Both are not equal because there is mixing loss in HTG and absorber. The exergy analysis of component and the system are carried out by using the F-P-L relationships and summarizes in Table 2. The results shows that maximum exergy destruction is 85.59 kW in HTG with Y_d (exergy destruction ratio) of 45.81%.The DHE has second largest exergy destruction of 56.01 kW with Y_d of 29.98%. They are followed by evaporator assembly where the exergy destruction is 35.39 kW with Y_d of 18.94%, heat reclaimer is 20.81 kW with Y_d 11.14%, LTG is 7.55 kW with Y_d of 4.05%,SHX-1 is 2.46 kW with Y_d of 1.32%,SHX-2 is 2.12 kW with Y_d of 1.13% .The same trend is found for Y^*_d in the system. Exergy loss is 70 kW in evaporator assembly which is carried by cooling water with Y_d of 26%. The exergy destruction of the system is 186.83 kW with exergetic efficiency of 15.43% which is very low. The destruction, loss and product of exergy are represented by pie- charts in figure 2. The destruction in generator is high because of improper mixing of the refrigerant and Libr solution. Second the higher heat is required to liberate the refrigerant from Libr because of weak solution of Libr. The refrigerant vapor is in superheated condition at 710mm of Hg. So, dissipation rate is also high. The destruction in DHE is also high. In DHE the refrigerant from LTG enters in vapour form which loses the latent heat in DHE. The 15% of the flow from absorber which is weak solution having concentration of 57% passed through the DHE. So, the heat transfer between the refrigerant and the solution is poor.

The temperature of generator is important parameter for exergy destruction. As the temperature increases the COP of the system the decreases, which is shown in Fig.3. As the generator temperature increases exergy destruction alsoincreases. So temperature should be optimized. Second thing child water temperature inlet to system increases the COP increases but increased load on absorber may give the problem of crystallisation problem. It can be graphically plotted in Fig.2 by using EES software.

CONCLUSION

The analysis presented in this paper demonstrates the exergy analysis approach to the double effect parallel flow VARS to find out the actual productive exergy for the system. From our analysis we can observe that exergy destruction in the HTG is high which can be optimized, also the temp. Of HTG is optimized, so that C.O.P. of system also improved. In DHE the destruction is also higher which can be also optimized. So that destruction of exergy is minimized and ultimately cost.

FIGURES

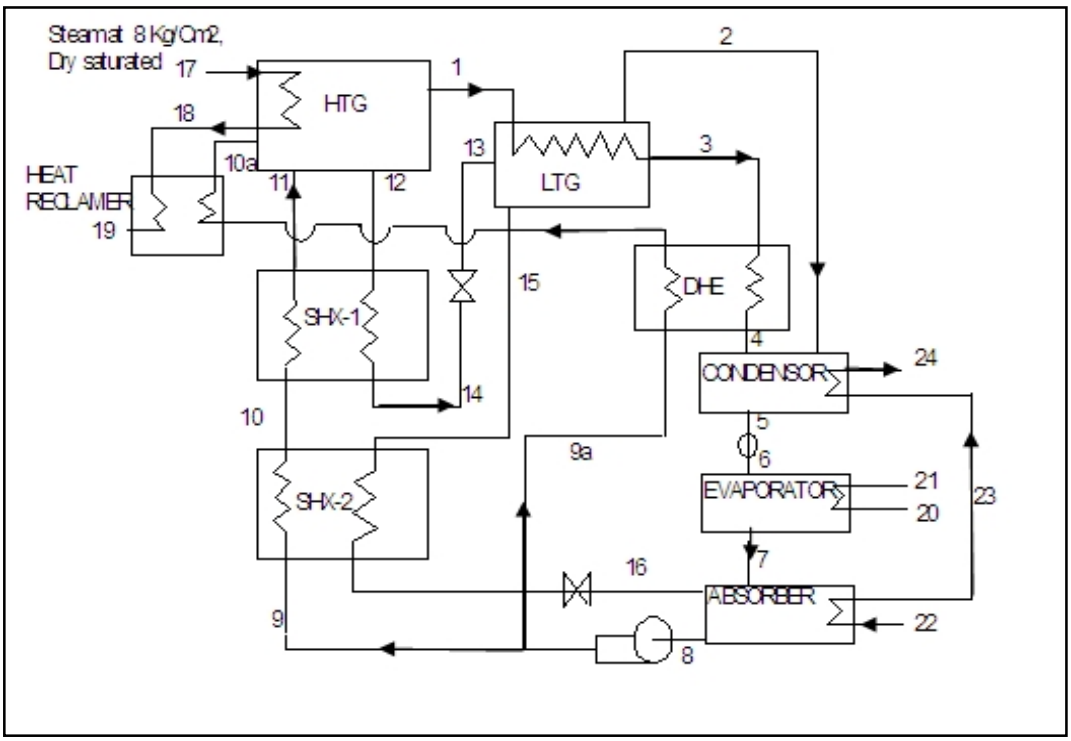
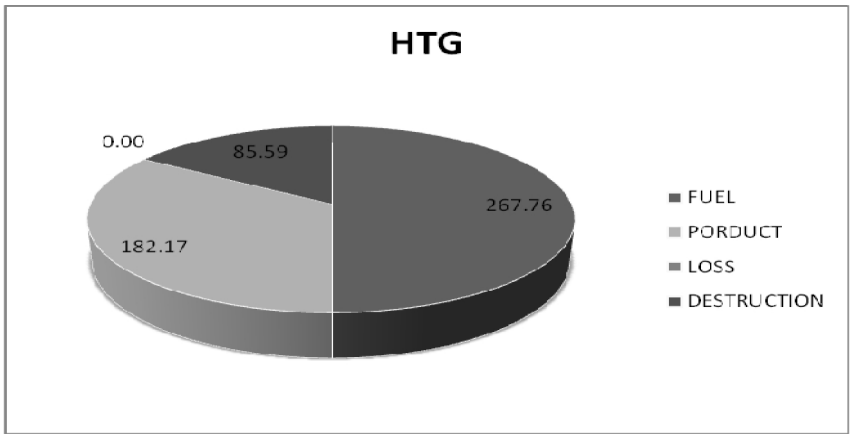
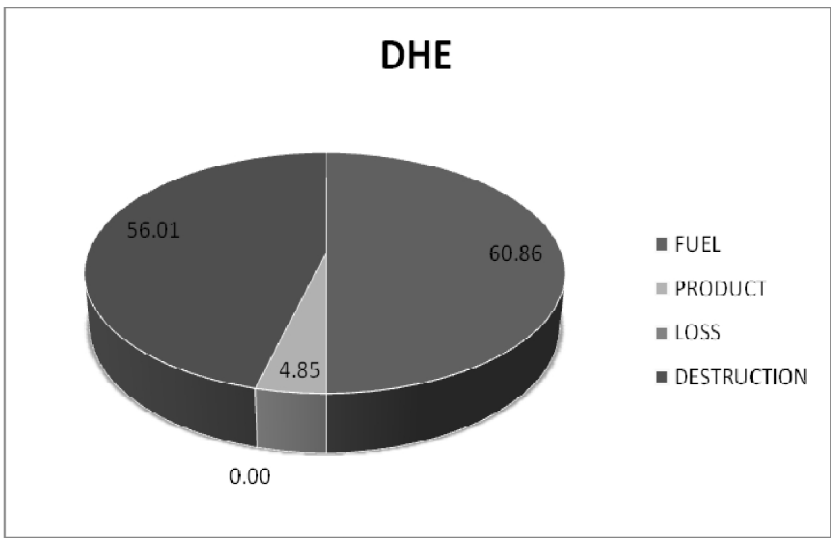
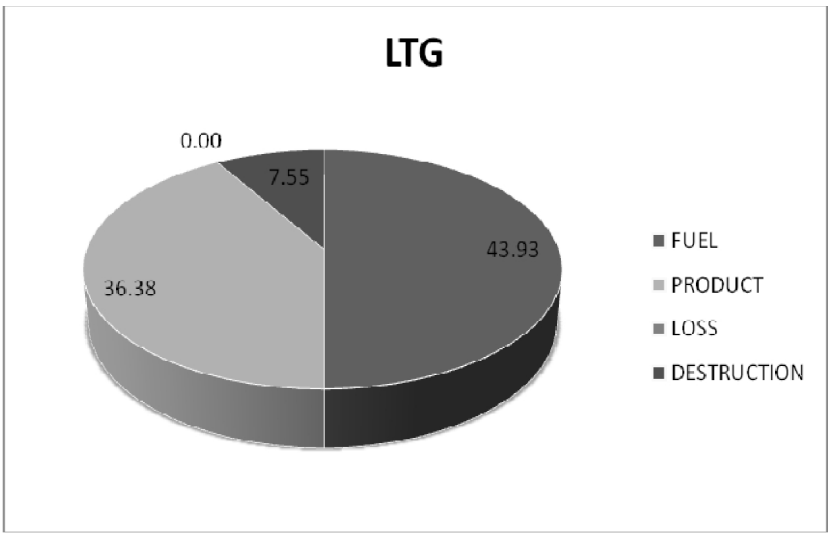


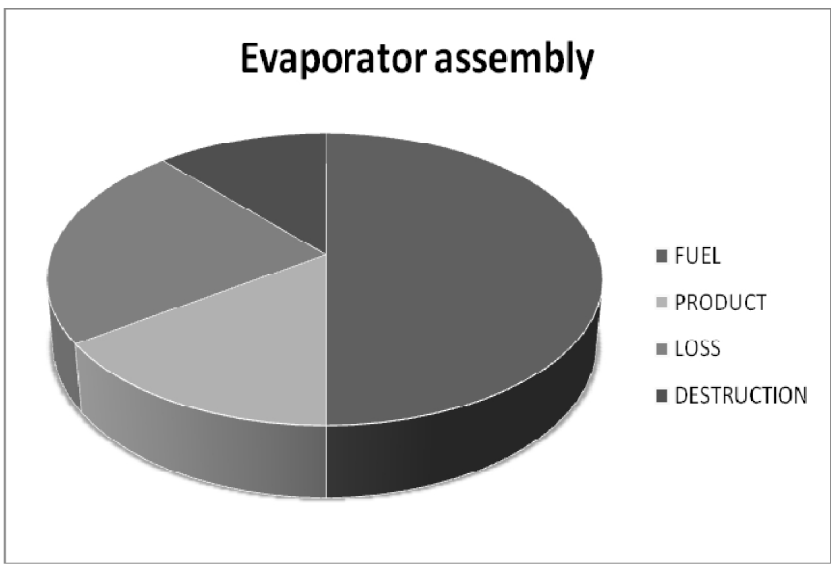
Fig. 1 Schematic diagram for double effect parallel flow LiBr/H_2O vapour absorption refrigeration system

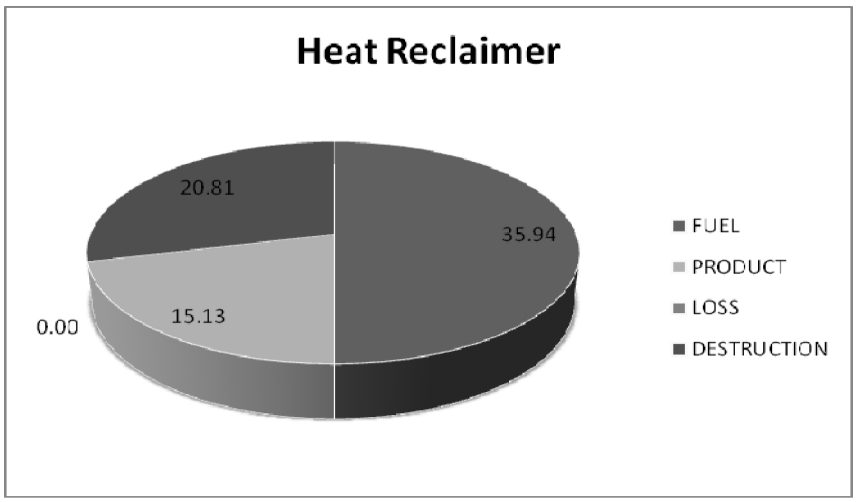
Fig.2 Pie chart of energy and overall system of VAR

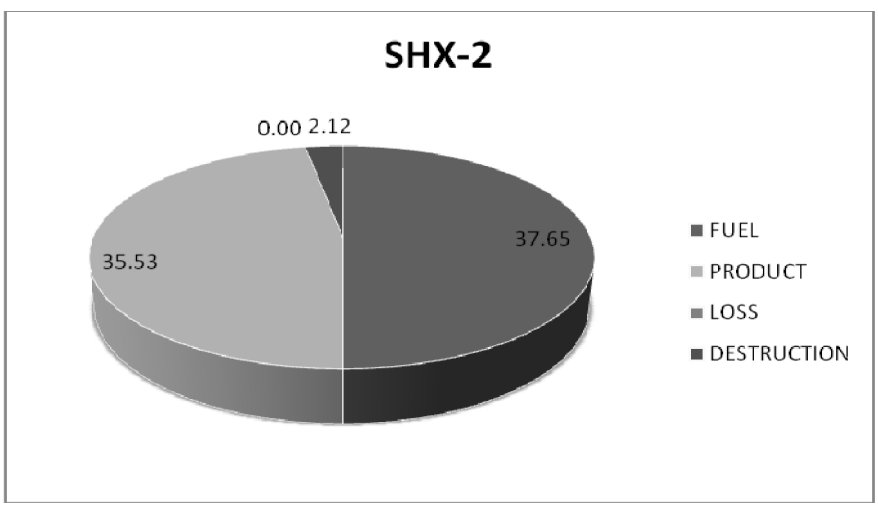

HTG
0.00
85.59
267.76
182.17
FUEL
PORDUCT
LOSS
DESTRUCTION

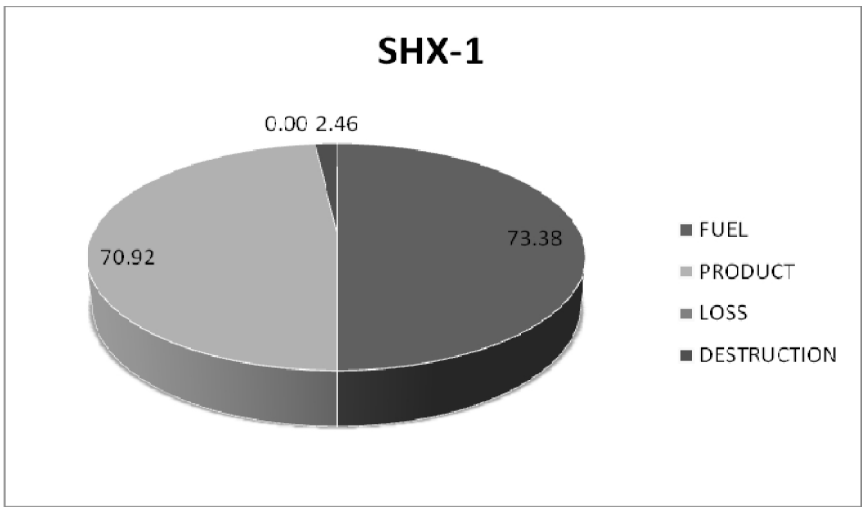

DHE
56.01
60.86
4.85
0.00
FUEL
PRODUCT
LOSS
DESTRUCTION

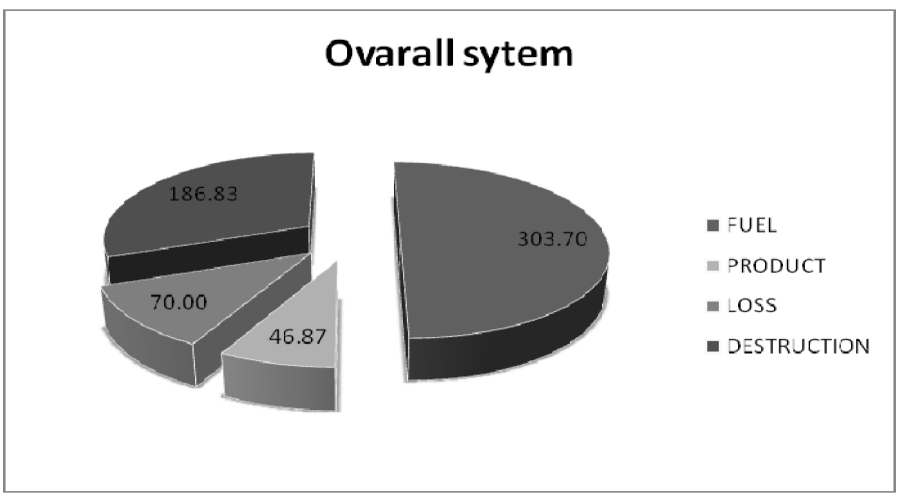

LTG
0.00
7.55
43.93
36.38
FUEL
PRODUCT
LOSS
DESTRUCTION


Evaporator assembly
FUEL
PRODUCT
LOSS
DESTRUCTION


Heat Reclaimer
20.81
35.94
0.00
15.13
FUEL
PRODUCT
LOSS
DESTRUCTION


SHX-2
0.00
2.12
37.65
35.53
FUEL
PRODUCT
LOSS
DESTRUCTION


SHX-1
0.00 2.46
73.38
70.92
FUEL
PRODUCT
LOSS
DESTRUCTION


Ovarall sytem
186.83
303.70
70.00
46.87
FUEL
PRODUCT
LOSS
DESTRUCTION

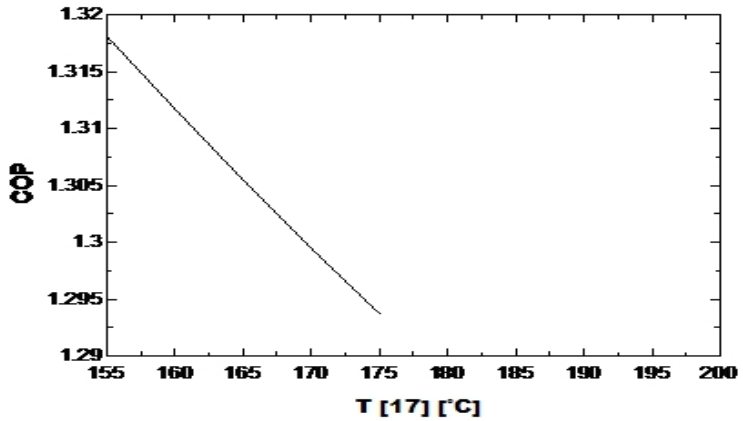


Fig.12 Effect of generator temperature on COP

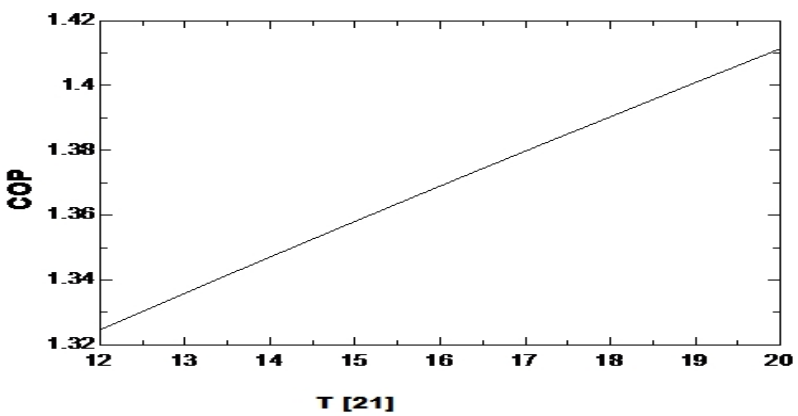


Fig.4Effect of chilled water temperature on COP

Table.1 Thermodynamic analysis results

State point	t('C)	p(mm)	m(kg/s)	X(%)	h(kJ/Kg)	S(kJ/KGK)	Exergy	
							E(kJ/KG)	Ė(KW)
1	155	710	0.30	0	2776.00	7.613	511.77	151.12
2	98	710	0.30	0	2687.50	7.775	375.00	110.73
3	45	55	0.30	0	2395.20	7.526	156.90	46.33
4	39	55	0.30	0	163.40	0.559	1.26	0.37
5	4.5	6	0.30	0	18.00	0.559	-144.14	-42.56
6	4.5	6	0.30	0	2509.00	9.039	-180.18	-53.20
7	38	6	2.27	57	65.27	0.280	-13.72	-31.20
8	38	6	2.27	57	65.27	0.280	-13.72	-31.20
9	90		2.27	57	59.27	0.580	-109.12	-248.11
10	145		2.27	57	59.27	0.850	-189.58	-431.06
11	145		2.27	57	59.27	0.850	-189.58	-431.06
12	155	710	2.38	61	773.38	0.870	518.57	1235.47
13	100	55	2.38	61	118.38	0.620	-61.93	-147.55
14	84	55	2.38	61	118.38	0.550	-41.07	-97.85
15	92	55	2.38	64	121.38	0.570	-44.03	-104.90
16	50	6	2.38	64	72.38	0.360	-30.45	-72.55
17	170		0.39	0	2767.50	6.600	805.15	312.40
18	170		0.39	0	720.00	2.045	115.04	44.63
19	85		0.39	0	355.90	1.134	22.41	8.70
20	12		34.72	0	50.40	0.181	0.91	31.53
21	7		34.72	0	29.40	0.106	2.26	78.40

22	32		69.44	0	134.20	0.464	0.37	25.97
23	35		69.44	0	146.70	0.505	0.66	45.55
24	37.4		69.44	0	155.84	0.539	-0.34	-23.33
8a	38	55	0.40	57	97.37	0.290	15.40	6.18
9a	75	710	0.40	57	172.05	0.500	27.50	11.03
10a	145	710	0.40	57	314.05	0.850	65.20	26.16

Table.2 Exergy analysis results of double effect parallel flow VARS

Component	**Fuel**	**Product**	**Loss**	**Destruction**	**ε(%)**	**Y_d(%)**	**Y_d^*(%)**	**Y_l(%)**
HTG	267.76	182.17	0.00	85.59	68.03	45.81	31.97	0
Heat Reclaimer	35.94	15.13	0.00	20.81	42.09	11.14	7.77	0
LTG	43.93	36.38	0.00	7.55	82.81	4.04	2.82	0
SHX-1	73.38	70.92	0.00	2.46	96.65	1.32	0.92	0
SHX-2	37.65	35.53	0.00	2.12	94.37	1.13	0.79	0
DHE	60.86	4.85	0.00	56.01	7.97	29.98	20.92	0
Evaporator assembly	152.26	46.87	70.00	35.39	30.8	18.94	13.22	26.14
Overall system	303.70	46.87	70.00	186.83	15.43	100.00	69.77	26.14

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An Efficient Adaptive Digital Predistortion Framework to Achieve Optimal Linearization of Power Amplifier

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ABSTRACT

In the era of modern communication systems, to achieve optimal linearity of power amplifiers without sacrificing energy efficiency has become more challenging especially in the case of orthonormal frequency division multiplexing (OFDM) signals. Most of the recent communication signals such as (OFDM signals, RF signals) usually have large peak-to-average power ratio. Therefore, it has an impact on enhancing non-linearity of signals with orthonormality. Also, it maximizes the in-band distortion and out band spectral re growth. This paper aims to develop a novel adaptive approach to formulating orthonormal basis functions, which have been further carried out to enhance the numerical instability during evaluation of coefficients. The experimental prototype based proposed framework also focuses on exhibiting computational complexities associated with least square methods. Also, it keeps maintaining the convergence speed of Recursive Least Squares Algorithms (RLS). A comparative analysis in between the proposed framework and conventional systems (RLS, LMS) has been performed by obtaining the experimental and simulation results which further exhibits and validates the effectiveness of the proposed framework.

Keywords: Adaptive Digital Predistortion Framework, Energy Efficiency, Orthonormal Basic Functions, Low Complexity, Power Amplifiers.

1. INTRODUCTION

A power amplifier is a kind of amplifier which amplifies the input signal power and control the output device. In a terrestrial broadcasting scenario, a power amplifier (PA) can be used to evaluate the overall power efficiency of a communication system. However, linear characteristics of a power amplifier only can be estimated for a small range of input values, and the overall performance pay-off of communication system exploits that PA doesn't achieve suitable power efficiency for a small range of liner values *(1, 2)*. To achieve power efficiency PA usually generates a nonlinear relationship on a nonlinear domain. The

non-linearly associated with PA can degrade the overall system performance by escalating both in-band distortions and out of band spectral re growth (3). Therefore, the in-band distortion can have an impact on breaking down the error vector magnitude (EVM), which is considered as a useful component to quantify the performance metric of a digital radio transceiver and the out band components can cause adjacent channel interference (ACI) where incomplete filtering of unwanted modulated components happen*(4, 5)*. PA's nonlinear characteristics can differ on various factors such as temperature, time, etc. The recent advancements in the field of information and communication technologies configure the PA's mostly on dynamic situations, for example, PA's dynamic characteristics are adopted in any heterogeneous networks (HetNet) *(6)*. There are different kinds of scenarios that dynamically alter the PA characteristics such as transitions of different carrier frequency signals on multi-career base stations, antenna impedance variations that affect the PA characteristics in real time *(7)*.

There are so many existing PA linearization techniques such as feedback, feed forward and prediction algorithm, etc. which have been studied to improve the energy efficiency of a communication system *(8, 9)*. Among all the power amplifier linearization techniques adaptive digital distortion (DPD) techniques have been found to be more precise regarding linearization performance, cost effectiveness and complexity reducing, therefore, Adaptive DPD models have been widely improvised into modern real-time communication applications *(10,11)*.

The polynomial model that has been widely used in the adaptive DPD systems has become quite popular for formulating DPD coefficients and nonlinear characteristics of different PA's *(12)*. The model also highlights the fact that how numerical instability and computational complexity become so much difficult during the coefficient estimation. The study of *(13)* established an orthonormal basis function that improve the numerical instability as well as enhance the linearization performance. The proposed study is highly motivated by the above-stated scenario where the use of orthonormal basis function eases the improvement of numerical instability. In this paper, a novel framework based on adaptive digital distortion has been introduced which derives the orthonormal basis functions efficiently to improve the numerical stability during coefficient evaluation *(14)*. The proposed algorithm achieves fast convergence and also consumes very less amount of hardware recourses as compare to the conventional coefficient algorithms such as recursive least square (RLS), least means square (LMS), etc. Considering the formulation of orthonormal basis function the proposed system achieves very low computational complexity as LMS algorithm whereas retains very fast convergence speed like RLS algorithm. The paper is organized as follows Section 2 discusses the recent studies towards energy efficient digital distortion techniques which are followed by problem statement in Section 3. Section 4 discusses proposed system followed by a discussion of algorithm implementation in Section 5. Section 6 discusses the result analysis followed by conclusion in Section 7.

2. RELATED WORK

This section discusses the existing studies that has been carried out in the past regarding achieving optimal linearization of power amplifier using digital distortion The discussion in this section is carried out with respect to recent work being carried out and study of standard DPD techniques that achieve optimal linearization of power amplifiers also consumes very less amount of energy.

The study of Wang et al. *(15)* presented a force ampuler given envelope following (ET) with application to the WLAN 802.11g framework. Baseband pre-mutilation is actualized to enhance the linearity. A high-effectiveness wide-transfer speed envelope speaker and a GaAs MESFET Class AB RFPA are planned and actualized for the framework. A general framework channel effectiveness of 30% is gotten, for an OFDM yield force of 20 dBm at 2.4 GEIz; the concentrate likewise depicted the outline framework approach and exploratory consequences of an exceedingly proficient 802.1 lg OFDM speaker.

In the study of Zenteno et al. *(16)*, a non-parametric system for displaying the conduct of power amplifiers is exhibited. The proposed system depends on the standards of thickness estimation utilizing

the portion technique and is suited for use in force intensifier displaying. The proposed approach changes the information space into an orthogonal memory area. The outcomes demonstrate that the technique can be utilized to model power amplifiers, in that yielding mistake execution like cutting edge parametric models. Besides, a parameter-proficient model structure with six coefficients was inferred for a Doherty power intensifier, in that altogether diminishing the sending's computational many-sided quality. At last, the procedure can likewise be all around misused in advanced linearization systems.

The study of Zhang et al. *(17)* proposed a band-isolated DPD strategy, in which the confined criticism data transfer capacity is partitioned into two sections in recurrence space focus and fringe areas of PA's band-constrained yield. These two sections are portrayed independently utilizing two distinct premise capacities with a band-partitioned memory polynomial (BDMP) model. It gives higher adaptability in selecting the advanced band-constraining channel's transmission capacity furthermore decreases the channel request. The estimation results for a class-AB GaN PA with orthogonal recurrence division multiplexing sign of 60-MHz transmission capacity have demonstrated the predominance of BDMP DPD over the past cutting edge.

Ma et al. *(18)* proposed a novel strategy, which permits the utilization of low-speed ADCs by acquainting unearthly extrapolation with the band-restricted criticism sign, is proposed. This permits effective usage of DPD for extremely wideband signs. Test results show that the data transfer capacity of the securing way can be even not exactly the transmission capacity of unique sign applying the proposed procedure. What's more, acceptable linearization execution has been accomplished utilizing wideband signs up to 160 MHz data transfer capacity.

The study of Liu et al. *(19)* exhibited a general DPD building design for wideband PA frameworks with compelled input transfer speed. By utilizing straight operations to cross out the transfer speed jumble between the proposed model and the PA criticism flag, the full-band PA model parameters can be assessed with data transmission constrained perceptions. This assessed PA model is therefore utilized with the PA data sign to remove the DPD capacity by applying the immediate learning calculations. The proposed DPD construction modeling diminishes the criticism transmission capacity to under two times that of the data signal, while it keeps up its linearization execution, as in the full-band case. Tests are performed on the 20-and 100-MHz long haul development propelled signs to show the viability of the proposed PA conduct displaying and DPD linearization exhibitions with restricted input data transfer capacity.

Liu et al. *(20)* presented another computerized distortion (DPD) solution for wideband force intensifiers (PAs) with confined input data transfer capacity and low testing rate. By coordinating a microwave cavity channel into the input way, the criticism data transfer capacity is decreased productively. A PA parameter extraction system is then proposed to distinguish the PA model, utilizing the transmission capacity obliged signs. By applying the extricated parameters to the immediate learning structural planning, the DPD capacity can be gotten, which can linearize nonlinear mutilation over the examining data transfer capacity. Tests exhibit that a 22 dB adjoining channel spillage proportion change is gained for a 100 MHz Long Term Evolution-propelled signal, notwithstanding when the input transfer speed is limited from 500 MHz to 100 MHz, which amazingly decreases the ADC examining rate from 1105.92 Mbps to 368.64 Mbps.

Jeong et al. *(21)* presented another system to diminish the data transmission of the dynamic power supply waveform utilized as a part of wideband envelope following force enhancers (PAs). At the point when the envelope the following strategy is connected to broadband flags, for example, WCDMA and 3GPP LTE, the wide data transmission of the envelope signal makes it hard to execute the dynamic supply modulator effectively and precisely. We appear here a strategy to lessen the data transmission of the force supply waveform, consequently permitting better effectiveness for the supply modulator; and a linearization system for rectifying the nonlinearity brought on by the transfer speed decrease. The achievability of this method is exhibited for a solitary transporter WCDMA signal with a 7.6-dB top to-normal force proportion utilizing a GaAs high-voltage HBT PA.

In the study of Son et al. *(22)* the envelope following system is connected to both the drive and principle power amplifiers to enhance the effectiveness of the drive intensifier, which is normally worked at a low proficiency. The trial results demonstrate that the productivity of the aggregate framework is enhanced by 2.1% while the proficiency of the drive intensifier is expanded by 8%. What's more, to defeat a genuine non-linearity because of the double envelope following the operation, another consecutive computerized distortion building design is proposed.

3. PROBLEM STATEMENT

The previous section introduced various existing studies towards power amplifier linearization where various framework to maximize the power efficiency have been introduced. The conventional techniques such as RLS, LMS, etc. gained a wide recognition in past for achieving the power amplifiers nonlinearity in the case of transmission signals (OFDM, RF signals). However the biggest problems associated with this mechanism are a repetition of nature, and very less amount of methods have defined which can optimize the in-band distortion of out band spectral re growth without sacrificing the power efficiency.

The conventional LMS algorithm is computationally very much effective but attains very less amount of convergence speed where as RLS algorithm achieves the very efficient amount of convergence speed but consumes a huge amount of computation recourses thus more power is wasted. Firstly majority of the recent techniques only focuses on efficient linearization of power amplifiers, but they do not consider energy consumption issues caused by the numerical instability of the conventional algorithms. Secondly most of the studies only discuses the theoretical background of the problem they have not formulated any kinds of enhanced adaptive distortion algorithm to improve the numerical instability that can further reduce the computational overhead from the communication system. Very few studies have been found to discuss the in-depth discussion and respective solution towards nonlinearity of PA's which leads to degrading the performance of Error Vector Magnitude (EVM) as well as Adjacent Channel Interference (ACI) by generating in band deformation. Thirdly it can also be observed that there are very less amount of existing studies which talks about how to control the power amplifier linearization during any kinds of dynamic scenario or in case of heterogeneous network as PA's output power or the non linear coefficients can vary with respect to the temperature, time and the variation associated with the input signals.

Fourthly the conventional PA linearization techniques such as feedback, feed forward and distortion only focused on gaining energy efficiency but their mathematical modeling such as adopted DPD technique does not offer a good compromise on complexity, linearization performance as well as cost effectiveness. To achieve the above-stated problem solutions an efficient and novel framework that also uses the concept of adaptive DPD technique but here an adaptive orthogonal basis function has been formulated to improve the convergence speed as well the processing speed of the algorithm during the coefficient estimation. Hence, the problem statement of the proposed study can be defined as "Designing a computationally efficient adaptive digital distortion framework to enhance the convergence speed during coefficient estimation to enhance the communication system performance".

The next section introduces the design specifications associated with the proposed system followed by the implementation and the result analysis respectively.

4. PROPOSED SYSTEM

The proposed study aims to develop a novel Adaptive Digital Predistortion Framework to achieve optimal linearization of power amplifiers without sacrificing the energy efficiency. Firstly the proposed system considers the model coefficient estimation during the formulation of orthonormal basis function where orthonormal basis function improves the numerical instability during the process of coefficient estimation. It can be seen by evaluating many conventional studies that polynomial model has been highly applied in adaptive DPD systems to evaluate the nonlinear characteristics of PA's. The memory

less polynomial framework is used to derive the PA model as well as distorter design. Finally, the simulation has been carried out on a real-time prototype by obtaining the values of NMSE (the normalized mean square error), and power spectral density of each signal.

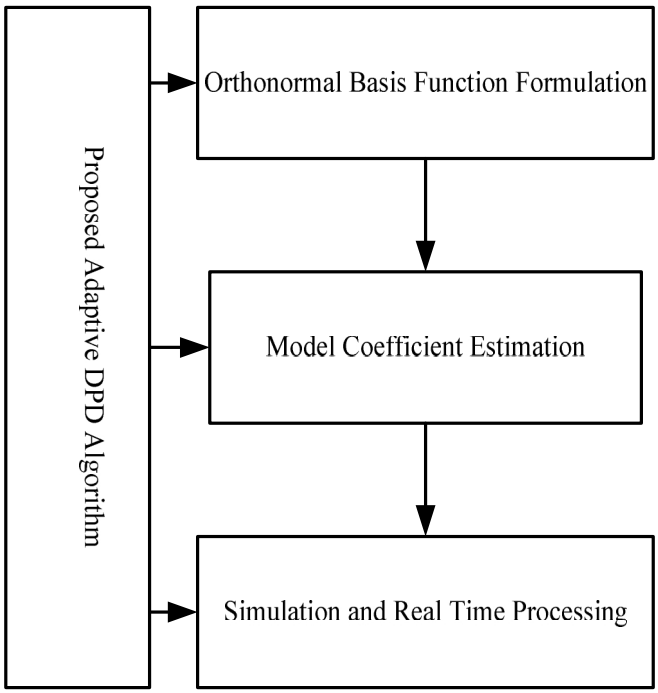


Fig. 1. Schematic Diagram of the Proposed System

A comparative analysis also has been highlighted where iteration number, PSD (dB), NMSE (dB), frequency are considered as performance parameters. The comparative analysis and the simulation result shows how convergence metrics associated with RLS, LMS, and the proposed algorithm has been obtained and how the proposed system ensures effectiveness regarding less computational complexity and high convergence speed. The above-stated figure 1 represents a schematic diagram of the proposed system where three different types of modules are considered and evaluated. The main objective of the proposed adaptive digital distortion framework is to achieve very low computational complexity like LMS algorithm whereas very fast convergence speeds like RLS algorithm. The design specification of the proposed system highlights that it converges quickly like RLS but addition of conventional orthonormal basis function enhance the numerical stability where as the performance metrics of the proposed algorithm shows that it works almost same as the conventional RLS algorithm, but the computational complexity associated with the proposed system is O (N) per iteration whereas the computational complexity of the RLS algorithm is on the order of O (N2) per iteration. The development of the proposed system is done in an analytical approach to ensure better evidential ground.

5. IMPLEMENTATION

The implementation of the proposed system has been carried out using MATLAB, where three different type of modules such as 1. Orthonormal basis function formulation using adaptive DPD, 2. Model coefficient estimation and 3. Simulation and comparative analysis have been introduced. The orthonormal basis function formulation that is further utilized to improve the coefficient estimation is discussed below.

The nonlinearity of a PA changes with respect to time, temperature and the energy level defined in the input signal. The proposed system adaptive DPD technique followed by orthonormal basis function

formulation to track and recompense the varying nonlinearity of a PA. The proposed system considers a pre-distorter block in between the input baseband signal and the PA input. The band pass signals are sampled, and an attenuator is used to evaluate the power of PA output. Therefore, the error signal is calculated by considering input signal and the output of the estimation block. The model coefficients are defined in the distorter block and also can be frequently updated using model coefficients.

A polynomial model has been used in the adaptive DPD systems to formulate PA modelling. The polynomial model also describes the non linear characteristics of a power amplifier. The following mathematical modelling highlights the framework for coefficient estimation. The following is the (2n-1) polynomial basis function.

$$Y(n)= \sum B.A \text{ where } B=\mu_{2n-1},\ A=\beta_{2n-1}(n) \qquad (1)$$

The above equation 1 has been formulated by combining two different type of polynomial model. In the above-stated equation the μ_{2n-1} is the (2n-1)-th model coefficient and $\beta_{2n-1}(n)$ is a polynomial basis function.

The second module of the proposed system calculates the error signal which is further applied to formulate the objective function which minimizes the power of the error signal E (N). The objective function is defined by evaluating the following equation

$$\text{ArgMin} \sum | E (N) |^2 \qquad (2)$$

Above mentioned equation 2 requires an LS solution where as Hermitian transpose metrics has been defined. By using the Hermitian transpose metrics, the probability density function of the signal amplitude is computed. The experimental prototyping considers that the input signals are highly correlated. The Matrix is computed by considering individual PSDs of signal components. The proposed system also highlights that condition numbers of the matrix grows exponentially on a number of polynomial terms. The next module of the proposed system formulates orthonormal basis functions that help to ease the numerical instability issues during the coefficient evaluation matrix.

The orthonormal basis function is defined as follows.$F [\acute{\alpha}_{2n-1}(K). \alpha_{2P-1}(L)] = \begin{cases} 0 & \phi n = P \\ 1 & \phi n \neq P \end{cases}$ (3)

The above-mentioned orthogonal basis function also can be achieved by a linear combination of the conventional polynomial basis functions.

An upper tri angular matrix also formed which further utilized to obtain the orthonormal basis functions. Therefore, the signal amplitude also considered to determine the power spectral density which is used in the LU factorization to evaluate the upper triangular matrix. The flowing diagram shows the process of the LU factorization.

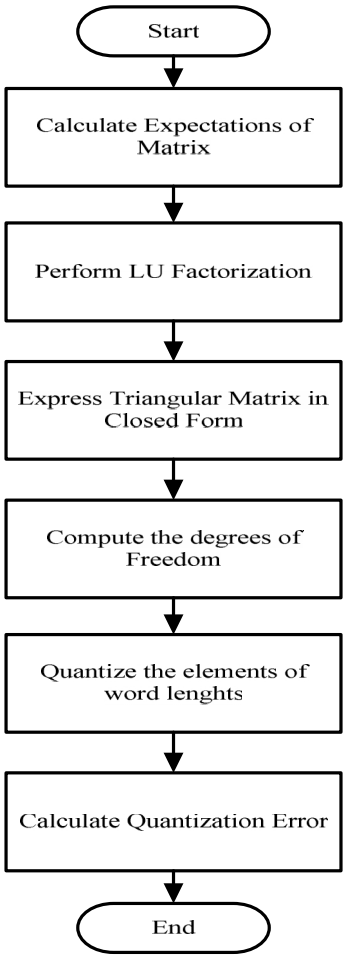


Fig. 2. Formulation of Orthonormal Basis Function

In the above-stated figure, an internal process of formulating orthonormal basis function has been highlighted where it can be seen that the quantization errors for orthonormal basis function can be significantly optimized. To achieve fast convergence speed the proposed method performed some division operation where the precise function μ (K) deviates on the step size 1/n. The next section discusses the performance analysis of the proposed method.

6. RESULT DISCUSSION

This section discusses the important findings of the proposed study. It also highlights a comparative analysis in between the proposed system and the conventional LMS and RLS algorithms and hence similar simulation parameters are considered for evaluating the performance metrics of the proposed system. The following Table one illustrates the comparison of the different adaptive approaches.

Table.1. Comparison in between different adaptive models

Methods	Multiplications	Iteration	Time (T)
RLS	74	458	3467
LMS	36	1024	5350
Proposed System	25	315	2489

The above-stated Table 1 shows the comparison in between the existing conventional method (LMS, RLS) with the proposed system where the second column highlights the multiplication required for evaluating each method. The third column shows the iteration required to compute each algorithm. In the last column, the computational time which is considered as CPU clock time has been highlighted.

Table. 2. Comparison in between different adaptive models

APCR Values (dBc)	Simulation		Experiment	
Algorithms	L	H	L	H
RLS	42.34	41.24	40.56	40.78
LMS	41.29	40.23	39.67	40.24
Proposed Method	41.20	40.18	39.12	39.50

The above stated table 2 also highlights the corresponding adjacent channel power ratio for different types of algorithms. The RLS, LMS and proposed method have been applied to linearize the Power amplifier; Figure 3 shows the comparative analysis of the proposed system where the normalized mean square error and iteration have been considered as performance parameters. It also shows the convergence seed of all algorithms. The green line denotes the convergence curve of the Recursive least square (RLS) algorithm. The convergence curve of the RLS algorithm has been derived from formulating the polynomial equations. The Black solid line here is considered as the convergence curve of the conventional LMS algorithm where a piecewise function (ϕ=0.005) has been considered. The blue solid line denotes the convergence curve of the proposed system (1/n). The convergence curve of the proposed system also has been formulated by evaluating the above stated equations. Figure 3 also highlights that normalized mean square error (NMSE) has been calculated for all the power amplifier linearization method. The comparative analysis highlights that proposed algorithm achieves almost similar convergence speed as compare to the RLS algorithm and mean square error in the proposed algorithm is very less as compare to the LMS algorithm.

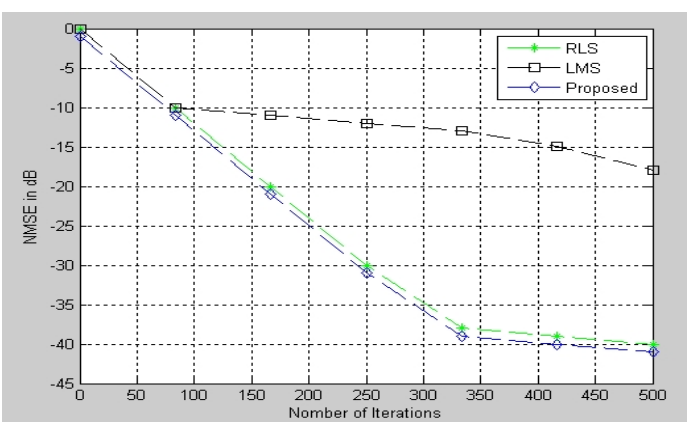


Fig. 3. Comparative Analysis of all Algorithms

Considering the orthonormality defined in the above-stated equations 1 and 2, the proposed system also utilizes the concept of RLS algorithm and develops an efficient adaptive algorithm that converges quickly

and also consumes very less amount of hardware resources. The orthonormality has been defined using a statistical coefficient estimation of sample signals.

To achieve the linearization of PA the proposed system an adaptive distorter with the polynomial model has been integrated at the transmitter and the receiver end. The sample size has been considered in between 500, and the number of polynomial terms has been taken as n= 5. The base band signal (IEEE802.11) has been considered to have 30 MHz bandwidth and 64QAM modulation as a configuration and channel properties. The existing studies highlight that majority of the OFDM signals leads to complexities of Gaussian domains.

The experimental prototype applies RLS algorithm. LMS algorithm and the proposed system for linearization of the power spectral density characteristics associated with the power amplifier. The proposed system also computes how it is more effective on minimizing the quantization error. The normalized min square error has been computed by observing the iteration numbers associated with each algorithm highlighted in table 1. The proposed system indicates that it converges very quickly as the formulation of orthonormal basis function improves the numerical stability and reduces the computational complexity. The proposed system with a piecewise model ϕ(n) converges bit slowly as compare to (1/n).

7. CONCLUSION

Power amplifier linearization without sacrificing energy efficiency in modern heterogeneous networks have become one of the most challenging issues thus the application of adaptive digital distortion algorithms to obtain better quality signal sort out the above-stated issue in some extent. The proposed study introduces a novel digital distortion framework to achieve the linearity of the power amplifier; it also consumes very less amount of energy as the algorithm requires very less computing recourses. The proposed algorithm achieves an efficient convergence speed while formulating the orthonormal basis function during coefficient estimation also reduce maximum quantization error in the context of power spectral density of a signal. A comparative analysis has been drawn to highlight the effectiveness of the proposed system. It has been also highlighted that the proposed system ensures better quality performance as compare to the conventional RLS and LMS algorithms.

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Permeability Test on Stabilized Black Cotton Soil with Cement Waste

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ABSTRACT

A series of permeability test was conducted on black cotton soil of Saurashtra region stabilized with Cement Waste and performance of black cotton soil was evaluated. Cement Waste, used, is a waste-dust of cement plant. As per IS recommendations, Falling Head Permeability test method was adopted to assess soil. Tests were performed immediately after addition of stabilizer and subsequently at 7 and 28 days of curing period. Variation in permeability was observed with and without stabilizer and investigation was made for suitability of stabilizer. It is observed that permeability was relatively high in non-stabilized soil samples and in stabilized form; it was decreased with the increase in curing time period. The trends observed in permeability were going lower and lower with the increase in the amount of cement waste percentage and curing time period. Satisfactory result was obtained from samples stabilized with 10% cement waste at 28 days of curing.

SUMMARY

Cement Waste is an appropriate driving force, as a stabilizer, for black cotton soil of Saurashtra region to get better the engineering properties like permeability and other for its positive performance in road construction.

Keywords: Stabilization, Black Cotton Soil, Cement Wast, Permeability.

INTRODUCTION

Black Cotton Soil*(1)*

Black cotton soil is widely available in *Saurashtra*area. According to the analysis done by experts of mineralogy, montmorillonite is present in huge quantity in black cotton soil. Chemical equation of montmorillonite can be given as $(OH)_2Al_4Si_8O_20.nH_2O$. This shows much attraction to water*(2)*. Sandwich hexagonal layers of two Si and one Al sheets form ionic structure of montmorillonite. O-O interlayer separates these sandwich layers of Si and Al. This oxygen bond is very weak and allows passing of water through it. These property results cracks in soil without any warning. As shown in Fig-1, Depth of the cracks may every so often extent rigorous border of one and half inch to twelve inches. Construction of roads on such soil leads to serious damage to the work as well as life of road. It may prove to be wastage of public money and as a result razor-sharp impact on the national economy. Climatic changes have influence on the behaviour of black cotton soil. Consequently it is bad for pavement construction. To stabilize such soil is the best solution to gear up the soil properties, by mechanical or chemical means. By means of stabilization there may be increase in strength and durability of black cotton soil. Main object of the stabilization is to achieve the expected design aspects of soil beneath the pavement which may withstand the whole assembly *(3)*.Design engineers have to be very careful in selecting stabilization technique as they are the key persons for success and/or failure of the process.Some of stabilization techniques are based on trial and error process but even though such modus operandi leads to exact on the goal point.We know that worldwide soils types are not same and at the same time soil properties also differ. To attain a remarkable success in stabilization work, laboratory testing of soil the on way. Before allowing any procedure at large scale, soils should pass through laboratory test every time. Black cottonsoils, in India, have moderate to heavy compressible nature due to presence of inorganic clays *(4)*.Mainly such soils are characterized by heavy shrinkage and swelling properties. Approximately 20% of total area of India, mainly the central and western region is covered with black cotton soil.High swelling and shrinkage characteristics of the black cotton soil are the main rival of transportation engineers. The black cotton soil is very hard when dry, but loses its strength completely when in wet condition.On drying, the BC Soil develops cracks of varying depth. This wetting and drying process result in to the vertical movement in the soil mass *(5)*.Such movement is a dangerous phenomenon as it leads to pavement failure,settlement, heavy depression, cracking and unevenness of pavement.

Permeability of Soil

Permeability is the property of soil pertains to the water accessibility in the soil. A soil sample allowing more water to pass through itself is considered to be permeable soil. And the soil, having very strict access for water, comes under the category of less permeable soil. A rock like strata of soil doesn't give acquiescence to water to make a way through it. Such soil is grouped as an impermeable soil. According to Standards association of Australia, permeability of black cotton soil is ranging from 0.01×10^{-2} to 0.01×10^{-5} mm/sec, depending upon the minuscule variation in grain size of the particles of soil. As per Indian Standard methods for test of soils, laboratory test for determination of permeability for a fine grain soil, falling head method is suitable to get the reliable results. Set up for the same is as in Fig.2. Permeability is a factor varies with the grain size. In soil like black cotton soil, sometimes permeability is affected by water bonding within clay particles. It is to be measure with more care in such state of affairs. In many cases permeability has proven to be a decisive property. False permeability is measured by chance when some impurities are present in water, which may lead to wrong calculation of behaviour of soil. Also in some test, organic matters and entrapped air block the way of water and hence permeability estimation goes wide of the mark. Such possibilities should be checked when a precise assessment is required.

According to Indian Standards, IS Code 2720-1986 (Part-17)*(12)*, falling head permeability test apparatus(Fig-3), specimen and procedure is adopted to evaluate co-efficient of permeability, k, for a black cotton soil sample. Value of k measured at temperature T° C is required to be corrected for a room temperature of 27° C.

MATERIALS AND METHODS

Characteristics of Black Cotton Soil:

As discussed earlier, Black cotton soil is a very poor type of soil having black and dark brown particles with very fine grains. 85 to 100 percentage of particles are smaller than 75 micron size. And these particles are pure clay particles. Normally, this soil has very low bearing capacity and high swelling and shrinkage characteristics. Due to its abnormal characteristics,it performs very poor materials for foundation and road construction.

Characteristics of Cement Waste:

Cement Waste used in the test is provided by Saurashtra Cement & Chemicals Limited, Ranavav Dist. Porbandar (Renowned by the Brand- HATHI CEMENT). It is an industrial waste, in a dust form, collected from packing department as dust settled on the floor during the automatic packing of bags of Portland cement. The cement dust possesses the chemical properties as tabulated in Table-1. As it is a dust formation, properties differs slightly than the basic properties of cement.

Falling Head Permeability Test Procedure for Modified Black cotton soil

Very first step for laboratory test on Black Cotton Soil is pulverization of black cotton soil. The soil was pulverized, soaked in water for 24 hours and the vegetation'swere removed. After drying in oven, the Soil was than tested in its natural state for the permeability, according to relevant IS Code. Maximum dry density and Optimum moisture content were considered to prepare the sample for permeability test. MDD and OMC were preliminary tested to use it for main test. Table-2 is showing the basic natural values for the Black cotton soil*(10,11)*. After obtaining the natural value of permeability for virgin black cotton soil, stabilizing agent namely cement waste was added into the soil and again a series of test of permeability was conducted. The addition of stabilizing agent was gradually increased in percentage ranging from 5% to 10% of total soil mass used. Total numbers of sample prepared were 6*3= 18. A net weight of 2.5 kilogram for each soil sample was measured and percentagewise stabilizer was thoroughly mixed in it. Whole sample was than compacted properly in proctor mould with three layers. The proctor mould size was observed as 127 mm height and 100 mm inner-diameter. Out of the total samples, 6 were tested immediately after removing from proctor mould i.e. no time of curing was given. Another 6 samples were tested after 7 days of curing and remaining 6 were tested on 28^{th} day of curing. Curing of samples was done with wrapping of jute bags over the samples, giving periodical sprinkling of water on them to maintain the moisture. Permeability for each sample was derived using standard procedure prescribed by IS 2720-1986 (Part-17)*(12)*.

RESULTS AND DISCUSSION

Cement waste has bonding nature as well as mixture is compacted at maximum dry density level, this helps the black cotton soils particles to bind together, resulting reduction in voids in soil. To lower down the permeability, chemical compositions of cement do the leading job. 20% of Sio_2 produces viscous gel. This gel will help to bind the particles of black cotton soil. Due to this, plasticity of the soil will increase and thin films of gel will bind the tiny grains of black cotton soil. Beta form is the diamond part of

stabilization. MgO at 0.8% and Al_2O_3at 5.7% help the mix to become durable and stiff. Addition of cement waste in black cotton soil gives pleasing results in the Laboratory. As the quantity of stabilizer increases, the permeability values gradually slow down. From 5% to 9% of cement waste, soil behavior for accepting water was step by step becoming sluggish. At 9% and 10% time for change in water level is mostly same. Saturated soil allows water to pass through but the molecules of cement waste in soil, progressively lower down the passage for water. Further, curing on samples gives marginal change in flow of water through the sample. While taking observation, it was observed that the full saturation of sample is taking place after 48 hours and change in water heads starts at 50 hours. At 28 days of curing, samples become more stiff and durable. Curing period of 28 days generates truss in to the samples which is must require for improvement in impermeability of black cotton soil. Table- 3 to 5,in order (1) without curing (2) 7 days of curing and (3) 28 days of curing, express gradual drop off in the permeability co-efficient value. Graphically, gradient lines for each numeric value convey the relation between co-efficient of permeability and percentage of cement waste added (Fig-4 to 7).In entire laboratory effort, it is to be taken care about the rigorous mixing of two materials and also over curing of the samples to be avoided. These may certainly damage to the final outcome.

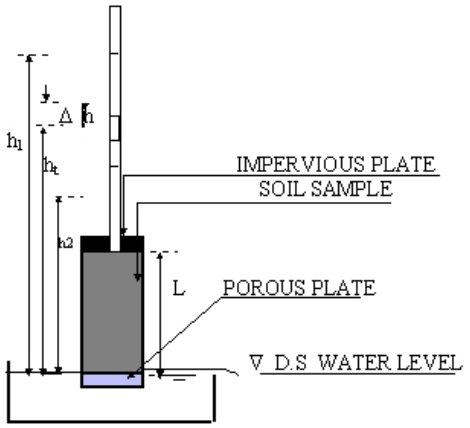
CONCLUSION

Overall, looking to the results of experiments, it can be finished off with minimum 9 to 10% of cement waste as stabilizing material, which seems to be a good fusion for black cotton soil of Saurashtra region under laboratory environment. The results obtained are to be had in graphical manner to read between the lines, effortlessly, in a view to right use. Unambiguous data leads to optimal and worth use of cement waste as stabilizing agent to perk up watertight grade of black cotton soil.

FIGURES



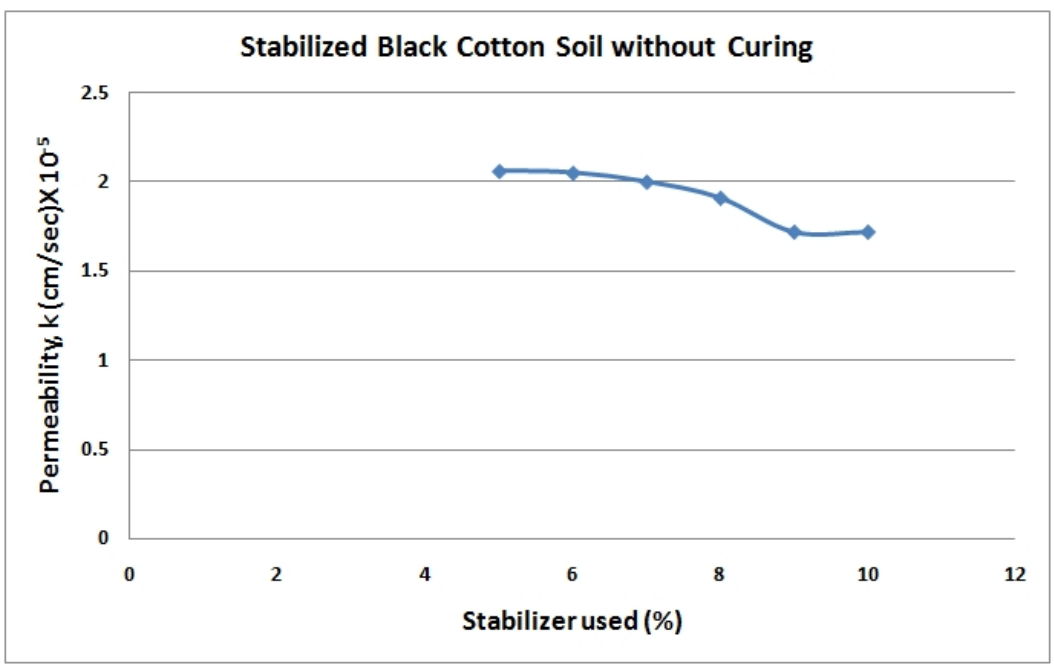
"**Fig. 1.**(a) Behaviour of Black Cotton Soil (b) Sample Collection of Black Cotton Soil"



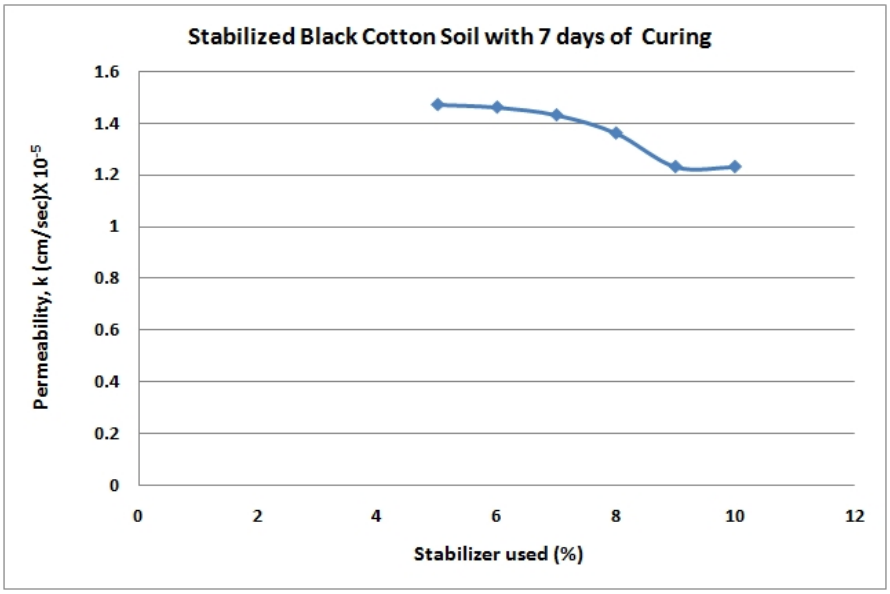
"**Fig. 2**. Falling head permeability setup"



"**Fig. 3**. Permeability apparatus"



"**Fig. 4**. Relation Established for Permeability and Use of percentage Stabilizer (Cement Waste) without curing."



"**Fig. 5.**Relation Established for Permeability and Use of percentage Stabilizer (Cement Waste) after 7 days of curing."

"**Fig. 6.**Relation Established for Permeability and Use of percentage Stabilizer (Cement Waste) after 28 days of curing."

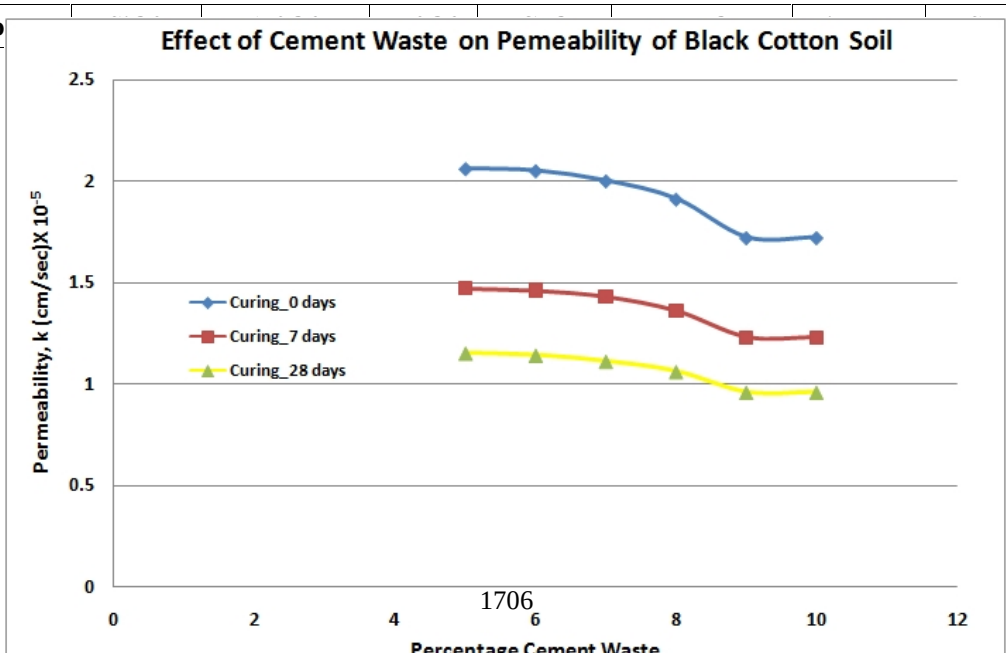
"**Fig. 7.**Aggregate relation for Permeability and Use of percentage Stabilizer (Cement Waste)"

TABLES

Sr.No.	Property of Black Cotton Soil	Value
1	Maximum Dry Density (N/mm^2)	1.449
2	Optimum Moisture Content (%)	16
3	Co-efficient of Permeability (cm/sec)	2.07×10^{-5}

"**Table 1**. Natural Properties of Black Cotton Soil"

Prop



of Cement Waste	(Silicon Dioxide)	(Aluminium Oxide)	(Iron Oxide)	(Calcium Oxide)	(Magnesium Oxide)	(%)	Gravity
% by weight	18.5	13.5	0.62	14.02	0.84	94.33	2.86
		# As per test certificate of NSIC No. 1583 24/8/12, Rajkot					

Sr No	Item	Nat_Cond	C_Waste 5%	C_Waste 6%	C_Waste 7%	C_Waste 8%	C_Waste 9%	C_Waste 10%
1	Co-efficient of Permeability (cm/sec)	2.07×10^{-5}	2.06×10^{-5}	2.05×10^{-5}	2.00×10^{-5}	1.91×10^{-5}	1.71×10^{-5}	1.71×10^{-5}

"Table 2. Properties of Cement Waste"*(1,14)*

"Table 3. Value of Permeability Test of samples of Black Cotton Soil with Cement Waste without Curing"

Sr No	Item	Nat_Cond	C_Waste 5%	C_Waste 6%	C_Waste 7%	C_Waste 8%	C_Waste 9%	C_Waste 10%
1	Co-efficient of Permeability (cm/sec)	2.07×10^{-5}	1.47×10^{-5}	1.46×10^{-5}	1.43×10^{-5}	1.36×10^{-5}	1.23×10^{-5}	1.23×10^{-5}

"Table4. Value of Permeability Test of samples of Black Cotton Soil with Cement Waste after 7 days ofCuring"

Sr No	Item	Nat_Cond	C_Waste 5%	C_Waste 6%	C_Waste 7%	C_Waste 8%	C_Waste 9%	C_Waste 10%
1	Co-efficient of Permeability (cm/sec)	2.07×10^{-5}	1.15×10^{-5}	1.14×10^{-5}	1.11×10^{-5}	1.06×10^{-5}	0.96×10^{-5}	0.956×10^{-5}

Table 5. Value of Permeability Test of samples of Black Cotton Soil with Cement Waste after 28 days of Curing"

ACKNOWLEDGEMENT

Our special thanks to the Deputy Manager Mr. Maulesh Oza, Saurashtra Cement and Chemicals Ltd, Ranavav for allowing us the use of cement Waste and providing the same without any cost *(1)*. We offer our special gratitude towards the students of Diploma Civil 5th Sem_2015 of Government Polytechnic, Rajkot for assisting in experimental Work.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

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RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

Category Analysis of Truck Trips Observed at Samakhiyali Check Post, Gujarat

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ABSTRACT

Widely used transportation modes are through air, water and land. Each of them has its own inherent characteristics. Historically, several cities have grown around sea throughout the world. Even today, the trade activity around ports is significant. The general trend of activity at several ports shows that the freight related activity at ports has continued to grow over the years. The freight activity at ports has a direct correlation to the economies of the areas surrounding the port and an impact at regional level also. Much of the work done in the field of modeling is on modeling personalized transport. Research of freight transport is scarce. In the recent years, interest in modeling the freight activity at ports has increased throughout the world. In the present study, category analysis of truck trips observed at Samakhiyali Check-Post, Gujarat is made for the modeling of freight transport at regional level which is generated by the Kandla Port activities.

SUMMARY

In present study category analysis of data collection for modeling of freight transport is presented.

Keywords: freight transport, modeling, data.

1. INTRODUCTION

Infrastructure is a key backbone of any nation for development. With the help of efficient infrastructure network and development only a country can make progress. In the era of globalization, foreign trade has also expanded in a great form. Internationally, goods and commodities can be move with the help of widespread network of infrastructure and success of a nation in international trade is much dependent on efficient infrastructure network. Two major means of movement of goods at international level is through Air and Water. 90% of the international cargo is transported through ships(Planning commission, India). Ports are no behind in contribution of development of nation. But movement of commodities through port is again dependent of several characteristics like freight demand, land use characteristics, socio-economic factors and location (Deshmukh, 2004). More demand of goods affects capacity of port in terms of expansion of existing roadway and railway infrastructure. Travel demand is the primary input for transportation planning and analysis. Each port is different depending on its size, the type of commodities and the volume of commodities, and may also include seasonal variation of demand.

A port may attract cargo based on its functioning, its hinterland connectivity, reliability in operation and entire supply chain. Shippers choose a port based on the network performance, cost-effectiveness, overall reduction in operating cost and increase in profit.

Transport sector accounts for a share of 6.4% in India's Gross Domestic Product (GDP). Road transport has emerged as the dominant segment in India's transportation sector with a share of 5.4% in India's GDP. Road transport demand is expected to grow by around 10% per annum in the backdrop of a targeted annual GDP growth of 9% during the Eleventh Five Year Plan (IRC).

Study of travel demand is essential for transportation planning. Important parameters to be included in freight travel demand modelling are connectivity of hinterland, access to freight terminal and travel time/ delay.

A large number of studies on data collection, selection of port, modelling etc. are made. In the next section study of methods of data collection for freight transport is made.

2. LITERATURE REVIEW

Pioneer work was done by Safwat (1982) who introduced the Simultaneous Transportation Equilibrium Model (STEM). In that model, the generation of trips in a region is incorporated by a specific nonlinear function that includes transportation costs [17]. In his study, equations were derived for delivery cost which depends on time value, unit price of commodity, time spend in Administration Logistics cost, and tariff to deliver a commodity. Separate model was also developed to work out operational cost and cost of link. In link cost, variables considered were mode, flow and length.

Study was conducted by Lorenzo Masiero and David A. Hensher (2012) [18] to identify the parameters related to freight transport modeling. Within a freight transport context, the origin-destination distance and the weight of the shipment play an important role in the decision of the most preferred transport service and in the way logistics managers evaluate the transport service's attributes.

Allen Browne and Cherrett (2012) [19] did investigation on relationships between road freight transport, facility location, logistics management and urban form. Their findings suggest that several geographical, spatial and land use factors have important influences on freight activity in urban areas.

Nir et al. (2003) in their finding stated that good or bad points of ports service system may affect the user's port choice behaviour, or even influence the cost of the whole fleet or shipper, so port choice is an important part of port transportation demand behavior [5]. International and inter-regional freight demand is determined by the spatial distribution of production and consumption activities [8].

The choice of the modality or mode of transport (road, rail, water, air) is the most discussed point of intervention for freight transport policies. This is in sharp contrast with the industry, however, where the decision is often taken implicitly, or without much contemplation. This can be explained by the fact that, given the dependence on available infrastructures and the transport requirements of the goods, the number of realistic choices for a firm is often limited (Jordans et al., 2006).

A roadside intercept survey was conducted by Hsing-Chung Chu (2009), from August to October 2009 to collect truck movement data in the Kaohsiung metropolitan area, which is particularly connected with the major truck-trip generator the port. A valid sample of 6,000 heavy-duty truck drivers carrying TEU(Twenty Foot Equivalent Unit) containers completed the questionnaire. The interviews were conducted at the locations of terminals at the port (2,200), warehouses/distribution centers (2,000), and freeway truck stops (1,800).

Patil (2014) developed macro level models based on GDP in Indian condition for in bound and out bound truck trips. The variables considered for model development are GDP, crude oil production and food grain production. Data from the statistical department was used for the modeling.

G. Ramadurai et al. (2014) did case study on Urban Freight Trip Generation of Chennai City. Data collected and develop freight trip generation models. Sources of data were Websites like Yellow Pages, Sulekha, Just Dial, Chennai Corporation, Commercial Taxes Department and Economic Census.

IRC SP-19: 2001 indicates collection of vehicular, commodity, trip and route related characteristics for freight traffic O-D survey such as registration number, vehicle type, commodity type, weight, origin, destination, trip length and route adopted.

3. Problem Statement and Need of study

Freight transport is an essential part of our economy as it fulfils a unique service within supply chains, bridging the distances between spatially separated places of supply and demand. As is the case with passenger transport, accessibility of places for freight is vital or the economic development of society [7]. There is a significant increase in commodity import/export movement from ports in last few decades. Moreover research in freight transport is scarce. So, an attempt is made through this study.
The major objective of this study is to develop a truck trip generation model for highway networks connecting to port. The model can be used to understand the freight traffic and to determine the significant parameters for truck trip generation for Kandla port. The minor objectives of this study are:
1. To find present O-D scenario of truck trips estimation.
2. To determine the effect of import/export commodity on truck trip generation.
3. To study the effect of parameters generating truck trips from port.

4. Study Area Selection

In the proposed research, study is be made keeping Kandla as origin and destination of goods trip. Kandla Port is a seaport in Kutch District of Gujarat state in western India, near the city of Gandhidham. Located on the Gulf of Kutch, it is one of major ports on west coast.
Samakhiyali is considered as entrance of Kutch region. It is the only means of movement to and from the port of Kandla. All goods vehicles moving to/fro Kandla has to pass RTO and/or Sales Tax check post.

5. Variables for Freight Data collection

Proposed variables identified for category analysis are as follows:

- Based on characteristics of the mode which include travel time and distance has important effect. Hence, the parameters related to it are,
  - Type of vehicle
  - Origin of trip
  - Destination of trip
  - Overall journey time
  - Distance

- Based on characteristics of the goods including shipment type is considered for goods related variables.
    - Type of commodity carried
    - Quantity of commodity carried(Gross weight)
    - Package characteristics

All the above variables can be constant, additive or multiplier to a function in modeling. Owing to the complexity of the goods movement, no single method of data collection could cover complete goods movement and its characteristics. So, manual data collection method is employed for this study.

6. Data Collection :

As discussed in the section 5, it is aimed to collect data in form of truck trip diary and personal interview survey. For freight modeling seasonal data is required. The work is divided among three seasons i.e. monsoon, winter and summer. However, data collected by personal goods interview survey before monsoon is presented. Data is collected from location where truck drivers are compelled to stop i.e. a check post. Samakhiyali check post located near village Samakhiyali was selected for the survey. In the vicinity of RTO check post, another check post of Commercial tax department is located. Both of this check posts were selected for data collection by the prior approval of authorities. 5 enumerators were employed for data collection purpose. The format of data collection is given in Annexure-II.

6.1 Geographical Location :

Details of survey spot:

- Type of road : 6 lane divided national highway (Toll road)
- National Highway No. : 8-A
- Pavement condition : Bituminous road with good riding surface
- Road Number : 161
- Distance of both check posts from Kandla port : 60 Kms.
- Type of traffic passing through : Mixed traffic

The satellite image of survey location is shown in figure 1 and actual photograph is shown in figure 2. Personal interview for freight traffic was carried out on July 5 (Sunday) and July 11 (Saturday), 2015.

The survey was conducted at Samakhiyali RTO check post (RTO) and Commercial Sales Tax (CST) check post. The format used for data collection is shown in Appendix I at the end of report.



Fig. 6.1 : Satellite image of Samakhiyali check post (Source : Google Earth)



Fig. 6. 2 : Samakhiyali check post

Following types of data are collected
1.Vehicular details like registration number, type of vehicle based on Axle.
2.Commodity related information i.e. name of commodity

3.Shipment characteristics (Loose, Packaged or Tanker)
4.Trip load (per trip)
5.Origin and destination of trip based on which distance is worked out.
6.Journey hours
Trips are divided into two categories i.e. Inward trips (trips towards Kandla) and Outward trips (trips going from Kandla). Details of data collection are given in the table below:

Table 1: Details of data collection

Date	Inward data	Outward data
05-07-2015	43 RTO	82 RTO+56 CST =138
11-07-2015	37 RTO	103 RTO+85 CST=188
Total	80 RTO	185 RTO+141 CST=326

7. RESULTS AND DISCUSSION

Category wise truck trip data is very useful for planning of highway facility and road networks. The data can be helpful at state and regional level planning and to researchers for study and modeling. Collected data is analyzed with respect to total number of truck trips per day, percentage share of trips from Kandla, average trip length, average time, average load, classification based on axle, packaging characteristics, commodity wise share of trips, state wise share of trip and distance wise trips. Each of these parameters is presented in terms of graph below :

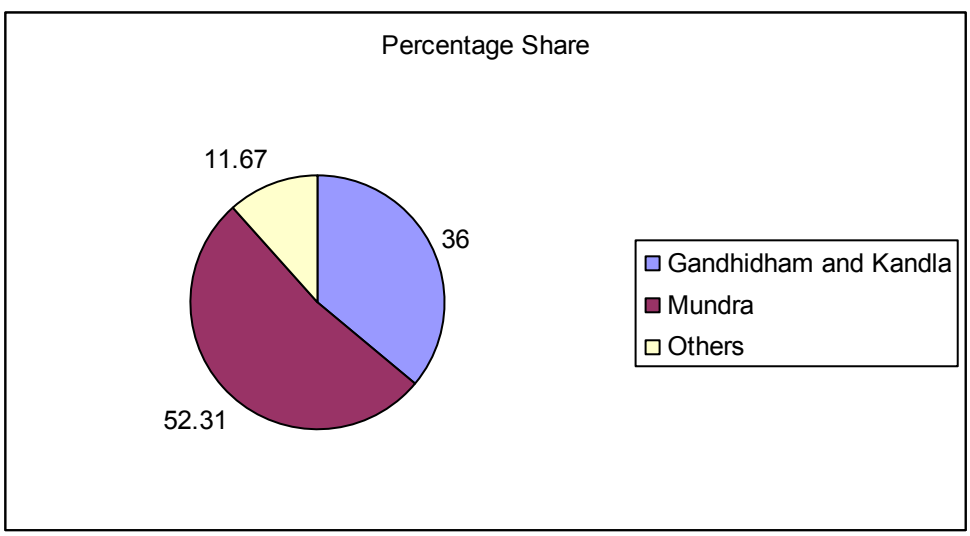


Fig. 7.1 : Percentage share of trips from Kandla

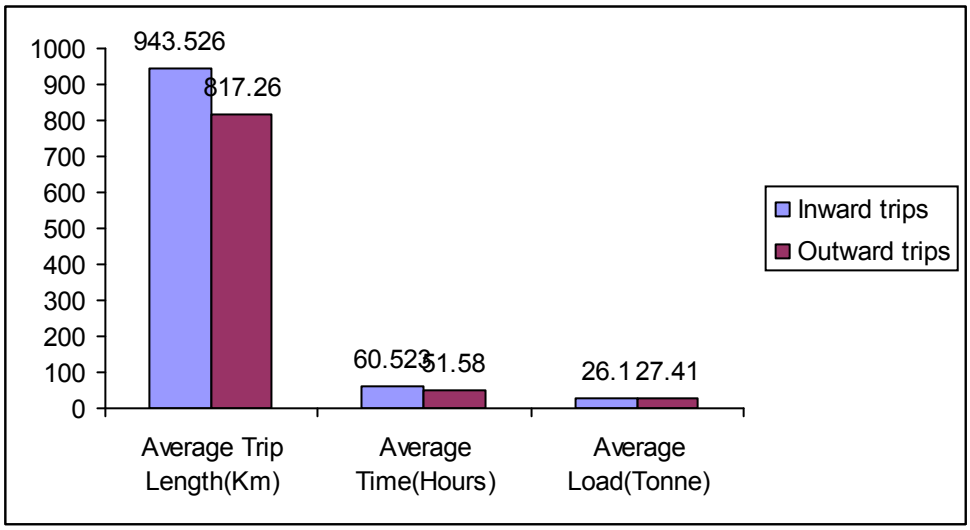


Fig. 7.2 : Average of trip length, travel time and load

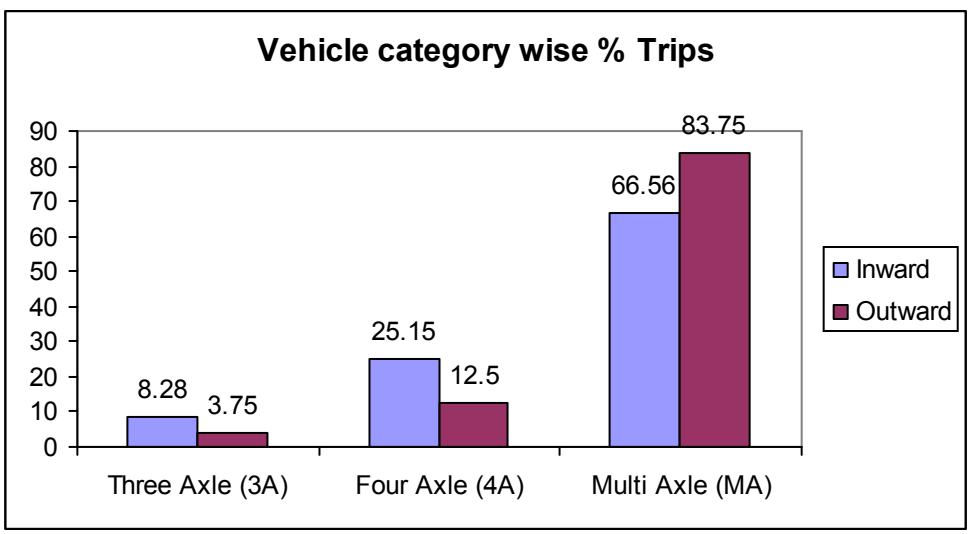


Fig. 7.3 : Classification based on vehicle (axle)

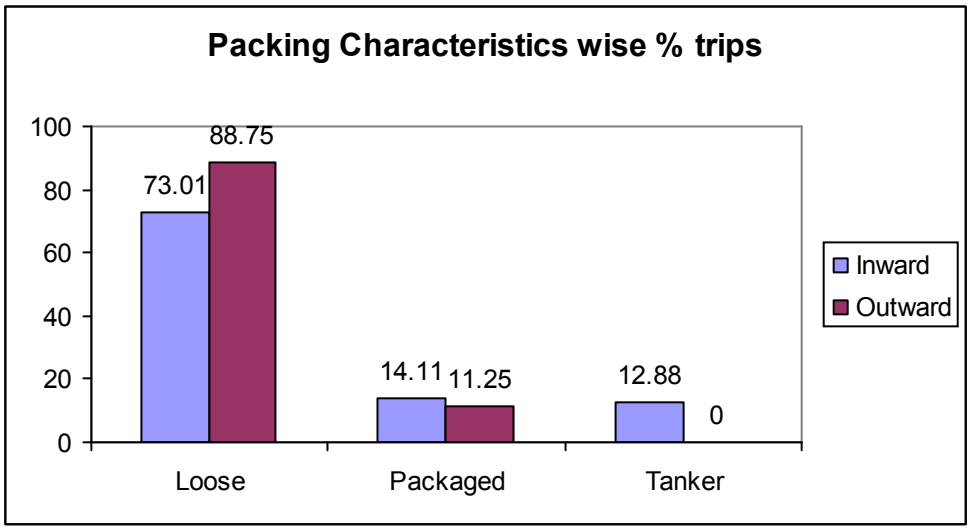


Fig. 7.4 : : Classification based on packaging characteristics of goods.

Table – 7.1 : State wise classification of trips

State	Percentage
Rajasthan	32.21
Hariyana	11.96
Punjab	11.96
Delhi	11.35
Gujarat	9.82
UP	7.98
Jammu &Kashmir	5.21
Maharashtra	4.29
MP	1.84
Himachal Pradesh	1.23
Karnataka	0.92
Odisa	0.92
Tamilnadu	0.31

Above data of tabular format is presented in terms of a Desire line diagram below:



Fig. 7.6 : Desire line diagram of trips

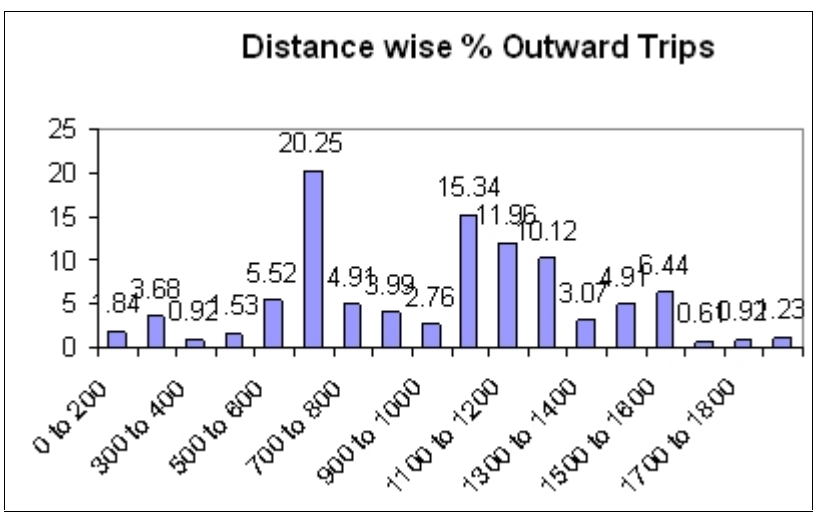


Fig. 7.7 : : Distance wise classification of trips (Outward)

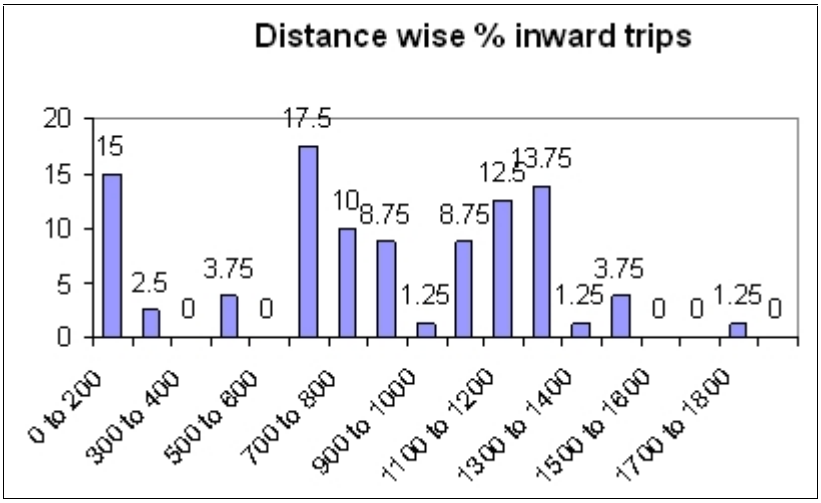


Fig. 7.8 : : Distance wise classification of trips (Inward)

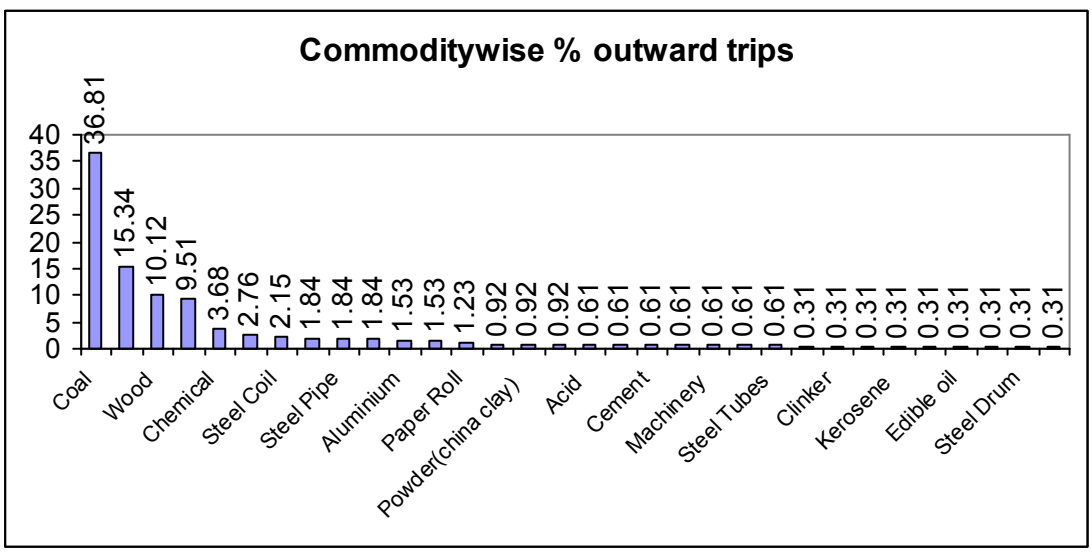


Fig. 7.9 : Commodity wise classification of Outward trips

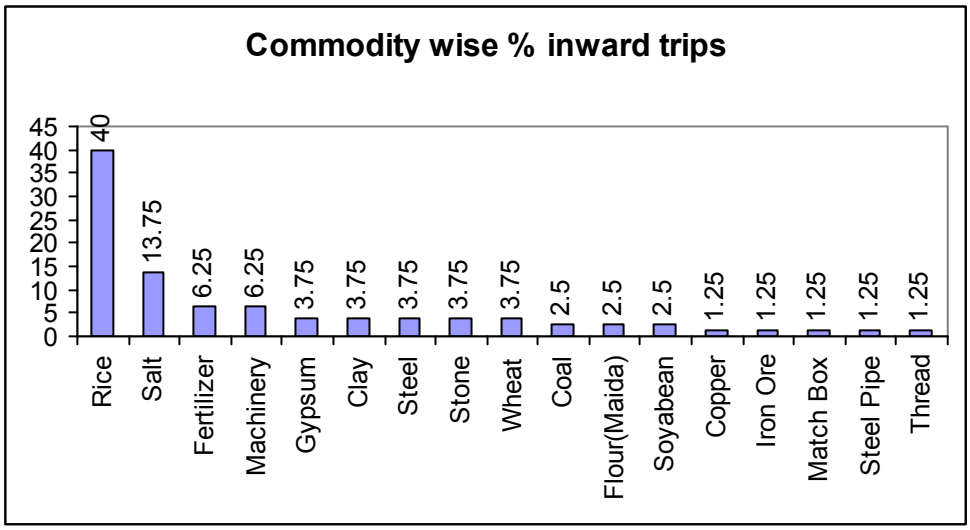


Fig. 7.10 : Commodity wise classification of Inward trips

8. CONCLUSION

Category analysis is one of the aspects of trip generation for transport modeling. In the present study, collected data is presented considering various parameters. For freight modeling yearly data is required i.e. before and after monsoon and dry season. The collected data before monsoon is presented here. The study is carried out in terms of two types of trips i.e. Inward trips(moving towards port) and Outward trips(moving from port).

- % share of outward trips is found significant. 80% of trips were found as inward trips and 20% outward trips.
- Hinterland of Kandla port is extended up to Jammu and Kashmir. It is observed an average trip length of 943.5 Km for inward trip and 817.2 for Outward trip with an average of travel time of 60.5 and 51.5 hours respectively for inward and outward trips.
- Average vehicle load is found at par with permissible limits of 26 tonnes.
- Share of multi axle vehicle is the highest followed by four and three axle.
- According to packaging characteristic, it is observed that mostly commodity is carried in Loose. (73% trips inward and 88% outward trips).
- State wise classification is shown in tabular format and in also in terms of Desire line diagram.
- Majority of the trips are from Rajasthan, followed by Hariyana, Delhi and Punjab which lies in range of 700 km to 1400 km.
- Commodity like Coal, Oil and wood is imported as their share is higher and Rice is exported during the period of data collection.

In present data collection, many constraints were faced like lack of cooperation from local agencies, permission related issues, data is not available in electronic form, security and confidentiality of data**.**

ACKNOWLEDGEMENT

The authors are thankful to the UG students of final year of civil engineering for their constant help in data collection and showing their willing to work as enumerator. Authors are also thankful to regulatory bodies (RTO and Commercial Sales Tax department) for giving permission to collect data at their check post Samakhiyali.

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ANNEXURE-I

ROADSIDE GOODS INTERVIEW SURVEY FORMAT

Sr. No.	Vehicle Reg. No.	Type of Vehicle 2Axle/ 3Axle/ 4Axle/ MultiAxle	Commodity carried	Gross Weight (Tonnage)	Origin	Destination	Distance (km)	Journey Time (Hrs.)
1	2	3	4	5	6	7	8	9

Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th& Jan. 6th, 2016)

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Parametric Optimization of Wire Electric Discharge Machining Process for Aluminum Metal Matrix Composite

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ABSTRACT

WEDM process is non-conventional machining method which uses spark erosion method for the material removal while machining using high current. This method has high dimensional accuracy but provides low machining rate. During this competitive age of modern manufacturing it is required that a machine should meet multiple purposes and hence the study for higher MRR and Surface finish with dimensional accuracy using this process has been very well presented in this paper. The paper describes about the mathematical modeling for the WEDM Process for the variables pulse ON/OFF time, current and bed speed. The paper also details about minimization of iterations with the use of optimization technique used and the convergence graphs have been plotted in Minitab software before and after the application of optimization technique.

SUMMARY

This research work describes about the application of optimization methodology for non-conventional processes like WEDM.

Keywords: MRR, Surface Roughness, Pulse ON/OFF time, Current Intensity, Bed Speed

INTRODUCTION

WEDM process is modified type of the Electric Discharge machining process in which the electrode used is the current carrying wire which can be feed according to the requirement of the process via wire feed system and that also maintains the tension in the wire by suitable arrangements. This process works on the principle of spark erosion due to high current flowing through the wire. This process is highly suitable for the hard to machine type materials like ceramics, composites, metal matrix composites etc. with complex and intricate shapes (*Garg et al. 2012*).These materials are advanced materials and can be alternatives for many present materials when concerned properties are light-weight, high strength to weight ratio, corrosion resistance, and high strength of materials required in the automobile, defense, aerospace, and other industries. It has industrial applications in recent past as high-technology materials due to their exceptional properties which includes better strength-to-weight ratio, high toughness and low value of coefficient of thermal expansion, light weight and can be operated at elevated temperatures without fail compared to conventional and present materials. Processed MMCs and composites can cause serious tool wear due to the presence of abrasive particles in the metal matrix in non-homogeneous state and hence reduces tool life when machined using conventional methods (*R. A. Kapgate et al. 2013*). Wire Electric Discharge Machining (WEDM) process is quite successful for machining of MMCs. WEDM seems to be a better choice due to easy control and it can machine intricate and complex shapes and profiles. The general WEDM process contains of a power source which may be inverter, transformer or generator which may be AC or DC type. Cutting tool may be a wire which is fed through wire feed mechanism. The actual process will require drilling of a hole in the work piece and the wire is made to pass through it which will then act as the tool for machining. The material is then removed from the cutting area by means of flushing of the dielectric fluid with dielectric feed system. The process parameters are pulse on/off time and voltage with wire feed system with wire tension and wire feed rate and flushing pressure of the dielectric fluid for the WEDM process. The detail of WEDM cutting process is shown in Figure 1.

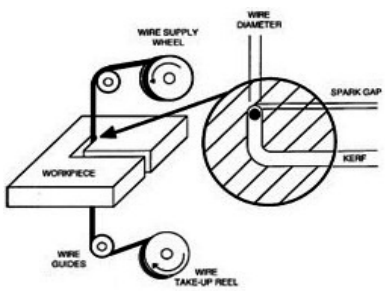
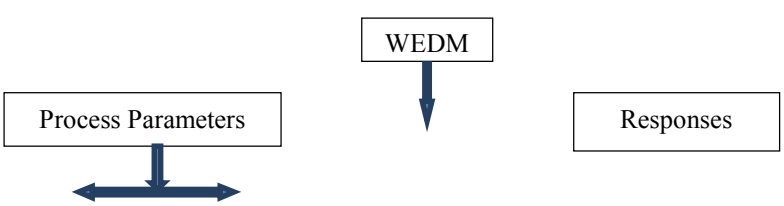


Fig. 1. Schematic view of WEDM Process

The responses from the process are considered as the surface roughness, dimensional accuracy with kerf characteristics, material removal rate, tool wear rate etc. These are thus required to be improved for the better machining as higher surface finish and dimensional accuracy are the desired requirements of some industrial and medical applications. In surgical instruments higher dimensional accuracy and surface finish is required. Figure 2 shows the schematic diagram of process parameters with responses.



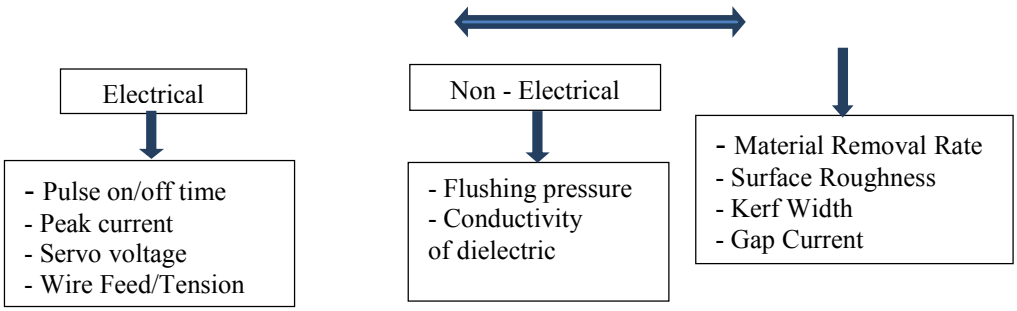


Fig. 2.WEDM Process Parameters and Responses

LITERATURE REVIEW

Some researchers already worked on WEDM process and which can be act as guideline for the further work. Garg et al. 2012 (*1)* have showed that the WEDM process gives better surface finish for intricate shapes. Lokeshwar Rao T. et al.(*2*) have performed experiments for the optimum set of parameters for Ti6Al4V alloy using WEDM process. They have considered that machining efficiency can be improved by Volume material removal rate and surface roughness improvement. The parameters considered for that are pulse on time, pulse off time, peak current, wire tension, servo voltage and servo feed. Thus these are the variables used for the study and we can consider those parameters B. H. Yan et al. (*3*) experimented on WEDM by using Al6061/Al2O3 of 20% by volume as work material to find out reason for wire breaking during machining and they found that, high flushing rate with higher wire speed are required for the prevention of wire breakage and that with providing sufficient wire tension will yield good surface finish. Jaganjeet Singh et al.(*4*) have used Minitab software for Design of Experiments and regression analysis has shown that Minitab software can be useful for the study. They have conducted DOE using Taguchi method using Taguchi's orthogonal arrays. From literature review it is obvious that the general procedure for any type of optimization follows the designing of number of experiments (*5*) required then conducting the experiments using the process and work material. The next step is to gather the data from the experiments by analyzing and then measuring the different responses. Next step is to form the mathematical relations or the model (*6, 7*) with best suitable and available mathematics equations with suitable methods and then validating that model (*8*). After that the final step arrives, which is of choosing a suitable method of optimization G. Ugrasena et al.(*9*) have worked with WEDM process considering Pulse on/off time, Current, Bed Speed as process parameters or variables and responses are Accuracy, VMRR and Surface roughness for their study. We have utilized their equation for the optimization purpose with the same range of variables as used by them for the mathematical modelling.

DESIGN OF EXPERIMENTS

Design of experiments are required to be carried out for the number of minimum experiments required to get the data and then that data can be used for the generation of mathematical model or relations between input parameters and output responses by many of the available methods.The design of experiments can be carried out using some software like Design expert, Minitab and sometimes by manually also. This software uses some techniques like full factorial method, partial factorial method, Response surface method, Taguchi method and sometimes mixture method for planningof Design of experiments and thusgives the table of experiments to be carried out with set of input variables which are required to be fed while planning the design of experiments.The full factorial design is based on the number of variable factors and the number of experiments will increase quite alarmingly as the variable increases. It uses the following formula for the Design of Experiments.

Number of Experiments = (Number of factors)$^{\text{Level of variables}}$

Consider Four variable three level design = $(4)^3$ = 64 Runs.

In partial factorial design the number of experiments is of a part of full factorial design as ½ factorial ¾ factorial etc. in the factorial form. Like half factorial design of above will yield as 32 Experiments. While in case of Response surface method the design is based on two other sub parts of design as central composite method and Box-Behnken method uses a three dimensional box and its surface as base for the design of experiments.Generally Taguchi method is used for Design of Experiments as it reduces the n umber of experiments without reducing the significance and mathematical relations thus formed. Taguchi's design of experiments are represented as orthogonal arrays like L8, L9, L16, L27 as numbered by the number of required experiments to be carried out. For 3-4 variables mostly used Design of Experiments are L16 andL27 orthogonal arrays. Thus we will use the experimental plans of L16 and L27 orthogonal arrays for design of experiments.

The Design of Experiment tables for the study are as below and the data is used from the G. Ugrasena et al. (*9*) for the comparison is shown in table 1.

Table 1. Taguchi's L16 Orthogonal array DOE and Experimental Data (*1*)

Run	Pulse - on (µs)	Pulse - off (µs)	Current (Amps)	Bed Speed (µm/s)	Surface Roughness (µm)	VMRR (mm^3/min)	Accuracy (µm)
1	16	4	3	20	2.39	5.52	12
2	16	6	4	25	2.38	5.50	11
3	16	8	5	30	2.53	5.36	9
4	16	10	6	35	2.79	7.22	20
5	20	4	4	30	2.63	6.80	19
6	20	6	3	35	2.54	6.24	18
7	20	8	6	20	2.84	7.35	21
8	20	10	5	25	2.89	7.44	23
9	24	4	5	35	3.32	9.73	28
10	24	6	6	30	3.28	8.63	24
11	24	8	3	25	2.48	6.06	13
12	24	10	4	20	2.25	5.20	11
13	28	4	6	25	3.02	8.59	26
14	28	6	5	20	2.59	6.38	17
15	28	8	4	35	2.48	6.09	15
16	28	10	3	30	2.35	5.38	10

MATHEMATICAL MODELING

The mathematical equations or mathematical model or relationship between input variables and output responses are required for the solution of those equations. These equations are made by using mathematical modelling techniques, statistical analysis and regression analysis whichever the best suitable is used and equations thus formed are validated and after that those equations are used for the optimization purpose. We have used regression analysis for the relationship between input variables and output responses.

B. Naga Raju et al. (*6*) have developed the RSM model for the Aluminium metal matrix composite study with following Limits to the parameters is shown in table 2.

Table 2. Parameters with limits

Parameters	Minimum	Maximum
T on	100	108
T off	45	55
IP (Peak Current)	10	12

The mathematical equations used for the optimization are as below.

Ra = -11.87 + 0.210 Ton + 0.0653 Toff - 0.164 IP - 0.000686 Ton*Ton + 0.000369 Toff*Toff + 0.0010 IP*IP -0.001111 Ton*Toff + 0.00259 Ton*IP - 0.00180 Toff*IP (1)

MRR = -19.05+ 0.3077 Ton + 0.1068 Toff + 0.382 IP - 0.001266 Ton*Ton+ 0.000242 Toff*Toff- 0.00501 IP*IP - 0.001056 Ton*Toff - 0.00266 Ton*IP + 0.000557 Toff*IP (2)

G. Ugrasena et al. (*9*) have experimented using different set of variables and get the results are used to develop the following mathematical model. The range of variables is as listed below in table 3.

Table 3. Range of variables

Variable	Minimum	Maximum
T on	16	28
T off	4	10
Current	3	06
Bed Speed	20	35

SR = 2.65 + 0.2122 p on - 0.01 p off + 0.1537 current + 0.0695 B speed - 0.299 p on*p off + 0.2987 p on*current - 0.0012 p on*B speed + 0.353 p off*current + 0.031 p off*B speed + 0.3821 current*B speed - 0.524 p on*p off*current + 0.2226 p on*p off*B speed + 0.9133 p on*current*B speed - 0.6085 p off*current*B speed - 0.5938 p on*p off*current*B speed (3)

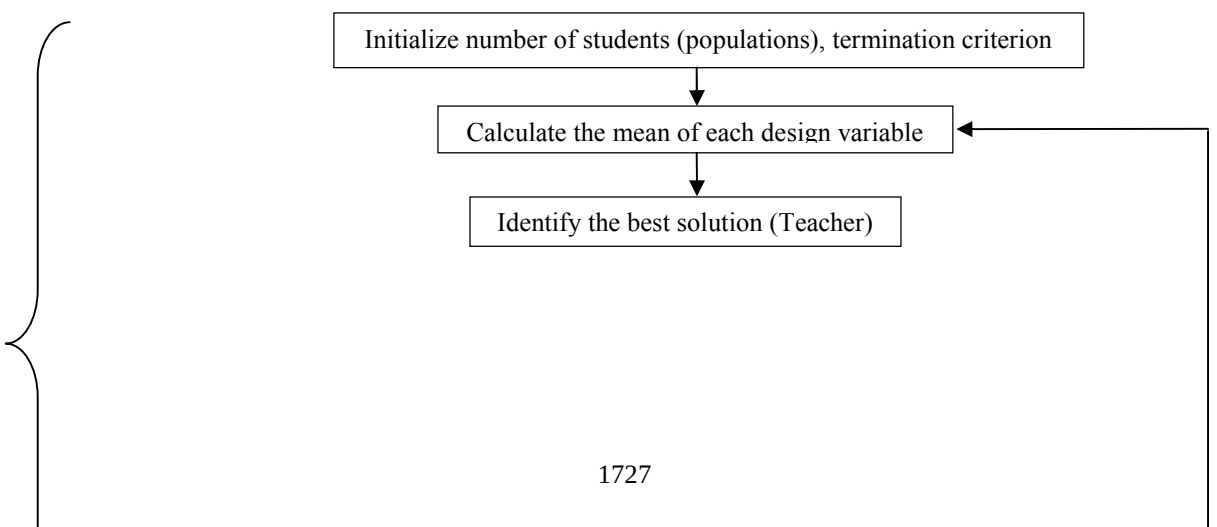
VMRR = 6.414 + 1.099 p on - 0.423 p off + 0.223 current - 0.148 Bed speed - 1.716 p on* p off + 1.122 p on*current + 0.586 p on*Bed speed + 1.707 p off*current – 0.462 p off*Bed speed + 1.437 current*Bed speed -2.551 p on*p off*current + 0.974 p on*p off*Bed speed + 3.204 p on*current*Bed speed – 2.006 p off*current*Bed speed -3.194 p on*p off*current*Bed speed (4)

Accuracy= 15.18 + 5.93 p on - 0.83 p off + 0.58 current - 1.02 Bed speed - 7.56 p on*p off + 7.26 p on*current + 2.90 p on*B speed + 12.33 p off*current - 3.94 p off*Bed speed + 6.41 current*B speed - 14.58 p on*p off*current + 8.46 p on*p off*Bed speed + 14.17 p on*current*Bed speed - 7.49 p off*current*Bed speed - 15.36 p on*p off*current*Bed speed (5)

Now we will use TLBO for the parametric optimization as it is recently developed and improved method for the optimization. It is specifically used for this study due to its applications for optimization and parametric study of non-conventional machining processes R. V. Rao et al. (*10, 11, and 12*).

TEACHING LEARNING BASED OPTIMIZATION TECHNIQUE

Optimization means to obtain the desired results with maximum use of the available variables in such a way that it will not compromise the significance of other variable. This can be achieved by various methods and techniques in engineering field. Some of them are Taguchi method, Artificial Bee Colony, Particle Sworm Optimization, Artificial Neural Network, Genetic Algorithm and Teaching Learning Based Optimization. TLBO is newly emerged optimization technique and that can be used to solve many engineering optimization problems and processes. TLBO is based on the real life classroom teaching process in which there are students and teacher. Similarly TLBO also has teacher phase and learner or student phase as shown in below algorithm. Figure 3 shows the flow chart of TLBO.



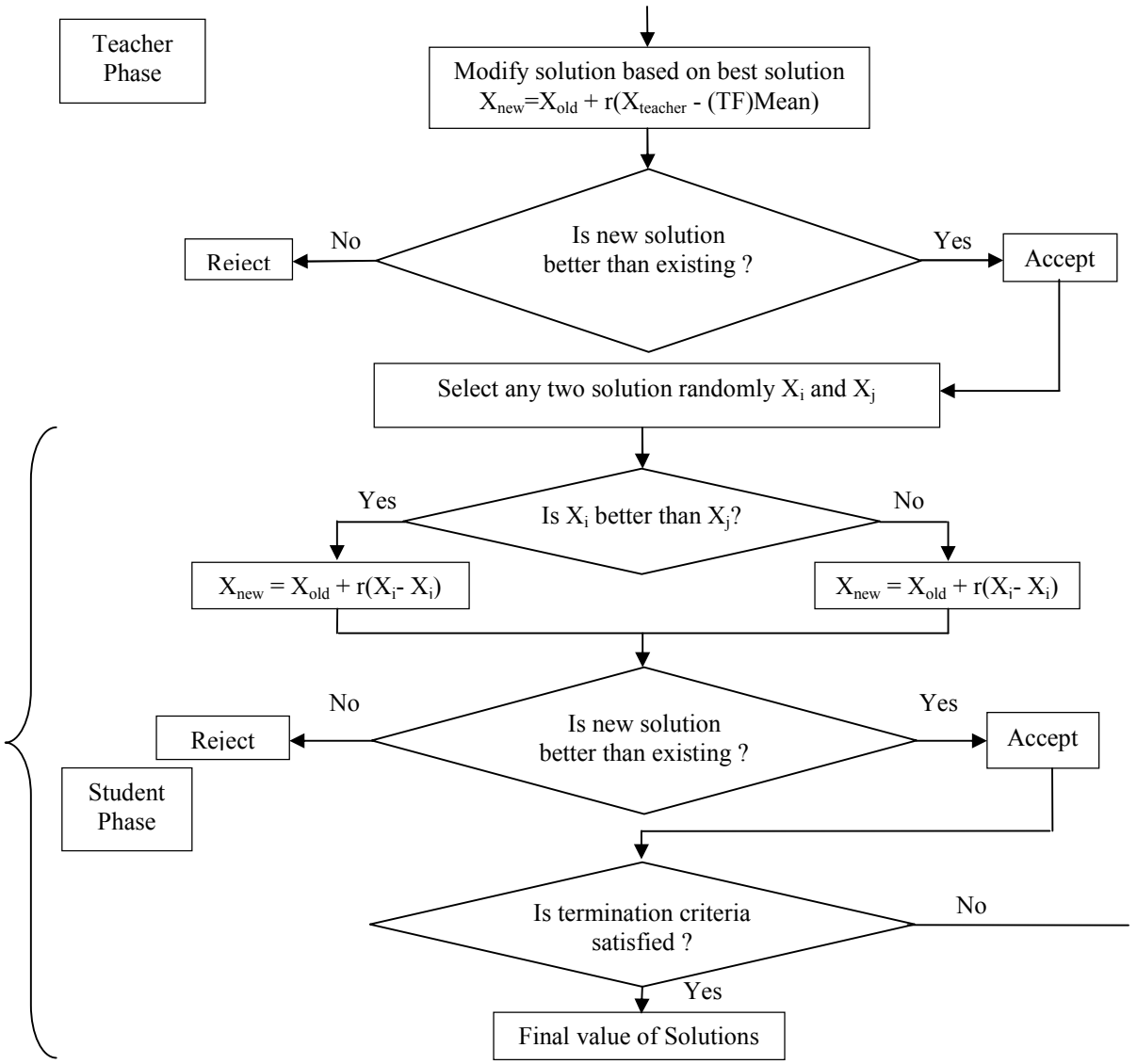


Fig. 3. Flow chart showing the working of TLBO algorithm

The process of working of TLBO is divided into two parts. The first part consists of Teacher Phase and the second part consists of Learner Phase.

- **Teacher Phase:**

In this phase the teacher tries to transfer the knowledge to the students as teacher has high knowledge and the teacher can bring the average knowledge of the classroom up to teacher's knowledge level. But teacher cannot raise the knowledge of each student as some of students are slow learners. Thus the average result is improved in TLBO. Let M_i be the mean and T_i be the teacher for i^{th} iteration then new solution with improved M_N will be given by the difference between the old and new mean given by following equation.

$$\text{Difference Mean}_i = r_i*(M_N - T_F*M_i) \quad (6)$$

where T_F is teaching factor and r_i is random number between (0,1)

The difference changes existing solution as following:
$X_{Ni} = X_{Oi}$ + Difference $mean_i$(7)

- **Learner Phase:**

In this phase the interaction between the students takes place and the weak students will learn from students having the higher knowledge and thus the knowledge transfer takes place at the student level and mean knowledge level of classroom increase. Here the interaction may be between two random students and the knowledge transfer takes place as following criterion.

For i = 1: PN
Randomly select two learners X_i and X_j, where i / = j
If f (X_i) < f (X_j)
$X_{Ni} = X_{Oi} + r_i (X_i - X_j)$ (8)
Else
$X_{Ni} = X_{Oi} + r_i (X_j - X_i)$ (9)

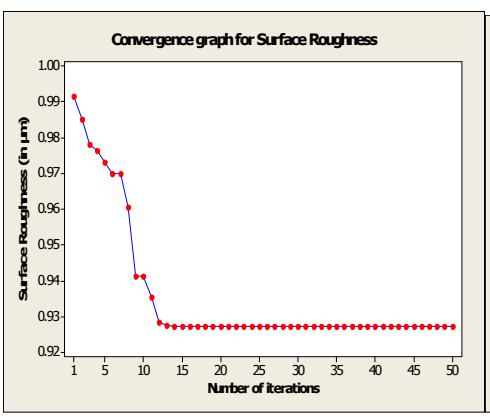
The following equation is obtained from the research work of B. Naga Raju et al. (*6*). They have developed the equation using the Response Surface Method for mathematical relations.

Ra = -11.87 + 0.210 Ton + 0.0653 Toff - 0.164 IP - 0.000686 Ton*Ton + 0.000369 Toff*Toff + 0.0010 IP*IP -0.001111 Ton*Toff + 0.00259 Ton*IP - 0.00180 Toff*IP (A)

The following equation is obtained by using data from G. Ugrasena et al. (*9*) and is developed by Minitab software. The experimental data is fed to the software and then regression analysis is carried out. Simple regression analysis doesn't give significant RSquare value so the regression analysis is carried out using Factorial fit analysis which gives significant relations between the variables and thus developed equation is used for the optimization as below.

SR = 2.65 + 0.2122 p on - 0.01 p off + 0.1537 current + 0.0695 B speed - 0.299 p on*p off + 0.2987 p on*current - 0.0012 p on*B speed + 0.353 p off*current + 0.031 p off*B speed + 0.3821 current*B speed - 0.524 p on*p off*current + 0.2226 p on*p off*B speed + 0.9133 p on*current*B speed - 0.6085 p off*current*B speed - 0.5938 p on*p off*current*B speed (B)

After applying TLBO for optimization below convergence graphs are obtained for the Surface roughness as in equations (A) and (B) as shown below in Figure 4 & Figure 5.



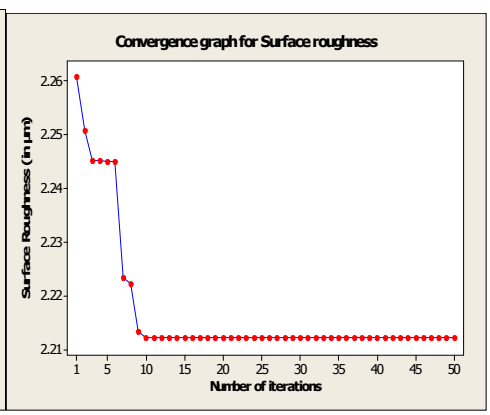


Fig. 4. Graph for SR using equation (A) **Fig. 5.**Graph for SR using equation (B)

CONCLUSIONS

- The significant parameters for the WEDM process are pulse on/off time, Wire tension, peak current, servo voltage, servo feed. Out of all those only four or five parameters are considered for the study at a time and thus are highly significant parameters which are pulse on time, servo voltage, servo feed, wire tension and peak current.
- We have formed the model for the factorial fit analysis and then used the relations and interactions for the generation of equations for the responses and got improved results in each of the three cases. The VMRR is found to be7.42 mm^3/min and corresponding Surface roughness and accuracy is found as 2.21 µm and 23.41 µm respectively and are better compared to 2.3µm and 23 µm.
- We have used the mathematical model formed by the B. Naga Raju et al using RSM and the results for the MRR and Surface roughness are found to be 0.91 mm^3/min and 0.927µm respectively. And are better compared to 0.879 mm^3/min and 0.964µm using Minitab response optimizer tool obtained by B. Naga Raju et al.
- From the literature review and above results its quite clear that TLBO is modern method for the optimization and will give better results than almost methods and can be explored further for the different machining and other processes. TLBO is highly suited to the multi-objective functions and thus can be utilized for that also.
- From the graphs plotted it's clear that TLBO will give better results at lower number of iterations and thus is quick process of optimization compared to other time consuming processes conventionally utilized for the optimization purpose.

ACKNOWLEDGEMENT

I express my deep sense of gratitude to my guide Dr.A.V.Gohil, Associate Professor & Head, Shantilal Shah Engineering College at Bhavnagar for providing valuable guidance for the development of this research work.

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Real-time moving object detection using a hybrid approach

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ABSTRACT

Object detection plays an important role in advanced video analysis. This paper introduces basis of some approaches for object tracking and detection. Image features and motion can be more useful to track any real time moving object. There are many application areas where we can use object detection like, Human-computer interaction, Medical imaging, Traffic control, Robot vision, Vehicle navigation, Animation etc. For moving object detection many methods are used like Background subtraction, Temporal differencing, Gaussian mixture model, Spatio-temporal smoothing transform, Optical flow and many more. By combining the advantages of Gaussian mixture model and Optical Flow, object detection can be done with Hybrid approach to get complete object tracking in quick time.

SUMMARY

We are going to detect real time moving object using hybrid approach by combining Gaussian Mixture model and Optical Flow method.

Keywords: Moving object detection, Gaussian Mixture model, Optical Flow, Motion detection, Digital image processing

INTRODUCTION

In current era, many investigations and analysis have been performed in object detection.Identifying an object in image or video frames is known as object detection. (XIII). Object detection is used to identify interesting object from video. Background complexity, noise, and illumination variations are the factors, due to it is also used in many fields for monitoring and security purpose. Strategies or Methods for detection of movingobject must be accurate and robust so that complex video systems can operate successfully. Moving object detection handles separation of moving object and background. In moving object detection, movement of human is the important part of human detection and motion analysis. Object detection is performed to check existence of objects in video frames (II). After detecting moving object, it can be classified in various categories like humans, vehicles and moving objects. Now a days, object detection for moving vehicles are nothing but the temporal difference between two consecutive frames (III). In computer vision, an object tracking problem is viewed as a two-part problem: motion prediction and object matching problem (IV). Many different strategies have been developed to solve object matching problem like, extracting features, calculating optical flows, using rigid shapes, and matching deformable contours (IV). There are many approaches which have been proposed for object detection like, feature-based object detection, template-based object detection and background subtraction or inter-frame difference-based detection (V). A very initial step in any of the tracking process is to detect an initial instance of moving object and then identifying that image object repeatedly in consecutive frames (VI).Object detection and object classifications are the two steps for detection process. Background subtraction, optical flow and spatio-temporal filtering can be used to perform object detection (XIV).
Object classification is divided into three categories like, shape based, motion based and texture based. In shape based approach, it deals with points, boxes and blobs of moving objects.

OBJECT DETECTION AND TRACKING

To establish object parts between sequential frames of video, we need to detect an object. For object tracking two most common approaches are with us one relates to correspondence matching and other one carries out explicit tracking with the help ofmotion estimation or position prediction (VI). Point detectors, segmentation and background subtraction are the methods of object detection. Object tracking is used to generate a temporary path followed by object which is moving over time by locating its position in each and every frame of image and video [VIII].

APPLICATIONS OF MOVING OBJECT DETECTION

Human-computer interaction: It includes human gesture recognition and eye gaze tracking for data input to computers (VIII).
Vehicle navigation: It is used for Video-based path planning and it provides obstacle avoidance capabilities with the help of object detection (VIII).
Medical-Imaging: Detect & identify tumor or other affected area in human body (VIII).
Automated surveillance: It includes monitoring a scene to identify and detect unlikely events in shopping malls, offices, banks, railway-stations, metro stations, border-security, corridor, fields or forests etc. (VIII).
Traffic monitoring: It is used for real-time gathering of traffic statistics to direct traffic flow (VIII).
Robot-Vision: It involves monitoring and handling robot activities (VIII).
Animation: Detect and track the actions of animated object (VIII).

After identifying the object of interest, we need to cluster pixels of these object (VII). For moving object detection many approaches are used like, background subtraction, temporal differencing, frame differencing, Gaussian mixture model and optical flow. Detailed explanations for various methods are given below.

Background subtraction

First step for background subtraction is background modelling (VIII). In background subtraction, it needs to subtract gray level value or foreground object of the current frame from the corresponding background (VIII). In this process, if corresponding pixels gray-level value is lower than the thresholding, the object is considered static, otherwise it can be considered as dynamic or moving. This technique uses reference model, each video frame is compared with this model to identify possible variation (VII). Background subtraction uses mean filter and median filter to realize background modelling (VII). This process contains two steps like, background and update model (IX).

Frame differencing

It is also known as adjacent frame difference method. It relates to a very small time intervals of two images before and after the pixel based on the difference (XII). It uses different methods to calculate difference between image frames like, differential, and the negative differential and fully differential (XII). In actual, it just checks the difference between two video frames (XI).

Gaussian mixture model

Most of the tacking system focused on single class of targets (I). Gaussian mixture model provides high level of performance. It uses Gaussian probability density function (IX). Each pixels function has its own mean, standard deviation and weight. Weights can be calculated from the corresponding Gaussian model of the frequency. If the higher frequency is there then, we need to find the maximum weight on the function (IX). The value of the function for means pixel is background image. It gives complete result but incomplete object tracking. It takes very long computation time and gives more noise. It also uses morphological and median filtering to avoid noise.

Sptio temporal filter

The action and motion is identified through the entire 3D spatio-temporal data volume consumed by the moving person in the image sequence. This method considers the motion as a whole (XIV). On the temporal axis, due to derivative operation, this filter shows the high responses at motion regions. This method is fast and very easy to implement because it uses simple convolution operations. This method is able to capture spatial and temporal information of motion (XIV). Advantages of this method is easy implementation and low computational complexity.

Optical flow

This technique calculates motion between two sequential image frames which are taken at consecutive times at every position (IX). It works on velocity of an object and calculates velocity for each pixel and that is why it is known as vector based approach. By considering brightness and smoothness, it is used to describe some features between image frames. This method is used to subtract successive images (IX). It gives quick calculations. It doesn't give complete object tracking. It takes very long computing time and

provides more noise. It is robust to simultaneous cameras. To implement optical flow, it requires a specialized hardware due to its complexity of an algorithm (XIV).

Temporal differencing

It calculates difference between two consecutive frames and then it compares those two images to find the moving object (X). It includes three sub modules like, block alarm module, background modelling module and object extraction module (XIV).

Flow chart for moving object detection using temporal differencing (X)

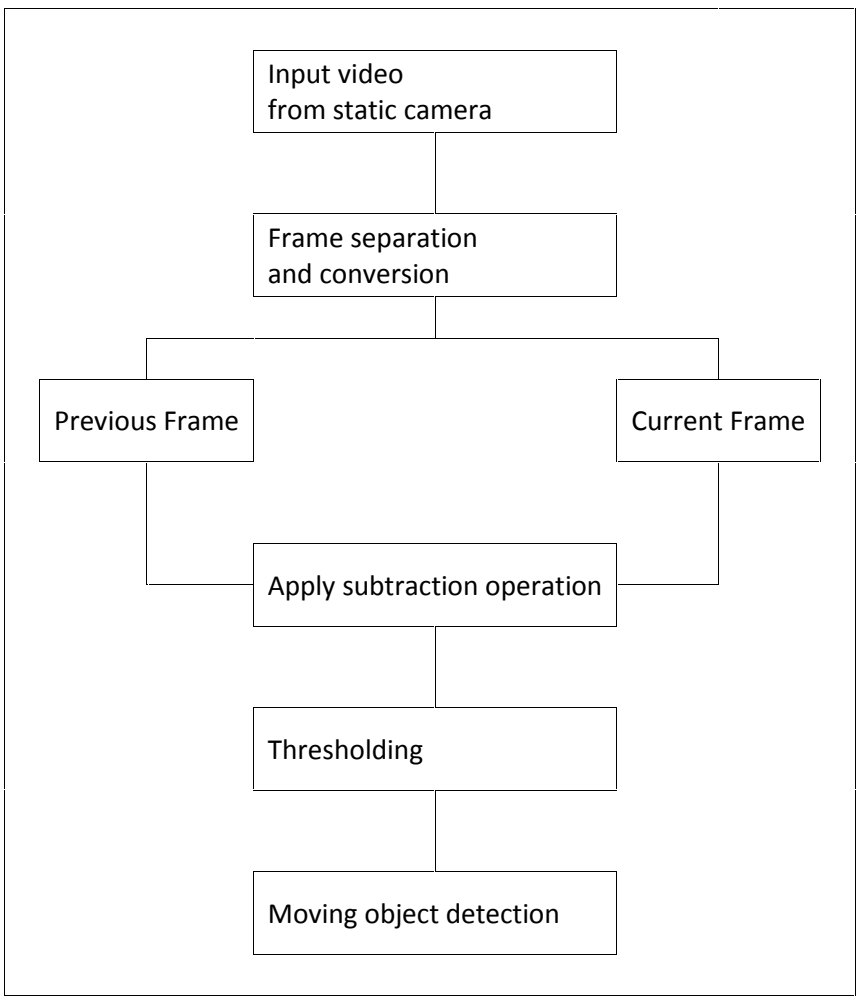


Fig – 1. Moving object detection using temporal differencing.

In this technique, it subtracts previous frames from the current frame. If it founds pixel difference greater than the threshold value then it gives pixels of moving object else it gives pixels of background.

COMPARATIVE STUDY OF DIFFERENT APPROACHES

Methods	Advantages	Disadvantages
Background subtraction	- Simple and easy to realize. - More accurate. - Provides fast recovery. - Low memory requirement.	- For known background only, it gives more applicable result. - Can't deal with quick changes. - It gives false positives.
Optical Flow	- Gives good performance under moving camera. - Produces complete object tracking.	- Very complex, - Computation takes much time.
Frame differencing	- Provides better result for static background.	- It needs static background.
Temporal differencing	- Simple and easy to implement. - Very adaptive to dynamic scene changes	- Doesn't detect whole relevant pixels. - Required specialized hardware.
Gaussian mixture model	- Requires less memory. - Gives good result in quick time.	- Doesn't work with multimodal background.

PROPOSED WORK

As we have discussed many methods to detect and track real time moving object. And also, we have seen the comparative analysis of each and every methods with its advantages and disadvantages. In few of the methods, complete object tracking is not there. In other methods, complete object tracking is there but it takes too long computational time. By seeing this kind of merits and demerits, we can do some innovative things like, we can combine any of the two or more methods and can club advantages of both. By observing the above analysis and by referring many journals and papers, our aim is to combine Gaussian mixture model and Optical Flow to get object detection and to track the object. Gaussian probability density function will play an important role in this hybrid approach as, it is having three components like, weights, mean and standard deviation.

CONCLUSION

Moving object detection is highly effective in many applications. Various approaches have been explained and compared. Each and every approach has its merits and demerits. By analyzing all the approaches, we came to know that background subtraction is easy to implement with less calculation. After detecting an object, it needs to be classified using classification techniques. All the methods of object detection, object classification and object tracking are explained with comparative analysis. We may combine one or more methods to get more fruitful result with very less amount of noise and to get complete object tracking. Gaussian mixture model and Optical Flow methods are the best matching combination for object detection.

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14. International Journal of Electrical, Robotics, Electronics and Communications Engineering VOLUME-7 ISSUE-9, 2013 "Optical Flow Based Moving Object Detection and Tracking for traffic surveillance Sepeher aslani, Homayoun Mahdavi Nsab

Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th& Jan. 6th, 2016)

(Proceedings available for download at rku.ac.in/icre)

RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

Securing AODV Reactive Routing against External Attacker using Link Encryption in MANET

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ABSTRACT

AsMANET is self organized, it is vulnerable from internal and external attacks and may cause the security risks during route discovery in the form of active and passive attack. In this paper, we proposed a scheme to secure the reactive route discovery in AODV routing protocol from AODV flooding attack, eavesdropping attack, sinkhole attack, grayhole and blackhole (packet dropper) attack, disclosing the routing information to external attacker. We used the concept of link encryption to secure routing process which is implemented using Blowfish symmetric encryption algorithm and the secret sharing key used in Blowfish is exchanged using steganography during neighbor discovery. The result shows that the proposed scheme can protect AODV message of route discovery from external attacker while maintaing confidentiality, authentication (node to node) and availability without use of trusted third party (TTP).

SUMMARY

The reactive AODV routing protocol is attempt to secure using link encryption against external attacker for MANET without use of TTP.

Keywords: Blowfish, Secret sharing key, Steganography, External attacker, AODV, Link encryption,Symmetric encryption

INTRODUCTION

MANET is a self organized network that form temporary wireless network with mobile nodes without infrastructure. In such network, all mobile nodes work as a router and host (*1*) in which the information deliver from one node to another node. MANET has unique characteristics such as dynamic topology, infrastructure less network, resource constrained and untrusted mobile nodes, low level security, variability of the radio channel, multi-hop communications, limited bandwidth, shared wireless medium, node mobility,multi-hop routes etc. Due to the nature of MANET, it is vulnerable and may cause the security risks (*2,3*) from internal and external attack (*4*) in the form of active and passive attack (*5*). In MANET, there are two types of data: control information and actual information. Control information is used for route discovery and actual information is an actual data being transferred from original source node to its final destination node via discovered route. Hence, security is required during route discovery and/or during the transmission of actual information in MANET.

In this paper, we proposed a scheme to secure the reactive route discovery process in AODV routing protocol from external attacker with confidentiality, authentication (node to node) and availability without use of TTP (Trusted Third Party). We used the Blowfish symmetric encryption algorithm for link encryption. The secret sharing key of Blowfish is exchanged with steganography at the time of neighbor discovery process. The proposed scheme is implemented and simulated in NS2.34 environment with modified AODV reactive routing protocol to provide the protection against AODV flooding attack, eavesdropping attack, sinkhole attack, grayhole and blackhole (packet dropper) attack, disclosing the routing information to external attacker.

The rest of paper is organized as follows: Section II discusses theoretical background for proposed scheme. Section III briefs the related work for securing the AODV routing protocol. Section IV gives the development and operation of proposed scheme. Section V describes the simulation and test results of proposed scheme. Section VI provides the security investigation of attacks and finally concluded this paper in last section.

BACKGROUND THEORY

Place of Encryption:Cryptography is one of the field which performs the encryption and decryption operations with various cryptographic based algorithms. Encryption is the process in which the meaningful information converted into coding form in such a way that only authorised parties can read it. It provides the prevention from leakage/steeling information. Decryption is the reverse of encryption process. The cryptographic algorithms are either symmetric or asymmetric (*6*). Asymmetric cryptography is costly and difficult in MANET because it required trusted third party or key management. Symmetric cryptography is less resource consuming compare to asymmetric (*7, 8*). Further, there are two places where the encryption/decryption process can be performed: link encryption (link to link) and end to end encryption (*9*). In link encryption, the encryption/decryption operation is performed at every node (links) while in end to end encryption, the origin node performs the encryption and destination node only performs the decryption operation.

Steganography: Steganography is an art in which secret information is embedded into digital media like image, text, video, packet, audio etc. (*10-13*). Embedded information is not readable by a third party until the place of hidden information is known. Because of this, the main advantage of steganography is that the intended secret information does not attract attention to itself as an object of examination.

Ad hoc On-demand Distance Vector:AODV (Ad hoc On-demand Distance Vector) (*14*) is a reactive routing protocol that discovers the routing information by AODV query and AODV reply. AODV uses four control messages for AODV query and AODV reply. The control messages are RREQ (Routing REQuest): message broadcasted to other nodes for finding a route to destination, RREP (Routing REPly): response message received from node, RERR (Routing ERRor): message for link failure notification and HELLO: message for the links evaluation and detection. The route request is flooded through the network for route discovery in AODV. All intermediate nodes forward route requests and create reverse route for destination node. The destination node will generate a route reply which is unicast back to the source (*15*, *16*). In this process, the node only knows its next hops towards the source or destination of active paths. This protocol works under the assumption that all nodes in the network are friendly and cooperative (*17*).

Blowfish:Blowfish is a secret key varying block cipher. The key length can be varying from 32 bits to 448 bits and 64 bits fixed block size. Blowfish has two modules: one for data encryption/decryption that performs the sixteen rounds based on Feistel network for encryption/decryption process and second for key expansion that converts key bits into several subkey arrays. In Blowfish, all subkeys must be initialize first before to perform any data encryption /decryption operation. It consist eighteen 32 bits P subkey array and four 32 bits S boxes with 256 entries of each. Each Blowfish round is a key & data dependent substitution and a key dependent permutation. Decryption process of Blowfish is exactly the same as encryption only subkey array P_1, P_2 ..... P_{18} are used in reverse order (*18*).

Subkeys Generation: Initialize the P array and S boxes. XOR P array with the key bits, example: P_1 XOR (first 32 bits of key), P_2 XOR (second 32 bits of key). Encrypt 64 bit all zero block with the blowfish algorithm and resulted 64 bits cipher text is P_1 (32bits) + P_2 (32bits) = 64bits. Replace P_1 and P_2 with the output.Encrypt the output of all zero block with the modified subkeys and replace P_3 and P_4 with the output.This output is now P_3 and P_4. Repeat above in order to calculate new subkeys for the P-array (P_1 to P_{18}) and the four S-boxes (S_1 to S_4)

Blowfish Data encryption algorithm:

Step1. Input: X=64 bit data
Step2. Divide X into two 32 bit Divide X into two halves say XL and XR
Step3. XL = XL XOR P_i (i=1to16)
Divide XL into four eight bit quarters say a, b, c, and d
$F(XL) = ((S_{1,a} + S_{2,b} \bmod 2^{32}) \text{ XOR } S_{3,c}) + S_{4,d} \bmod 2^{32}$
XR = [Round Function F(XL)] XOR XR
Swap XL and XR
Step 4. Repeat step 3, 16 times.
Step 5. Swap XL and XR
Step 6. XR = XR XOR P_{17}
Step 7. XL = XL XOR P_{18}
Step 8. Combine XL and XR

RELATED WORK

In paper (*19*) preformed analysis to find the vulnerability against network layer routing protocol attacks of AODV and Secure AODV (SAODV) and proposed the security scheme called Robust SAODV (R-SAODV). RSAODV incorporated time stamping of SAODV. The time stamping addressed the replay attacks by using the freshness of the exchanged control messages. They proved that the new proposed scheme gives similar performance as SAODV. In (*20*), they proposed a trust based routing scheme for

securing the ADOV in MANET which referred as FrAODV. FrADOV provides robust environment and it is simulated with NS2 and real test bed JADHOC. They used some features to evaluate the trust for friendship. Hence friendship based algorithm used to evaluate the AODV nodes trust. In (*21*), they presented the survey on network layer attacks and existing schemes for defending AODV in MANET against the network layer attacks. In (*22*) introduced the novel approach to secure the AODV from packet drop attack blackhole. They detected the attack based on the available neighbor's information. The scheme is validate route reply with propagation of forged information in the network. The scheme is analysed in NS2 that shows that it can detect the black hole attack with slight delay on the network. In paper (*23*) presented trust based scheme for defending the DOS and blackhole attacks for AODV routing protocol. They used local and global trust along with security mechanism. They proved that the proposed scheme gives good performance in terms of end to end delay, routing overhead and packet delivery ratio. In (*24*), they developed Secure Efficient Ad-hoc Routing (SEAR) protocol for MANET. It is used symmetric cryptography and asymmetric cryptography. Asymmetric cryptography used for initial key. They proved that SEAR gives significant security with very less overhead compare to other existing secure AODV protocols. In (*25*) proposed an approach based on the Hash Compression Function (HCF) for securing AODV against wormhole attack in MANET. They used HCF to compute hash value for route request message. They proved that the proposed approach is promising solution to other existing solutions. However the proposed scheme improves the security of AODV but down the performance of AODV due to computing the hash value at every node. In paper (*26*) proposed RBDR (Rank Based Data Routing) scheme for detecting the malicious path for packet drop attack: blackhole and grayhole. They used AOMDV multipath routing protocol for detecting malicious path and avoid the path for data delivery. The scheme used the multipath disjoint routing protocol AOMDV. Due to AOMDV the packet delivery ratio improved in the presence of packet drop attack. In (*27*) proposed the technique for the detection of the blackhole/malicious node in AODV routing protocol. They generated a new request which is a kind of trap process for detecting the blackhole attack. When the blackhole attack detected, the scheme generate an alert using alarming process to aware the other nodes.

OUR CONTRIBUTION

To secure the AODV routing protocol, we used the Blowfish symmetric block cipher for encrypting the header fields of AODV. In MANET, at the time of route discovery every intermediate participated node must able to read and handle the AODV message. All AODV routing control packets are encrypted using Blowfish block cipher. If nodes wish to participate in the network, it must know the key called link secret key for Blowfish cipher. This link secret key is use for link encryption. Link encryption is the technique in which encryption/decryption takes place between nodes that prevents from external attacks. For secret sharing key exchange, we used our proposed work (*28*) in which link key is shared among the discovered neighbor nodes using steganography.

Secret Sharing Key Exchange using Steganography

As shown in figure 1 (*28*), node 1 broadcast one hop control message (Hello Packet) to discover its neighbor nodes. The neighbor nodes 2, 3, 4 and 5 received the broadcasted message from node 1. After that neighbor node 2, 3, 4 and 5 unicast one hop message to node 1 with hidden link key. When node 1 receives the one hop unicast message from neighbors 2, 3, 4 and 5, it take out the hidden secret link key from received control message. In this case, broadcaster node 1 received four unicast messages one from each neighbor and extracts the four different hidden key from received unicast messages such as shared secret key between node 1 and node 2, a shared secret key between node 1 and 3, a shared secret key between node 1 and 4, and a shared secret key between node 1 and 5.

Securing AODV Request and Reply Messages

At the time of node discovery, the link key exchanged with neighbor nodes. Hence all nodes have their link key for Blowfish algorithm. Every node encrypts the header fields of AODV route request message before flooding the request. When the intermediate node receives the route request, only authenticated node i.e. participated into the process of link key exchange can decrypt it. Therefore the other intermediate nodes such as external node or outside of network nodes cannot decrypt it. As shown in figure 2, there are total six nodes labelled with 0, 1, 2, 3, 4 and 5. Considered the node 0 wishes to communicate node 5. So, node 0 is referred as a source node and node 5 is referred as a destination node. Initially, all the nodes have shared the secret sharing key during the neighbor discovery phase as listed in table 1. Node 0 has two secret sharing keys: one is use to communicate node 1 and second key is used to communicate node 3. Same way all the nodes have different secret sharing key as many neighbors. Note that the secret sharing key to communicate from node 0 to node 1 and node 1 to node 0 are distinct which will enhance the security level.

Table 1. Secret sharing keys

Neighbor Nodes	Secret Key
0' Neighbor 1	0→1
0' Neighbor 3	0→3
1' Neighbor 0	1→0
1' Neighbor 2	1→2
2' Neighbor 1	2→1
2'Neighbor 5	2→5
3'Neighbor 0	3→0
3'Neighbor 4	3→4
4'Neighbor 3	4→3
4'Neighbor 5	4→5
5'Neighbor 4	5→4
5'Neighbor 2	5→2

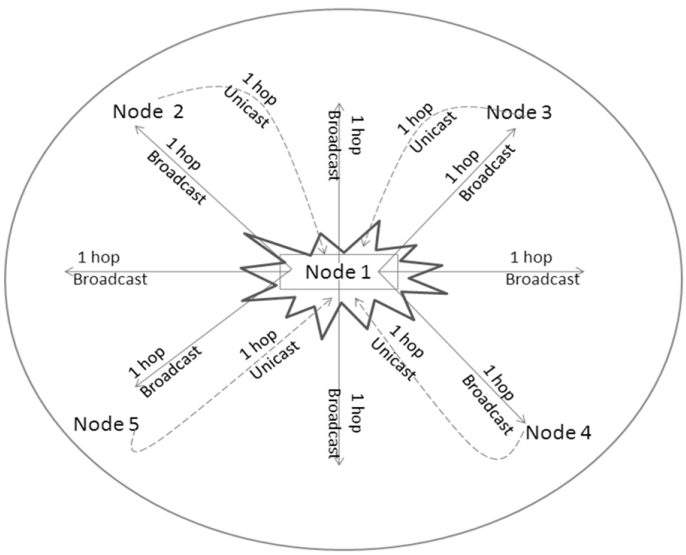


Fig.1. Link key exchange

Now, source node 0 encrypts the mutable fields or all header fields of AODV route request message using Blowfish algorithm and send it to node 1. The key being used for encryption is secret sharing key of node 0 to communicate node 1. Sameway, node 0 sends the encrypted message to node 3 which is encrypted with the key being used to communicate node 3. Once the intermediate node 1 and node 3 receive the encrypted route request, they decrypt request with specified key. Then after both intermediate nodes are act and perform the same operations as flooded route request perform in normal AODV. The node 1 or node 3 may reply back to node 0 if they have fresh path information otherwise they further flood the route request. Before reply back to node 0, node 1 or node 3 perform the encryption of AODV route reply using respective secret sharing key. If the node 1 or node 3 has to flood the route request, they encrypt route request with their shared secret key. Node 1 send the encrypted route request to node 2 using the secret sharing key node 1 to 2. Node 3 send the encrypted route request to node 4 using the secret sharing key node 3 to 4. Nodes 2 and 4 act same as node 1 and 3 with their neighbors.

When destination node 5 receives the encrypted message from node 2, it will decrypt it using the secret sharing key node 2 to 5. If the node received the encrypted message from node 4, it will decrypt it using the secret sharing key node 4 to 5. Finally, node 5 receives the route request message and decrypt it same as in normal AODV. After that node 5 replies with encrypted AODV route reply using the secret sharing key 5 to 2. Node 2 decrypts reply using secret sharing key 5 to 2. Now, node 2 acts and performs the same operation as in normal AODV. Now node 2 encrypts route reply using secret sharing key node 2 to 1. Node 1 performs the same as node 2 for route reply with their neighbor secret shared key. Finally node 0 decrypts the reply message using secret sharing key node 1 to 0. The complete process is demonstrated in figure 2.

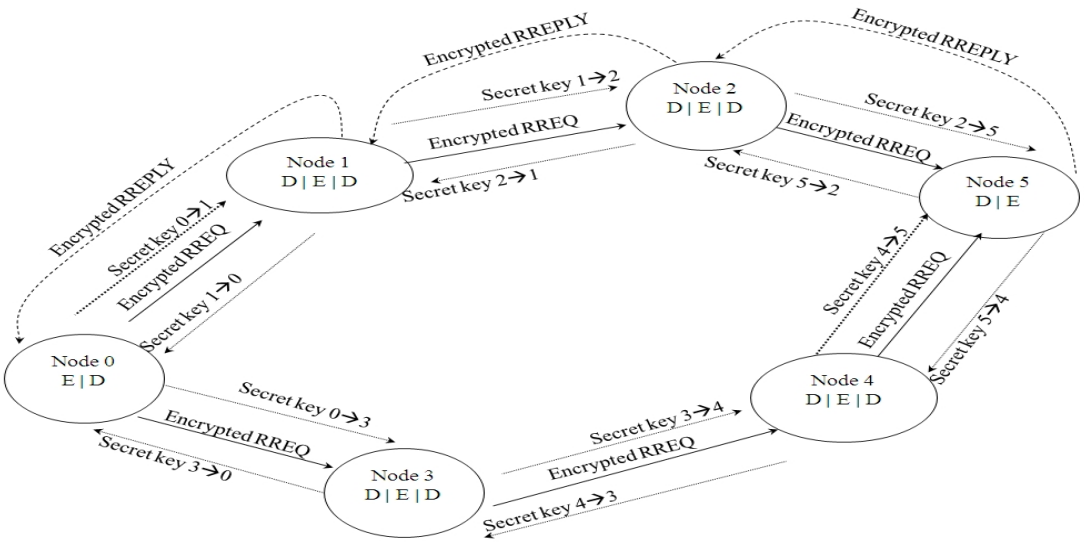


Fig.2. Securing AODV request and reply message

SIMULATION AND TEST RESULTS OF PROPOSED SCHEME

We have considered simulation parameters as mentioned in table 2 with AODV RFC in NS2. Figure 3 represents position of nodes as initial stage before the AODV discovered the route for FTP traffic. Table 3 shows the discovered secret sharing keys (link keys) for the nodes shown in figure 3. Figure 3 also shows complete procedures for securing AODV route discovering among six nodes. In this destination sequence number and hop count field of AODV route request message are encrypted using Blowfish block cipher at node 0 (source node) for sending it to node 0's neighbor node 1 and node 3. Node 1 and node 3 receives the encrypted fields and performed the decryption and reencryption operation to send the route request to their neighbor nodes (node 1 sends it to neighbor node 2 and node 3 sends it to neighbor node 4.) At last node 5 (destination) received encrypted the route request. Node 5 replies with encrypted destination sequence and hop count fields to neighbor node 2. This node 2 decrypts these fields and reencrypts the updated fields for forwarding it to its neigbhor node 1. Same as node 2, node 1 performs and forward this encrypted reply message to source node 0. Finally node 0 received the secure path information.

Based on simulation, figure 4 illustrate that the original AODV takes 15.1ms and our proposed scheme takes 25ms for route discovery. Hence the proposed scheme takes 9.9ms more compare to original AODV for route discovery. Because every nodes perform at least one time encryption and decryption operation for securing route discovery process. Figure 5 shows that the average consumed energy for existing

AODV is 0.87J and 0.93J for proposed scheme. Figure 6 demonstrate that the total energy consumed by network. Original AODV consumes 5.29J and proposed scheme consumes 5.59J.

Table 2. Simulation parameters

MAC Type	802.11 (WLAN)
Area	500x500 square meter
Number of nodes	6
Routing Protocol	AODV
Traffic type	FTP
Simulation time	15 S
Initial Energy	100J
txPower	0.6 W
rxPowre	0.2 W
Neighbor Purge time (Proposed scheme)	10S
Neighbor Purge time (Existing AODV)	1 S
Hello interval time (Proposed scheme)	10 S
Hello interval time (Existing AODV)	1S
Simulator	NS2 version 2.35
Processor	Intel Pentium Dual with 2.20GHz
Main Memory	1GB RAM
Operating System	32 bits Ubuntu 12.04 LTS
Encryption Algorithm	Blowfish

Table 3. Shared secret keys

Neighbor Node	Secret Key
0's Neighbor 1	0X01358090
0's Neighbor 3	0X00642448
1's Neighbor 0	0X020A39BC
1's Neighbor 2	0X00CCCE53
2's Neighbor 1	0X007057D0
2's Neighbor 5	0X012F154E
3's Neighbor 0	0X02F14789
3's Neighbor 4	0X00797CC0
4's Neighbor 3	0X027F72E9
4's Neighbor 5	0X018B906F
5's Neighbor 4	0X00D90C89
5's Neighbor 2	0X00CCCE58

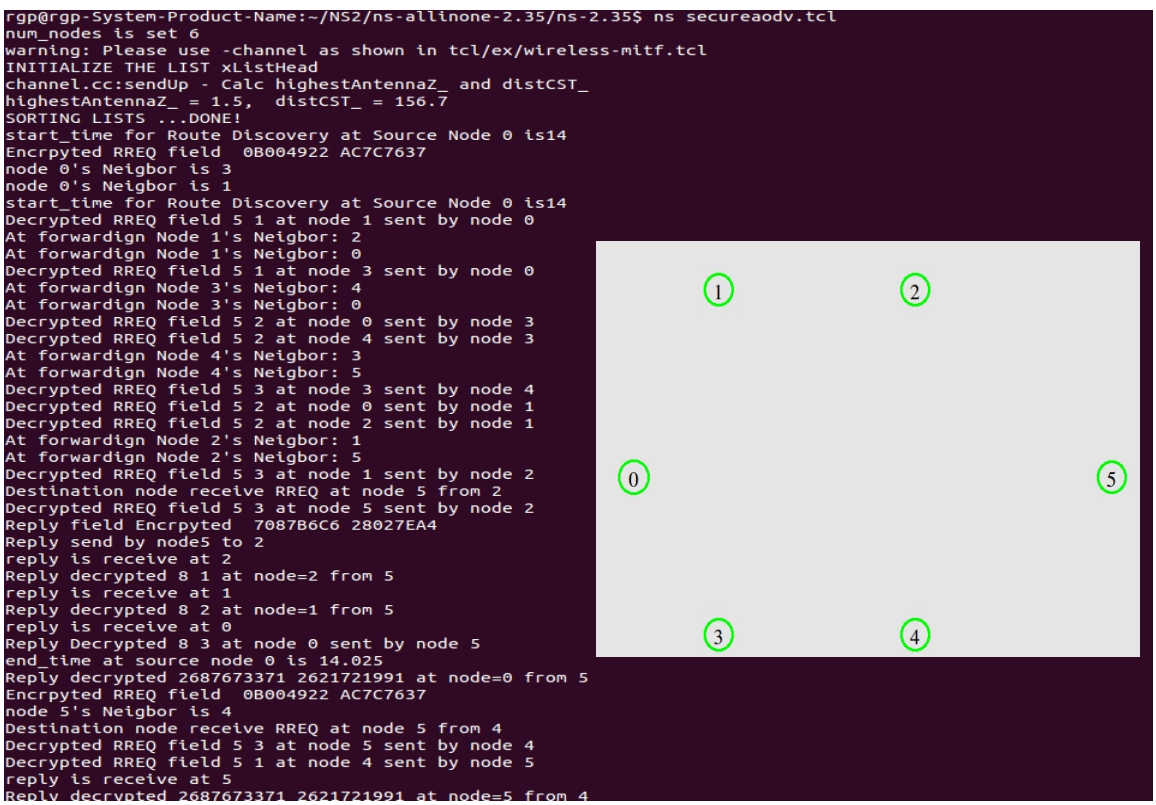


Fig. 3. Securing AODV RREQ and RREP

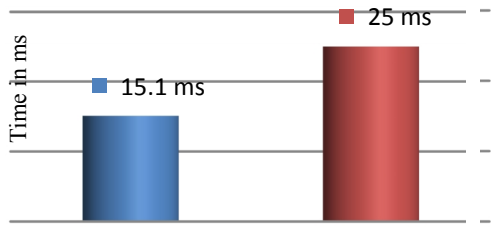


Fig. 4. Route discovery time

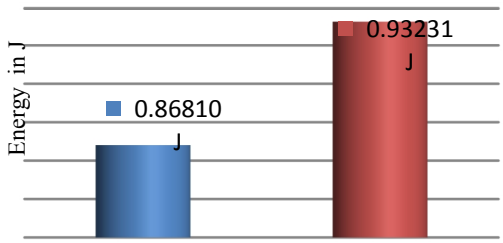


Fig.5. Average energy

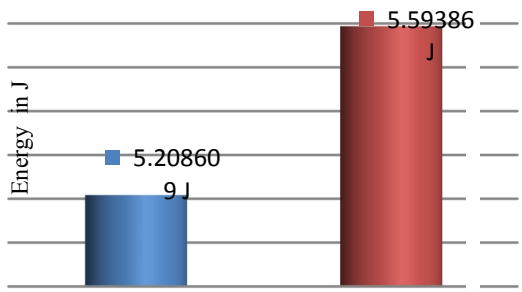


Fig.6. Total energy

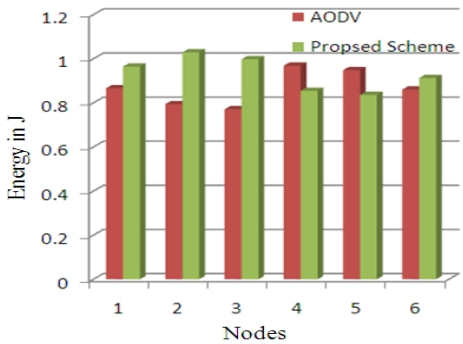


Fig.7. Utilized energy by node during route discovery

SECURITY INVESTIGATION

Assumption:During security analysis, we considered that the link secret sharing key has been securely delivered to secure the AODV routing protocol from external attacker. The attack may be from external or internal attacker in the form of active or passive. We considered the role of only external attacker in the form of active or passive for below listed attacks. External attacker is the outsider node of established network that do not have knowledge to compromise the established security in network. Security based encryption and authentication schemes can accomplish the prevention of external attacks (*29*). Following attacks (either active or passive) are considered by proposed scheme.

- *AODV Flooding attack:* Attacker can send the number of AODV RREQs within sort period of time for destination who does not exist.
  Resistant: Normally, AODV broadcast the RREQ. But in our scheme, we use the unicast RREQs for destination node. Hence, once node receives the RREQs from external attacker, they immediately ignore the request before to proceed further due to broadcast address.
- *Eavesdropping attack:* It is a passive attack in which attacker is try to listen communication without interfering or injecting the communication.
  Resistant: During the communication, AODV message fields are encrypted which can only read or decrypt by participated or internal nodes. Hence the outside of network node cannot do it.
- *Sinkhole attack*: A node attack to neighbor nodes by informing the fake route entry (it has route for destination). Hence the attracted node send data and the sink node simply drop the data.
  Resistant: In our scheme, the link to link (node to node) communication uses the encrypted fields for exchange. The sinknode simply send the fake route entry to attack the nodes. These sent messages

by sinknode are normal message for neighbor nodes. The neighbor nodes do not process because the message are in the form of plain text. Hence, the neighbor nodes ignore the received message.

- *Grayhole and blackhole (Packet Dropper) attack*: Packet dropper node attract to other nodes by claiming that it has shortest path. After that the nodes are prefer shortest path which is suggested by packet dropper node. When the packet dropper node receive data packet simply drop all data packet (grayhole) or randomly drop some packet (blackhole).
  Resistant: Packet dropper cannot attract to source node because the all routing communication packets are coded or encrypted. Even if the packet dropper node cannot know the value of coded header fields such as destination sequence number, hop count. Due to this packet dropper cannot suggest shortest path for attracting the other nodes.
- *Disclosing the routing information:* External malicious node try to disclose or latter to use the information for monitoring, disclosing node information etc.
  Resistant: External malicious node cannot understand the encrypted header fields of packet. Hence simply it can only disclose the traffic analysis without getting much more information.

Our scheme provides confidentiality due to encrypted fields of AODV messages. The encryption takes place between links or node to node, hence the scheme gives the node to node authentication. The scheme follows the link encryption for one hop, due to that the packets are always available. We use the concept of neighbor discovery with steganography for sharing secret key. So it does not required to use TTP or public key mechanism for secret key distribution. Table 4 summarizes security services provided by proposed scheme.

Table 4. Security services

Security Services	**Provided**
Confidentiality	Yes
Authentication	Yes
Integrity	No
Availability	Yes
TTP	Not required

CONCLUSION

MANET is vulnerable and may cause the security risks from internal and external attack in the form of active and passive attack. We proposed a scheme to secure the reactive route discovery process in AODV routing protocol against AODV flooding attack, eavesdropping attack, sinkhole attack, grayhole and blackhole (packet dropper) attack, disclosing the routing information from external attacker. The proposed scheme is implemented and simulated in NS2.34 environment with modified AODV reactive routing protocol. The proposed scheme provides confidentiality, authentication and availability of AODV messages among links without use of TTP.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

(Proceedings available for download at rku.ac.in/icre)

RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

Isobaric Vapour-Liquid Equilibrium Data Prediction for a Binary System of 2-Methyltetrahydrofuran and Acetic acid using Group Contribution Methods

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ABSTRACT

Isobaric vapour-liquid equilibrium data are predicted for the binary system of 2-Methyltetrahydrofuran and acetic acid at atmospheric pressure using group contribution methods. The data prediction is done by UNIFAC method and modified UNIFAC Dortmund method. The predicted data are checked for the thermodynamic consistency by the Herington test. The activity coefficients have been correlated using Van Laar, Wilson and NRTL equations. The binary interaction parameters of the models have been obtained by regression. The predicted VLE data of UNIFAC method and modified UNIFAC Dortmund method are compared and conclusions are made.

SUMMARY

The binary VLE data useful for designing the separation equipment for the green solvent 2-MeTHF and acetic acid are obtained by group contribution methods.

Keywords: vapour-liquid equilibrium, 2-Methyltetrahydrofuran, acetic acid, group contribution methods, UNIFAC method, modified UNIFAC Dortmund method

INTRODUCTION

Solvents play a crucial role in the synthesis of many chemicals, pharmaceutical intermediates, and other petrochemical products. They are also significant regarding their impact on the environment because a majority of the solvents are volatile, flammable and toxic which raise concerns related to safety and health of human being and animals. The search for so-called green solvents is always on as they have no effect or minimal effect on the health and environment due to their less toxic nature and other desirable characteristics. These green solvents offer an alternative to the conventional solvents. The acetic acid recovery from water has become the problem of industrial importance because of its influence on the economy and environmental pollution. Hongxun Zhang, Guangyu Liu, Chen Li, et al. measured liquid-liquid equilibria of water, acetic acid, and Cyclopentyl methyl ether (CPME) mixtures at different temperatures and indicated that CPME would be a good replacement for conventional organic solvents to separate acetic acid from water (*1*). 2-Methyltetrahydrofuran (2-MeTHF) is also an ethereal solvent considered to be one of the green solvents. 2-MeTHF is truly a green alternative to Dichloromethane and Tetrahydrofuran (*2*). The solvent 2-MeTHF is being increasingly used as an alternative to its more conventional analogues such as tetrahydrofuran and tert-butyl methyl ether within the academic and industrial chemical communities (*3*). It guarantees superior versatility, efficiency and reactivity in Grignard and organometallic reactions (*4*). The additional advantage that 2-MeTHF has over CPME is that 2-MeTHF is derived from renewable sources. It is a versatile and eco-friendly solvent that is derived from a variety of agricultural byproducts such as corncobs and bagasse (*5-7*). The predicted vapour-liquid equilibrium (VLE) data for the system of CPME and acetic acid are available in the literature (*8*). But VLE data for 2-MeTHF and acetic acid system that is essential for the design of distillation column for separating 2-MeTHF and acetic acid from their mixture does not exist in the literature. So exploration of VLE data of this binary system becomes necessary. The experimental determination of VLE data is a time-consuming and costly procedure that requires sophisticated instruments such as VLE apparatus, gas chromatograph, refractometer, spectrophotometer, etc. The need for VLE data generation of systems involving green solvents and outline of the procedure for generating VLE data is described in the literature(*9*). Numerical computations using group contribution methods provide an alternative to experimental measurement of VLE data. The predicted VLE data can be used for the preliminary design of distillation columns. The aim of this paper is to predict VLE data for 2-MeTHF and acetic acid system at atmospheric pressure using group contribution methods.

ADVANCED GROUP CONTRIBUTION METHODS:

Reliable knowledge of properties of materials is essential for the design of chemical processes. The information of physical properties of fluids is critical in the design of many kinds of products, processes, and industrial equipments (*10*). The VLE data are vital for the optimal design of chemical and separation processes. When experimental VLE data are available, the equilibrium behaviour is modelled with the equations of state and excess Gibbs energy models. In the absence of experimental data, group contribution (GC) methods can be used to predict the phase equilibrium behaviour (*11, 12*). Some examples of GC methods that have been developed for the estimation of properties of pure compounds include those published by Joback and Reid (*13*), Lydersen (*14*), Ambrose (*15*), Constantinou and Gani (*16*) and Marrero and Gani (*17, 18*). On the other hand, many group contribution based property models have also been generated to predict properties of mixtures mainly to predict the non-ideality of the liquid phase using activity coefficients which include ASOG (*19, 20*), Original UNIFAC(*21*), Modified UNIFAC Dortmund (*22*) and PSRK (*23*). In the present work, well known and established GC methods

such as UNIFAC method that is also known as original UNIFAC and modified UNIFAC Dortmund method are employed to predict liquid phase activity coefficients for binary mixtures of 2-MeTHF and acetic acid.

Original UNIFAC and modified UNIFAC Dortmund methods:

The general UNIFAC equation for the calculation of activity coefficient is as follows with the combinatorial and residual contributions:

$$\ln \gamma_i = \ln \gamma_{i(combinatorial)} + \ln \gamma_{i(residual)} \quad (1)$$

The combinatorial part,

$$\ln \gamma_{i(combinatorial)} = \ln \frac{\Phi_i}{x_i} + \frac{z}{2} q_i \ln \frac{\theta_i}{\Phi_i} + l_i - \frac{\Phi_i}{x_i} \sum_j x_j l_j \quad (2)$$

where,

$$l_i = \frac{z}{2}(r_i - q_i) - (r_i - 1) \quad (3)$$

$$\theta_i = \frac{q_i x_i}{\sum_j q_j x_j} \quad (4)$$

$$\Phi_i = \frac{r_i x_i}{\sum_j r_j x_j} \quad (5)$$

$$r_i = \sum \nu_k^{(i)} R_k \quad (6)$$

$$q_i = \sum \nu_k^{(i)} Q_k \quad (7)$$

$$R_k = \frac{V_{wk}}{15.17} \quad (8)$$

$$Q_k = \frac{A_{wk}}{2.5 \times 10^9} \quad (9)$$

Where, $\nu_k^{(i)}$, an integer, is the number of groups of type k in molecule i. R_k and Q_k are group parameters that are obtained from the van der Waals group volume and surface areas V_{wk} and A_{wk}, given by Bondi (1968) (*10*). The value of parameter Z is taken as 10.

And the residual part,

$$\ln \gamma_{i(residual)} = \sum_k \nu_k^{(i)} \left(\ln \Gamma_k - \ln \Gamma_k^{(i)} \right) \quad (10)$$

where,

$$\ln \Gamma_k = Q_k \left(1 - \ln \left(\sum_m \theta_m \Psi_{mk} \right) - \sum_m \frac{\theta_m \Psi_{km}}{\sum_n \theta_n \Psi_{nm}} \right) \quad (11)$$

where, the group area fraction θ_m and group mole fraction X_m are given by the following equations:

$$\theta_m = \frac{Q_m X_m}{\sum_n Q_n X_n} \tag{12}$$

$$X_m = \frac{\sum_j \nu_m^{(j)} x_j}{\sum_j \sum_n \nu_n^{(j)} x_j} \tag{13}$$

where, the group-interaction parameter Ψ_{mn} is given by the following equation:

$$\Psi_{mn} = \exp\left(-\frac{U_{mn} - U_{nn}}{RT}\right) = \exp\left(-\frac{a_{mn}}{T}\right) \tag{14}$$

where, U_{mn} is a measure of the energy of interaction between group m and n. Note that a_{mn} has a unit of Kelvin and $a_{mn} \neq a_{nm}$.

Some weaknesses of original UNIFAC method are well known. For example, quantitive predictions of excess enthalpies H^E can not be obtained due to the independence of model parameters from temperature.To overcome this and other weaknesses, the modified UNIFAC Dortmund method was introduced. In this new model, the combinatorial part of original UNIFAC method was revised to improve predictions of asymmetric systems. In the residual part also logarithmic and quadratic temperature-dependent interaction parameters were introduced to allow improved predictions of VLE, H^E and γ_i^∞. This improved version of original UNIFAC method can also extrapolate reliably the predictions of VLE in a wide temperature range (*24, 25*). The differences between UNIFAC and modified UNIFAC Dortmund method are given in the following equations. In modified UNIFAC Dortmund method, Eq. (2) and Eq. (14) of UNIFAC method are replaced by Eq. (15) and Eq. (19) as described below.

$$\ln \gamma_{i(\text{combinatorial})} = 1 - V_i' + \ln V_i' - 5q_i\left(1 - \frac{V_i}{F_i} + \ln\left(\frac{V_i}{F_i}\right)\right) \tag{15}$$

where,

$$V_i = \frac{r_i}{\sum_j x_j r_j} \tag{16}$$

$$F_i = \frac{q_i}{\sum_j x_j q_j} \tag{17}$$

$$V_i' = \frac{r_i^{3/4}}{\sum_j x_j r_j^{3/4}} \tag{18}$$

$$\text{and } \Psi_{mn} = \exp\left(-\frac{a_{mn} + b_{mn}T + c_{mn}T^2}{T}\right) \tag{19}$$

Group identification of the compounds:
Correct identification of groups of the compounds is the first and the most important step towards the prediction of reliable property parameters because group contribution model calculations rely on type and number of groups existing in the compounds. The group identification is done using the data given in the literature (*10*) for UNIFAC method. For modified UNIFAC Dortmund method, data given in the literature (*24*) are used. The identified groups are presented in Table 1 and Table 2 respectively.

Binary interaction parameters (BIPs):
Binary interaction parameters (a_{mn}) for UNIFAC method have been taken from the literature (*10*). The BIPS (a_{mn}, b_{mn}, and c_{mn}) for modified UNIFAC Dortmund method have been taken from the literature (*24-26*). All BIPS are presented in Table 3 and Table 4 respectively.

Spread sheet Calculation of VLE data:
The group contribution method models are used to calculate activity coefficients. Temperature T and liquid phase composition x_1 are given as input and γ_1 and γ_2 are calculated. Using Antoine Eq. (20), p_1^{sat} and p_2^{sat} are calculated, then total pressure P is calculated, and then correct temperature T is found out by regression using Eq. (23). The tables 5 and 6 present calculated data for UNIFAC method and modified UNIFAC Dortmund method respectively.

The Antoine equation,

$$\ln p_i^{sat} = A_i - \frac{B_i}{T + C_i} \qquad (20)$$

Where, pressure is in kPa and temperature is in Kelvin. The constants A, B, and C of Antoine equations of 2-MeTHF and acetic acid are listed in Table 7.

THERMODYNAMIC CONSISTENCY TEST:

The thermodynamic consistency of the predicted vapour liquid equilibrium data is checked by semi-empirical Herington test (*27*). In this method, the values for D and J are found out by Eq. (21) and Eq. (22) respectively. If the value of D – J is not larger than 10 then the predicted VLE data are said to be thermodynamically consistent. The values of $|D - J|$ for the binary system are listed in Table 8.

$$D = 100\,\frac{\int_{x_1=0}^{x_1=1} \ln\frac{\gamma_1}{\gamma_2}\,dx_1}{\int_{x_1=0}^{x_1=1} \ln\left|\frac{\gamma_1}{\gamma_2}\right|dx_1} \qquad (21)$$

$$J = 150\,\frac{T_{max} - T_{min}}{T_{min}} \qquad (22)$$

DATA REDUCTION USING g^EMODELS:

The predicted VLE data are correlated with various models such as Van Laar, Wilson and NRTL *(28, 29)*. The Antoine equation with the parameters given in Table 7 is used to calculate vapor pressures of pure

components.By the minimization of the objective function %AAD ∑(δP), the binary interaction parameters are obtained for these models that are used to minimize error by the regression procedure. (%AAD = absolute average deviation and n represent no. of predicted data points). Similarly, AAD ∑(δT) and AAD ∑(δy) are calculated by Eq. (24) and Eq. (25) respectively. The "pre" and "cal" subscripts represent the predicted and calculated values respectively.

$$\%AAD\ \sum(\delta P) = \frac{100}{n}\sum_{i=1}^{n}\frac{\left|P_{i,pre.} - P_{i,cal.}\right|}{P_{i,pre.}} \qquad (23)$$

$$AAD\ \sum(\delta T) = \frac{1}{n}\sum_{i=1}^{n}\left|T_{i,pre.} - T_{i,cal.}\right| \qquad (24)$$

$$AAD\ \sum(\delta y) = \frac{1}{n}\sum_{i=1}^{n}\left|y_{i,pre.} - y_{i,cal.}\right| \qquad (25)$$

Table 9 and 10 show the binary interaction parameters, correlated from predicted VLE data by UNIFAC method and modified UNIFAC Dortmund methodrespectively. α which is a characteristic constant of the non-randomness for the binary system is recommended as 0.3 for this binary system because it belongs to type I system according to the definition given in the literature (*29*). The comparison of predicted data by UNIFAC method and modified UNIFAC Dortmund method with calculated T-x_1-y_1 data by Van Laar, Wilson, and NRTL models for the binary system 2-MeTHF (1) + acetic acid (2) at atmospheric pressure is given inFig. 1 to Fig. 6.From the figures, it can be seen that isobaric VLE data predicted by UNIFAC method and modified UNIFAC Dortmund method for the system 2-MeTHF and Acetic acid are very well represented by Van Laar, Wilson and NRTL models.

CONCLUSION

The VLE data for the binary system 2-MeTHF with acetic acid have been predicted at atmospheric pressure using UNIFAC method and modified UNIFAC Dortmund method. The activity coefficient models Van Laar, Wilson and NRTL have been found capable of accurately fitting the predicted VLE data by UNIFAC method and modified UNIFAC Dortmund method. The value of $|D - J|$ is 8.70 for the UNIFAC method and 38.68 for the modified UNIFAC Dortmund method which should be less than 10 for the correctness of thermodynamic consistency. Despite several modifications done in modified UNIFAC Dortmund method, it fails to predict VLE data to the desired extent. The possible reason for the failure of this method can be attributed to the inability of interaction parameters to predict the accurate VLE behaviour of the binary system. Azeotrope formation is not found for this system.

FIGURES

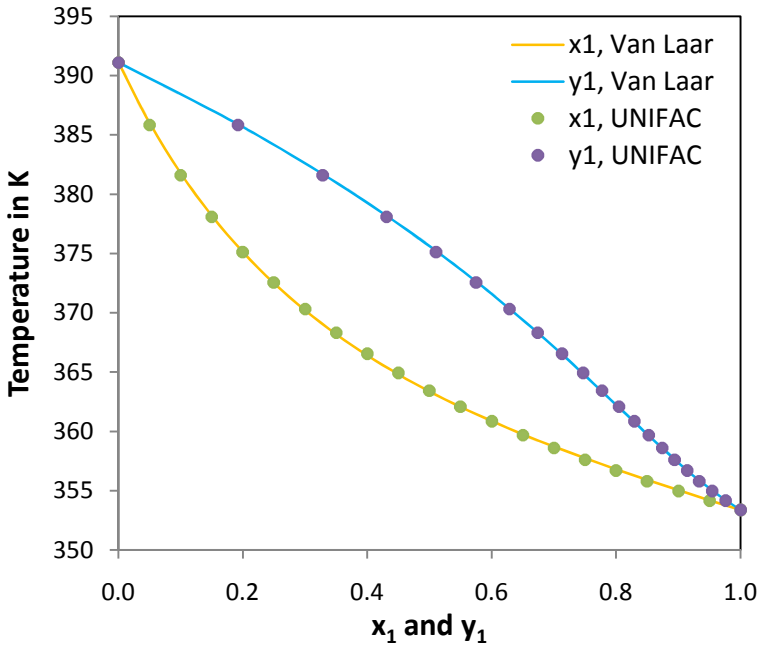


Fig. 1. T-x_1-y_1 diagram calculated by Van Laar and predicted by UNIFAC method

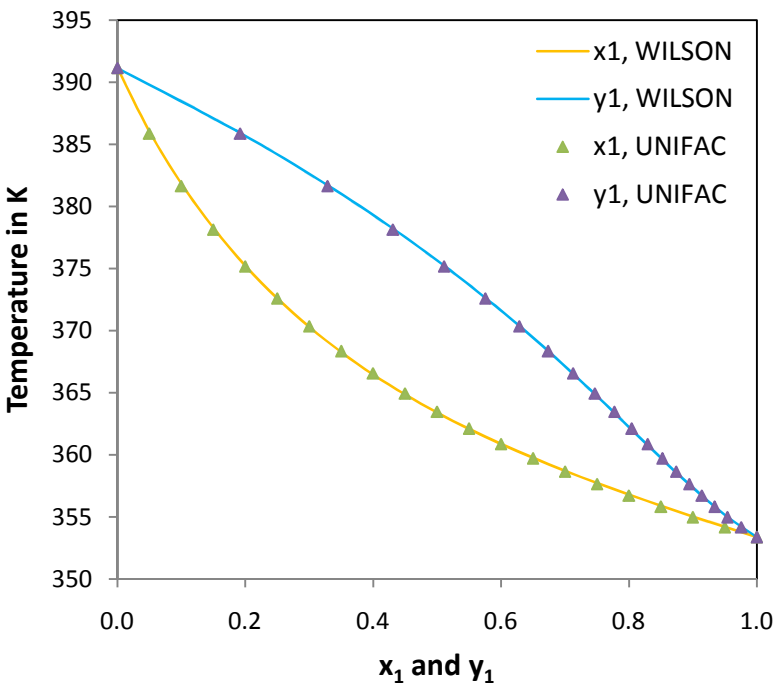


Fig. 2. T-x_1-y_1 diagram calculated by Wilson and predicted by UNIFAC method

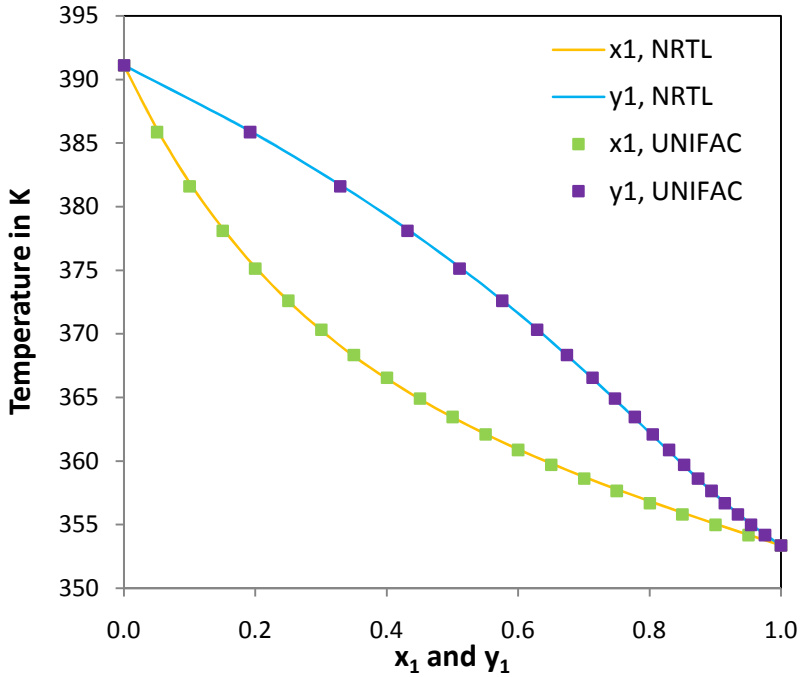


Fig. 3. T-x_1-y_1 diagram calculated by NRTL and predicted by UNIFAC method

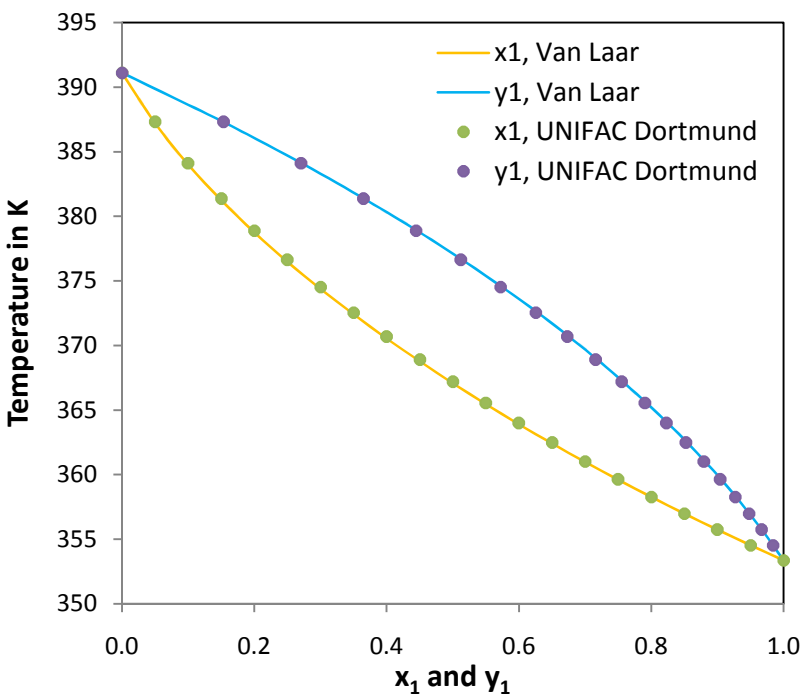


Fig. 4. T-x_1-y_1 diagram calculated by Van Laar and predicted by modified UNIFAC Dortmund method

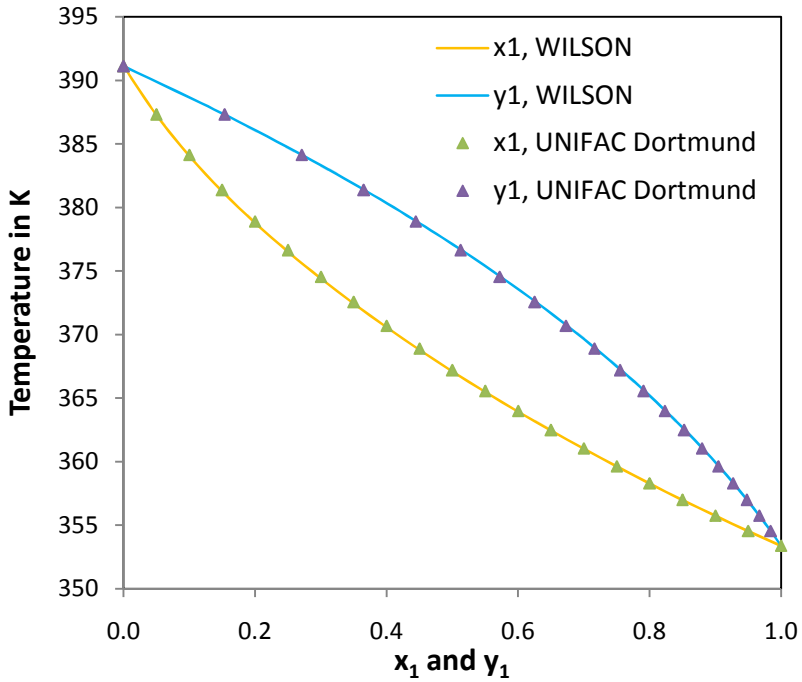


Fig. 5. T-x_1-y_1 diagram calculated by Wilson and predicted by modified UNIFAC Dortmund method

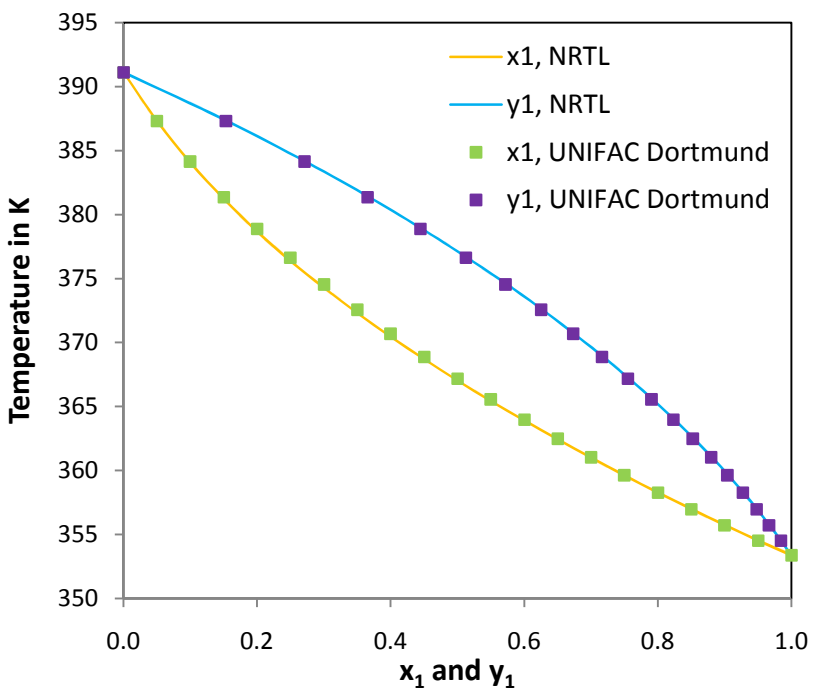


Fig. 6. T-x_1-y_1 diagram calculated by NRTL and predicted by modified UNIFAC Dortmund method

TABLES

Table 1. Group identification for 2-MeTHF and acetic acid for UNIFAC method

Molecule (i)	Name	Group No.		$v_k^{(i)}$	R_k	Q_k
		Main Group No.	Secondary Group No.			
2-MeTHF (1)	CH_3	1	1	1	0.9011	0.848
	CH_2	1	2	2	0.6744	0.540
	CH	1	3	1	0.4469	0.228
	CH_2O	13	27	1	0.9183	1.100
Acetic acid (2)	CH_3	1	1	1	0.9011	0.848
	COOH	20	42	1	1.3013	1.224

Table 2. Group identification for 2-MeTHF and acetic acid for modified UNIFAC Dortmund method

Molecule (i)	Name	Group No.		Group No.	R_k	Q_k
		Main Group No.	Main Group No.			
2MeTHF(1)	CH_3	1	1	1	0.6325	1.0608
	cy-CH_2O	43	27	1	1.7023	1.8784
	cy-CH_2	42	78	2	0.7136	0.8635
	c-CH	42	79	1	0.3479	0.1071
Acetic acid (2)	CH_3	1	1	1	0.6325	1.0608
	COOH	20	42	1	0.8000	0.9215

Table 3. BIPs for 2-MeTHF and acetic acid for UNIFAC method

Group	CH_3	CH_2	CH	CH_2O	COOH
CH_3	0.0	0.0	0.0	251.5	663.5
CH_2	0.0	0.0	0.0	251.5	663.5
CH	0.0	0.0	0.0	251.5	663.5
CH_2O	83.36	83.36	83.36	0.0	664.6
COOH	315.3	315.3	315.3	-338.5	0.0

Table 4. BIPs for2-MeTHF and acetic acid for modified UNIFAC Dortmund method

Group		m	n	a_{mn}	b_{mn}	c_{mn}	a_{nm}	b_{nm}	c_{nm}
CH_3	COOH	1	20	1182.2	-3.2647	0.009198	2017.7	-9.0933	0.01024
CH_3	cy-CH_2, cy-CH	1	42	-680.95	4.0194	-0.006878	1020.8	-6.0746	0.01015
CH_3	cy-CH_2O	1	43	79.507	0.7089	-0.002098	186.71	-1.3546	0.002402
COOH	cy-CH_2, cy-CH	20	42	1169.3	-3.0737	0	582.81	1.4976	0
COOH	cy-CH_2O	20	43	720.45	-1.5187	0	-140.77	0.309	0
cy-CH_2O	cy-CH_2, cy-CH	43	42	20.834	-0.3472	0	242.49	-0.03832	0

Table 5. VLE data for 2-MeTHF and acetic acid binary system at atmospheric pressure by UNIFAC method

T/K	x_1	y_1	γ_1	γ_2
353.37	1.0000	1.0000	1.0000	----
354.16	0.9500	0.9757	1.0027	1.7231

354.97	0.9000	0.9541	1.0099	1.5766
355.81	0.8500	0.9338	1.0205	1.4661
356.70	0.8000	0.9141	1.0336	1.3805
357.63	0.7500	0.8942	1.0490	1.3126
358.63	0.7000	0.8738	1.0663	1.2577
359.70	0.6500	0.8523	1.0854	1.2126
360.85	0.6000	0.8293	1.1064	1.1751
362.10	0.5500	0.8044	1.1295	1.1434
363.45	0.5000	0.7771	1.1547	1.1165
364.93	0.4500	0.7468	1.1823	1.0935
366.55	0.4000	0.7127	1.2129	1.0737
368.34	0.3500	0.6738	1.2468	1.0567
370.34	0.3000	0.6286	1.2847	1.0421
372.58	0.2500	0.5753	1.3275	1.0297
375.15	0.2000	0.5109	1.3762	1.0195
378.11	0.1500	0.4310	1.4322	1.0113
381.62	0.1000	0.3287	1.4973	1.0052
385.85	0.0500	0.1921	1.5738	1.0013
391.12	0.0000	0.0000	----	1.0000

Table 6. VLE data for 2-MeTHF and acetic acid binary system at atmospheric pressure by modified UNIFAC Dortmund method

T/K	x_1	y_1	γ_1	γ_2
353.37	1.0000	1.0000	1.0000	1.1140
354.53	0.9500	0.9840	1.0000	1.1128
355.73	0.9000	0.9667	0.9999	1.1111
356.98	0.8500	0.9477	1.0000	1.1087
358.28	0.8000	0.9270	1.0001	1.1057
359.63	0.7500	0.9044	1.0004	1.1021
361.02	0.7000	0.8796	1.0010	1.0978
362.48	0.6500	0.8526	1.0021	1.0928
363.98	0.6000	0.8230	1.0037	1.0872
365.55	0.5500	0.7906	1.0062	1.0809
367.19	0.5000	0.7551	1.0098	1.0739
368.89	0.4500	0.7160	1.0149	1.0663
370.68	0.4000	0.6729	1.0219	1.0582
372.55	0.3500	0.6252	1.0314	1.0496
374.53	0.3000	0.5721	1.0443	1.0407
376.63	0.2500	0.5123	1.0617	1.0316
378.90	0.2000	0.4443	1.0850	1.0228
381.37	0.1500	0.3652	1.1162	1.0145
384.13	0.1000	0.2708	1.1580	1.0073
387.31	0.0500	0.1536	1.2138	1.0021
391.12	0.0000	0.0000	1.2881	1.0000

Table 7. Antoine constants

Compound	Antoine constants			temperature range/K
	A	B	C	
2-MeTHF	13.7005	2706.71	-55.3	357 to 395
AA	15.0694	3580.79	-48.5	375 to 395

Table 8. Thermodynamic consistency check

D	J	D-J	Method
7.33	16.03	8.70	UNIFAC
54.71	16.03	38.68	modified UNIFAC Dortmund

Table 9. Correlated model BIPs from predicted data by UNIFAC method

Model	Binary Parameter		AAD (ΔT)	AAD (Δy)
Van Laar	A_{12}	A_{21}	0.0881	0.0017
	0.4581	0.5943		
Wilson	a_{12}	a_{21}	0.0763	0.0016
	-1566.039	3352.219		
NRTL	b_{12}	b_{21}	0.0933	0.0019
	1874.248	-128.502		

Table 10. Correlated model BIPs from predicted data by modified UNIFAC Dortmund method

Model	Binary Parameter		AAD (ΔT)	AAD (Δy)
Van Laar	A_{12}	A_{21}	0.0732	0.0027
	0.2798	0.1001		
Wilson	a_{12}	a_{21}	0.0584	0.0018
	1385.071	55.218		
NRTL	b_{12}	b_{21}	0.0970	0.0039
	-2090.326	3299.729		

ACKNOWLEDGEMENT

We wish to thank the management of V.V.P. Engineering College, Rajkot for theinfrastructural support and Gujarat Council on Science and Technology (GUJCOST), Gandhinagar for the financial assistance.

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Nomenclature

P - Absolute pressure, kPa

T - Absolute temperature, K

θ_i - Surface area fraction of compound i

Φ_i - Volume fraction of compound i

r_i - Relative Van der Waals volume of compound i
q_i - Relative Van der Waals surface area of compound i
Q_k - Relative Van der Waals surface area of subgroup k
R_k - Relative Van der Waals volume of subgroup k
Γ - Temperature dependant integration constant
θ_m - Surface area fraction of subgroup m
X_m - Mole fraction of subgroup m
Ψ - Group-interaction parameter
V_i - Volume/mole fraction of compound i in the mixture
F_i - Surface area fraction of compound i in the mixture
V'_i - Modified volume/mole fraction of compound i in themixture (modified UNIFAC Dortmund method)
ln - Natural logarithm (base e)
log - Logarithm (base 10)
x_i - Liquid phase mole fraction of i^{th} species
y_i - Vapor phase mole fraction of i^{th} species
γ_i - Activity coefficient of i^{th} species
H^E - Excess enthalpy
A_{ij} - Adjustable parameter (Van Laar Model)
λ_{ij} - Interaction parameter (Wilson Model)
Λ_{ij} - Adjustable parameter (Wilson Model)
α_{ij} -The non-randomness of the fluid empirical parameter
τ_{ij} - Adjustable parameter (NRTL Model)
A, B, C- Antoine equation constants

Superscripts
E - Excess property
sat - Saturated property value
∞ - Property at infinite dilution concentration

Subscripts
1 - Component 1
2 - Component 2
i - Property of i^{th} species
j - Property of j^{th} species

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th& Jan. 6th, 2016)

(Proceedings available for download at rku.ac.in/icre)

A Comparison Of The Enhanced Greedy Perimeter Stateless Routing Methods In Mobile Ad-Hoc Networks

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ABSTRACT

Mobile ad-hoc network is a self-configured and infrastructure less networks in wireless. In MANET the nodes are move anywhere in the network. So, mobility updation is more important to accurate the communication between nodes. For the military purposes, communication is very important for the operation planners and commanders. MANET represents the Greedy Perimeter Stateless Routing (GPSR) protocol to support such situation. This paper presents the works and performance study of location-based GPSR protocol. Two forwarding methods used in GPSR are: Greedy Forwarding and Perimeter Forwarding. This paper represents the some techniques which improved the GPSR are Location-update-strategy, Energy aware routing, Adaptive position update strategy, Ferry-Assisted GPS, Double-hand rule, Buffer zone technique, etc. These techniques are used to improve the performance of GPSR.

SUMMARY

Improve the energy efficiency and increase the node's existence by GPSR protocol in MANET.

Keywords: GPSR, Mobile ad-hoc network , Location-based.

1. INTRODUCTION

Mobile ad-hoc network(MANET) is dynamically distributed and infrastructure less networks. MANET is based on two types of routing. One of them is topology based routing and another is position based routing (*1*). Further topology based routing has two types: 1) Proactive: in this method node keep updating their routing table in periodically manner. 2) Reactive: in this method node update their routing table only when they want to communicate with others. In topology based routing nodes keep to update and maintain the table which contains the information of other nodes. This limitation is discovered by

location based routing. In location based routing nodes does not keep to maintain the topology table. Also, they do not need for root establishment (*2*). This advantage is improve the performance of routing protocol. Node keeps only their own id and position. Greedy Perimeter Stateless Routing (GPSR) is one of the positions based routing protocol in MANET.

1.1 Greedy Perimeter Stateless Routing(GPSR)

Greedy Perimeter Stateless Routing is location based routing protocol in MANET. GPSR used two mechanisms for forwarding the packet. One is the Greedy forwarding and another is perimeter forwarding(*3)*.

Greedy Forwarding: In this method the source node forward the packet to that node which is nearest to the sink node. The source node forward the packet with the one hope distance. Fig 1 show the example of greedy method(*3*).In certain situation greedy forwarding is fail to forward the packet. Then it comes into the perimeter mode.

Perimeter Forwarding: Sometimes greedy is fail to deliver the packet. It happens when sink node is out of range for the source node. Then they enter into the perimeter mode for recover the greedy mode. So it is also called the recovery mode(*1*). In this they used the Right-Hand-Rule mechanism for forward the packet. Fig 2 show the example of perimeter method(*3*).

As shown in Fig 2 there is a hole in the network than it is fail to the send the packet directly to the sink node. Using right hand rule it can take only the edge of the node and forward the packet to the destination. The greedy forwarding method is executed again to achieve the destination. in Fig 2 path for forward the packet will be X->Y->Z->D.

Original GPSR algorithm flow chart is shown in the Fig 3.

2. RELATED WORK

The main aim of the research in GPSR is to identify and improve the issue. The energy efficiency and link reliability are the most critical issue for the network. The main aim of this work is:

- Update the position of the node for long duration of the network.
- Saves the energy mechanism.

2.1 Advanced GPSR

Kim Kyu Seok and Navrati Saxena(*4*) has discuss about the energy. The energy is most important in MANET. They discuss about the advance GPSR to resolve the energy regarding problem. In this technique first check the residual energy of the node and than decide the next hope node. So this concept is used to increase the lifetime of the node. It also increases the throughput of the network.

2.2 Location Update Strategy

Mrs. B.Manimegalai and Mrs. D. Geetha(*5*) has discuss like that: In position based routing location update is most important for end to end delay. They used the Adaptive Location Update (ALU) strategy. In this strategy they used 3 updation beacon scheme. 1) Adaptive Beacon: If a node changes its speed, direction and distance than that node broadcast the beacon packet to the other node.2) Reactive Beacon:

The nodes update the beacon only when they want to communicate with the other nodes.3) Periodic Beacon: In this the node broadcast the Beacon packet at specific intervals.The packet delivery ratio can improve using this three updation scheme.

2.3 Becoan Update Strategy

Dhanarasan P and Gopi S.(*6*) presents the one strategy for beacon update. This strategy is called Adaptive Position Update(APU). In APU 2 scheme is used. One is Mobility Prediction(MP) and another is On-Demand Learning(ODL) .1) Mobility Prediction (MP): MP used when the information of the location broadcast in the earlier beacon becomes incorrect. The next beacon is broadcast only if the predicted error in the location estimate is greater than the certain amount of threshold.2) On-demand Learning(ODL): a node broadcast the beacon only when it hears the communication of the packet from the neighbor nodes.So it is used to save the energy and bandwidth from the periodically update of the beacon.

2.4 Patrol Seeking Mode

In basic GPSR, they used the two modes for forwarding the packet. And if the packet does not reach till the destination for certain reason than it drop the packet. Al-Roqi and Yasser Stylianos Papanastasiou and EvtimPeytchev (*1*) has discussed the another mode. This mode is called The Petrol Seeking Mode. In the Patrol Seeking Mode one node chooses randomly as a patrol node. Patrol caches the destination id and pre-defined path to reach the destination. Patrol node also used two modes like: ferry-greedy and ferry-perimeter. This two mode works same as the in standard GPSR. So, this helps to improve the packet delivery ratio and end to end delay.

2.5 Bufferzone Based Greedy Forwarding Strategy (BZGFS)

In Greedy Forwarding Strategy(GFS) the sender is sent to the packet which is nearest to the sink node. However, the node which is selected may not exist in the range of the forwarding node due to the movements of nodes. Thus, packets are dropped due to the link break. This is not recognized for the forwarding node. This matter is called Temporary Communication Blindness (TCB).W. Wei and Z. H. Yang (*7*)overcomes the problem of the TCB problem by adding the buffer zone at the radio margin with suitable radius. This buffer zone is based on the function of the maximum velocity and beacon period. When the node is moving into the buffer zone is still comes into the radio range of the forwarding node. So, it is recognized by the next beacon packet, and sender chooses the another next neighbor node. So, the packet loss during Greedy Forwarding Strategy(GFS) is decrease.

2.6 Left And Right Rule

In standard GPSR, when the node comes into the perimeter mode they used the Right-Hand rule to traverse the packet. But sometimes it fails to reach to the destination when it goes into the loop direction.T. Guoming and X. Yi and T. Daquan and T. Jiuyang presents the two rules: one is the Right-Hand rule and another is the Left-Hand rule. The packet is traversing in according to both rules and choose the optimal route from them. And thus it increases end to end delay and the packet delivery ratio.

Comparison Table:
Here, this table presents the different different method that improve the GPSR protocol. We are found some advantages and disadvantage for each method. Among them some methods are best for improve the GPSR which has not as much of drawback. In the Table 1 we show the comparison between methods.

3. RESULTS AND DISCUSSION

As we are shown the different methods and we will choose the best method to improve the GPSR like Patrol seeking mode because of its lesser amount of weakness. In the Patrol seeking mode node is selected randomly. In the proposed, this method will be improve using energy mechanism. As well as we know energy is most important for the wireless network. We will count the residual energy for individual nodes. And for the data forwarding we will choose the node which has more energy. So, it will decrease the packet loss and improve the delivery ratio for the network.

We will be improve this proposed method using NS-2 simulation.

4. CONCLUSION

In MANET some challenges are comes like Energy Efficiency, Scalability, Quality Of Service etc. GPSR try to improve the some challenges. This paper presents the comparison between GPSR'methods with some advantages and disadvantages. And among them we will improve the one GPSR method using energy mechanism as discuss in above section.

5. FIGURES

Fig 1: Example of greedy forwarding(*3*)

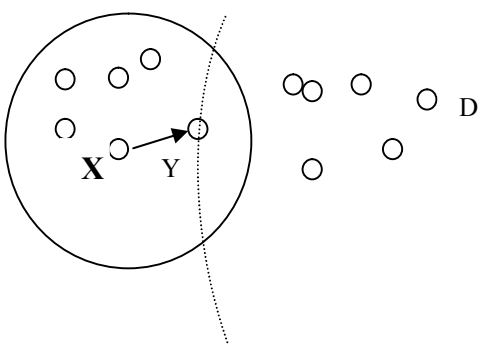


FIG 2: Example of perimeter forwarding(*3*)

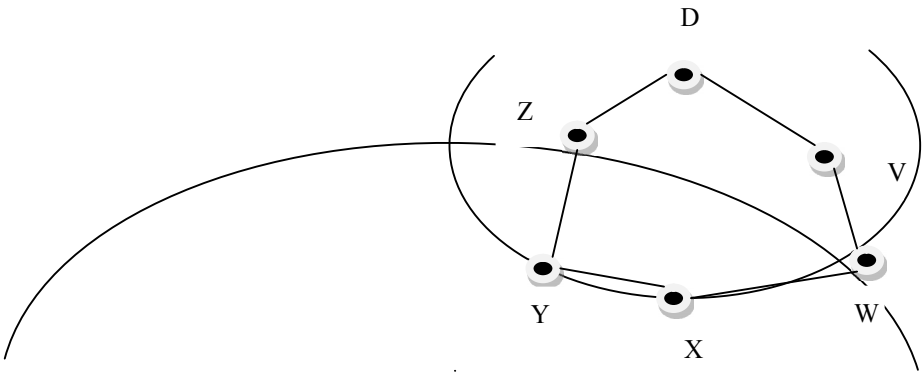
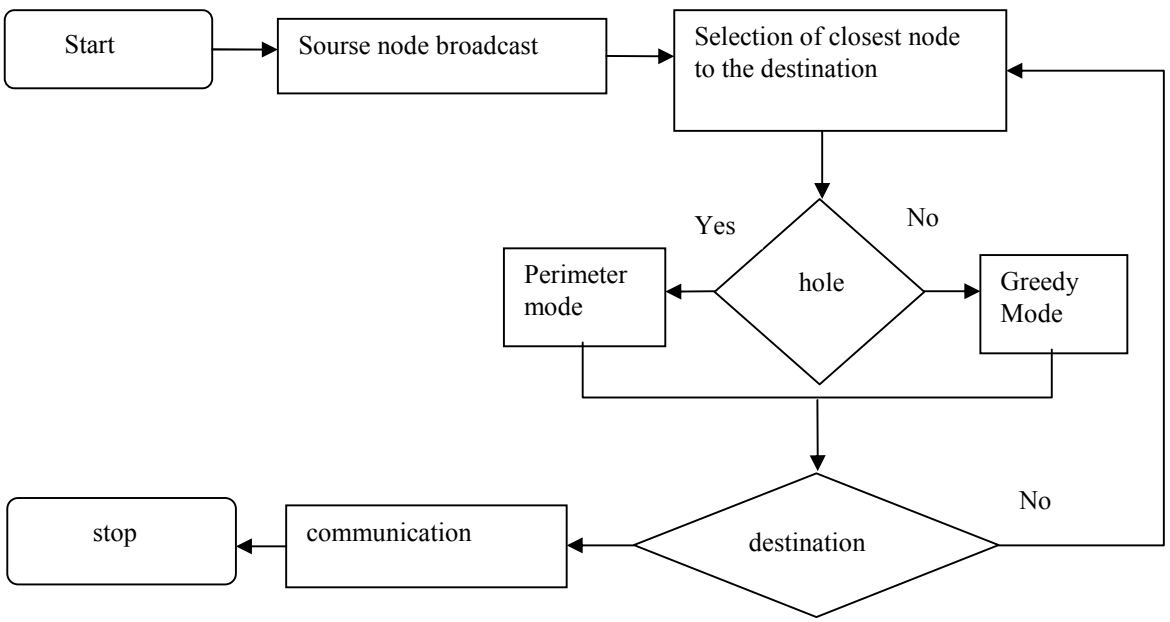


FIG 3: Flowchart for GPSR algorithm (*4*).



6. TABLES

Table 1: Comparison between Methods.

No.	Methods	Advantages	Disadvantages
1	Advanced GPSR	Efficiently saves the energy , improve lifetime of the network.	If the range is bigger than nodes need more energy.
2	Location update strategy	The packet delivery ratio is increased,less energy Consumption	If the network is dense then we cannot use this scheme.

3	Becoan update strategy	Save the energy and bandwidth	If the network is dense then sometimes it is difficult.
4	Patrol seeking mode	Packet delivery ratio increased,Also increase end to end delay	Length of the path for communication is become longer than standard GPSR .
5	Buffer zone based greedyforwarding strategy (BZGFS)	The packet delivery ratio is increased.	When mobility is very high than it is difficult.
6	Left and Right both rules	Increase the packet delivery ratio , and short the routing path	For highly scalable network it consumes more time

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

(Proceedings available for download at rku.ac.in/icre)

RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

Pictorial Presentation for Thermodynamic Analysis of a 77 MW Power Plant by use of Pi-Chart

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ABSTRACT

Cogeneration is a technique of energy conservation that involves the production of two forms of energy heat and work from a single thermal power plant. Main purpose of this paper is to present energy and exergy analysis of 77MW cogeneration steam power plant by use of Pi-Chart. A case study has been done at the Vadinar Power Company Ltd. (VPCL) in Jamnagar, India. Energy and Exergy analysis for all the thermal energy systems of power plant are presented with use of Pi-Chart. The energy loss mainly occurred in the condenser about 45.5% is lost into environment while main exergy loss occurred in the steam boiler with 80.65%. During combustion process large amount of entropy generates in the combustion chamber of the boiler and this is the major source of exergy destruction.

SUMMARY

This research mainly focuses on Energy and exergy analysis of cogeneration power plant ant various losses are presented by using Pi-Chart.

Keywords: Energy,Exergy,Power plant

INTRODUCTION

For the purpose of energy conservation and management, Cogeneration is most energy efficient technology as compared to other conventional power plant for heat and power generation with the use of single power plant. Now a days exergy becomes more popular tool for design, assessment, and

improvement of the existing power plant systems. Base of exergy analysis is Second Law of Thermodynamics and base of energy analysis is First Law of Thermodynamics.

For this purpose numerous researchers have suggested that exergy analysis can be used to help decision making for ecological consideration in place of energy analysis (*1, 2 & 3*). Exergy analysis found as useful tool in the design optimization, estimation, and up gradation of thermal power plants systems. It determines the value, place & reasons for irreversibility in the power plant, but also gives more significant evaluation of plant individual element efficiency (*4, 5*). Kanoglu and Dincer made comparison for various cogeneration power plant for their energetic & exergetic efficiency (*6*). T. Ganapathy presented evaluation of the exergy losses in the sub systems and for entire power plant (*9*). In the research of Kamate, boiler was the main system causative exergy loss in the power plant (*8*).Mehmet Kopac et al. presented the exergy investigation for the determination of the effect of reference ambient temperature on the efficiency of sub systems and entire power plant (*10*).

The most important objective of this paper is to examine VPCL plant from an energetic & exergetic perspective such as component wise energy losses and exergy destruction rate have been calculated and presented with use of Pi-Chart.

MATERIALS AND METHODS

I.DESCRIPTION OF COGENERATION POWER PLANT

Vadinar Power Company was firmed in the year of 1998 along with refinery to manage inner steam & power necessity. The capacity of the plant is 120MW equivalent175TPH*3 oil fired boilers, 38.5MW*2 steam turbines, feed water and steam supply to refinery. The industrial data for the plant is given in TABLE-I.

II.METHOD FOR ANALYSIS

By using the data obtained from both plants managers and field inspectors, process flow diagram has been made including major components such as boiler (combustion chamber, economiser, super heaters), steam turbine, condenser, dearetor, SCAPH, turbo drives (BFP, CFP, AEP).Following assumptions have been made for the assessment of power plant.

- Changes in Potential & Kinetic are neglected.
- Reference state of environment is pressure 1.01325 bar and temperature 25℃.

Thermodynamic analysis has been done based on First law of thermodynamics and second law of thermodynamics For a steady state flow process, with the use of mass balance, energy balance and exergy balance energy loss, exergy destruction can be evaluated.

$$\Sigma \dot{m}_{in} = \Sigma \dot{m}_{out} \quad (1)$$

$$Q + \dot{W} = \Sigma \dot{m}_o \; h_o \; - \Sigma \dot{m}_{ii} \, h_{ii} \quad (2)$$

$$\dot{E}_{x,he} \; - \dot{W} = \Sigma \dot{E}_{x,o} \; + \Sigma \dot{E}_{x,ii} \; + \; \dot{I}_D \quad (3)$$

Where subscripts ‘in’ and ‘out’ referred to streams at inlet and outlet from the control volume, respectively. The exergy destructionand exergy loss are referred as$\dot{I}_D$and Ėx respectively.

$$\dot{E}_{x,h} \; = 0 \text{ (For single components)} \quad (4)$$

Exergy of the system is

$\dot{E}_x = \dot{E}_{x,p} + \dot{E}_{x,c} + \dot{E}_{x,k} + \dot{E}_{x,p}$ (5)

Where Ėxph, Ėxch, Ėxkn, Ėxpeare physical exergy, chemical exergy, kinetic exergy& potential exergy.(Bejan et al.1996) Exergy expressions for different energy stream are as under:

For Steam, $\dot{E}_x = \dot{m}[(h - h_0) - T_0(S - S_0)]$ (6)

For Liquid fuel, $\dot{E}_x = \Psi \,.\, LHV$ (7)

Where Ψ= Exergy factor=0.9 for furnace oil

For a Flue Gas, $\dot{E}_x = \dot{E}_{x,p} + \dot{E}_{x,c}$ (8)

$$= \dot{m}\Sigma x_i \left[Cp^h(T - T_0) - T_0 Cp^s \ln\left(\frac{T}{T_0}\right)\right] + RT_0 \ln\left(\frac{P}{P_0}\right) + \dot{m}\Sigma\left[x_i e_x^c + RT_0 x_i \ln x_i\right]$$

Exergy destruction rate (İD) of each component for the cogeneration plant can be found as follows:

For Boiler :

$\dot{I}_B = \dot{E}_{x,f+}\Sigma\dot{E}_{x,i\imath,b} - \Sigma\dot{E}_{x,o\ ,b}$ (9)

For Steam turbine and Turbo drive:

$\dot{I}_T = \Sigma\dot{E}_{x,i\imath,t} - \Sigma\dot{E}_{x,o\ ,t} - \dot{W}_t$ (10)

For Condenser :

$\dot{I}_C = \Sigma\dot{E}_{x,i\imath,c} - \Sigma\dot{E}_{x,o\ ,c}$ (11)

For SCAPH :

$\dot{I}_S = \Sigma\dot{E}_{x,i\imath,s} - \Sigma\dot{E}_{x,o\ ,s}$ (12)

For Dearetor :

$\dot{I}_{D/A} = \Sigma\dot{E}_{x,i\imath,d} - \Sigma\dot{E}_{x,o\ ,d}$ (13)

RESULTS AND DISCUSSION

For the purpose of analysis, operating data at inlet and outlet condition of each component of plant at different state are taken as shown in TABLE-II. The energy efficiency of cogeneration plant is calculated 68.90%. This efficiency was based upon LHV of supplied fuel to incorporate losses taking place in furnace of boiler system due to energy lost with exhaust gases, deficient combustion, radiation and convection losses, etc.

In the energy analysis point of view, highest energy loss takes place into the condenser. It contributes about 45.5% of total energy loss because of rejected heat by condenser is exhausted directly into atmosphere which is totally useless. These losses of energy can be large in quantity but these losses are thermodynamically not important due to low quality.

In the exergy analysis point of view, maximum exergy destruction takes place in steam boiler system with 80.65% of total exergy destruction of plant, while the exergy destruction in condenser is 0.7% only. However, the major loss takes place in the boiler system where entropy generated. Boiler operates with 40.08%exergetic efficiency only. The exergetic efficiency of cogeneration plant is calculated 34.69% which is higher than conventional power plants.

CONCLUSION

Energy conservation and efficient use of energy becomes a major challenge for developing countries. For this, energy system must be analyzed to measure its performance criteria.A case study for energy and exergy analysis of cogeneration plant is carried out at VPCL,in Jamnagar. From the analysis result, steam condenser is the major source of energy loss while steam boiler is contributing major exergy loss as compared to other energy systems. Exergetic efficiency of boiler is 40.08%..Plant operates with thermal efficiency of 68.90% and exergetic efficiency of 34.69%.

FIGURES

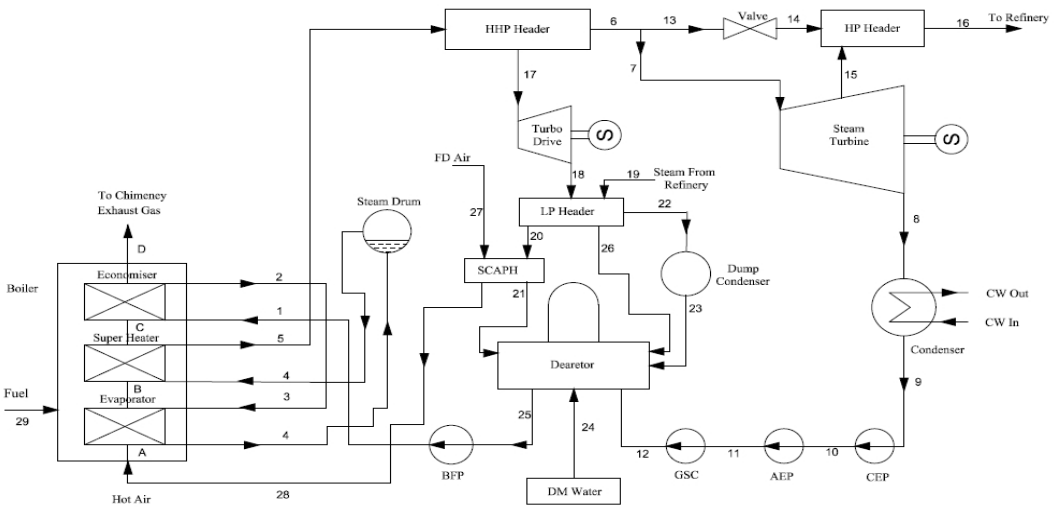


Fig. 1 77 MW Cogeneration Steam Power Plant diagram.

AEP:Air Extraction Pump ,CEP: Condensate Extraction Pump,BFP:Boiler Feed Pump ,GSC:Gland Steam Condenser

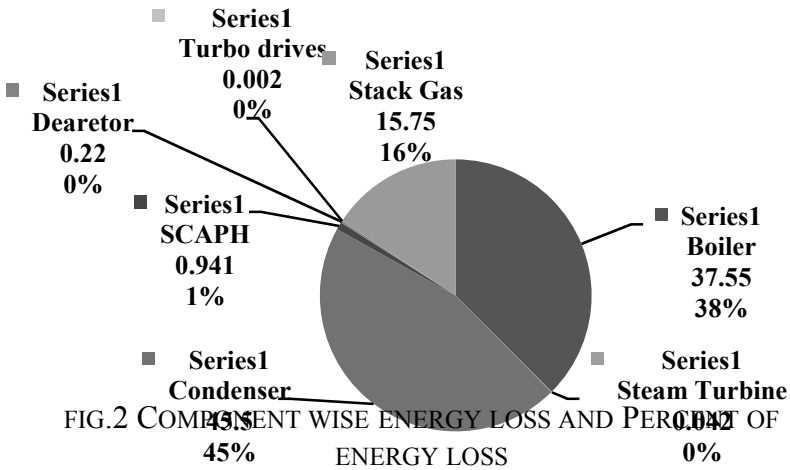


FIG.2 COMPONENT WISE ENERGY LOSS AND PERCENT OF ENERGY LOSS

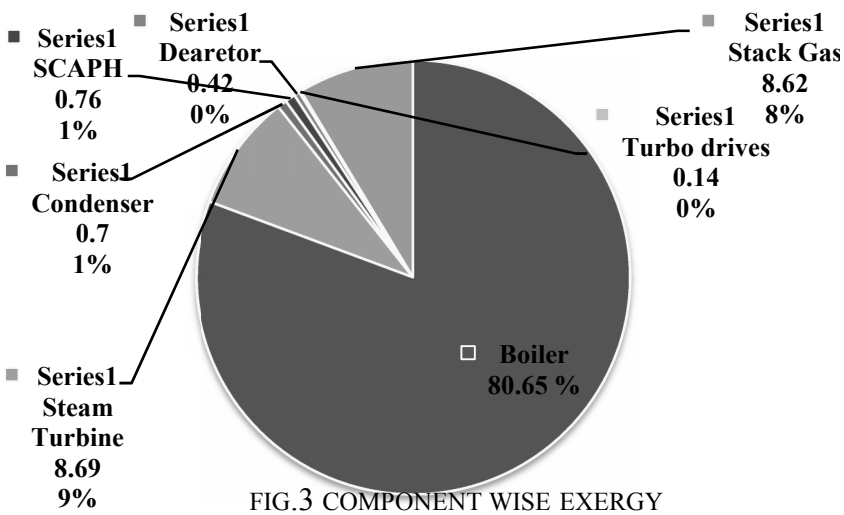


FIG.3 COMPONENT WISE EXERGY DESTRUCTION, PERCENT OF EXERGY DESTRUCTION

TABLES

TABLE-I
Plant Technical Specifications

Parameters	Value
Output Power	77 MW
Steam generation Rate	230 TPH
Temperature ,Pressure, & Mass flow rate of main steam	453˚C/61.75 bar/486 kg/s
Temperature ,Pressure, & Mass flow rate of Extracted steam	407˚C/ 40.15 bar/44.72 kg/s
Condenser temperature and pressure	39˚C /0.062 bar
Temperature of flue gas	190˚C
Type of Fuel	Furnace oil
LHVof fuel	10,000 kcal/kg

TABLE II
Value of thermodynamics properties at different state

State	Pressure (bar)	Temp (C0)	Mass (kg/S)	Specific Enthalpy (kJ/kg)	Specific Entropy (kJ/kg)	Specific Exergy (kJ/kg)	Exergy (MW)	Energy (MW)
1	61.751	152	135	646	1.52	192.936	26.05	87.21
2,3	61.751	237	135	1007.25	1.975	418.596	56.51	135.98
4	61.751	277	135	2783	5.88	1029.166	138.93	375.705
5	61.751	453	135	3380	6.6	1343.096	190.78	456.3
6	61.751	453	135	3380	6.6	1343.096	190.78	456.3
7	61.751	453	95.56	3380	6.6	1343.096	135.03	322.99
8	0.062	39	50.84	2569	8.315	91.026	4.652	131.30
9	0.062	39	51.84	165.75	0.532	7.11	0.36	8.47
10	7.563	40	51.108	170	0.5431	8.0522	0.41	8.69
11	7.563	41	51.108	174.25	0.5549	8.7858	0.45	8.91
12	1.169	42	51.108	178.5	0.57	8.536	0.44	9.12
13	61.751	453	36.67	3380	6.6	1343.096	49.251	121.38
14	40.155	407	36.67	3260	6.8	1233.496	45.23	119.54
15	40.155	407	44.722	3260	6.8	1217.336	55.16	145.79
16	40.155	407	81.39	3260	6.8	1233.496	100.39	265.33
17	61.751	453	2.78	3380	6.6	1343.096	3.9284	9.397
18	4.463	200	2.78	2880	7.12	758.136	2.11	8.00
19	4.463	200	34.73	2880	7.12	758.136	26.33	100.02
20	4.463	200	6.945	2880	7.12	758.136	5.27	20.00
21	1.472	110	6.945	462.8	1.423	38.642	0.27	3.21
22	4.463	200	13.89	2880	7.12	758.136	10.53	40.00

23	4.463	145	13.89	616.25	1.4743	176.8046	2.46	8.565
24	2.098	52	77.5	218.4	0.672	18.04	1.40	16.93
25	1.289	106	144.5	450.4	1.21	89.716	12.96	65.08
26	2.48	110	13.89	2860	7.4	654.696	9.09	39.73
27	1.031	35	202.942	34.84	---	0.84	0.17	7.07
28	1.031	115	202.942	115.01	---	15.82	3.21	23.34
29	Fuel	---	11.67	41488.43	---	37340.19	435.76	484.17
30	2.58	30	1500	130.05	0.428	2.402	3.603	195.075
31	2.58	34.3	1500	145.78	0.474	4.424	6.636	218.67
A	1.044132	1325	214.622	1313.93	---	922.56	241.71	344.25
B	1.02453	700	214.622	638.931	---	392.56	102.85	167.41
C	1.01874	500	214.622	430.534	---	254.695	66.73	112.8
D	1.0128	200	214.622	155.73	---	101.76	26.66	40.88

ACKNOWLEDGEMENT

The authors gratefully acknowledge to Mr. K.B.Makadia Head of Vadinar power company Ltd for allowing us to conduct above study by using operating data obtained from the plant and also appreciate the plant management and engineers for their co-operation throughout this research.

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DESIGN AND ANALYSIS OF TWO-DIMENSIONAL LINEAR COMPRESSOR CASCADE FLOWS

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ABSTRACT

Axial flow compressor is used to get the compressed pressurized air as an input the gas turbine. Generally it is used in the aircraft engine and the industrial application. Compressor is composed with the rotor and stator blades at each stage. Aerodynamic shapes of the blades play important role to improve the efficiency of the compressor. The objective of work presented is to design Axial flow compressor by using mean line method for a given mass flow rate and required pressure ratio.The parameters determined also include thermodynamic properties of the working fluid, stage efficiency, number of rotor and stator blades, tip and hub diameters, blade dimensions (chord, length and space) for both rotor and stator, Mach number, flow and blade angles (blade twist).The same parameters are also determined for all five stages. Then the work analysing the flow behaviour through a compressor cascade with the help of Computational Fluid Dynamics using ANSYS FLUENT.

SUMMARY

Aerodynamic Design of Axial Flow Compressor for small scale gas turbine engine with assuming some specified data.

Keywords:ANSYS FLUENT, ANSYS WORKBENCH, MATLAB

INTRODUCTION

The axial flow compressor being used in aircraft gas turbine power plant. Although in past turbojet engines incorporated the centrifugal compressor, but at recent trend use axial flow compressor for high speed and long range application. Thisdominance is a result of the ability of the axial-flow compressor to satisfy the basic requirements of the aircraft gas turbine. For aircraft gas turbine basic requirements of

compressors include high efficiency, high airflow capacity per unit frontal area, and high pressure ratio per stage. For wide range operation and rapidly acceleration and for high level of aerodynamic performance. Compressor should have minimum length and weight. Its design should be simple so we can reduce manufacturing time and cost. Its structure should be mechanically rugged and reliable. Mean line prediction calculation, through flow calculation, and blading procedures are the different aerodynamic compressor design processes. The mean line prediction very simple one dimensional calculation of flow where measured all dimension at mid height line of compressor and it is the first step within compressor where global parameters as the annulus geometry, the number of stages, and the stage pressure ratios are scaled (1).

The mean line process is very quick and reliable method for compressor design. The preliminary process results are obtained by time consuming manual parameter studies based on engineering intuition or experience. The final one dimensional solution is used as an initial guess for the subsequent design process, e.g. for through flow calculations (2).

Analytical Design Process

The steps which followed in design of axial flow compressor is Shown in figure 1. Suitable design point under sea level staticconditions [3], given overall pressure ratio is 3 and Air mass flow rate is 0.8 kg/sec.and Axial velocity Ca= 130 m/s considered.

Stagnation and static properties can be found by simple thermodynamic equations and the Enthalpy-Entropy diagram which is shown in figure.2 Once P0, T0 and P, T at entry and exit have been found.

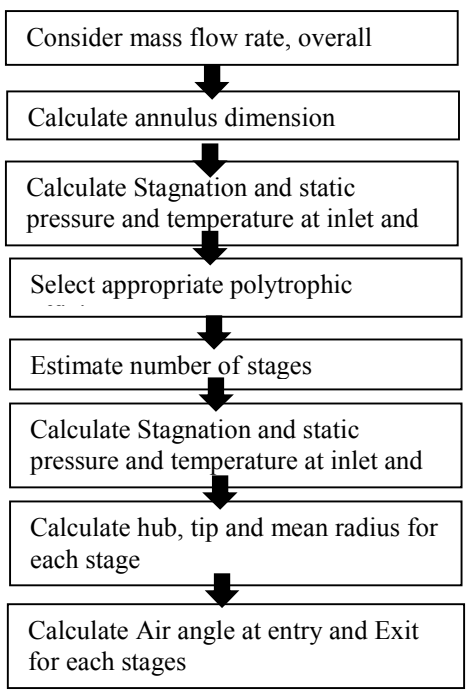


Figure1:Aerodynamic Design input and Steps

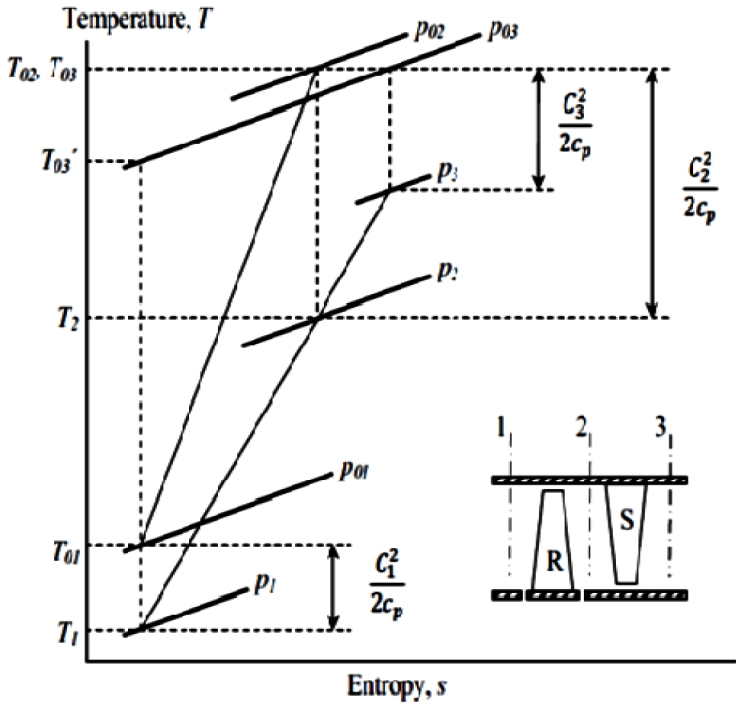


Figure 2: CompressorT-S diagram (4)

1.3 Formula used in calculation

1.The area of axial flow compressor

$$A = \frac{m\sqrt{T_t}}{P_t M_n \sqrt{\frac{\gamma}{R\left(1+\frac{\gamma-1}{2}(M_n)^2\right)^{\frac{\gamma+1}{\gamma-1}}}}}$$

2.Density at inlet

$$\rho_0 = \rho_1 \left[1 + \left(\frac{\gamma-1}{2}\right) M^2\right]^{\frac{1}{\gamma-1}}$$

3.Absolute Axial velocity

$$C_a = \frac{m}{A*\rho}$$

4.Tip radius

$$R_t = \sqrt{\frac{A}{\pi\left[1-\left\{\frac{h}{t}\right\}^2\right]}}$$

5. Hub radius

$$R_h = \frac{h}{t} R_t$$

6.Blade height

$$Hb = Rt - Rh$$

7.Compressor efficiency

$$\eta_c = \frac{P^{\frac{\gamma-1}{\gamma}} - 1}{P^{\frac{\gamma-1}{\gamma\eta_p}} - 1}$$

8. Mean Relative Velocity

$$U_m = \frac{C_a}{\Phi}$$

9. Absolute tangential velocity

$$C_t = C_a * \tan\alpha$$

10. Relative tangential velocity

$$W_t = U_m - C_t$$

11. Rotation speed

$$N = \frac{U_m * 30}{\pi * R}$$

12. Mach Number

$$M = \frac{C}{\sqrt{\gamma}}$$

13. Relative Air angle

$$\beta = \tan^{-1}\frac{W_t}{C_a}$$

14. Absolute Air angle

$$\alpha = \tan^{-1}\frac{W}{U_m * C_a}$$

As from above calculation using MATLAB software find out the result data as following

Table 1: Result of Calculation.

Stage	Area (cm^2)	Hub Radius (cm)	Tip Radius (cm)	Blade Height (cm)	Mean Radius (cm)	Relative Air angle at entry (°)	Absolute Air angle at exit (°)
1	59.2407	5.7899	7.2374	1.4474	6.5538	51.34	46.70
2	55.8315	5.8829	7.2374	1.3545	6.5957	51.51	48.55
3	47.4331	6.1058	7.2374	1.1315	6.6956	51.93	50.14
4	40.3392	6.2880	7.2374	0.9493	6.7793	51.28	51.80
5	34.3388	6.4381	7.2374	0.7799	6.8494	52.56	53.48

1.4 Analysis model

Based on calculated dimension, cascade geometry of different rotor and stator blade done in workbench FLUENT software in design modeler.We have done coupled field analysis using different mesh size and found with the increase in mesh density pressure distribution and flow separation increase. Then wehave done triangular meshing with specified element size.in meshing 32903 nodes and64243 elements generate.

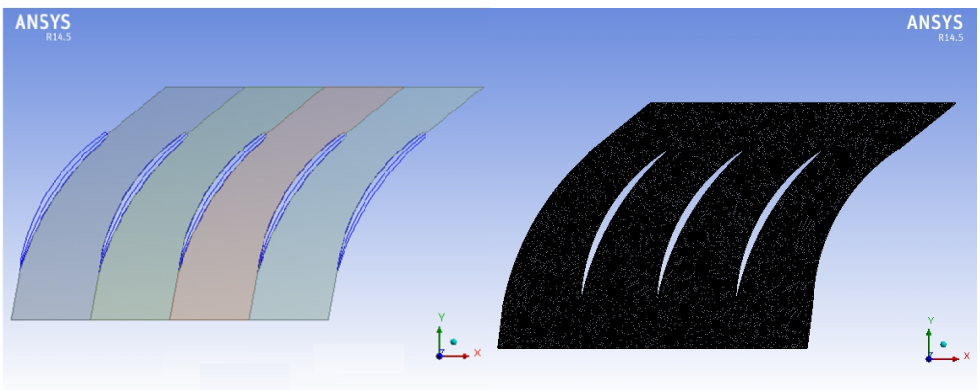


Figure 3: Cascade Geometry of BladeFigure 4:Meshing of Blade

In our problem, the boundary conditions given to our two dimensional linear compressor cascade flow are shown as follow:

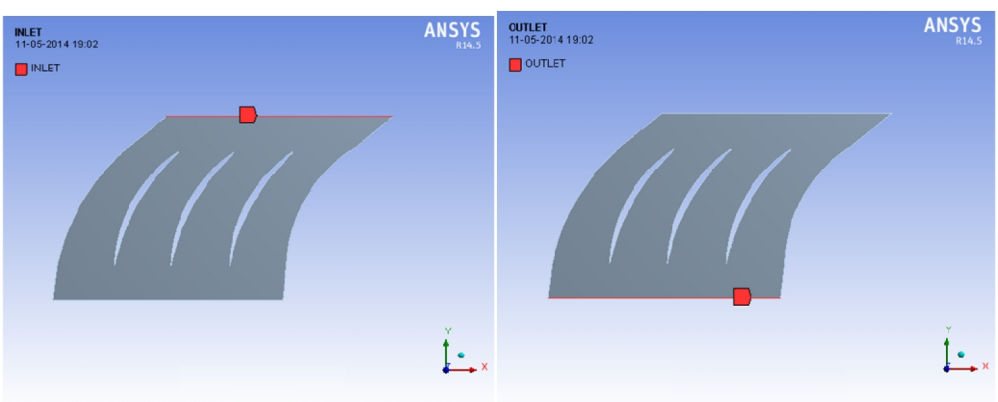
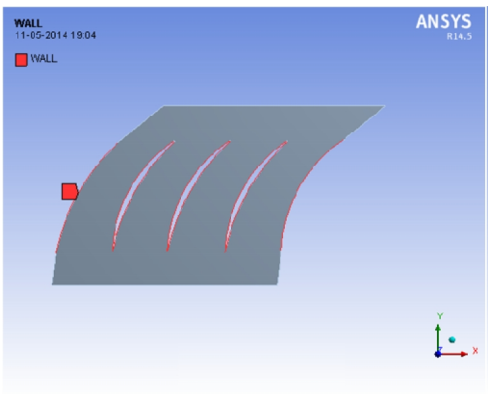


Figure 5: Velocity Inlet Boundary Condition Figure 6:Pressure Outlet Boundary Condition



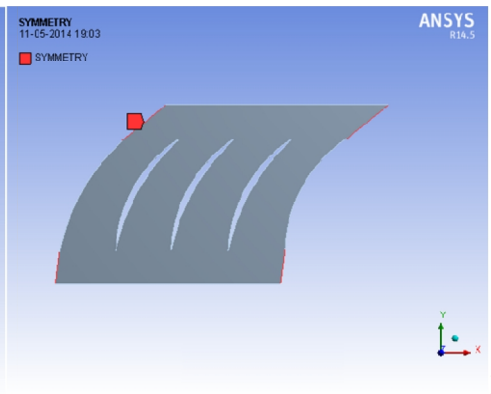
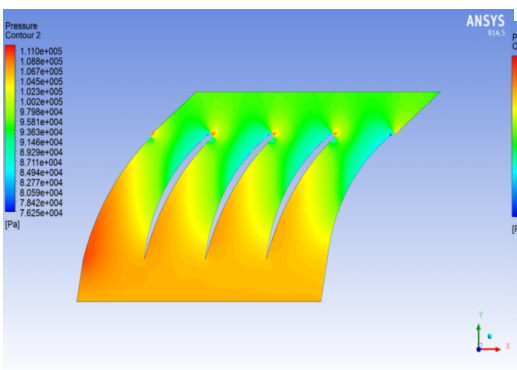


Figure 7: Wall Boundary Condition Figure 8: Symmetry Boundary Condition

CFD Analysis

Contour Plot of Pressure Analysis



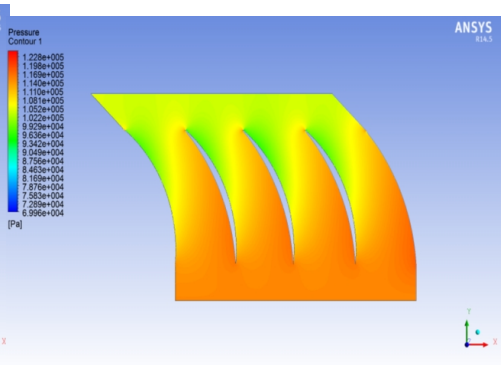


Figure 9: Contour Plot of Pressure for Rotor Stage 1 at β1=51°& β2=10° Figure 10: Contour Plot of Pressure for Stator stage 1 at α2=46°&α3=0°

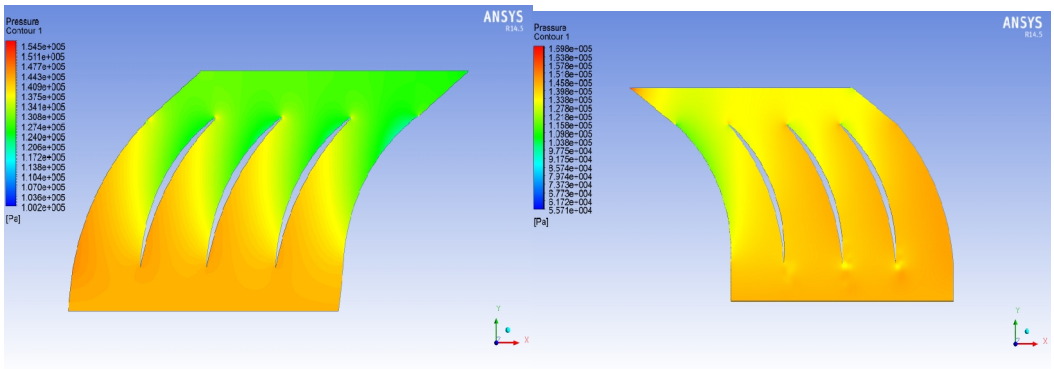


Figure 11: Contour Plot of Pressure for RotorFigure 12: Contour Plot of Pressure for Stator Stage 2 at β3=51°& β4=7° stage 2 at α4=48°&α5=0°

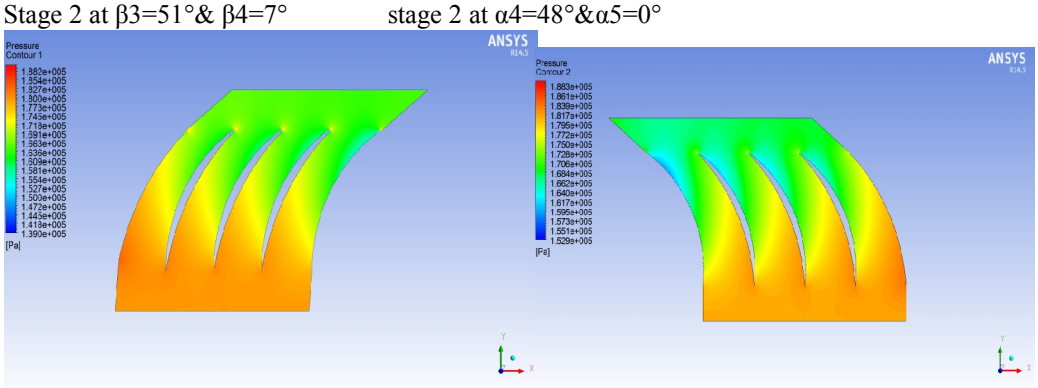


Figure 13: Contour Plot of Pressure for RotorFigure 14: Contour Plot of Pressure for Stator Stage 3 at β5=51°& β6=4° stage 3 at α6=50°&α7=0°

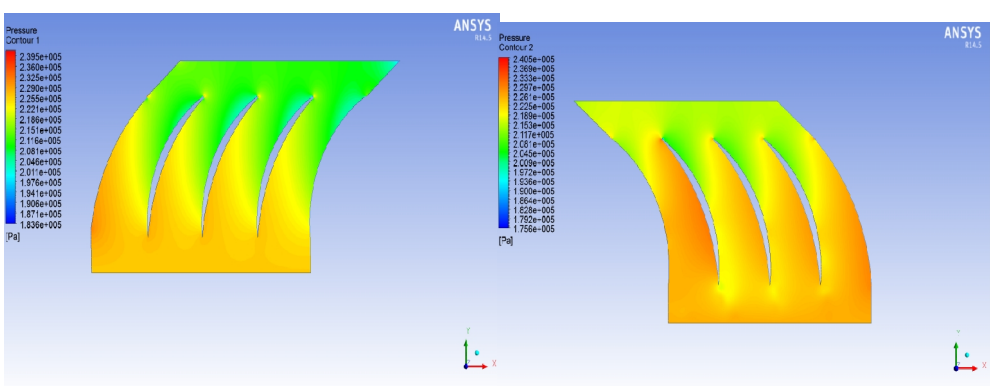


Figure 15: Contour Plot of Pressure for RotorFigure 16: Contour Plot of Pressure for Stator Stage 4 at β7=52°& β8=2° stage 4 at α8=51°&α9=0°

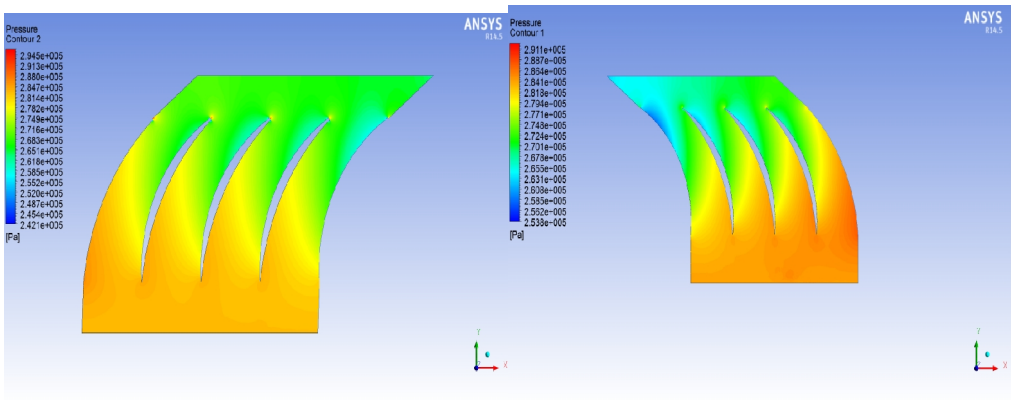


Figure 17: Contour Plot of Pressure for Rotor Stage 5 at β9=53°& β10=1°

Figure 18: Contour Plot of Pressure for Stator stage 5 at α10=53°&α11=0°

RESULTS AND DISCUSSION

By analyzing these CFD Analysis for different stages of axial flow compressor we canconclude that pressure on compressor blade increases towards its tail with decreasein velocity of air which is also seen in the calculated results of chapter no 4, it is alsoObserved that minor flow separation occurs towards tail of rotor and stator blades.From these, we can say that cascade geometry prepared on basis of the calculateddata is matching with these CFD analysis results.

The Analytical value and software value of total Pressure is as below
Analytical result pressure rise = 198961
Software result pressure rise =187682
Pressure Variation between both result is = 5.06%.

CONCLUSION

In this paper an axial flow compressor design process has been studied. Also the efficiency optimization of single stage and multistage axial-flow compressor has been studied using different flow-theory. With available data i.e. Mass flow rate, Pressure ratio and Pressure at given Altitude. The Blade profile has been generated for both Rotor and Stator analytically.The other possibility of flow separation is also checked and observed that minor flowseparation takes place towards tip of both stator and rotor. Calculation spread sheetis prepared, so by putting values one can get required parameters to generate blade geometry. Analytical design results are validated with CFD analysis results by pressure contour, velocity profile and velocity vector and it is observed that the CFD analysis results are differing by 5% from analytical results, which is under acceptable range.Thus this two dimensional cascade geometry gives matched results for calculated dataand CFD analysis.

FIGURES

Fig.1. Aerodynamic Design input and Steps
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Fig.3.Cascade Geometry of Blade
Fig.4. Meshing of Blade
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Fig.10. Contour Plot of Pressure for Stator stage 1 at α2=46°&α3=0°
Fig.11. Contour Plot of Pressure for Rotor stage 2at β3=51°& β4=7°
Fig.12. Contour Plot of Pressure for Stator stage 2 at α4=48°&α5=0°
Fig.13. Contour Plot of Pressure for Rotor stage 3 at β5=51°& β6=4°
Fig.14. Contour Plot of Pressure for stator stage 3 at α6=50°&α7=0°
Fig.15. Contour Plot of Pressure for Rotor stage 4 at β7=52°& β8=2°
Fig.16. Contour Plot of Pressure for stator stage 4 at α8=51°&α9=0°
Fig.17. Contour Plot of Pressure for Rotor stage 5at β9=53°& β10=1°
Fig.18. Contour Plot of Pressure for Rotor stage 5α10=53°&α11=0°

TABLES

Table 1. Result of Calculation.

ACKNOWLEDGEMENT

First and foremost I offer my sincerest gratitude to my mentor, supervisor Prof.Vinal.G.Virani, who has supported me throughout my research work with his patience and knowledge whilst allowing me the room to work in my own way. I would also like to thanks Dr.KartikD.Kothari, Head of Department, Mechanical Engineering, to help me in doing this project. I am also thankful to all the other staffs of workshop, library and department for their cooperation, help and guidance provided me for my research work. I am also thankful to my all colleagues for building good team work to make the success of my work. Last but not the least; I would like to thank God Almighty, my parents, my family members and friends for their love, support and excellent co-operation to build my moral during the work.

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Partial Discharge Detection Techniques in Power Transformer

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ABSTRACT

Partial Discharge (PD) occurring in the insulation systems of transformer are an important indicator of their deterioration. Insulation degradation is a well-known source of power transformer failure. Many methods have been realized to measure PD, including electrical, chemical, acoustic and UHF methods. A survey of current research reveals the continued interest in the application of advanced techniques for partial discharge diagnostic in transformer. This paper conducts a literature survey and reveals general backgrounds of research and developments in the field of PD diagnostic. Physics of PD has been briefly explained to get insight of PD phenomena. Different techniques used for PD detection and measurement have been explained and compared.

SUMMARY

This paper highlights history and current trends in partial discharge detection techniques and its comparison.

Keywords: Partial discharge, Acoustic,UHF

INTRODUCTION:

Power transformers represent the largest portion of capital investment in power system. In 2010 India had a total market share of 20.2%, during the forecast period 2011-2020, India's market size is expected to increase at a Compound Annual Growth Rate (CAGR) of 12.5% (*1*). Reliability of transformer affects

economical operation of utility. Normally, faults in power transformers are caused by the decreasing of dielectric strength and dielectric breakdown during operation due to the cumulative effect of the thermal, electrical and mechanical stresses (*2*). Dielectric breakdown in transformers is most frequently initiated by partial discharges (*3*). The consequences of these types of occurrences can be hazardous if not detected in a timely fashion. Regular PD analysis and bushing monitoring give an accurate indication of the status of the deterioration process. So it is possible to foretell developing fault condition by online monitoring and precautionary tests. It is very much essential to have information of PD level and location to plan maintenance of electrical equipment.

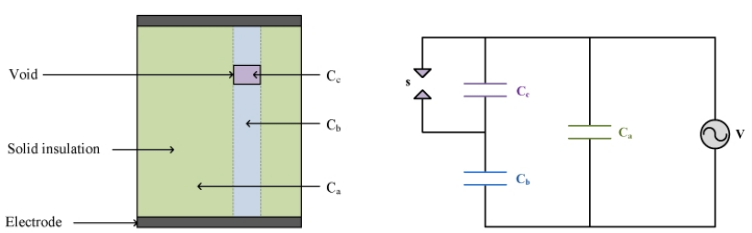
A famous method of understanding the health of the transformer is by studying the partial discharge signals. Monitoring of transformer can be either online or offline. The primary established techniques for electrical PD detection by measuring current or Radio Frequency (RF) pulses, as detailed in the standards (*3*), are performed when the transformer is off-line and preferably within a shielded enclosure to eliminate electrical interference. Suppression of interference is one of the main challenges in detecting PDs, either while the transformer is off-line or on-line in a noisy environment. The off-line PD detection methods only provide snapshots in time of part of the transformer's condition. On the other hand, no standards have yet been developed for on-line electrical monitoring of PDs.

PARTIAL DISCHARGE BASICS:

The term "Partial Discharge (PD)" is defined by International Electro technical Commission (IEC) 60270 (Partial Discharge Measurements) as a localized electrical discharge that only partially bridges the insulation between conductors and which may or may not occur adjacent to a conductor (*4*). A full discharge would be a complete fault between a conductor at line potential and ground. The pulses occur at high frequencies; therefore, they attenuate quickly as they pass through a short distance. The discharges are effectively small sparks occurring within the insulation system, therefore deteriorating the insulation, and can eventually result in complete insulation failure (*5*).

Physics of Partial Discharge:

A partial discharge in HV transformer occurs when the electric field in a localized area exceeds the breakdown strength. This localized breakdown manifests itself as an electrical pulse. The most likely sources of PD are floating components, corona, and voids. However, the detection of PD created by floating components and corona does not yield any useful information about the insulation because their appearance is not directly related to the condition of the insulation. Gas bubbles in liquid dielectric and cracks in solid dielectric are defined as voids.



(a) Physical modeling of void (b) Equivalent circuit

Fig. 1 PD Model

The void region has lower dielectric constant and because of that it has different capacitance value in comparison to surrounding material. PD occurs when electric field across void exceeds breakdown field strength. Two criteria must be fulfilled for occurrence of partial discharge, one is availability of free electron within volume and second is higher electric field across void. If above said both conditions are met, than current streamer development causes current flow across void. This phenomenon may take few minutes to few hours to reach breakdown field strength. Due to this reason it is highly impulsive phenomena. Gemant Philipoff's 'a-b-c' model (*7*), is the most accepted model for studying the behavior of internal discharges at AC voltage.

As shown in Figure 1, in this model, the cavity is represented by a capacitance. Model uses three capacitances, C_c for the void, $C_b$for the part of dielectric in series with the void and $C_a$for the remaining sound insulation.

A free electron in gap is accelerated by externally applied electric field. Free electrons get multiplied in the gap due to ionisation by collision. New free electron again accelerated in the gap due to electric field and produces free electron. The number of free electron increases and electron avalanche takes place as this process is cumulative. The electron avalanche produces streamers, as it grows sufficiently large.

A relationship between the discharge and the physical phenomena was initiated by Kreuger (*8*). He suggested that the discharge may be given as a measure of q in the form

$$q \cong 9 \cdot \varepsilon \cdot A \cdot \Delta V \cdot \left(\frac{1}{d}\right) \quad \text{.....(1)}$$

Where q is the dielectric constant of the insulation, A is the area of the discharge site in square millimetres, V is the applied voltage in KV and d is the insulation thickness in millimetres.

The drawback in the use charge (q) to quantify discharge is the fact that it is inversely proportional to the insulation thickness. Hence, requiring higher voltages to initiate breakdown in small voids during physical modelling. Nevertheless, q is said to be a useful indication of the charges, since it is directly related to the energy in the discharge and the size of the defect and can be readily measured by a discharge detector. Meek (*9*) proposed a single quantity criterion to estimate the electric field that transforms an avalanche into a streamer.

$$E_r = 5.27 \times 10^{-7} \cdot \left(\frac{\alpha \cdot e^{\alpha x}}{\sqrt{x/p}}\right) V/cm \quad \text{.....(2)}$$

Where E_r is space charge field at radius r, α is Townsend's first ionization coefficient, x is the distance to which the streamer has extended in the gap and p is the gas pressure in torr.

Bartnikas (*10*), has cited a method to determine the inception or critical breakdown voltage of cavities for air voids in solid insulation. This formula, as shown below indicates that the critical breakdown (E_{cb}) of voids can be given by the relationship between its size in relation to the insulation structure, as well as the inception point of the applied voltage (V_i) and the relative permittivity (ε_r). Hence,

$$V_i = E_{cb} \cdot t \left\{1 + \frac{1}{s_r}\left(\frac{d}{t} - 1\right)\right\} \quad \text{.....(3)}$$

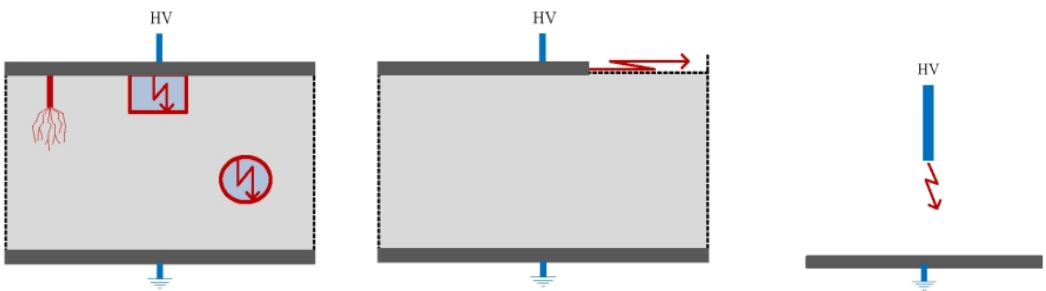
Where d is thickness of insulation and t is thickness cavity.

Van Brunt (*11*), outlines that surface emission process causes free electron in the gap. In surface emission, mainly field emission and field induced ejection of surface charge produce free electron. Other possible process to generate free electron are ionization of gas by radiation and electron bombardment.

Niemeyer (*12*), stated that the relationship between electron emission intensity and external electric field is exponential. It depends on surface roughness, polarity, material and contamination on the cavity. The generation of free electrons depends on the time difference between a discharge and the subsequent one. If time lag between discharges is long than it leads to decay discharges by products due to different factors like recombination, ion drift in field and diffusion(*6*).

Classification of PD:

According to the literature, partial discharges are classified into three categories depending on their location and mechanism; namely, internal discharge, surface discharge, and corona discharge. Internal PD occurs in cavities of dielectric; surface PD occur at the surface of a dielectric; corona discharges occur in the non uniform field around a sharp point of an electrode.



(a) Internal Discharge (b) Surface Discharge (c) Corona Discharge

Fig. 2 Types of Partial Discharge

Fig. 2 illustrates the three basic classes of partial discharge.

Internal Discharge:

Internal discharges occur in cavities wholly within a solid dielectric material or bounded at one side by an electrode as shown in Fig. 2(a). The original PD in the cavity has caused development of an electrical tree. Discharge in the electrical tree may also be considered as an internal discharge. The internal PD usually results from a high electrical stress across a small void or air gap causing ionization of gas in the void. Voltage gradient results in the high electrical stress (*5*). The electrical stress in the gas void is amplified by the ratio (ε_{r_solid} /ε_{r_air}) of the dielectric properties.

Surface Discharge:

Discharges at the boundary of two substances are commonly called surface discharge. The surface discharge phenomenon is illustrated in Fig. 2(b). Large stress component parallel to the

dielectric surface causes this type of discharge. The large stress component may appear at the edge of the high voltage electrode. The addition of contamination and moisture may initiate and support the surface discharge phenomena.

Corona Discharge:

Corona discharges occur around the sharp point or edges of the high voltage electrodes in presence of strong in-homogeneous electric fields. Corona discharges can also occur at sharp protrusion from a conductor at early potential or floating material, such as small-thin wire or loose bolt.

PARTIAL DISCHARGE DETECTION:

The measurement and location of partial discharge phenomena has been of interest since at least the early 1940's (*13*). Recording facility was not feasible in early days because of the apparatus and technology limitation. In last four decades several methods have been developed to detect PD within power transformer on the basis of long historical development.

Year	Development
1777	First time *Lichtenberg* has demonstrated surface discharge by dust figures
1873	*Maxwell* published his work on physical model and instrumentation design of PD phenomena
1896	*Hertz* conducted experiments on electromagnetic wave propagation in space and time
1919	*Schering* developed loss factor bridge which is the first device used for PD detection
1925	*Schwaiger* recognized corona discharges
1928	*Lloyd and Starr* displayed PD process on Braun tube
	Byrstlyn has introduced equivalent circuit for PD loss. With this concept *Gemant & Philipoff* introduced very famous 'a-b-c' model for PD (Figure1)
1954	*Mole* has designed first commercial edition of PD detector having frequency band below 1 MHz.
1978	*Tanaka and Okamoto* developed computer based system for PD pattern analysis
1980	*Lekme* designed wide band PD detectors

Table 1: Historical Development in PD detection Technique

Later on new development added to improve sensitivity by addressing problems of external interferences. Nowadays there is a growing trend in development of PD detection and analysis techniques due to availability of powerful computer and computational techniques. Present research trend is online PD detection, development of noise rejection techniques, reliability improvement, advanced PD sensor

development and PD source location identification with different algorithms. Simultaneously wide ranges of software programs are also available for PD data handling and analysis.

It is well known that the occurrence of discharge results in discharge current or voltage pulse, electromagnetic impulse radiation, ultrasonic impulse radiation and visible or ultraviolet light emission. Accordingly, there are several detection methods that have been developed to measure those phenomena respectively.

- Electrical detection technique
- Chemical detection technique
- Acoustic detection technique
- UHF detection technique

Electrical Detection:

Current streamer generates electrical pulses in the void. Electrical detection method targets capturing of these electrical pulses (*14*). Electrical PD detectiontechniques are more user friendly, standardized, vulnerable, effortless implementation compared to other technique. An electrical detection system is efficient to measure the internal discharge, surface discharge and corona discharge. Signal intensity, pulse shape and its relative phase location within AC cycle carries information of type and severity of PD discharge. The electrical PD detection methods can be further divided into two different groups: intrusive and non-intrusive methods. To detect PD pulses by intrusive method, sensing element needs to be put inside the power equipment. In contrast, with the non intrusive technique, the sensor is located outside the equipment. Correspondingly, the most commonly used two techniques in the field power transformer PD testing are direct probing and RF emission testing. Capacitive coupler used in direct probing method and antenna used in RF emission testing method.

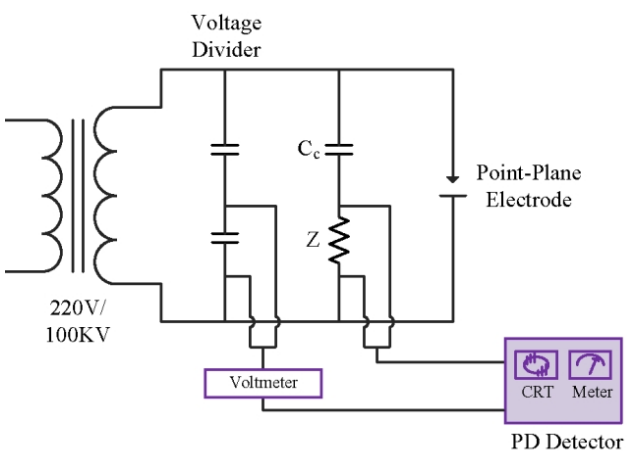


Fig. 3 Electrical PD Detection Technique

The direct coupling method is the most common means of PD measurement. It detects the PD current by using a high voltage capacitor connected to the high voltage terminal of the test object as shown in Figure 3 (*15*). A standard test circuit mainly includes a test object (point plane electrode), a coupling capacitor *Cc*, measuring impedance *Z* and the PD display and recording unit. Measuring impedance is connected either in series with the test specimen or with the coupling capacitor.

If the sample capacitance is large, the measuring impedance is connected in series with the coupling capacitor to avoid the passage of large charging current through it (*16*). A voltage divider is used to produce a suitable fraction of the test voltage for measurement. Here capacitive voltage divider is connected in parallel with the supply voltage. This typically has a ratio of 10,000:1, so that the reduced low voltage is used for instrumentation purpose.

However, this method is predominantly an offline method. It is also vulnerable to electrical disturbances and noise during onsite measurement.

Chemical Detection:

In PD detection by chemical method, current streamer across the void may leads to chemical decomposition. Dissolved Gas Analysis (DGA) and High Performance Liquid Chromatography (HPLC) are two acclaimed methods for further analysis of different chemical components. Fundamentalpart of DGA is periodic oil sampling. Primary diagnostic gases have been identified ashydrogen (H_2), ethylene (C_2H_4), ethane (CH_4), methane (C_2H_6), acetylene (C_2H_2), carbon monoxide (CO) and carbon dioxide (CO_2) (*17*). Gassing characteristic is greatly reliant on size and structure of transformer, loading of transformer and history of maintenance. DGA is thus considered as "art" instead of "science". Most of the harmful PD occurs close to the paper insulation. Hence CO_2 emitted from the cellulose is considered to be an indicator for presence of PD.

Acoustic Detection:

Hot current streamer in void vaporized surrounding materials which causes sudden increase of mechanical energy in terms of acoustic waves. PD generates acoustic waves in range of 20 kHz to 1 MHz. External system and internal system are two categories of acoustic detection techniques based on sensor location in transformer. External system is widely accepted as sensors are mounted outside of the transformer. In external system typically Lead Zirconium Titanate (PZT) types of sensors is used.

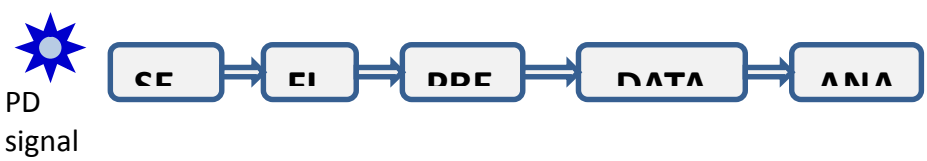


Fig. 4 Acoustic Detection Technique Block Diagram

Figure 4 shows Acoustic emission detection technique block diagram. Wall mounted acoustic emission sensors senses vibrations and convert it into electrical signals. After filtration process, signals amplified in preamplifier unit. Data acquisition unit stored data and sends for further analysis.

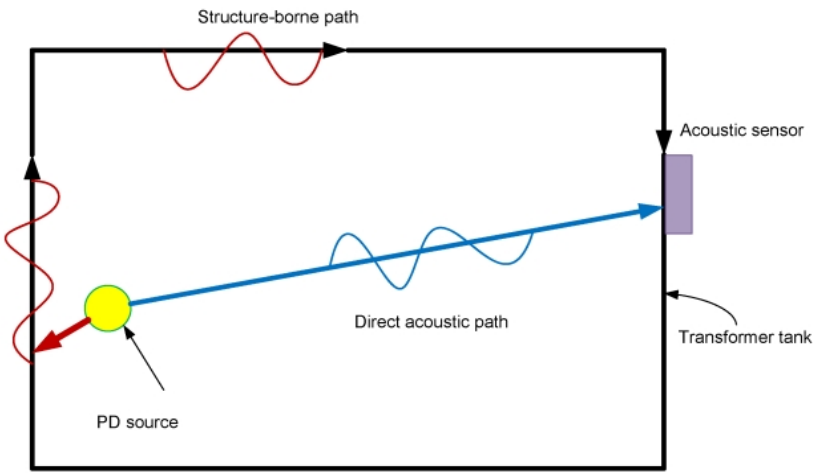
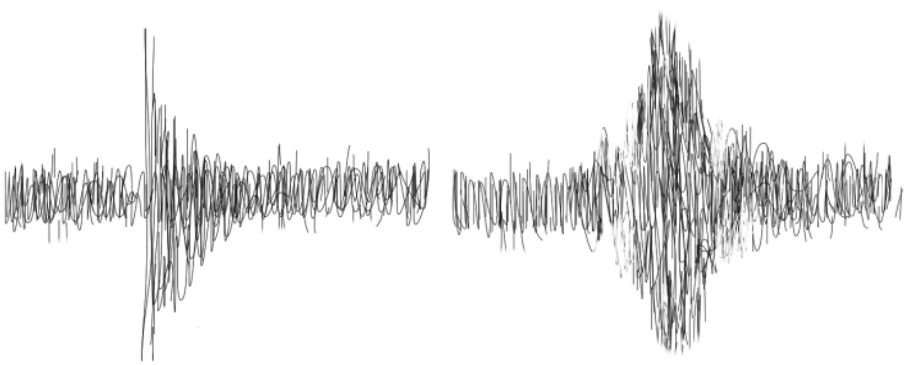


Fig. 5 Acoustic PD Signals Propagation Path *(19)*

Figure 5 shows PD source location and acoustic signal propagation path. One is direct propagation between a PD source and the sensor, second is structure born indirect path of propagation. The travelling waves speed through transformer oil is 1500 m/s and on the transformer tank surface (metal/steel) is 6000 m/s (*19*).

Propagation of acoustic wave is depends on media. So acoustic wave propagation path in different structures and material give us information of physical process occurring.



(a) Arrow Head Shaped Signal (b) Egg Shaped Signal

Fig. 6 Shape of Signals

Shape of captured signal by sensor is affected by types of sensor, propagation path and source. In general signals obtained are of arrow head shaped and egg shaped as shown in Fig. 6. Arrow head-shaped signals obtained in low attenuation (oil path). Egg-shaped signals obtained in solid material propagation (metal/steel path).

Apart from the detection of PD, acoustic method can be used for estimating location of PD site. The waves impregnate over insulation material and then hit the equipment enclosure. Acoustic signal is devitalized by (i) spreading of geometric wave-front, (ii) Wave distribution due to multiple propagation paths (iii) transmission losses due to change of medium and break within the structure, and (iv) absorption in different materials. PD source localisation can be done by finding highest amplitude of signal, by observation of rise time of signal and from total time taken by signals to reach sensor.

Acoustic emission detection technique is very famous in Gas Insulated Substation (GIS). An obvious advantage of the acoustic method is that it can locate the site of a PD by algorithms. Electromagnetic interference may cause corruption of signals captured by piezoelectric sensors. Also, acoustic signals decay relatively fast in solid insulation.

UHF Detection:

UHF detection is advanced technique for PD detection. Discharges are exceptionally fast and transient pulse. Pulse emission energy is in Giga Hertz range. PD is detected by means of electromagnetic transient signals frequency range of 300-3000 MHz. UHF detection is famous due to various reasons like online monitoring, less noise interference. The UHF detection technique was first implemented in Gas Insulated Switchgear (GIS) (*21*)and later on used to power transformers (*21*). Amplitude of UHF pulses and time interval between two pulses are very important parameter for detailed understanding of PD activity. Basically UHF sensor (antenna) will capture PD signal, which are subsequently connected to preamplifier, multiplexer and digital oscilloscope. Capacitive sensor and inductive sensor are two broad categories of UHF sensor. Capacitive sensor has higher sensitivity than inductive (*22*). Mainly four types of sensors are widely in use (i) Window sensor (ii) Internal disk sensor (iii) Barrier sensor and (iv)Oil valve sensor. Other than these wave guide sensor, hatch sensor, directional electromagnetic sensor, etc. are also in use. In transformer localisation of PD is possible by installing more number of sensors. For this purpose provision for sensor has to make at the time of transformer manufacturing.

COMPARISON OF PD DETECTION TECHNIQUES:

After searching and reviewing a number of text books, research papers and journals on the topic of Partial Discharge Detection techniques, following inferences can be drawn.

Electrical detection:

- Standardized, widely accepted, sensitive and easy implementation technique.
- Vulnerable to electrical disturbance, noise.
- Offline method of PD detection.
- Used for laboratory/factory testing only.

Chemical detection:

- Commonly used analytical technique, low sensitivity when used for the detection of partial discharges, and it does not identify the location of incipient faults in power transformers.
- Gassing characteristic of transformer is depends on structure, loading, maintenance record and manufacturer. So separate consideration is required for every transformer.
- Agreement is based on historical data. DGA is thus often referred to as “art” instead of “science”.

Acoustic detection:

- Able to locate PD site by phase delay/amplitude attenuation of wave.
- Acoustic PD detection is immune to electromagnetic interference.
- Measurement of PD is possible from the enclosure of the component so couplers installation not required.
- Acoustic PD detection is non-invasive.
- Investment costs for an acoustic PD measurement system are moderate
- Wave propagation is depending on material properties of medium
- Acoustic signal calibration is not possible
- Used for onsite testing.

UHF Detection:

- High signal to noise ratio compare to conventional method.
- Noise interference with the signal is lower at UHF range.
- Difficult to find location of PD source.
- Online detection technique.
- UHF method is less flexible.
- More expensive due to high frequency components.

COMBINATION OF DIFFERENT PD DETECTION TECHNIQUES:

Looking to strength and weaknesses of presently available PD detection techniques it can be seen that there is a very good scope for combination of different detection technique. Electrical detection technique can confirm presence of PD but unable to trace location of PD source within equipment because minimum four sensors are required for space coordinates and time information for localisation of PD. Generally it is difficult to mount more than three internal sensors in any equipment. In that case acoustic detection technique is very useful as there is no limitation for externally mounted sensors. At the same time acoustic sensors are very sensitive to external noise disturbances so if acoustic technique can be combined with electrical/UHF techniques than possibility of very good results. Combination of different detection techniques increases immunity to wrong results.

CONCLUSION:

This paper highlights physics of partial discharge and its detection techniques. The existing literature on the different types of PD detection methods including electrical, chemical, acoustic and UHF detection methods are discussed with their key features.

From the literature review, it is found that acoustic and UHF detection are better than conventional detection techniques. There is very good scope for combination of acoustic and UHF technique to get better results.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th& Jan. 6th, 2016)

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Solar Maximum Power Point Tracking by Incremental Conductance Algorithm Using Various Types of Grid Loads

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ABSTRACT
Solar energy is a spotless, green and endless wellspring of energy. Sunlight based cells by photovoltaic activity can change over the sun based vitality into electric current. The present commercial efficiency of sunlight based cells is not more prominent than 15%, and hence, the accessible proficiency is to be retrieved to the most extreme conceivable worth and the Maximum Power Point Tracking (MPPT) with the support of power electronics to sun oriented cluster can make this conceivable. In this research paper, an exercise is conducted to comprehend the essential usefulness of a standout amongst the most well-known algorithms viz. Incremental conductance algorithm. This algorithm is tested by mimicking a 100 kW solar power generating station connected to a grid having different types of load using the MATLAB Simulink software. MATLAB M-files are produced to comprehend MPPT and its reliance on insolation and temperature.

SUMMARY
The paper presents Incremental Conductance Algorithm for Maximum Power Point Tracking using the various grid connected loads and its effects on the performance of PV array parameters

Keywords: Incremental Conductance Algorithm, Power Grid, MPPT

INTRODUCTION

A global temperature alteration, air contamination, and rising fossil fuel costs have ended up mindboggling for the researchers, ecological architects and notwithstanding for the entire world today. The race of industrialization is demonstrating its astringent results. The discharges, particularly from thermal power plants and vehicles, are of significant concern. With the increasing populace, the vitality needs are expanding step by step, and that is to be satisfied by establishing new power plants. In this regards, Photovoltaic (PV) power generation has a crucial part to play because it is a green source. The only emanations connected with PV power generation are those from the creation of its segments. After their establishment, they create electrical vitality with the assistance of sun based light without transmitting nursery gasses. PV arrays can be introduced at the spots that, all in all, are of no utilization e.g. rooftops, pastries, remote areas, trench tops, a place that is known for no use and numerous more [1-6].

As per the maximum power transfer theorem, a power source will give its maximum power when its impedance becomes equal to the load impedance. For solar PV arrays, the batteries are not the ideal loads and this mismatch of the two results in efficiency loss. Consider a 12 Volt battery to be charged by a solar PV panel having maximum power point around 17 volts.

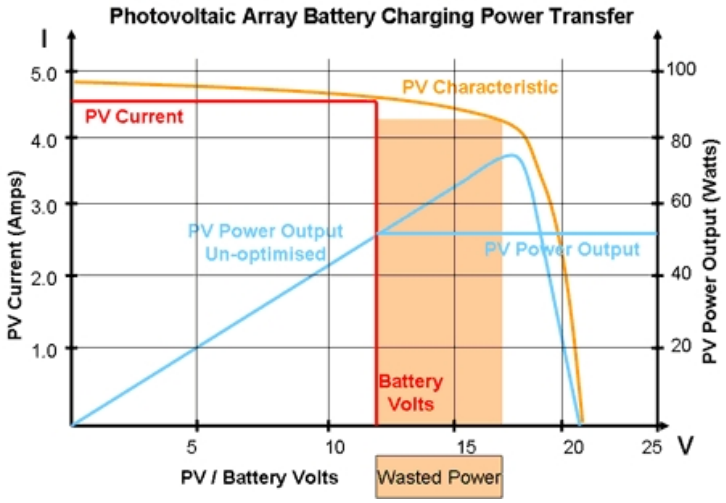


Fig.1. PV array charging the battery [7]

If PV array of 17 V, 4.4 A is directly connected to the battery, as shown in Figure 1, then it will deliver only 2.5 A to the battery at 12 V. In this situation, the PV array will supply only 50 Watts instead of 75 Watts (power loss of 75 – 50 /75 = 33.33%). Maximum Power Point Tracking (MPPT) can help to solve this problem. This MPPT, in the form of the voltage regulator, is placed between the battery and the PV array. The MPPT presents an ideal load to the PV array that will allow it to operate at 17 Volts, and it will supply 75 Watts irrespective of the battery voltage. A DC/DC converter in the MPPT module will adjust the DC output automatically, to match the battery voltage of 12 Volts. Now, as the voltage is stepped down by factor 12/17, the current will rise by the same factor, i.e. 17/12. Thus, a battery charging current will be 17/12 * 4.4 = 6.23 A. Hence, it is possible to supply 6.23 * 12 = 75 Watts. In the actual practice, considering a maximum of 10% converter losses, a significant efficiency improvement will be possible.

Due to atmospheric changes, the ambient temperature changes invariably which also makes the Maximum Power Point (MPP) switch its location. Thus, MPPT is a device, which will continuously monitor and adjust the operating point to retrieve the maximum power in the varying temperature and irradiance conditions [8].

1.1 BLOCK DIAGRAM OF MPPT

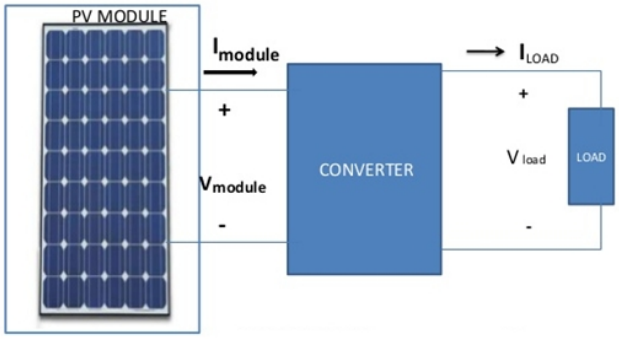


Fig. 2. MPPT Block Diagram [9]

The Maximum Power Point Tracking (MPPT) is a process to make source impedance equal to the load impedance. The impedance matching is achieved by a DC-DC Converter. By varying the duty cycle of the DC-DC converter,

the matching of two impedances is made possible, and hence, maximum power is delivered. The block diagram of MPPT is shown in Figure 2.

Considering DC-DC converter in buck mode, with input and output voltages as V_i and V_o respectively and the duty cycle D, the converter output is written as;

$V_o = D * V_i$ (1)

The transfer equation for output impedance R_o and an input impedance R_i will become

$R_o = D^2 * R_i$ (2)

$R_i = R_o / D^2$ (3)

Here, R_o, the output impedance remains constant but the input impedance seen by the source is made to change by varying the duty cycle D. This process makes the resistance of source retrieve the maximum power.

2. INCREMENTAL CONDUCTANCE ALGORITHM

Various algorithms are proposed by the different researchers to vary the duty cycle D of the DC-DC converter and extract the maximum power, like;

(1) Fractional Open Circuit Voltage (FOCV)
(2) Fractional Short Circuit Current (FSCC)
(3) Fuzzy Logic
(4) Perturb and Observe (P & O)
(5) Incremental Conductance (INC)

These algorithms have their own merits and demerits. One of the prevalent and useful algorithms, Incremental Conductance (INC) is discussed hereunder.

The Incremental Conductance (INC) is widely accepted algorithm because it gives nearly perfect MPP tracking without the higher circuit complexity. The algorithm like, Perturb and Observe (P & O) or Fractional Open Circuit Voltage (FOCV) are least complex, but P & O has the limitation of giving the non-exact output under varying atmospheric conditions, whereas, the FOCV doesn't provide the best MPPT efficiency due to inherent deficiencies of calculations. INC can come over both these limitations and hence, it is highly acceptable [10-11]. The fundamental INC algorithm is shown in Figure 3 and the flow chart is presented in Figure 4 respectively.

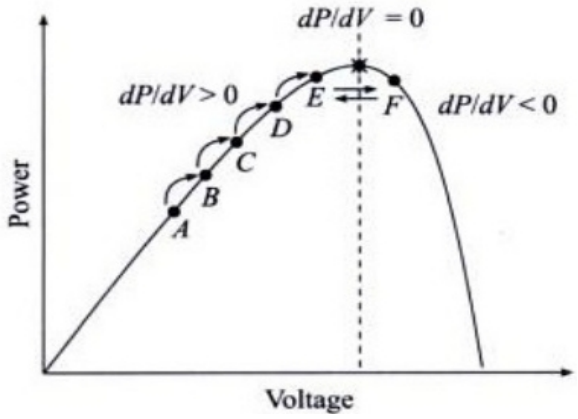


Fig. 3. INC Algorithm

For a PV array, the equation of power can be written as:

P=V*I (4)

(Where P= module power, V=module voltage, I=module current);

Differentiating about V

dP/dV = I + V*dI/dV (5)

The algorithm works the above equation. At peak power point,

dP/dV = 0 (6)

$dI/dV = -I/V$ (7)

If the operating point is to the right of the power curve, then we have

$dP/dV < 0$ (8)

$dI/dV < I/V$ (9)

If operating point is to the left of the power curve then we have

$dP/dV > 0$ (10)

$dI/dV > I/V$ (11)

Using equations (7), (9) and (10) the peak power can be tracked. This incremental conductance method is shown in Figure 3.

This algorithm has benefits over perturb and observe in that it can determine when the MPPT has touched the MPP, where perturb and observe oscillates around the MPP. Also, incremental conductance can follow rapidly increasing and reducing irradiance conditions with higher accuracy than perturb and observe. One drawback of this algorithm is the increased complexity when related to perturb and observe as it requires two sensors Viz. Current and Voltage whereas P & O method requires just one sensor and that is Voltage only [12-14]. Flow chart of incremental conductance method is displayed in Figure 4.

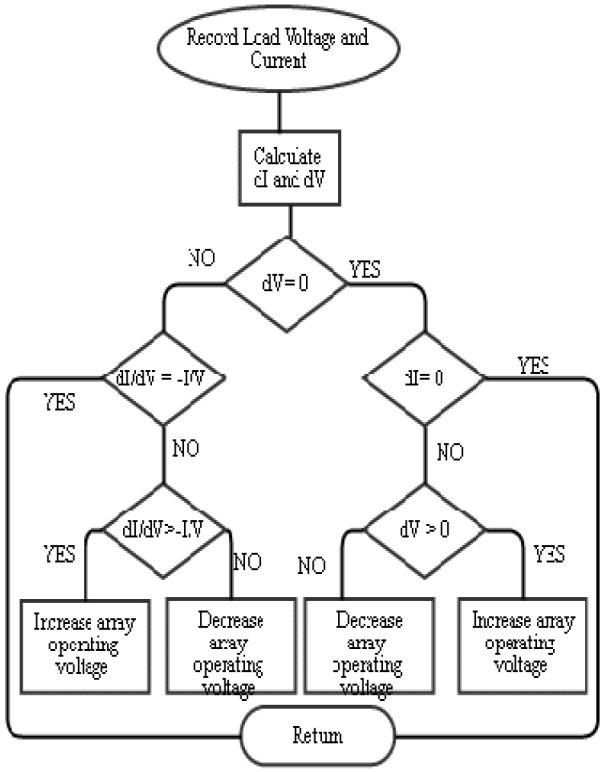


Fig. 4. Flow chart- Incremental Conductance (INC) Algorithm

3.1 THE SIMULATED SYSTEM AND DIFFERENT LOADS

Figure 6 presents the Pi section of the utility grid used for simulating the load side parameters. The system simulated in MATLAB/Simulink® environment, gave the results as shown in Figures 7 to 9. The load on grid side was made variable for the different cases of (a) (b) and (c).

Case (a) :2 MW at , 30 MW 2 MVAR in Pi section of grid as shown in the Figure 6.

Case (b) :5 MW at , 40 MW 10 MVAR in Pi section of grid similar to the Figure 6.

Case (c) :30 MW at , 20 MW 20 MVAR in Pi section of grid similar to the Figure 6.

3. SIMULATION OF 100 KW GRID CONNECTED SOLAR POWER PLANT

The 100 kW Grid Connected Solar Power Plant was simulated using MATLAB/Simulink® software. The parameters of the system are displayed in Table 1. The simulated system is shown in Figure 5.

Table 1. Parameters of the Simulated System

Solar panel model	Sun Power SPR-305
Solar panel wattage	305 Watt (Wp)
Open circuit voltage for panel	64.2 Volt
Voltage at MPP for panel	54.7 Volt
Short circuit current for panel	5.96 A
Current at MPP for panel	5.57 A
No. of panels in series	5
No. of panels in parallel	66
Total power of system at MPP	305 * 66*5 = 100.65 kW
Boost converter output voltage	500 Volt
Grid voltage (Through X'mer)	50 kVA, 500 V/25 kV
Boost converter inductance	5 mH
Boost converter switching frequency	5 kHZ.
Filter inductance	250 µH
Inverter	3 Level VSC
Grid length	19 km. (pi section)
Grid load	Variable with different cases

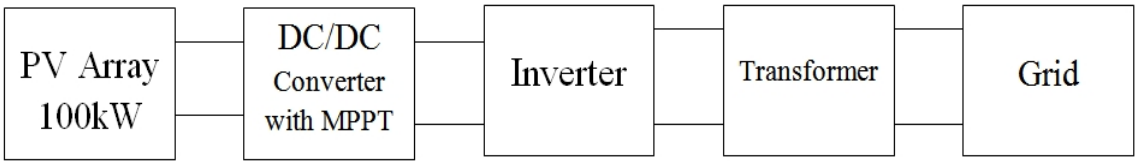


Fig. 5. Simulated System of 100 kW Grid Connected Solar Power Plant

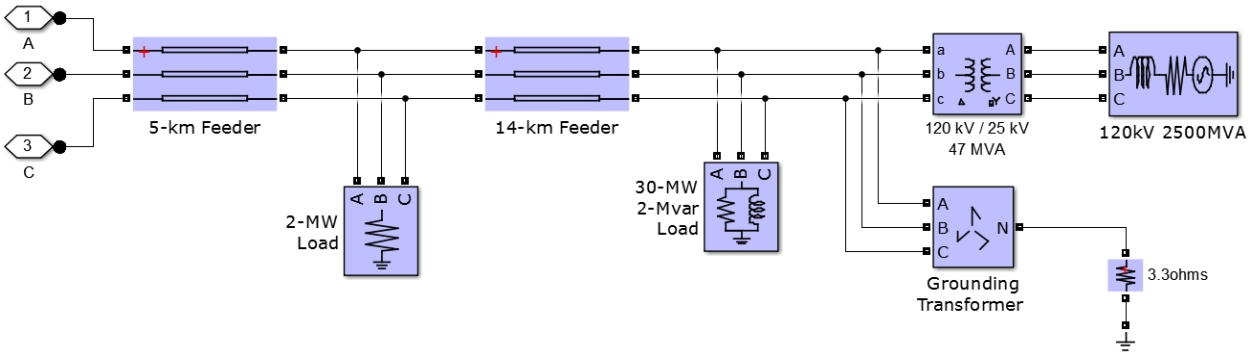


Fig.6. Pi Section of the utility grid and connected load

4.SIMULATION RESULTS AND DISCUSSION

Case (a):

In this case, the system was simulated using 2 MW, 30 MW 2 MVAR load in Pi section of grid. The total Pi section is divided in two parts. The total Pi section is divided in two parts. First 5 km feeder and the second 14 km feeder. The results of the simulation are presented in Figures 7(a) to 7(c). Figure 7 (a) presents the variations in irrdiance (insolation), temperature and P_{mean} (power generated bythe soalr array). These variations make it clear that the power generated by the solar panels is directly proportional to insolation level and inversely proportional to the

temperature. Figure 7 (b) indiacte variations in V_{mean} (solar array voltage) and duty cycle. The duty cycle varion is limite to 0.47 to 0.5. Again the temperature increment has negative effect on output volatge that can clearly be seen. Figure 7 (c) shows the variation in power supplied to the load (grid side) by the solar PV panels. The total loss in the converetrs, invertrs and transformers is around 1.5 kW. Again this feeding of laod varies with the irradiance and temperature conditions.

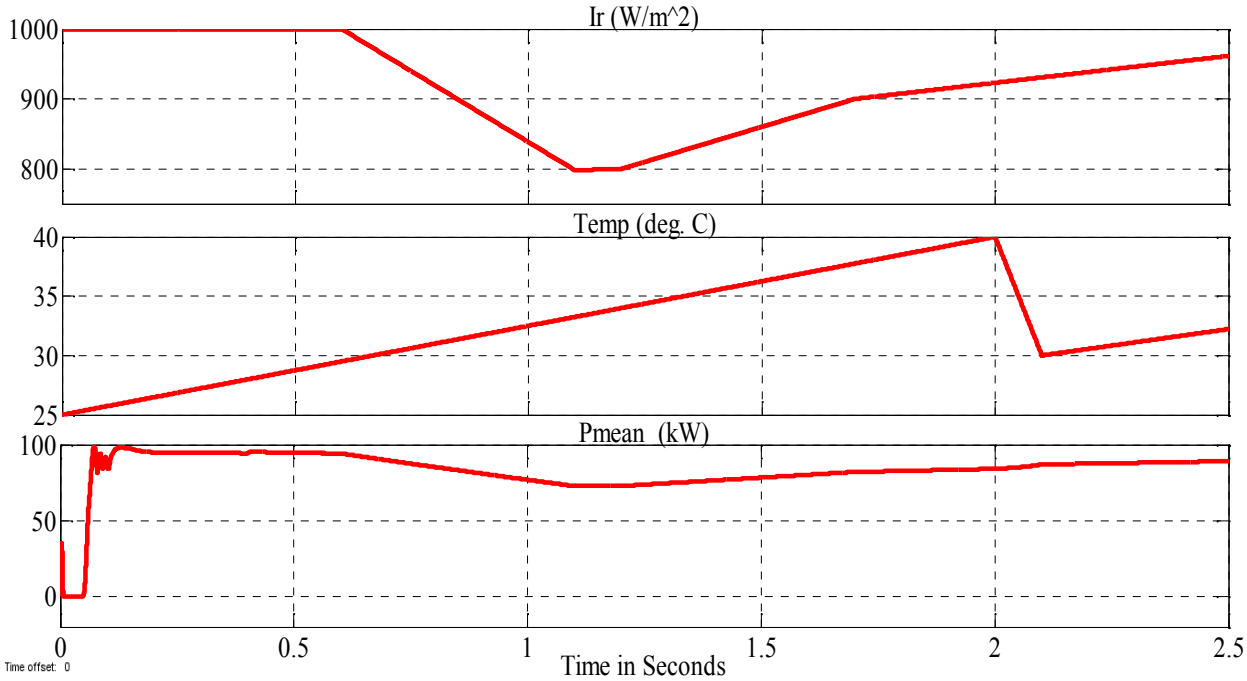


Fig. 7(a) Variations in Insolation, Temperature and P_{mean} with time for case (a)

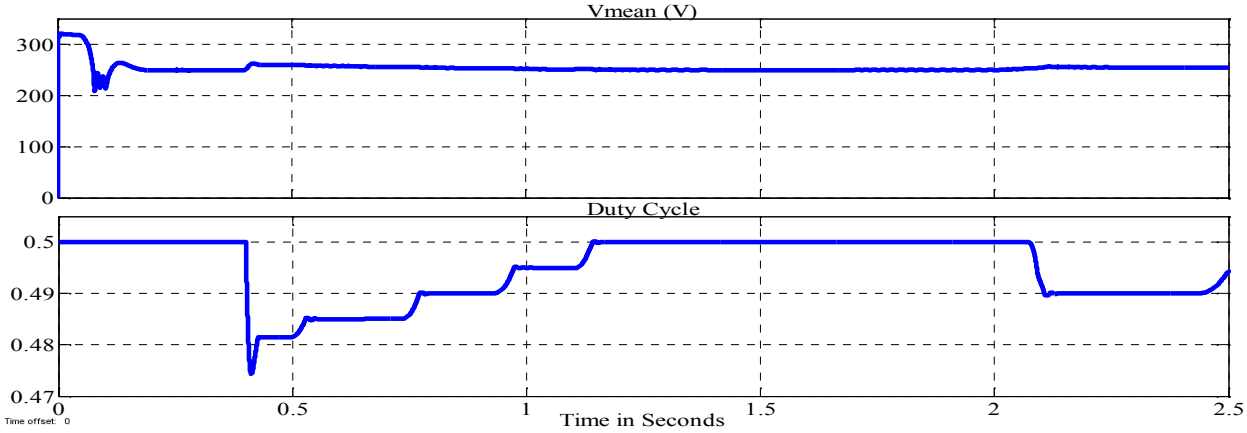


Fig. 7(b) Variations in V_{mean} and Duty with time for case (a)

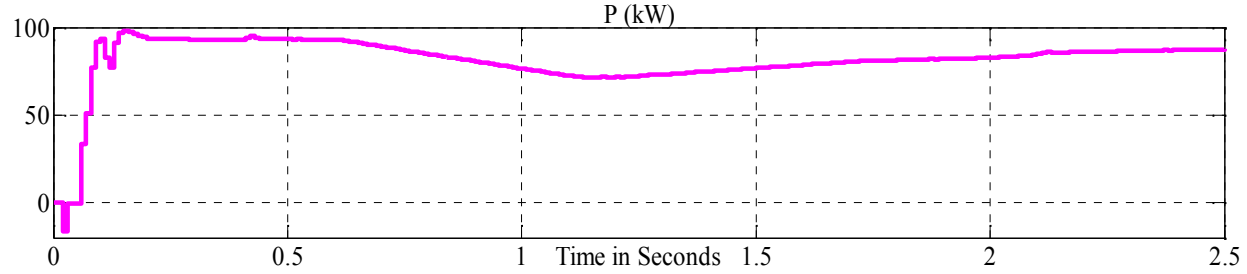


Fig. 7(c) Variation in power supplied to the grid for case (a)

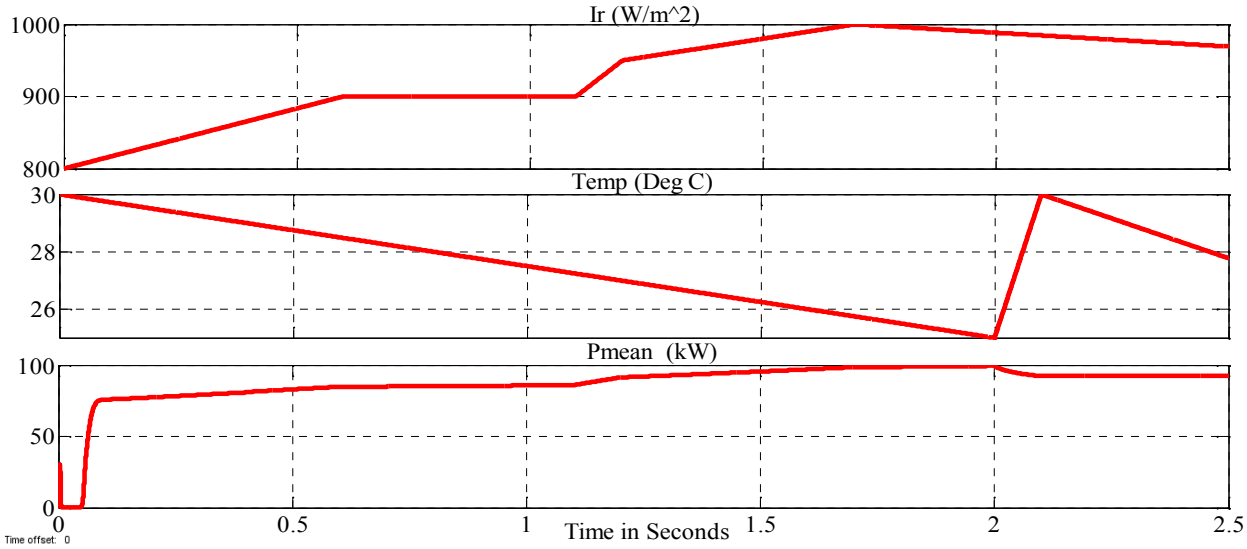


Fig.8(a) Variations in Insolation, Temperature and P_{mean} with time for case (b)

Case (b):

In this case, the system was simulated using 5 MW, 40 MW 10 MVAR load in Pi section of grid using the same Pi section as in case (a) apart from the load parameters. The irrdiance variation is made in widre range from 800 to 1000 W/m^2 at the different intervals. The temperature variation was made smaller. The results presented in Figure 8(a) shows that P_{mean} is increaded due to higher irrdiance average and lower temperature variations. Figure 8 (b) indiactes that voltage variation is also narrowed down due to limite temperature variations. Figure 8 (c) makes it clear that only during thea very small starting period the power output to the load is negative which becomes stable after 0.02 seconds.

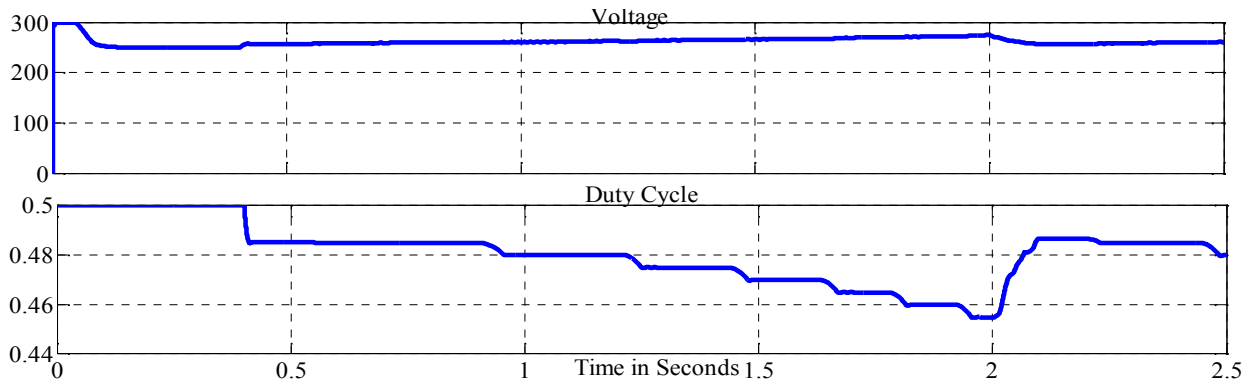


Fig. 8(b) Variations in V_{mean} and Duty with time for case (b)

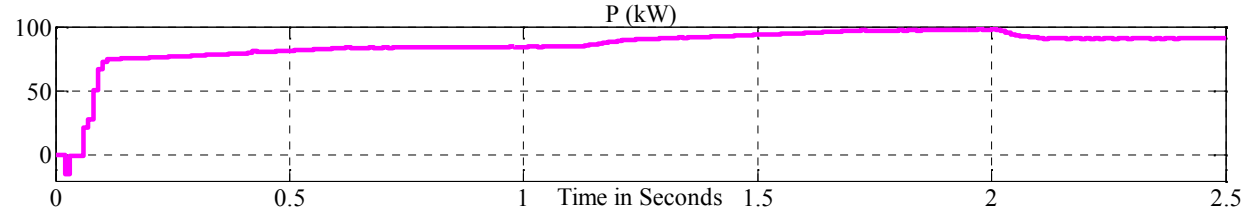


Fig.8(c) Variation in power supplied to the grid for case (b)

Case (c):

In this case, the system was simulated using 30 MW, 30 MW 20 MVAR load in Pi section of grid using the same Pi section as in case (a) apart from the load parameters. The irrdiance variation is made in widre range from

800 to 1000 W/m^2 at the different intervals. The temperature variation was made smaller. The testing of increment in the reactive power by large scale has negative effect on P_{mean} due to the fact that reactive power support from grid takes time (Figure 9(a)). The varaition in V_{mean} (Figure 9(b)) is also higher and Power delievered has a signifiacnt variation due to reactive power variation on the load side.

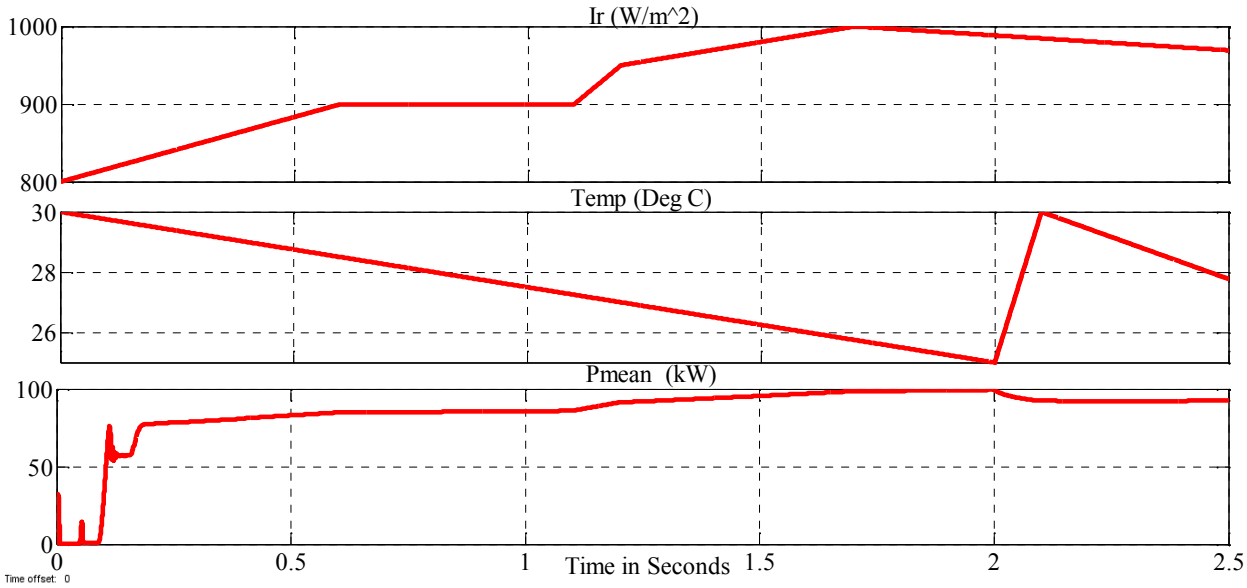


Fig.9(a) Variations in Insolation, Temperature and P_{mean} with time for case (c)

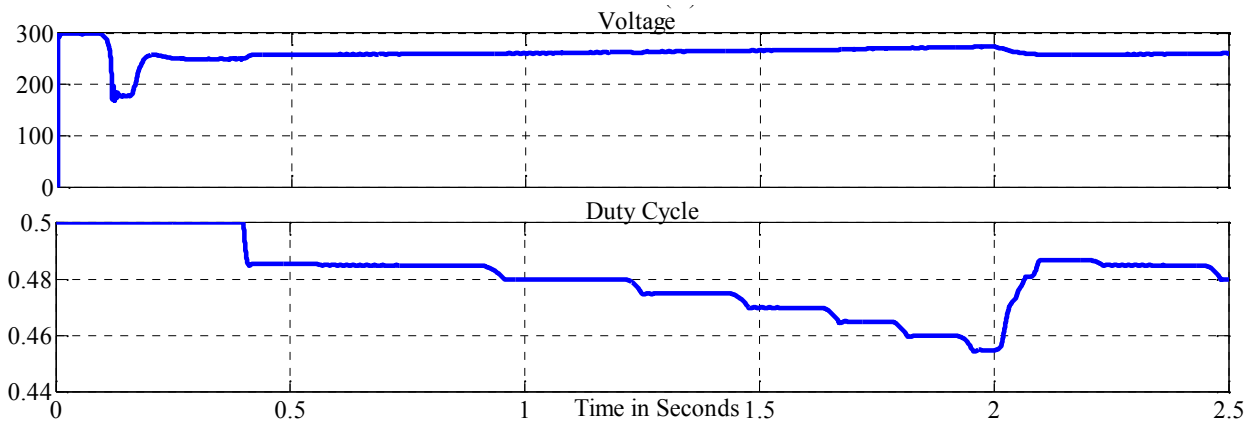


Fig. 9(b) Variations in V_{mean} and Duty with time for case (c)

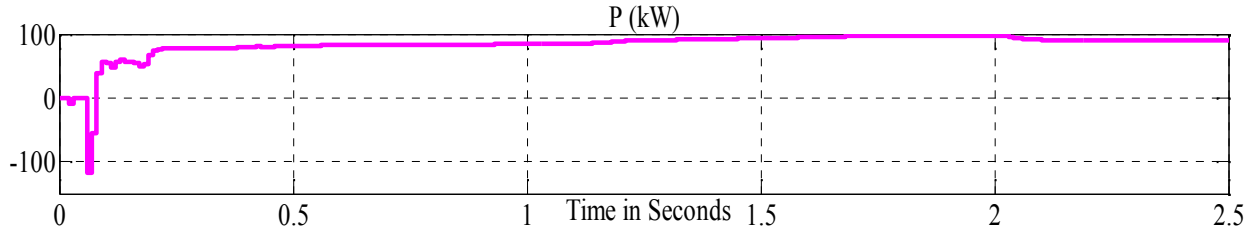


Fig. 9(c) Variation in power supplied to the grid for case (c)

5. CONCLUSION

The paper presents the fundamental principle of incremental conductance MPPT. The power generation by solar PV panels was retrieved at the maximum by using the MPPT. Different cases for the grid connected loads were taken and MATLAB/Simulink® is utilized to create the test bench. The simulated results present the variation in the insolation, temperature, power generated by the PV panels, voltage generated by the PV panels, duty cycle and the power delivered to the load. The variations in P_{mean} and $V_{mean,}$ has the inverse relation with the temperature increment and direct relation with the insolation level. The power delivered to the load has a large effect when reactive power magnitude is too high and in this case, the V_{mean} changes abruptly during the time elapsed to get the reactive power support given by the grid. The overall results verify the basic principles related to the solar power generation and the relation between the reactive power and the voltage profile.

ACKNOWLEDGEMENT

This work was sponsored by Gujarat Council on Science and Technology (GUJCOST), Gandhinagar, India, a Gujaratstate body to fund the science and technology research work.The Reference No. of the Grant is GUJCOST/MRP/2015-16/1116.

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Wide Area Measurement System Based on PMU for Voltage Stability Assessment

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ABSTRACT

The power systems are being enforced to operate nearer to its stability limit due to the current economic development and the intricacies to upgrade the existing grid infrastructure. By the impulsive increment of power demand, the voltage instability problem has become a key concern to the power system operator that generated the need of methods for prior determination and prevention of power system from collapse. In the present work, dynamic voltage stability is assessed with the use of Phasor measurement unit (PMU). The PMU receives real-time synchronized data for voltage or current which will be utilized to evaluate Voltage Stability Risk Index (VSRI) in real time. The proposed method evaluated for Western System Coordinating Council (WSCC) 3 - Machines; 9-Bus test system by creating a test bench in MATLAB/ Simulink® environment.

SUMMARY

The dynamic voltage stability is assessed with the use of Phasor Measurement Unit(PMU) in wide area measurement system by creating a test bench in MATLAB/ Simulink® environment.
Keywords: Phasor Measurement Unit, Synchrophasor, Voltage Stability Risk Index, Wide Area Measurement System

1. INTRODUCTION

The ability of power system to maintain acceptable voltages at all buses in the network during normal conditions as well as disturbances termed as voltage stability *(1)*. The voltage instability characterized by the slow decay of voltage followed by a sharp decline at any bus in the power system. As the power system becomes more complex and heavily loaded, voltage stability has become a serious problem *(1)*. In the modern days, the study of voltage stability has assumed importance, mainly due to numerous documented incidents of voltage collapse in the entire world. Because of line loadings, reactive power shortages, switching of components, voltage collapse is caused. Voltage instability involves an uncontrolled decrease in voltage produced by disturbances, leading to voltage collapse. Voltage collapse may be fast or slow that depends upon the nature of the event. The voltage instability normally results in monotonically decreasing voltages. Sometimes the voltage instability may manifest as negatively damped or undamped voltage oscillations prior to voltage collapse. Thus, nonachievement of post-disturbance equilibrium state leads to voltage collapse. However, characteristics of different components and system conditions are used to manipulate the procedure. Also, the magnitude of the voltage and its threshold are no more a dependable voltage instability indicator.

The study of voltage stability has been psychoanalyzed with many methods, but that can be separated into the dynamic and static analysis. Analysis of static voltage stability is based on steady state operation of the power flow model or a linearized dynamic model. At the same time, analysis of dynamic stability depends upon the algebraic equations and model that includes tap changing transformer, dynamics of generators, etc. through transient stability simulations *(2)*. Many works on indices are reported in the literature for calculating proximity to the voltage instability *(3-5)*. Either network admittance matrix or a Thevenin equivalent of the system is required for these indices calculation. Conventional voltage stability methods suffer from the drawback of sluggish approach and hence are not worthy for real-time applications *(6-10)*. However, direct measurement methods have become simpler than said methods, utilities are now utilizing them in power system network for protective device to prevent voltage collapse *(11-13)*.

Moreover, there are many known measures introduced to prevent voltage collapse, but the main issue was the lack of information related to the actual system state *(14)*. So, this constraint will lead the system to incorrect or delayed corrective actions, and that will accelerate a change of voltage instability. Nowadays, phasor measurement units having synchrophasor technology provide fast and efficient ways to improve the power system state estimation *(15)*. Phasor measurement units (PMUs) and fast communications networks have led to the evolution of synchronized phasor measurement technology which in turn has resulted in the development of wide area measurement system *(16-19)*. Within the short interval, the present network with PMUs takes samples of the power system variables. This method of wide area stability assessment provides preventive early detection of voltage instability. VSRI rather than using conventional indices for the voltage stability calculation utilizes time series data of voltage magnitude from all connected PMUs for evaluating voltage instability of the system. Hence, the said algorithm is used for PMUs, located at different buses to estimate the voltage stability of the WAMS network during transients.

The proposed scheme simulated in MATLAB for WSCC 9-bus system. The algorithm is implemented for PMU development in MATLAB to compute the phasor of voltage signals at all buses in the network. The

output of PMU gives the phasor data at the high sampling rate for the assessment of voltage stability in an event of the different disturbance. The paper organized as follows: The estimation of VSRI is presented in Section 2. The test system, results and discussion of the proposed approach are presented in 3 followed by the conclusion in Section 4.

2. ESTIMATION OF VOLTAGE STABILITY RISK INDEX

The instantaneous values of load bus voltages are the input to PMU. The PMU is capable of producing bus voltage magnitude and phase angle by processing the input data with very high sampling rate. In this simulation, the sampling rate of 30 frames per second is considered where each frame contains 60 samples. The algorithm is applied for extracting the voltage magnitude and phase angles of the load buses. These time series data of the load bus voltages used for the VSRI *(20-21)* calculation. The voltage stability can be predicted by observing the dynamic change in VSRI value as follow:

- The positive value of VSRI indicates that the system is in the stable state in normal conditions and afterhaving been subjected to any kind of disturbance.
- The negative value of VSRI indicates that system has a high risk of voltage instability.
- The highest variation in the voltages with respect to the pre-disturbance condition seen during the firstdips of VSRI.

3. TEST SYSTEM, RESULTS AND DISCUSSION

3.1 Test System

This section illustrates the use of VSRI to access the voltage stability of the test as shown in Fig. 1. For the calculation of VSRI, three PMUs are placed on all the load buses (Bus no. 5, 6 and 8). Two distinct types of disturbance are created; one result in post-disturbance stable state and other results post-disturbance unstable state. The types of disturbance are detailed in Table 1.

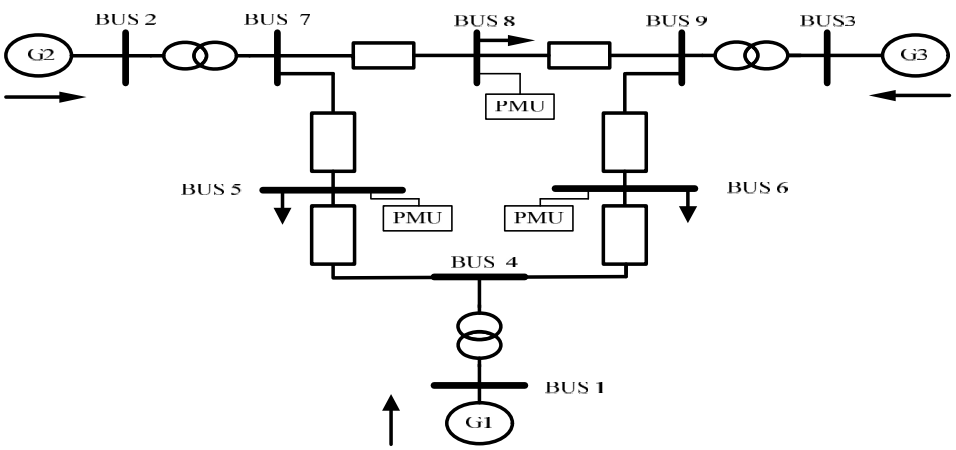


Fig. 1.WSCC 9-bus test system with PMUs connected on load bus

Table 1. Case studies for the simulation of VSRI

	Case No.	**Events**
Post disturbancestable case	Case-1	Increase of active load from 100MW to 200 MW for bus-8 at t=1sec
Post disturbance unstable case	Case-2	Increase of active load from 100MW to 200 MW for bus-8 at t=1sec Increase of active load from 100MW to 200 MW for bus-8 at t=1.02sec Increase of active load from 100MW to 200 MW for bus-8 at t=1.04sec
Post disturbance unstable case	Case-3	Increase of active load from 125MW to 225 MW for bus-5 at t=1sec Increase of active load from 100MW to 200 MW for bus-6 at t=1sec Increase of active load from 225MW to 425 MW for bus-5 at t=1.01sec Increase of active load from 425MW to 725 MW for bus-5 at t=1.02sec

3.2 Results and Discussion

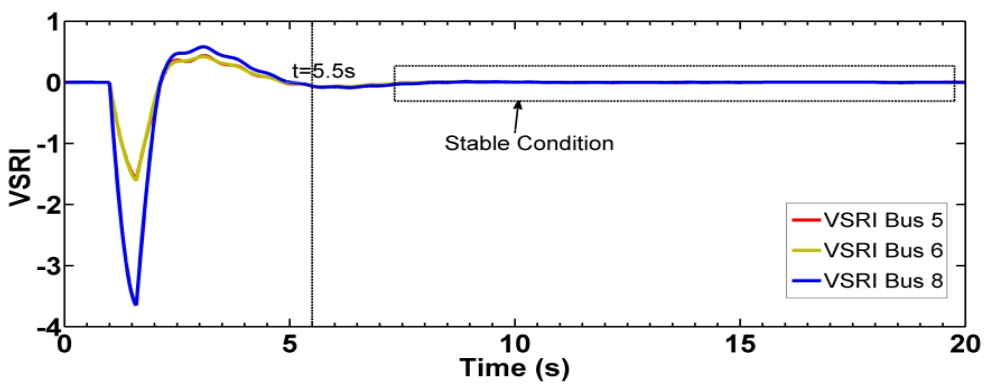


Fig. 2. Variations of VSRI for the load bus-8 for case no.1

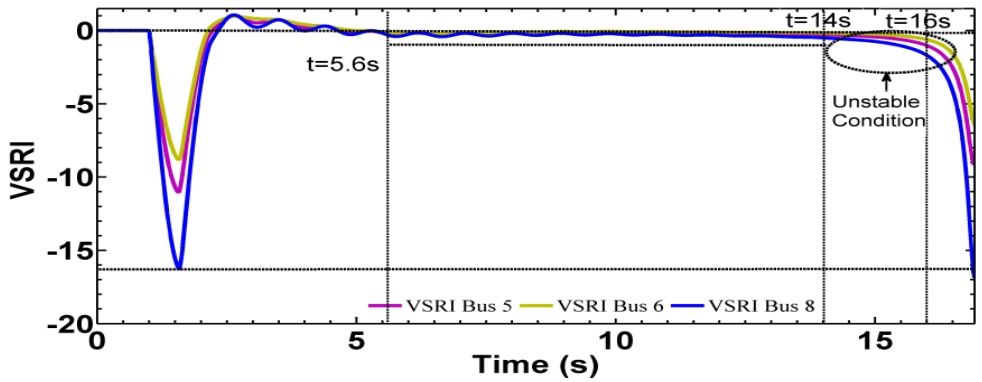


Fig. 3. Variations of VSRI for the load bus 5,6 and 8 for case no.2

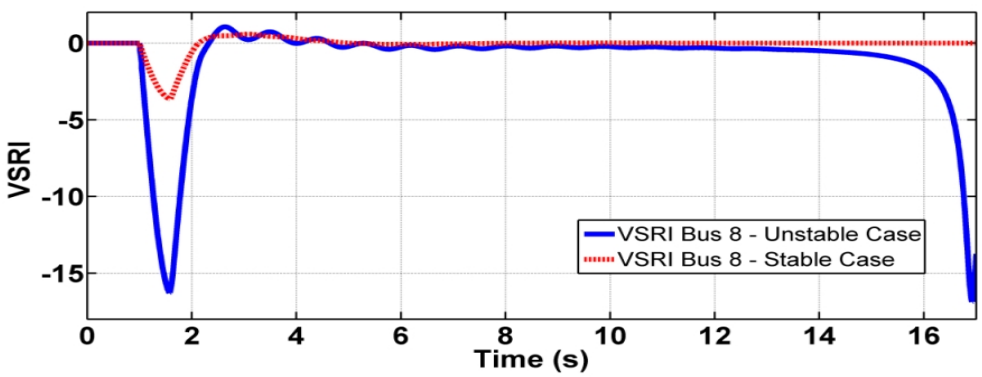


Fig. 4. Variation of VSRI at bus-8 during stable and unstable condition

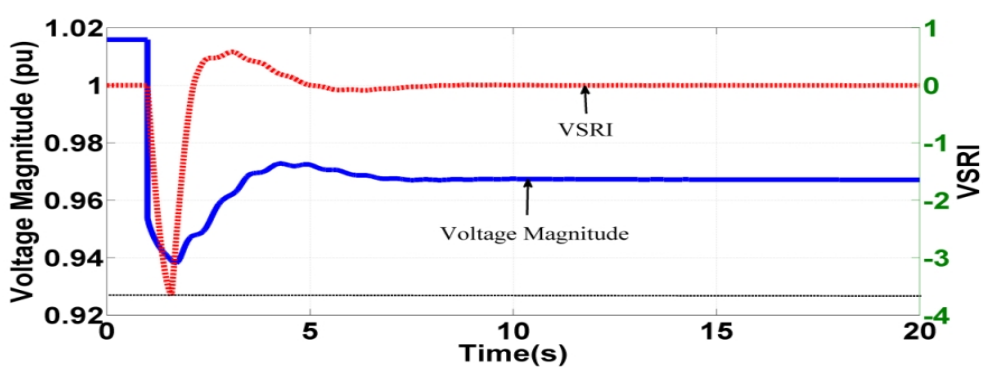


Fig. 5. Variations of VSRI and voltage magnitude for the load bus-8 for case no.1

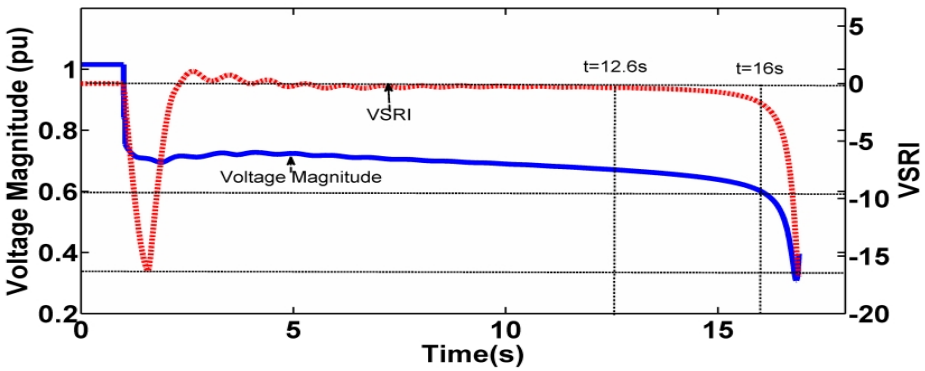


Fig. 6. Variations of VSRI and voltage magnitude for the load bus-8 for case no.2

In Fig. 2 the variation of VSRI is calculated for the case-1 given in Table 1. As shown in Fig. 2, a dip in VSRI can be observed at starting of the curve due to the application of load at bus 8. This initial negative dip of VSRI disappears with the subsequent settling of voltage magnitude of load buses in the post-disturbance state. The constant positive values of VSRI in the post-disturbance state indicates that the system works in voltage stable mode. From the Fig. 2 it has also been observed that VSRI value for load bus-8 shows the greater decrement as compared to bus-5 and 6. The results of Fig. 2, it is revealed that load bus-8 is more prone towards voltage instability. Fig. 3 shows the variation of VSRI for the case-2 given in Table 1 which has resulted in the post-disturbance unstable state as can been seen from the Fig. 3 that VSRI value continuously oscillating after the application of disturbance and does not settle to the constant value. This indicates that the voltage value of load buses have not restored back to the nominal values and continue to operate with the low voltage that is shown in Fig. 6. Hence, the progressive decline of the load bus voltages finally leads to voltage instability as shown in Fig. 3. Further, compare to Fig. 2 decrement in VSRI value is greater as the system is subjected to larger load disturbance. Fig. 4 shows the comparative variation of VSRI for the load bus-8 for the representing stable and unstable cases. Oscillatory values of the VSRI gives the information about the impending voltage instability so that the operators can take corrective actions to prevent voltage instability.

Also, voltage profile and VSRI for the load bus-8 is compared in Fig. 5. VSRI after 5s is settle to zero with the voltage magnitude of 0.96 p.u. on bus-8. On the other hand, the comparison of voltage profile and VSRI for bus-8 are shown in Fig. 6. The low voltage trip is set at 0.6 p.u. that occurs at t=16s, but the VSRI goes into negative direction just after t=12.6sec that gives the early warning about the impending voltage instability. From the Fig. 6, if the corrective action is taken between t=12.6s to t=16s like load shedding, then it can prevent the system from voltage collapse that is the key advantage of VSRI in WAMS based network for dynamic voltage stability assessment.

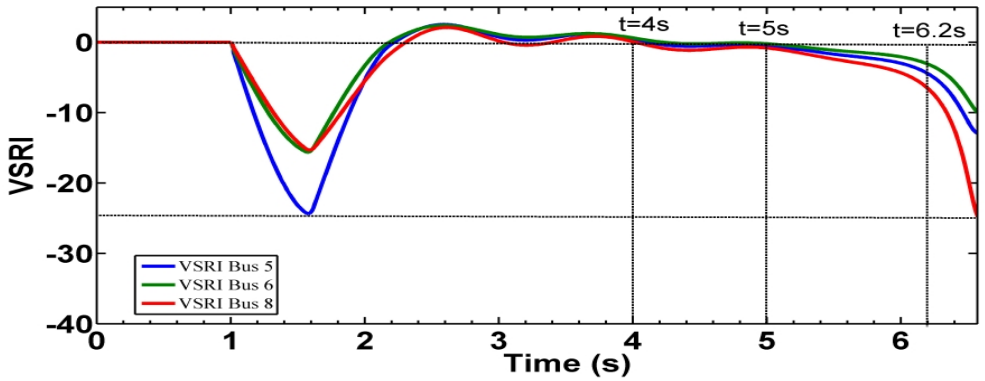


Fig. 7. Variations of VSRI for the load bus-5,6 and 8 for case no.3

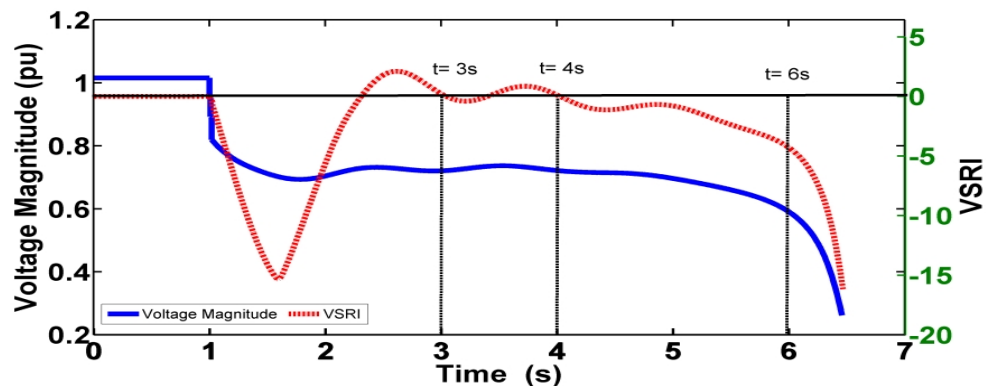


Fig. 8. Variations of VSRI and voltage magnitude for the load bus-8 for case no. 3

In the second case study, a voltage unstable case was simulated with further increase in load at bus-5 and bus-6 as per event data given in Table 1. The variation of VSRI for this case shown in Fig. 7. The variation in VSRI is the highest for bus-5 as the load is suddenly increased on that bus. However, with the sudden application of load on bus-5, the voltage of other load buses i.e. bus-6 and bus-8 are also affected. By the time, these load bus voltages continuously decline and resulted in voltage instability. The VSRI for the bus-8 is very sudden and sharp which needs more attention for the prevention of voltage instability that gives the indication about the initiation of corrective actions on bus-8 for prevention of voltage collapse. From Fig. 8, it is again confirmed that early detection of impending voltage of instability with the use of VSRI helps the system to remain stable.

4. CONCLUSION

The phasor estimation of the voltages available at the different load buses has been computed with the PMU. A scheme for detection of voltage stability with the use of VSRI is presented in this paper. The VSRI is calculated with the use of synchrophasor data obtained with proposed PMU located at different load buses. The higher sampling rate of the PMU as compared to conventional SCADA scheme results in

the quick calculation of VSRI for voltage instability determination. Real-time measurement through a synchrophasor based wide area measurement system (WAMS) is utilized in the present system to ensure voltage stability. The negative value of VSRI predicts the abnormal conditions much before the low voltage tripping threshold so that the corrective actions such as load curtailment may be initiated in well advance. This scheme also helps to identify the optimal locations for load shedding. The desired behavior of the proposed scheme is validated by its application to WSCC-9 bus system.

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Design and Development of Charging Mechanism for Electric Two Wheel Vehicle

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ABSTRACT

Electric vehicles have proved to be an efficient alternative for the fuel-powered vehicle. The problem of pollution is minimized by the electric vehicles. But the electric vehicles have their own drawbacks. The battery used in electric vehicle needs to be charged before driving the vehicle. It then gets discharge as we drive the vehicle. So there is always a fear that the battery will get discharged before reaching the destination. Also when the electricity is not available, then it is not possible to charge the battery. This drawback of the electric vehicle can be eliminated by the development of a "Charging Mechanism for Electric Two Wheel Vehicle". This mechanism can be equipped in the existing electric vehicle as an additional attachment. The main function of the mechanism is to generate the electric energy with the help of human effort. This electric energy can then be utilized to charge the vehicle.

SUMMARY

Electric two wheel vehicles are very popular now a days compare to fuel powered two wheel vehicles. Major advantages of electric two wheel vehicles over fuel powered vehicles like free from pollution and low operational cost. Limited range of drive is the major disadvantage of electric vehicles. Battery should be charged before the driving. During the long journey, battery may be discharged before reaching destination. In such situations using "charging mechanism" one can charge the battery instantly. Human effort is converted in to rotary motion of the alternator by proper mechanism and power generated by alternator is stored in battery.

Keywords: Electric, Vehicle, Battery, Charging, Mechanism

INTRODUCTION

Type Electric vehicles have emerged to be an efficient alternative for the fuel-powered vehicles. The fuel powered vehicles have many advantages like fuel is everywhere. The initial price of the fuel-powered vehicle is lower than the electric vehicle. However, a growing disadvantage is the price of fuel and the dwindling supply. The engines in the fuel-powered vehicles are fairly inefficient. It only converts a part of energy available from the fuel while the other is lost as heat. The cost of maintenance for the fuel-powered vehicle is high. The impact of the fuel-powered vehicle on the environment is a considerable point and so the electric vehicles have been introduced in the market to minimize the disadvantages of the fuel-powered vehicles (6).

The electric vehicles are propelled by an electric motor which is powered by rechargeable battery packs. The electric motor gets power from the controller and controller receives power from batteries. The electric vehicle operates on the electric or current principle. It uses battery packs to provide sufficient power to motor. The motor uses power to turn the transmission which rotates the wheels of vehicle (4).

Warren Vaz et.al.have defined two approaches to be considered; one considering the battery voltage constant and the other considering the battery voltage as a function of time. The predicted range was lower considering the second approach since the battery voltage is constantly reducing (4). The electrical vehicle consumption reduces with the increase in the motor efficiency. However it has less impact in case of smallervehicles, i.e. two-wheelers (3). It was seen that the main reason for the decline of the electric vehicle in India is the limited range offered by the electric vehicle against their cost (11).

DESIGN AND METHODOLOGY

The limited driving range is most considerable drawback for the electric vehicle which is due to discharging of the battery. There is a fear of discharging the battery during the journey. To overcome this problem it was needed to keep the batteries of the electric vehicles charged for a longer time than the presentones (6). This paper presents a mechanism for charging the batteries when the batteries of vehicle are discharged during travelling.

Many electric two wheelers are in market like Hero electric maxi, Hero electric optima, Hero electric photon, TVS Scooty Teenz, YO Xplor, YO Spark, YO ElectronER and YO EXL. Base on availability, charging mechanism of battery for YO Xplor is developed. Technical specifications for YO Xplor is listed in Table 1.

Most electric two-wheelers use a battery pack consisting of 4 identical batteries of 12V. The motor used in electric two-wheelers is a 48V motor with different power depending on the vehicle. So, the four batteries are connected in series to supply a total of 48V to the motor. The project is aimed at a BLDC hub motor of 48V and 250W power. A 250W motor of 48V draws (250/48) = 5.20A current from the battery pack according P = I*V. The main design constraint is to supply this current back to the battery to keep it charged over a operation of the vehicle (10).

To charge the batteries of the electric vehicle, it is needed to supply the electric power back to batteries. For this it is needed to generate this electric power remotely on the vehicle. To accomplish this, an alternator is used (9). To make the equipment cost effective, it is decided to use the alternator of Maruti 800 for the research work. The Maruti 800 alternator has 12V and 45A. The batteries of 12V are charged by connecting in parallel. This makes it possible for the alternator to provide the required 12V for all the batteries. Because of the parallel connection, the current coming from the alternator gets divided in four. Since one battery requires a 5.20A, a total of (5.20*4) = 20.8A current have to generated from the alternator.

Any battery is specified by two ratings: Voltage and Ampere-hour. Of these, the Ampere-hour rating of the battery decides the time for which it can supply a certain amount of current. e.g., a 20Ah battery can

supply 1A current for 20hr or 20A current for 1hr. The selected model of electric two-wheeler uses four 24Ah batteries. The motor used in it draws 5.20A current. The time for which the battery can provide current is the ratio of "Ampere-hour rating of battery" to the "Current drawn from the battery". Time t = 24/5.2 = 4.6 hours.

The output rpm required of the gear train are the rpm required as input to the alternator. From experiments by trial and error method, the rpm required to generate 20A current from the alternator was found to be around400 rpm. The input rpm supplied to the mechanism are given via pedals, i.e., human effort. After a number of trials, it was found that the average rpm in available by pedaling are 70 rpm. The gear ratio is given by,

$$\frac{N1}{N2} = \frac{D1}{D2}$$

Input rpm (N1) = 70
Output rpm (N2) = 400
Gear ratio = 400/70 = 5.71 ≈ 6
Now, this gear ratio cannot be achieved in one single step. So it was divided into two, i.e. 2 and 3.
The diameter of the flywheel is fixed.
The diameter of the flywheel = 18cm
The gear ratio to be achieved = 3
So, the diameter of sprocket required = 18/3 = 6cm
The gear ratio achieved at the flywheel is 3 and gear ratio required at the pulley is 2.
The diameter of the pulley at alternator = 7cm
So, the diameter of the other pulley = 14cm

PRACTICAL WORK

The practical model for charging mechanism of battery is developed using various components like alternator, battery, flywheel, sprocket, pulley, shaft and belts. The mechanism components are selected for modelling based on calculated design parameters. Technical specifications of various components are listed in Table 2.

The model is made to utilize the mechanical energy, enhance it and convert it to electric energy. The mechanical energy is provided to the model by the human effort. For this, pedal mechanism is provided in the model. In the analytical design, it was assumed that the average rpm available while pedalling is 70rpm. But practically, these were reduced to 60rpm. The pedal mechanism turns the flywheel attached to the pedals. The flywheel does the job of distributing the effort equally through the entire cycle. A sprocket is attached with the flywheel by a chain. A gear ratio is kept so that it can turn at greater rpm than the flywheel. A pulley is attached on the same shaft of the sprocket so that the same rpm are transferred to the pulley. This pulley finally turns the alternator pulley via belt drive. The belt is an 'A' size belt with V-threads. This turns the rotor of the alternator so as to generate the electric energy. Figure 3 shows the practical model of "Charging mechanism of battery for electric two wheeler".The practically speeds at flywheel, sprocket, pulley and alternate pulley are measured by performing an experiment. Tachometer was used to measure available speed.

At flywheel
Diameter of flywheel = 18cm
Rpm available = 60
At sprocket
Diameter of sprocket = 7.5cm
Gear ratio = 18/7.5 = 2.4
Rpm available = 60*2.4 = 144
At pulley
Diameter of pulley = 15.2cm

Rpm available = 144 (on the same shaft as sprocket)
At alternator pulley
Diameter of pulley = 7cm
Gear ratio = 15.2/7 = 2.17
Rpm available = 2.17*144 = 312.48

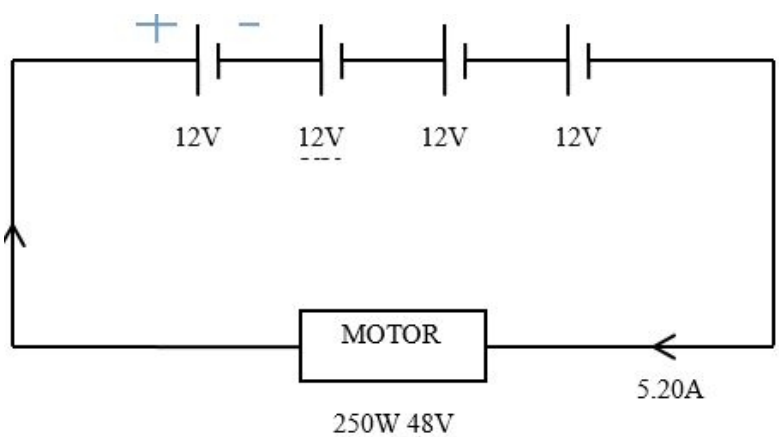
RESULTS AND DISCUSSION

The 20A current is required to charge the battery of YO Xplor electric two wheeler vehicle. Using trial and error method concluded that an alternator is required to rotate at 400 rpm for to produce 20A current. The measured rpm at alternator using tachometer is 313 rpm in developed practical mechanism. The final speed of 313rpm available at the alternator is less than the expected speed of 400rpm during the design of the mechanism. The theoretical speed at pulley is 210 rpm while measured practical speed is 144 rpm. The measured practical speed at sprocket is 144 rpm which is less than theoretical speed of 210 rpm at sprocket. The theoretical speed at flywheel is 70 rpm and measured practical speed at flywheel is 60 rpm. Table 3 shows the comparison between theoretical and practical speeds for flywheel, sprocket, pulley and alternator.

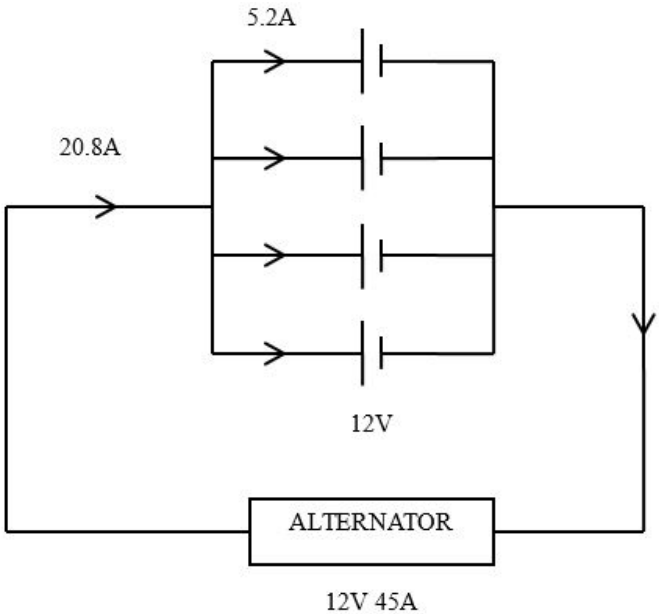
CONCLUSION

The measured practical speeds using tachometer are less than theoretical speeds for flywheel, sprocket, pulley and alternator. Practical speed for the alternator is not as per expected speed hence the current coming out the alternator is less than the expected current 20A. As a result, the mechanism would take more time in charging the battery than the expected time. The practical speed is less than theoretical speed due to the friction between the rotating parts and its misalignments.

FIGURES



"**Fig. 1.** Circuit diagram during discharging (8)."



"**Fig. 2.** Circuit diagram during charging (8)."



"**Fig.3.** Practical model."

TABLES

"**Table 1.** Technical specifications of YO Xplor (5)."

Vehicle Size and weight	
Length*Width*Height	1835mm*830mm*1155mm
Vehicle weight	91kg
Battery	
Type	VRLA deep discharge
Capacity	24Ah
Electricity consumption	1.25 units
Charging time	6 to 8 hours
Motor	
Type	BLDC hub motor
Output power	250Watt
Operational Specifications	
Max speed	up to 25km/hour
Max range	up to 60km/charge
Payload capacity	75kg

"**Table 2.**Technical specifications of components."

Components	**Specifications**
Alternator	12 V , 45 A
Batteries	12 V, 24 Ah
Flywheel	18 cm OD, 1 cm groove
Sprocket	8 cm OD, 1.5 cm groove and 18 Teeth
Pulleys	Bigger pulley has 15 cm OD and Smaller pulley has 7 cm OD
Shaft	Two shafts
Belt	A type timing Belt

"**Table 3.** Comparison between theoretical and practical speed."

Components	Theoretical speeds (rpm)	Practical speeds (rpm)
Flywheel	70	60
Sprocket	210	144
Pulley	210	144
Alternator	420	313

ACKNOWLEDGEMENT

We acknowledge to Institute of Technology and Management Universe Vadodara for providing the facility for experimental work. We heartily thanks to management of ITM Universe for sponsoring our research work costing.

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The application of cloud based casting simulation techniques to manufacture FG 250 Grey cast Iron casting

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ABSTRACT

Casting is a solidification process and simulation of such process is required in industry before it is actually manufactured. The defects like shrinkage, cavity, porosity; sink can be minimized by designing an appropriate feeding system to ensure directional solidification in casting. In this research paper a new gating system is developed to produce sound FG 250 Grey cast Iron casting by cloud based computer simulation. The simulation uses gradient vector method to compute solidification time and optimal riser combinations to obtain defect free casting. The experimental results hold good for producing defect free casting in foundry. Ultimately that results in better quality and better yield.

SUMMARY

Cloud based casting simulation is effective tool for producing quality casting.

Keywords: *Casting simulation, E. foundry, Algorithm, Shrinkage Porosity*

INTRODUCTION

Small and medium scale foundry industries in developing countries suffer from pair quality and productivity due to number of process parameters involved in casting process. It is observed that 90% casting defects are related to solidification and 10% casting defects are related to mould. So it is essential to know the solidification which is taking place inside mould cavity (*4*).

The entire study has been carried out at Krislur Concast Pvt. Ltd. Situated at Bhavnagar, Gujarat. This medium scale industry is doing job work for Lohia Corporation, Kanpur. The drawing of the component (upper ring) is shown in Figure 1. The maximum diameter is 1241 mm and minimum diameter is 1086 mm. the chemical composition of material FG 250 is shown in Table 1.

The part is produced by sand mould casting process. After a few batch of manufacturing, it was observed that a major defect in the casting is porosity. Figure 2 shows the shrinkage porosity observed inside the casting with existing methodology.

MATERIALS AND METHODS

I. Simulation Process:

Casting simulation provides a clear cut insight regarding solidification in mould cavity. This solidification behavior is useful for finding location and extent of internal defects. It helps in identifying high temperature zone (hot spots) (white yellow region) which manifest as shrinkage porosity defects. The defect is usually eliminated by changing the position and size of feeders (Risers) which are designed to solidify later than the hot spot. It supplies liquid metal to compensate the volumetric contraction at the hot spot.

Risers (feeders) are ultimately cut-off and recycled, hence its size and shape should be optimized to ensure high yield. The different solidification views are seen on the computer in graphical form, which help designers to visualize the defects in the process design stage. Further the modified gating designs can be tried without resorting to the actual production of tooling. Here casting process is simulated using cloud based computer simulation which uses Vector Element Method (VEM).

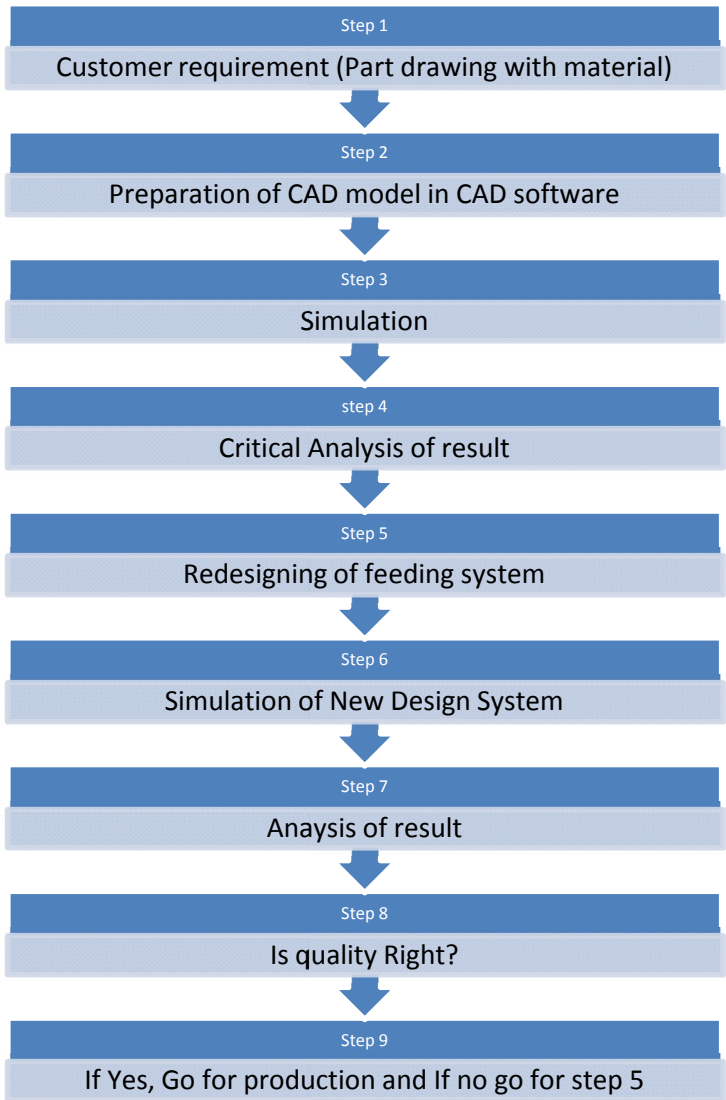
Cloud computing as a concept is very much applicable to casting simulation. Database of casting and casting simulation can be analyzed effectively with the help of cloud computing. In a global market e-resources and information technology can be integrated and it gets accurate results.

From the first simulation of upper ring without gating system, hotspots are observed over the circumference. Using existing gating system design simulation is carried out using cloud base computing system.

Solidification result shows feeder is slightly undersize. There are clear isolated hot spots inside casting, matching with the shrinkage porosity found in actual casting (Fig.2). The simulated result clearly indicates the need of increasing the number of risers as the circumference of the part is large.

II. New Design Methodology

In order to improve the existing methodology, a generic algorithm is prepared for the implementation of new design.



The basic requirements for the feeder system in the casting:

The feeder must be thermally adequate means the solidification time of metal in the feeder must be greater than the solidification time of metal in the mold, so that it can feed enough metal to the casting in order to compensate volumetric shrinkage during solidification (*12*).

According to Chvorinov's Equation
Solidification time : $\sqrt{t_s}$ = K (V/A)

Where t_s = Solidification time of casting in second

V= Volume of casting in mm^3

A= Surface area of casting in mm^2

K= Mould constant or solidification constant, which depends on the characteristics of the metal being cast and the mould material

K is found out by using formulae

$$K= \rho_{cast} (L+C_{cast} (T_{pour} – T_{sol})) / 1.128 (T_{int} – T_{amb}) \sqrt{K_{mould} \rho_{mould} C_{mould}}$$

Following values are taken for the calculation

ρ_{cast} = Density of the liquid metal = 7200 kg/m^3

L = Latent heat of metal = 272000 J/kg

C_{cast} = specific heat of cast metal = 510 J/kg K

T_{pour} = Pouring temperature = 1450°C

T_{sol} = Solidus temperature = 1390°C

T_{amb} = Ambienttemperature = 38°C

T_{int} = Interface temperature = $0.9 \times T_{sol} = 0.9 \times 1380 = 1242$°C

ρ_{mould} = Density of mould material = 1540 kg/m^3

K_{mould} = Thermal conductivity of mould material = 0.61 J/mKs

C_{mould} = Specific heat of mould material = 1170 J/kg K

Now

$K = 7200(272000+510(1450-1390)/1.128(1242-38) \sqrt{(0.61\times1540\times1170)} =$ **1530**

Riser shape:

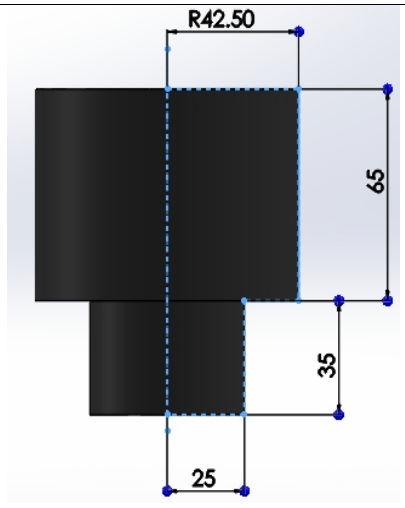
Spherical feeders are usually difficult to mould and it presents feeding problem, since the last metal to freeze would be near the centre of the sphere.The riser with cylindrical shapes is favourable as it presents the smallest possible surface-volume ratio.

Case 1: Upper ring 85 mm with One Gate and Three Risers

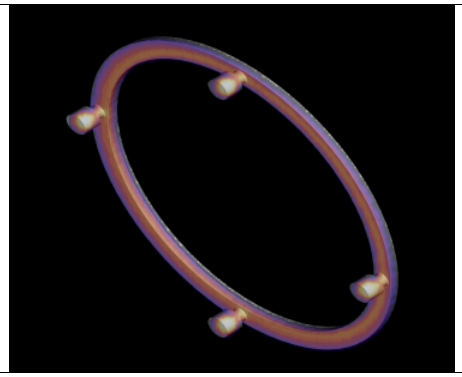
Surface Area A(mm^2) = 34204.0

Volume V (mm^3) = 437564.9

Modulus = Volume/Surface Area = 12.79 mm

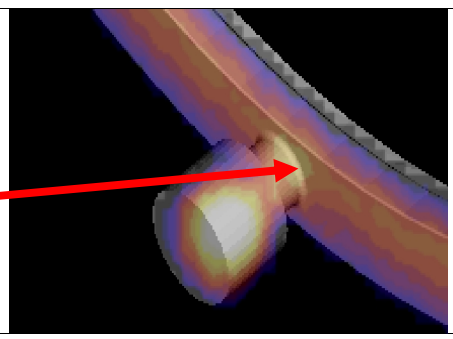


Gate and Riser Shape and Size



Simulated Result

Isolated Hotspots inside the casting indicate under sizing of riser



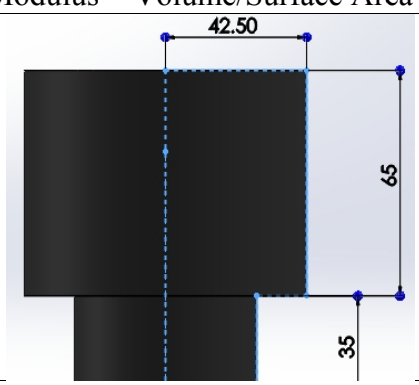
Enlarge View

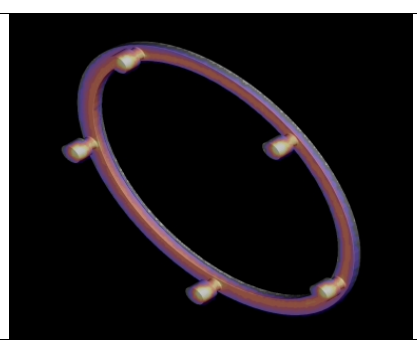
Case 2: Upper ring 85 mm with One Gate and Four Risers

Surface Area A (mm^2) = 34753.8

Volume V(mm^3) =451996.6

Modulus = Volume/Surface Area =13.0mm

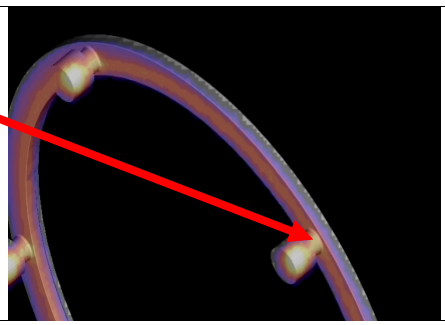




Simulated Result

Gate and Riser Shape and Size

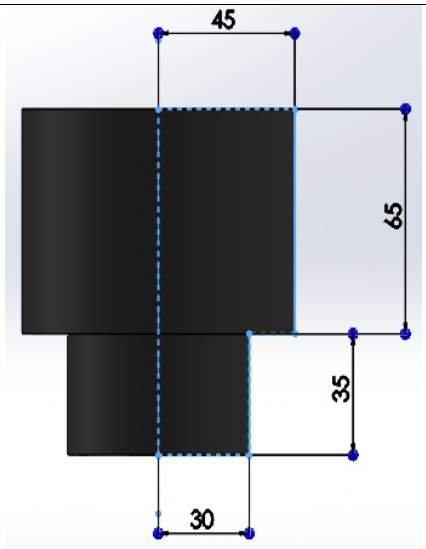
Hotspots are not completely shifted inside riser



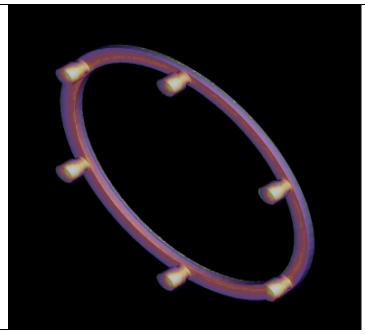
Enlarge View

Case 3: Upper ring 90 mm with One Gate and Five Risers

Surface Area A(mm^2) =37699.1
Volume V(mm^3) = 512472.3
Modulus = Volume/Surface Area =13.59mm

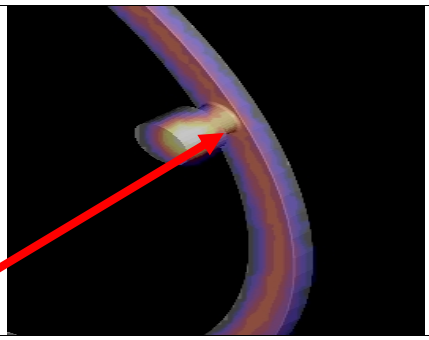


Gate and Riser Shape and Size



Simulated Result

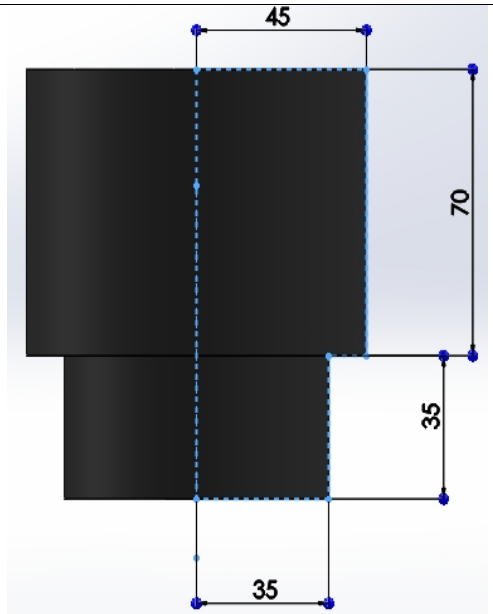
Hotspots are not completely shifted inside riser



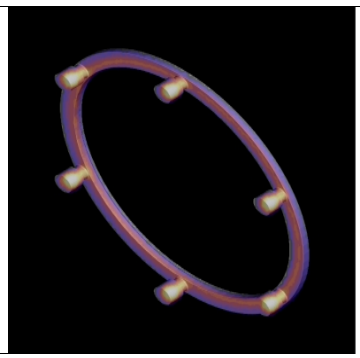
Enlarge View

Case 4: Upper ring 90 mm with One Gate and Five Risers

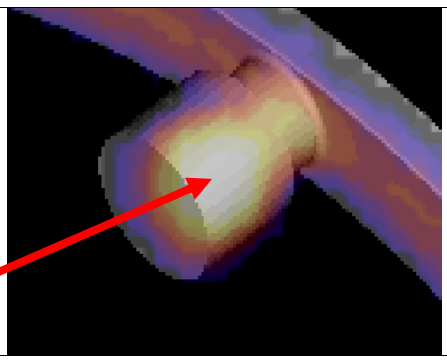
Surface Area A (mm^2) =40212.3
Volume V (mm^3) =580016.5
Modulus = Volume/Surface Area =14.42mm



Gate and Riser Shape and Size



Simulated Result



Enlarge View

Hotspots are completely shifted inside riser ensuring directional solidification

Calculation for Case 4

Solidification time for riser $\sqrt{t_s}$ = K (V/A) =1530*14.42*10^{-3}s

t_s= 486.7s=8.11min.

Solidification time for part $\sqrt{t_s}$ = K (V/A) = 1530*10.42*10^{-3}

t_s= 254.5s=4.24min.

Casting yield for last iteration

= vol. of the actual casting/(vol. of the casting + vol. of gating and riser system)

=10359140.30/(10359140.3 + 6(580016.5))

=75%

RESULTS AND DISCUSSION

From above simulated results it is observed that by varying the location and size of riser the hot spots position can be shifted. In last case 4 hot spots are completely shifted in risers and part becomes hot spot free. Ultimately that results in directional solidification. More over casting yield is 75% which is optimum.

By applying cloud based casting simulation, small and medium scale foundry can able to minimize the bottlenecks and no value added time in casting development, as it reduces the no. of trials casting required on the shopfloor.

CONCLUSION

From this work it is concluded that cloud based computer simulation gives insight about solidification in casting of any shape. Solidification related defects like shrinkage, porosity, cavity, and voids can be minimized by altering the method design.Small and medium scale foundries can increase their top line and bottom line by adopting new technology like cloud based casting simulation.

Figures

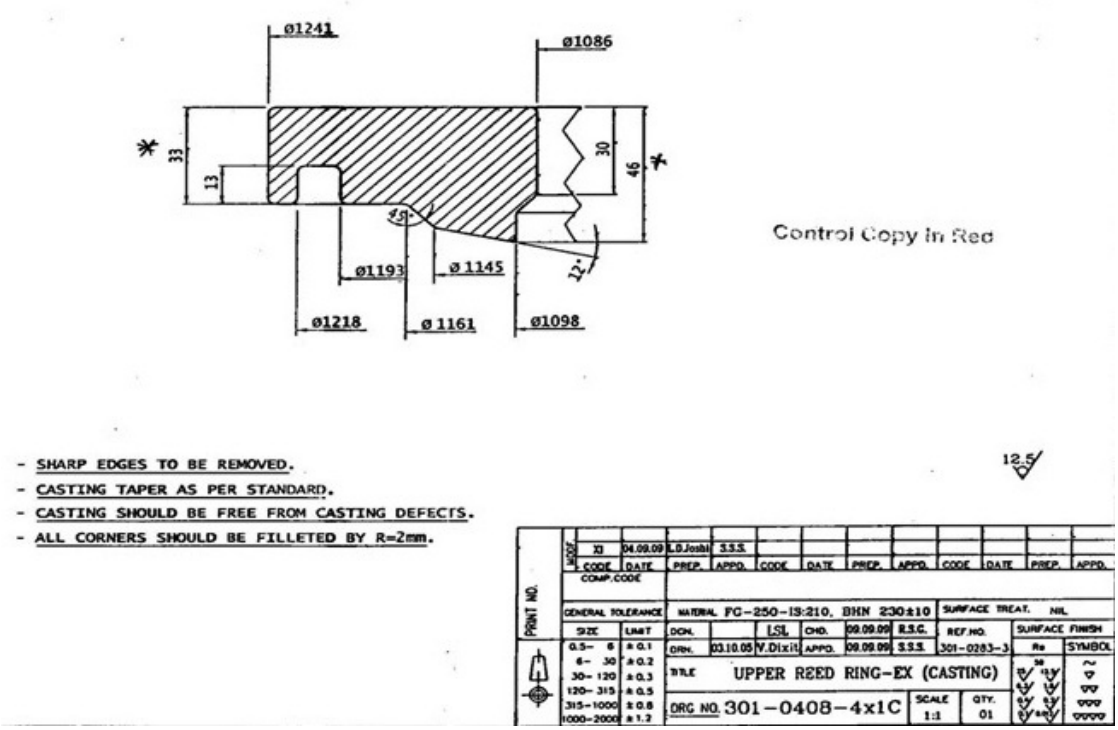


Fig.1: Component drawing (upper ring)



Fig. 2: Shrinkage porosity observed inside the casting at different locations

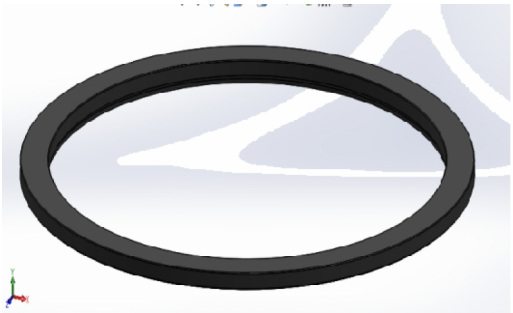


Fig. 3 CAD Model of part (upper ring)

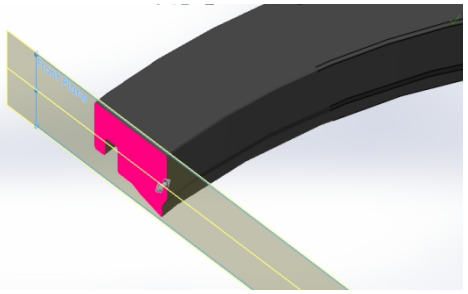
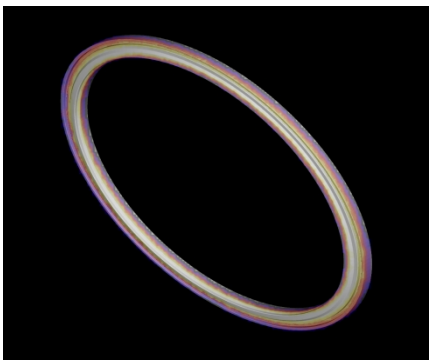
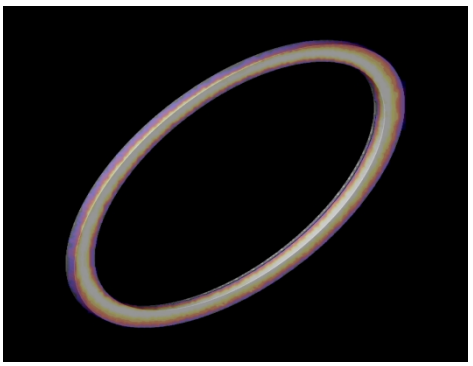


Fig. 4 Cross-section of part (upper ring)



Back View



Front View

Fig.5 Simulation of Upper Ring without Gating System

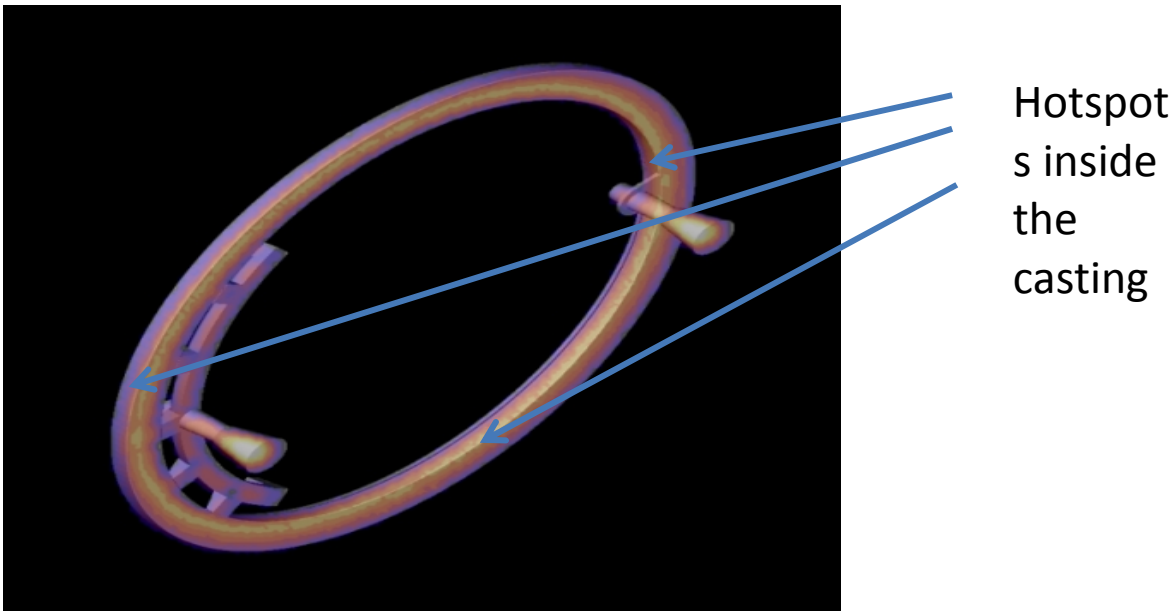


Fig.6 Simulated result of upper ring in existing gating system design



Fig.7 Mold filling for new methodology



Fig.8 Slotted View of Upper ring



Fig.9 closed view ofMachined Surface (part free from shrinkage porosity)

TABLES

Table1: Chemical composition of FG 250 Grey Cast-iron

Elements	Iron, Fe	Carbon, C	Silicon, Si	Magnesium Mn	Sulphur, S
Composition %	90	3.3	2.1	0.67	0.08

ACKNOWLEDGEMENT

I express my deep sense of gratitude to Dr. M.G.Bhatt Principal S.S.Engg.College Bhavnagar for providing guidance for the development of this work. I am also thankful to Shri Rajubhai Proprietor of Kslur concast Pvt. Ltd. Bhavanagar for providing the opportunity to carryout experiment work.

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Parallel (Edge) Coupled Microstrip filter for C-Band Applications

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ABSTRACT

This Parallel Coupled Microstrip Filter operating at 6 GHz for C-Band is mathematically designed and modelled, numerically analyzed, iteratively simulated, fabricated and tested through experimental measurements. Physical dimensions and parameters such as space, length, and width of the filter are found using the standard odd and even impedance method. These parameters are used to design and optimally simulate the CAD model using CST Microwave Studio. This filter is fabricated on flame retardant4 substrate considering no adverse electrical-effects and unknown variations in its permittivity when used for real time C-Band applications. Different electrical performance-parameters such as insertion loss and reflection loss of this parallel coupled microstrip filter are measured using a practical experimental set-up in the laboratory and compared the same with predicted analysis and results. The values obtained from simulations and measurements are encouraging in terms of enhancing the electrical behaviour and performance of this filter and are scientifically acceptable.

SUMMARY

Satisfactorily Designed and Realized Parallel Coupled Microstrip Filter for 6 GHz using Electromagnetic Numerical Analysis of CST Microwave Studio, Fabricated-Tested-Measured for Electrical Parameters to assure Performance in Comparison with International-Technical Standard.

Keywords: Flame retardant 4 (FR-4), Bandpass filter (BPF), Fractional bandwidth (FBW), Computer simulation technology (CST)

I. INTRODUCTION

A microstrip low profile filter is a circuit that facilitates transmission of microwave signals and energy within passband, and also rejection for stopband frequency range. This microstrip filter is a two port network circuit. This has been designed using Insertion Loss Method rather than using the Image Parameter method (1).

The image parameter method is less compatible for improvement of the design of the filter performance. So, mostly the insertion loss method is preferred as it has desired control over the passband and stopbandcharacteristics.

The designed parallel coupled microstrip bandpass filter allows frequencies above lower cutoff frequency and belowupper cutoff frequency. The frequency range between lowerand upper cutoff frequency is referred as passband frequency range. If we combine low pass filter and highpass filter, it will become bandpass filter. The lowpass filter is transformed into band pass filter because it functions as a good resonator. Mechanical parameters like size and weight can be reduced by using microstrip filter. Figure 1 shows the basic structure of the microstrip filter. It consists of a rectangular shape metallic sheet with dielectric constant (ε_r) and height (h) (1).

The microstrip filters are having small size, low cost and lighter weight. It is also giving better performance compared to other filters. Microstrip band pass filters can be designed with the help of many topologies like end-coupled, half-wavelength resonator filters, parallel-coupled, half-wavelength resonator filters, hairpin line bandpass Filters, interdigital bandpass filters, combine filters, pseudo combine filters, stub bandpass filters. Because of the relatively weak coupling micro-strip coupled line filters has been used to attain narrow bandwidth band pass filters (2, 3).

Parallel coupled Microstrip filter is used to get good scattering parameters and performance characteristics from its n+1 sections compared to single parallel coupled microstip bandpass filter. Parallel coupled Microstrip filter has planar structure and wide bandwidth so it is more popular and its design procedure is quite understandable compared to design of other filters. The design of this filter provides sufficient space gap between coupled sections for improvement of the coupling efficiency. This paper shows the applications of filter in C band which is widely used for satellite communication and wireless communication. This filter is designed for operating frequency of 6 GHz which lies in C band. Return loss and transmission loss of this filter is quite good compared to the result of other experimental research (7, 8).

Section II of this paper discusses about the design of this proposed edge coupled Microstrip filter. It also highlights the mathematical calculations for mechanical dimensions using known formulae and methods. The later part of this paper represents the research carried out through simulations of the prototype CAD model in the CST microwave studio and the electrical results are discussed in the next section IV of this paper. These results are compared with the measured results of this filter fabricated in the laboratory and discussed in the section V of this paper.

II. DESIGN OF PROPOSED FILTER

The flow of design procedure for this filter considered comprises of determination of attenuation characteristics, values of prototype elements, invertors values, odd and even impedance values, width, separation and length of each couple section.

Fig.2 shows the general structure of parallel coupled microstrip bandpass filter. The width of coupled section is indicated by W_1, W_2, W_s, W_{s+1} in fig. 2. The length of coupled line is mentioned by l_1, l_2, l_s, l_{s+1}. The spacing between coupled sections is given by S_1, S_2, S_s, S_{s+1}. The mathematical analysis is adopted for the calculation of width, length and space of the parallel coupled Microstrip filter.

The resonant frequency of design can be expressed in (1) to find the relation and dependency between ω_0, ω_1 and ω_2.

$$\omega_0 = \sqrt{\omega_1 \omega_2} \qquad (1)$$

Where, ω_0 is the center frequency and ω_1, ω_2 are the band edge frequencies. Re-expressing the frequency relations for FBW, the fractional bandwidth in (2) makes the concept clear.

$$\text{FBW} = \frac{w_2 - w_1}{w_0} \qquad (2)$$

The mathematical relations between the parameters for analyzing and designing the parallel coupled Microstrip bandpass filter are expressed in (3)-(5).

$$\frac{J_0}{Y_0} = \sqrt{\frac{\pi}{2} \times \frac{Fl}{g_0 - g_1}} \qquad (3)$$

$$\frac{J_{0\ +1}}{Y_0} = \frac{\pi}{2} \times \frac{\pi 1}{g_0 g_1} \text{ for j = 1 to n-1} \qquad (4)$$

$$\frac{J_{y\ 1+1}}{Y_0} = \sqrt{\frac{\pi}{2} \times \frac{Fl}{g_0 g_{1+1}}} \qquad (5)$$

Where g_0, g_1,g_n are the elements of a ladder – type low pass prototype with a normal cut off frequency ω_0 = 1, and FBW, the fractional bandwidth of band pass filter. $J_{j,\ j\ +\ 1}$ are the characteristic admittances of j inverters and Z_0 is the characteristic impedance of the terminating line. The even and odd mode characteristic impedances of the coupled strip line bandpass filter are determined using (6) and (7) (2, 3).

$$(Z_{oo})_{j,\ j\ +1} = \frac{1}{Y_0}\left[1 + \frac{J_{j,j+1}}{Y_0} + \left(1 + \frac{J_{j,j+1}}{Y_0}\right)^2\right] \qquad (6)$$

$$(Z_{oe})_{j,\ j\ +1} = \frac{1}{Y_0}\left[1 - \frac{J_{j,j+1}}{Y_0} + \left(1 + \frac{J_{j,j+1}}{Y_0}\right)^2\right] \qquad (7)$$

The values of Z_{oo}, Z_{oe}, length, width and line spacing for each coupled line are calculated and depicted in the Table I. The substrate used is a standard FR4 substrate with dielectric constant (ε_r) = 4.4, Height (h) = 1.56 mm, Thickness (t) = 0.035 mm and Loss tangent (Tanδ) = 0.025. The calculated values of width, length and space are rounded off to one decimal point to minimize the possibility of fabrication errors. Therefore, the test and measurement results when compared with the simulation and analysis results are found deviating in an acceptable range of less than 10% which is valid for microwave communication applications (4-6).

The table 1 presents the electrical parameters such as odd and even impedances for the values of n ranging between 1 – 4, while the other columns of the table I represent the mechanical parameters of the filter operating at 6 GHz such as width (W), Length (L) and Space (S). As obvious for the fabrication reasons, the values of these parameters shown in table 1 are rounded off by one decimal point.

III. SIMULATION OF THE PARALLEL COUPLED BANDPASS FILTER

The parameters of the filters have been designed and calculated as shown in table 1. From these data, the filter is realized in CST software and the simulated result is achieved. The simulated results show that insertion loss and reflection loss.

The dimensions of the designed filter are shown in figure 3. The length of the filter for different sections is 6 mm and widths are 4 mm and 2.4 mm. The size of the board is 32 mm*22.8 mm. The spacing between all the strips is 0.2 mm. The distance of 5 mm kept on both sides to apply the source signal.

The simulation result of the designed filter is shown in figure 4. The numerical value 1 in figure 4 indicates lower cutoff frequency for -5 dB and numerical value 2 indicates upper cutoff for -5 dB value of the magnitude of S parameter. Numerical value 3 indicates that at about 5 GHz the magnitude of the reflection coefficient is below -30 dB. The reflection loss of this filter is about -40 dB for 7 GHz as indicated by numerical value 4 and it is about -25 dB for 6 GHz indicated by numerical value 5. The value of return loss is achieved below -10 dB for three frequencies for this filter and transmission loss is also above -10 dB for this designed parallel coupled microstrip filter.

IV. FABRICATION AND MEASUREMENT OF THE PROPOSED FILTER

Figure 5 shows the realized filter which is fabricated on FR4 substrate. On both sides of this filter gold plated female pcb mounted SMA connector is connected. These SMA connectors are connected to scalar network analyzer to measure the S parameters of this parallel coupled microstrip filter. The size of the fabricated filter is 32 mm * 22.8 mm. This fabricated structure of parallel coupled microstrip filter is tested and measured using scalar network analyzer.

Test and measurement setup for parallel coupled bandpass filter is shown in figure 6. As depicted from the figure 6 that the HP8757scalar network analyzer, device under test (DUT), Frequency generator etc. are used for test and measurement. This fabricated filter is tested for operating frequency of 6 GHz. This filter is tested and measurement of transmission loss and return loss has been carried out for 6 GHz operating frequency. The notation A in figure 6 indicates the HP8757 scalar network analyzer which is used for scattering parameter measurement and it's used for measurement of 10 MHz to 100 GHz. The notation B indicates the sweep generator which is required to generate frequency of 3 GHz to 9 GHz for measurement. The notation C of figure 6 device under test (DUT) which is fabricated 6 GHz parallel coupled microstrip filter operated at 6 GHz and fabricated on FR4 substrate.

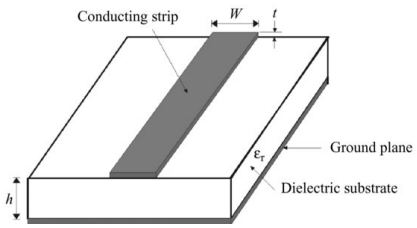
V. RESULT COMPARISON AND CONCLUSION

To obtain good electrical performances from the proposed filters, the designed filter was simulated using CST simulation software. The simulated results are shown in figure 4. The filter is realized on FR4 substrate and the result of fabricated filter are shown in figure 7 and figure 8. Figure 7 shows reflection loss is achieved about -36 dB. Figure 8 shows that the transmission loss is achieved about -9 dB. The results which are received at the desired operating frequency of 6 GHz are very good and useful for microwave applications. An HP8757A Scalar Network Analyzerwas used to measure the response of the proposed filter and the results are shown in Figures 7-8.

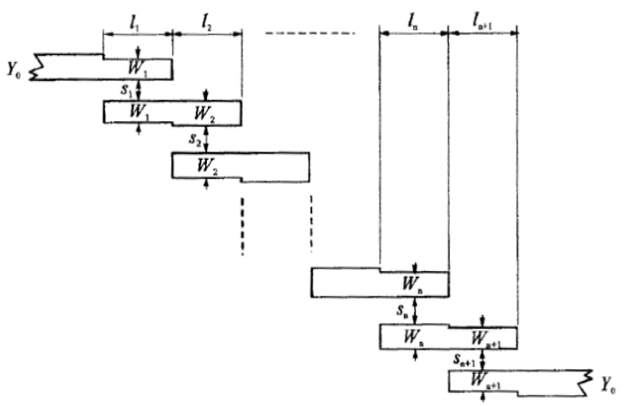
The scattering parameter S_{11} of fabricated microstrip filter is shown in figure 7. Figure 7 shows that the filter is tested and measured for the frequency range of 3 GHz to 9 GHz. The horizontal axis of the figure graph shown in figure 7 indicates frequency and vertical axis of the graph indicates magnitude of S parameter. The notation X indicates that at frequency of 5.96 GHz the value of magnitude of return loss S_{11} is -36 dB which is desirable and expected from the analysis and design of the filter.

Figure 8 shows the measurement of transmission loss (S_{12}) of fabricated parallel coupled microstrip bandpass filter operated at 6 GHz. This filter is tested and measurement of S_{12} is about -7 dB for frequency of 6.85 GHz as indicated by notation Y. The notation X indicates the measurement of S_{12} for frequency 6 GHz which is about -9 dB. Measurement is done for the frequency of 3 GHz to 9 GHz.

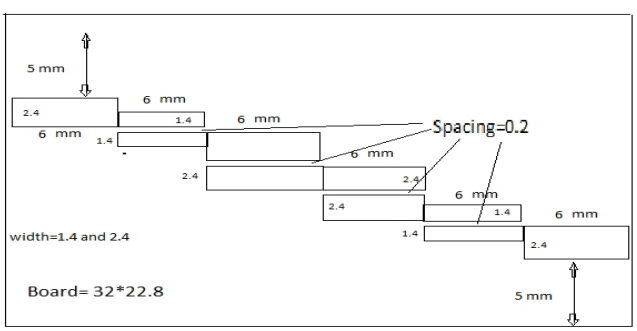
FIGURES



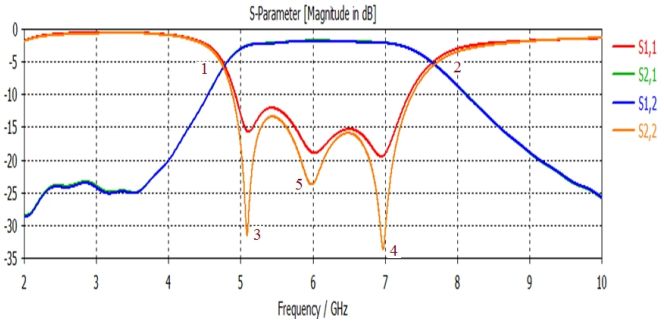
"**Fig. 1**. Basic structure model of a standard microstrip low profile filter" [1]



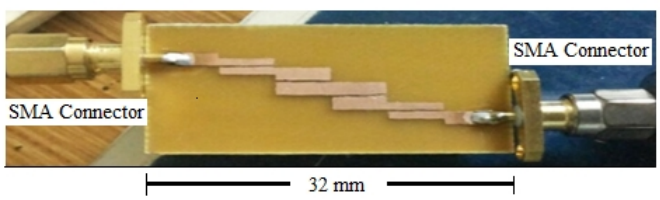
"**Fig.2**. General structure of parallel (edge) coupled microstrip bandpass filter" [2]



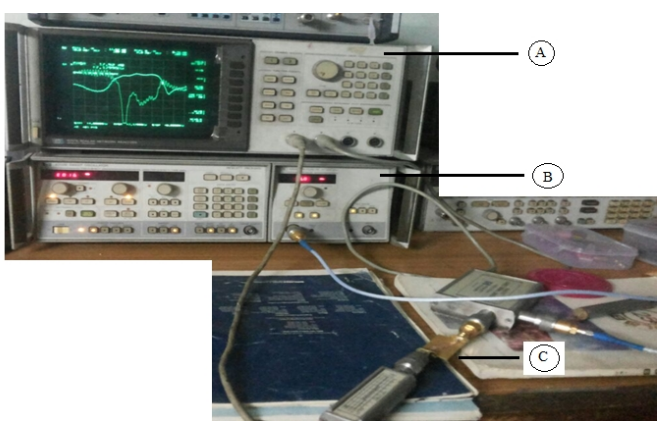
"**Fig. 3.**Dimension of Microstrip parallel coupled band-pass filter designed at 6 GHz"



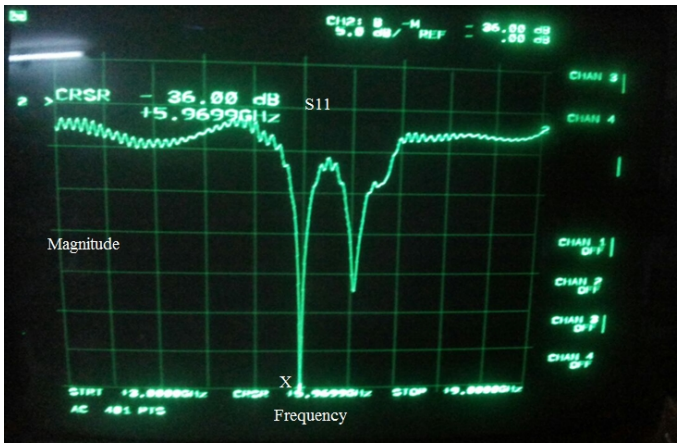
"**Fig.4.**Simulated Performance of threepole microstrip parallel coupled bandpass filer"



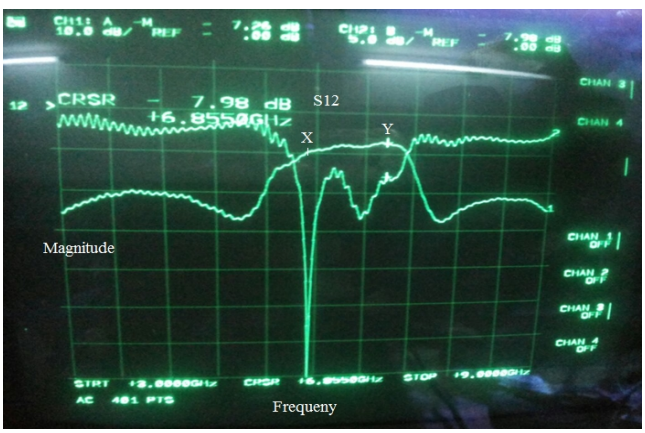
"**Fig. 5.** Fabricated structure of the parallel coupled bandpass filter at6GHz"



"**Fig. 6.** Measurement setup for parallel coupled bandpass filter at6GHz"



"**Fig. 7.** Measured Reflection loss (S_{11}) parallel coupledbandpass filter using Network Analyzer"



"**Fig. 8.**Measured transmission loss (S_{12}) of parallel coupledbandpass filter using Network Analyzer"

TABLE

"**Table 1.** Calculated electrical and mechanical parameters of parallel coupled bandpass filter for 6 GHz"

n	$Z_{oo}(\Omega)$	$Z_{oe}(\Omega)$	W(mm)	L(mm)	S(mm)
1	101.5	38.5	1.4	6	0.2
2	71	39	2.4	6	0.2
3	71	39	2.4	6	0.2
4	101.5	38.5	1.4	6	0.2

ACKNOWLEDGEMENT

The research scholar expresses his gratitude to MBICT (A CVM institution at Vallabh Vidyanagar, Gujarat, India) where he is serving as Assistant Professor, and the RK University, Rajkot, Gujarat, India fromwhere he is pursing for his Ph D, for the consistent encouragement and support.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

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ATC Enhancement by Optimal Placement of Facts Device Using Grey Wolf Optimizer Algorithm

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ABSTRACT

In this paper, impact of FACTS devices for available transfer capability (ATC) augmentation is studied by optimal placement and compensation of thyristor controlled series compensator (TCSC) device using meta-heuristic GWO optimization technique. The proposed technique finds the optimal location of TCSCs and compensation of each along with ATC. This algorithm is inspired by the leadership ranking and hunting method of grey wolves. The proposed method has been applied to standard 39-bus New England system to demonstrate the effectiveness, keeping all constraints within specified limits. For the comparison purpose, well-known genetic algorithm is also applied. Results show that GWO algorithm can also be a very effective tool for TCSC placement.

SUMMARY

The main contribution of this paper is to apply a new meta-heuristic grey wolf optimization (GWO) technique based on grey wolf's leadership ranking and hunting methodology for ATC enhancement, by optimal placement of TCSC facts device in a 39-bus New England system.

Keywords: Available transfer capability (ATC), Grey wolf optimizer (GWO) meta-heuristic algorithm, Thyrister controlled series compensator (TCSC), Genetic algorithm (GA).

INTRODUCTION

ATC calculation has been a research area of exponentially increasing interest, due to economic and reliability consideration, particularly in the past two decades. The aspire at the back of restructuring electricity market is to bring some form of competition among the market participants, open access to all, to provide options and benefits to the end user customers. Open access leads some undesirable impacts such as heavier line loadings and increased loop flows(*1*). This necessitates the calculation of ATC of transmission path (*2*) in deregulated electricity markets. Adequate ATC is needed to ensure market security and to accommodate probable market power bid transactions. However, various problems such as social, economic and environmental impose restriction for new transmission network construction. This situation forces and motivates us to use existing facilities at its best(*3*) using other ways such as optimal setting of transformer tap and generator voltage, rescheduling of real and reactive power (*4*), and application of FACTS devices(*5*). Introduction of FACTS devices changed drastically the way of power system operation.

In order to locate the competitive location for new TCSCs with its compensation, to use the existing transmission system efficiently, in a power system, prior study regarding its optimal compensation and location, and possible enhancement in ATC has to be carried out. Furthermore, as compared to deterministic optimization methods, meta-heuristics algorithm have superior abilities to avoid local optima entrapment due to its heuristic nature. Real world problems are very complex with huge number of local optima, so meta-heuristics are better options for optimizing these real world problems. The rapid development in the field of heuristic optimization motivates us to apply different techniques for same problem solution *(6)*.

The main objective of this paper is to apply a new meta-heuristic GWO technique for ATC enhancement by optimal placement of facts device in a 39-bus New England power system. The New England 39-bus system data is extracted from *(7)*. This paper is organized as follows: Section 1.1 demonstrate ATC calculation using RPF methodology; Section 1.2 provides brief view for meta-heuristic GWO Algorithm; Section 1.3 illustrate modeling of TCSC device; Section 1.4 provides ATC enhancement problem formulation by optimal placement of TCSC using GWO Algorithm; Result and Discussion section provides results obtained; and at the last, conclusion is provided.

MATERIALS AND METHODS

1.1. ATC Calculation Using RPF

North American Electric Reliability Council (NERC) defines ATC as a "measure of transfer capability remaining in the physical transmission network for further commercial activity over and above already committed uses (*1*).

Mathematically, ATC is defined as the total transfer capability (TTC) less the transmission reliability margin (TRM), less the sum of existing transmission commitments (ETC) (which includes retail customer service) and the capacity benefit margin (CBM)". ATC can be expressed as:

$$ATC = TTC - TRM - ETC - CBM \qquad (1)$$

In (*8*), G.C.Ejebe et al. reported a novel formulation of the ATC problem based on full AC power flow solution to incorporate the effects of reactive power flows, voltage limits and voltage collapse as well as thermal loading effects. RPF method implementation for ATC calculation is simple, and suitable for large-scale power systems. In RPF method, the system load and power generation, for specified bilateral

transaction buses, will be increased by a specified rate and continues until one of the system operating limits related to ATC is violated,as follow:

Maximize: ATC

Subjected to:

$$P_i - \sum_{j \in i} V_i V_j (G_{ij} \cos\theta_{ij} + B_{ij} \sin\theta_{ij}) = 0 \quad (2)$$

$$Q_i - \sum_{j \in i} V_i V_j (G_{ij} \sin\theta_{ij} - B_{ij} \cos\theta_{ij}) = 0 \quad (3)$$

$$P_i = P_{Gi} - P_{Di} \text{ and } Q_i = Q_{Gi} - Q_{Di} \quad (4)$$

$$P_{Gi}^{min} \leq P_{Gi} \leq P_{Gi}^{max} \quad (5)$$

$$Q_{Gi}^{min} \leq Q_{Gi} \leq Q_{Gi}^{max} \quad (6)$$

$$|V_i|^{min} \leq |V_i| \leq |V_i|^{max} \quad (7)$$

$$S_{ij} \leq S_{ij}^{max} \text{ and } S_{ji} \leq S_{ji}^{max} \quad (8)$$

ATC is a scalar representsincrease of load in MW at a demand bus above base case in the bilateral transaction. P_{Gi} is the real power generation at bus i in MW. Q_{Gi} is the reactive power generation at bus i in MVAr. P_{Di} is the real power demand at bus i in MW. Q_{Di} is the reactive power demand at bus i in MVAr. $|Vi|$ is the voltage magnitude at bus i. S_{ij} is the apparent power transfer from bus i to bus j and $S_{ij\text{-}max}$ is the maximum permissible apparent power transfer from bus i to bus j in line L_{ij}.

1.2. Grey Wolf Optimizer

At first Seyedali Mirjalili et al. (*9*) introduced meta-heuristic, Grey Wolf Optimizer (GWO) in 2014. This algorithm mimics the headship chain of command and hunting method of grey wolves. For leadership hierarchy simulation, four search agents (grey wolves) are used as follow:

- Alpha grey wolf (most dominant i.e. best solution/search agents)
- Beta grey wolf (obeys Alpha and orders omega i.e. second best solution/search agents)
- Delta grey wolf (obeys Alpha and orders omega i.e. third best solution/search agents)
- Omega grey wolf (least dominant and obeys all)

Three important steps for hunting mechanism are as follow:

- Searching for prey (i.e. solution)
- Encircling prey (i.e. identifying solution region)
- Attacking prey (i.e. moving toward solution)

Table 1. Pseudo code for GWO (*9*)

1.	Initialize the population Xi (i = 1, 2, ..., n)
2.	Initialize a, A, and C

```
3.      Calculate the fitness of each wolf i.e. search agent
4.      Xα=alpha wolf i.e. best solution
5.      Xβ=beta wolf i.e. second best solution
6.      Xδ=delta wolf i.e. third best solution
7.      while (t < Max_iter)
8.          for each search agent
9.              Update the position by equation (15)
10.         end for
11.         Update a, A, and C
12.         Calculate the fitness of all search agents
13.         Update Xα, Xβ, and Xδ s
14.         t=t+1
15.     end while
16.     return Xα
```

Various equations governing GWO algorithm are as follow:

$$\vec{D} = \left| \vec{C}.\overrightarrow{X_p}(t) - \vec{X}(t) \right| \quad (9)$$

$$\vec{X}(t+1) = \vec{X}_p(t) - \vec{A}.\vec{D} \quad (10)$$

$$\vec{A} = 2\vec{a}.\vec{r}_1 - \vec{a} \quad (11)$$

$$\vec{C} = 2.\vec{r}_2 \quad (12)$$

$$\vec{D}_\alpha = | \vec{C}_1.\vec{X}_\alpha - \vec{X} | .\vec{D}_\beta = | \vec{C}_2.\vec{X}_\beta - \vec{X} | .\vec{D}_\delta = | \vec{C}_3.\vec{X}_\delta - \vec{X} | \quad (13)$$

$$\vec{X}_1 = \vec{X}_\alpha - \vec{A}_1.(\vec{D}_\alpha).\vec{X}_2 = \vec{X}_\beta - \vec{A}_2(\vec{D}_\beta).\vec{X}_3 = \vec{X}_\delta - \vec{A}_3.(\vec{D}_\delta) \quad (14)$$

$$\vec{X}(t+1) = \frac{\vec{X}_1 + \vec{X}_2 + \vec{X}_3}{3} \quad (15)$$

Where, t is the current iteration, and $\vec{A}$ and $\vec{C}$ are coefficient, $\vec{X}_p$ is the position of the prey, and $\vec{X}$ indicates the position of a grey wolf. Where components of $\vec{a}$ are linearly decreased from 2 to 0 over the course of iterations and, $\vec{r}_1$ and $\vec{r}_2$ are random vectors in [0,1].

Equation (9) and (10) mathematically model the encircling behavior of the grey wolf during hunting. Equation (13), (14) and (15) model the hunting mechanism of grey wolf. When |A| < 1, it offers local search (exploitation) and for |A| > 1, it offers global search (exploration). The exploration, exploitation, local optima avoidance, and convergence capability of GWO is competitive than the other heuristic algorithms for various benchmarked functions (*9*).

1.3. TCSC Modeling

A TCSC is a series-controlled inductive or capacitive reactance "$\pm jX_{TCSC}$" inserted in a lumped π equivalent transmission line circuit as shown in **Error! Reference source not found.** and **Error!**

Reference source not found.(*10*). When TCSC is operated in capacitive reactance mode, it reduces the reactance of line thereby increase power transfer and during inductive reactance mode, it increases reactance of line thereby reduces power transfer. In this paper, TCSC is operated in capacitive and inductive mode whose value varies from $-0.4X_L$ to $+0.4X_L$ where $X_L$is the reactance of transmission line to which TCSC is connected.

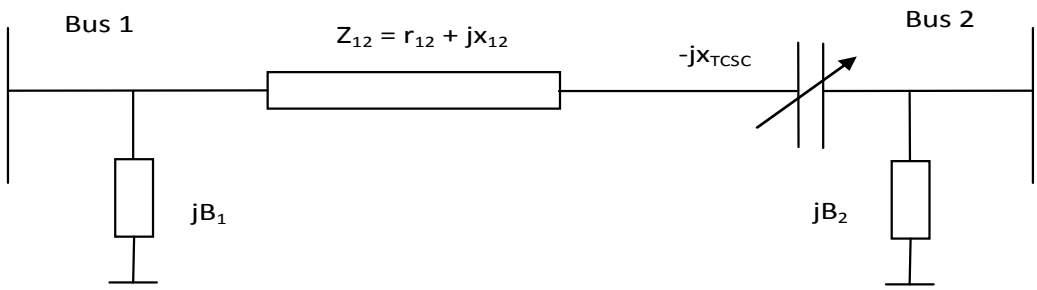


Fig. 1. TCSC model for capacitive operative Region

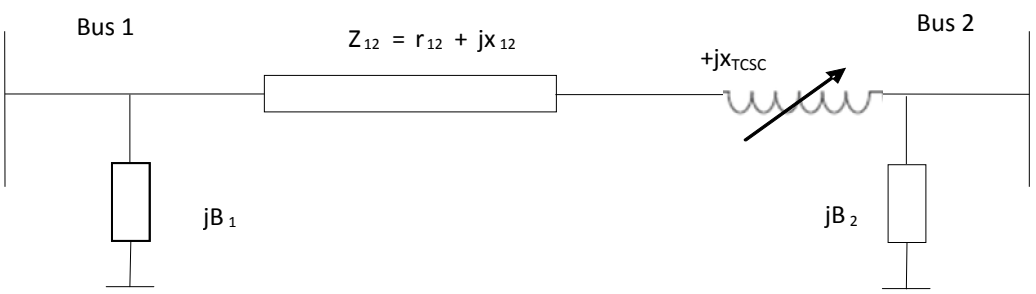


Fig. 2. TCSC model for inductive operative Region

1.4. Problem Formulation

ATC enhancement by optimal placement of TCSC is the power flow problem that gives the optimal setting of decision variables by maximizing ATC objective function. However, the source (positive power injection) and the sink (negative power injection) of identified bilateral transaction vary, while all real power parameters of other buses (total real generation and consumption at other buses) remains unaltered in a deregulated power market ATC calculation. ATC calculation using GWO uses the set of decision variables are mentioned in the following table.

Table 2. Decision Variables

1. Location of TCSCs , L_{TCSC}
2. Compensation level of each TCSC, $\pm jx_{TCSC}$

Various steps for ATC enhancement by optimal location of TCSC device, using GWO is given in Section 1.5.

1.5. GWO steps for Optimal TCSC placement for ATC enhancement

1. Read the base case (power system data) and its variable limits.
2. Read the decision variables.
3. Initialize GSO algorithm first generation.
4. Calculate ATC by RPF method (fitness).
5. Update decision variables.
6. Calculate ATC by RPF method (fitness).
7. Go to step 5 until stopping criteria met.
8. Display results.

ATC for the each of the stipulated source and sink is computed for 39-bus New England power system. ATC enhancement by optimal placement of one, two and three TCSC device with its optimal compensation has been studied. ATC values are calculated for base case, without contingency, and with selected line outage. The amount of compensation (reactance) offered by TCSC is +40% to -40% of transmission line in which it is placed.

RESULTS AND DISCUSSION

The whole study is divided in four stages:

1. Stage 1 (Setting up algorithm and its parameter)

At first the optimization tool genetic algorithm and GWO is tested for population size, maximum iteration and some algorithm specific parameters for optimal convergence.

2. Stage 2 (Base case ATC calculation)

For the chosen base case, different bilateral transactions with and without line outage are assumed for study. The results of the 39-bus New England power system are depicted in Table 3 and **Error! Reference source not found.**. Line i-j means line connecting bus i and bus j. Tables show that ATC is restricted by line flow violation; hence, TCSC can be engaged for ATC enhancement.

3. Stage 3 (ATC enhancement by GWO and GA)

There are various possibilities for numbers of TCSC placement and its compensation for ATC enhancement, hence, this is an optimization problem. The problem formulation is based on BAT and genetic algorithm, which gives optimal location of TCSC and its compensation with enhanced ATC value.

Table 5 and
Table 6 shows results of 39-bus New England system.

4. Stage 4 (Impact of single line outage)

Once after deciding the location of TCSC in the system, impact of one line outage is studied by allocating optimal compensation using GA and GWO.

Table 7 shows the results for 39-bus New Englandsystem.

Table 3.ATC for 39-bus system base case in absent of TCSC (using RPF method)

Source/sink bus no.	ATC (MW)	Violation constraint

38/7	510.99	28-29 line
31/7	632.73	6-7 line

Table 4. ATC for 39-bus system with single line outage in absent of TCSC (using RPF method)

Source/sink bus no.	ATC (MW)	Violation Constraint	Contingency
38/7	69.93	26-29	28-29 line
31/7	123.29	5-8	6-7 line
31/7	119.01	6-7	5-8 line

Table 5.ATC for 39-bus system base case in presence of one TCSC

Algorithm Used	Source/sink bus no.	ATC (MW)	ATC enhancement (MW)	TCSC location Line i-j	TCSC compensation	Violationconstraint
GA	38/7	547.37	36.38	26-29	-0.0088	6-11 line
GA	31/7	794.95	162.22	5-8	-0.0044	6-7 line
GWO	38/7	547.37	36.38	26-28	+0.0092	6-11 line
GWO	31/7	800.77	168.04	6-7	+0.0037	6-7 line

Table 6.ATC for 39-bus system base case in presence of two TCSC

Algorithm Used	Source/sink bus no.	ATC (MW)	ATC enhancement (MW)	TCSC location Line i-j	TCSC compensation	Violation constraint
GA	38/7	570.08	59.09	(1) 26-29	(1) -0.0236	2-25 line
				(2) 4-14	(2) -0.0052	
GA	31/7	793.29	160.56	(1) 5-8	(1) -0.0044	6-7 line
				(2) 3-4	(2) -0.0063	
GWO	38/7	562.41	51.42	(1) 4-14	(1) -0.0036	2-25 line
				(2) 26-28	(2) +0.019	
GWO	31/7	844.56	204.75	(1) 5-8	(1) -0.0045	6-7 line
				(2) 5-6	(2) -0.00081	

Table 7.ATC for 39-bus system with single line outage in presence of one TCSC

Algorithm used	Source/sink bus no.	Contingency Line i-j	TCSC location Line i-j	Compen-sation	ATC (MW)	ATC enhancement (MW)	Violation constraint
GA	38/7	28-29	26-29	-0.0204	72.16	2.23	26-29line
GA	31/7	6-7	5-8	-0.0063	142.38	19.09	5-8line
GWO	38/7	28-29	26-28	+0.018	71.19	1.26	26-29line
GWO	31/7	5-8	6-7	+0.0037	135.15	16.14	6-7line

Table 8. New England 39-bus system line parameters

Line no.	From bus i	To bus j	Series impedance		Shunt	Limit
			Rij	Xij	Bi	(MVA)

1	2	30	0	0.0181	0	900
2	6	31	0	0.025	0	1800
3	19	20	0.0007	0.0138	0	900
4	19	33	0.0007	0.0142	0	900
5	20	34	0.0009	0.018	0	900
6	10	32	0	0.02	0	900
7	12	11	0.0016	0.0435	0	500
8	12	13	0.0016	0.0435	0	500
9	22	35	0	0.0143	0	900
10	25	37	0.0006	0.0232	0	900
11	29	38	0.0008	0.0156	0	1200
12	29	38	0.0008	0.0156	0	1200
13	23	36	0.0005	0.0272	0	900
14	1	2	0.0035	0.0411	0.6987	600
15	1	39	0.001	0.025	0.75	1000
16	2	3	0.0013	0.0151	0.2572	500
17	2	25	0.007	0.0086	0.146	500
18	3	4	0.0013	0.0213	0.2214	500
19	3	18	0.0011	0.0133	0.2138	500
20	4	5	0.0008	0.0128	0.1342	600
21	4	14	0.0008	0.0129	0.1382	500
22	5	6	0.0002	0.0026	0.0434	1200
23	5	8	0.0008	0.0112	0.1476	900
24	6	7	0.0006	0.0092	0.113	900
25	6	11	0.0007	0.0082	0.1389	480
26	7	8	0.0004	0.0046	0.078	900
27	8	9	0.0023	0.0363	0.3804	900
28	9	39	0.001	0.025	1.2	900
29	10	11	0.0004	0.0043	0.0729	600
30	10	13	0.0004	0.0043	0.0729	600
31	13	14	0.0009	0.0101	0.1723	600
32	14	15	0.0018	0.0217	0.366	600
33	15	16	0.0009	0.0094	0.171	600
34	16	17	0.0007	0.0089	0.1342	600
35	16	19	0.0016	0.0195	0.304	600
36	16	21	0.0008	0.0135	0.2548	600
37	16	24	0.0003	0.0059	0.068	600
38	17	18	0.0007	0.0082	0.1319	600
39	17	27	0.0013	0.0173	0.3216	600
40	21	22	0.0008	0.014	0.2565	900
41	22	23	0.0006	0.0096	0.1846	600
42	23	24	0.0022	0.035	0.361	600
43	25	26	0.0032	0.0323	0.531	600
44	26	27	0.0014	0.0147	0.2396	600
45	26	28	0.0043	0.0474	0.7802	600
46	26	29	0.0057	0.0625	1.029	600

47	28	29	0.0014	0.0151	0.249	600

All in p.u. based on a 100MVA base.

Table 9. Base case data for the New England 39-bus system

Bus	V_{mag} [p.u.]	V_{angle}	P_{load}	Q_{load}	$P_{generation}$	$Q_{generation}$	$Q_{injection}$
1	1.039384	-13.5366	97.6	44.2	0	0	0
2	1.048494	-9.78527	0	0	0	0	0
3	1.030708	-12.2764	322	2.4	0	0	0
4	1.004460	-12.6267	500	184	0	0	0
5	1.006006	-11.1923	0	0	0	0	0
6	1.008226	-10.4083	0	0	0	0	0
7	0.998397	-12.7556	233.8	84	0	0	0
8	0.997872	-13.3358	522	176.6	0	0	0
9	1.038332	-14.1784	6.5	-66.6	0	0	0
10	1.017843	-8.17088	0	0	0	0	0
11	1.013386	-8.93697	0	0	0	0	0
12	1.000815	-8.99882	8.53	88	0	0	0
13	1.014923	-8.92993	0	0	0	0	0
14	1.012319	-10.7153	0	0	0	0	0
15	1.016185	-11.3454	320	153	0	0	0
16	1.032520	-10.0333	329	32.3	0	0	0
17	1.034237	-11.1164	0	0	0	0	0
18	1.031573	-11.9862	158	30	0	0	0
19	1.050107	-5.41007	0	0	0	0	0
20	0.991011	-6.82118	680	103	0	0	0
21	1.032319	-7.62875	274	115	0	0	0
22	1.050143	-3.18312	0	0	0	0	0
23	1.045145	-3.38128	247.5	84.6	0	0	0
24	1.038001	-9.91376	308.6	-92.2	0	0	0
25	1.057683	-8.36924	224	47.2	0	0	0
26	1.052561	-9.43877	139	17	0	0	0
27	1.038345	-11.3622	281	75.5	0	0	0
28	1.050374	-5.92836	206	27.6	0	0	0
29	1.050115	-3.16987	283.5	26.9	0	0	0
30	1.049900	-7.37047	0	0	250	161.762	0
31	0.982000	0	9.2	4.6	677.871	221.574	0
32	0.984100	-0.18844	0	0	650	206.965	0
33	0.997200	-0.19317	0	0	632	108.293	0
34	1.012300	-1.63112	0	0	508	166.688	0
35	1.049400	1.776507	0	0	650	210.661	0
36	1.063600	4.468437	0	0	560	100.165	0
37	1.027500	-1.5829	0	0	540	-1.36945	0
38	1.026500	3.892818	0	0	830	21.7327	0
39	1.030000	-14.5353	1104	250	1000	78.4674	0

All in p.u. based on a 100MVA base.

CONCLUSION

In this paper, a novel methodology is proposed for ATC enhancement by optimal placement of TCSC with its optimal compensation using GWO Algorithm. GWO and GA optimization algorithms are successfully applied to 39-bus New England system for ATC enhancement. Table 3 to 7 shows the results obtained thereof using the aforementioned methodology. The results obtained for chosen system using GWO are compared with GA methodology, which indicates that GWO algorithm can be a very effective tool for TCSC placement.

APPENDIX A

The New England 39-bus system data, See Table 8and
Table 9

ACKNOWLEDGEMENT

Authors acknowledge financial support extended by World Bank and Government of Gujarat through TEQIP-II project and RK University, Rajkot.

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Computer-Aided Analysis of Permanent Magnet Synchronous Motor Drives using Adaptive Neuro-Fuzzy Inference System

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ABSTRACT

The advancement of permanent magnet material and power electronic devices make Permanent Magnet Synchronous Motor (PMSM) is a better choice for motor control application which gives high torque to weight ratio, wide speed range and good dynamic torque control. An Adaptive Neuro-Fuzzy Inference System (ANFIS) on the place of Fuzzy-Logic-Controller (FLC) or Proportional-Integration (PI) controller is analysed for speed control of PMSM drive using field oriented control. This paper presents a simulation study of PMSM drive with different controller and results are compared with different parameter for steady state and dynamic performance. The computer-aided simulation study in MATLAB and Simulink environment indicates the merit of ANFIS as speed controller with the fast response and good robustness.

SUMMARY

An ANFIS controller for speed control of PMSM is analyzed and merits of ANFIS is explored.

Keywords: PMSM, ANFIS, fuzzy logic control, filed-oriented control, PI

I. INTRODUCTION

Permanent Magnet Synchronous Motor is now-a-days widely used as electromotive application where high-precision and robustness is prerequisite. The wide speed range, high efficiency since less rotor loss, faster dynamic response since doubly excitation, absence of brushes and high torque to weight ratio makes this motor is better choice for drive application. In this paper, the surface magnet PMSM motor is selected for this study where there is q-axis and d-axis inductance parameters are considered as the same. As in real application, the uncertainty and non-linearity of motor behaviour with conventional controller has limitation to give same response for every given condition over a long span of time. Intelligent controller has been proposed by (1-11), such as expert system, fuzzy logic and artificial intelligent to overcome the limitation of conventional control algorithm. These techniques give good and faster response with the given environment and it regains original condition with faster rate. The limitation of these techniques is problem with initial development and implementation with processor. These restrictions take more time and effort of a developer and sometime it intervene the focus of a designer from real application to be developed.

A 15 rule-base function torque adapted gain fuzzy inference system is presented in (1). The control algorithm was presented in this literature is to eliminate the trial and error method of adjusting the input and output gains of fuzzy controller which normally can be done manually. Flux-weakening control using fuzzy logic control to control the speed over a wide range is presented in (2). The d-axis current reference is generated from fuzzy logic controller which has been inputted the operation times of zero voltage space vector to produce the reference current. A digital signal processor (DSP) based Fuzzy Direct Torque Controller (DTC) is implemented and experiment results are compared for variable parameters in (3). The fuzzy controller reduces the ripple stator flux and developed torque reduces using the given algorithm is presented in this paper. In (4), ANFIS for speed control of induction motor is proposed. The proposed scheme was compared with Mamdani method and indicated that ANFIS gives fast and dynamic response with given plant input. A fuzzy adaptive PI controller to control servo motor based on decoupling control is presented in (5). The simulation results gave good dynamic performance and indicated adaptive fuzzy PI controller is superior in performance. Another paper (6) presents a model adaptive fuzzy control (MRAFC) is realised on PMSM drives. The proposed scheme is simulated and results proved that this method is efficient especially in drive application. An ANFIS estimator for DTC for PMSM is proposed in (7). With well-trained network, the system is implemented and it is shown that proposed scheme is independent of machine parameter and type. It is also shown that this technique is suitable for estimating torque, speed, position, flux and switching table of DTC too. An adaptive neuro fuzzy controller for switched reluctance motor (SRM) speed control is presented in (8). The adaptive nature of algorithm proves that controller give superior performance with parameter variation and external disturbances. A sensorless control with back emf sliding mode observer implemented using DSP is presented in (9). Adaptive fuzzy controller with space vector pulse width modulation (SVPWM) is integrated in DSP experimental results were given satisfactory performance under uncertainty condition.

II. MATHEMATICAL MODEL OF PMSM

In this section, mathematical model of PMSM is presented (12). Basically a three phase input is applied to PMSM motor. This three phase equivalent model is transferred to two phase system in alpha-beta transformation which is also referred as Clerk's transformation. The mathematical transformation from *abc to αβ* can be performed using following expressions.

Let v_a, v_b and v_c are the three phase supplied voltage at the stator terminal of the machine and i_a, i_b and i_c are the three phase current flowing though the machine.

$$v_\alpha = \frac{2}{3}\left(v_a - \frac{1}{2}v_b - \frac{1}{2}v_c \right) \tag{2.1}$$

$$v_\beta = \frac{2}{3}\left(-\frac{\sqrt{3}}{2}v_b + \frac{\sqrt{3}}{2}v_c \right) \tag{2.2}$$

Similarly,

$$i_\alpha = \frac{2}{3}\left(i_a - \frac{1}{2}i_b - \frac{1}{2}i_c \right) \tag{2.3}$$

$$i_\beta = \frac{2}{3}\left(-\frac{\sqrt{3}}{2}i_b + \frac{\sqrt{3}}{2}i_c \right) \tag{2.4}$$

Once the stator quantities are transformed into the alpha-beta transformation, now machine can be analyzed as two-phase machine. The expression of v_α and v_β of the machine can be expresses as

$$v_\alpha = R_s i_\alpha + L_\alpha \frac{di_\alpha}{dt} + e_\alpha \tag{2.5}$$

$$v_\beta = R_s i_\beta + L_\beta \frac{di_\beta}{dt} + e_\beta \tag{2.6}$$

Where $e_\alpha = -K_E \omega \sin\theta_e$ and $e_\beta = K_E \omega \cos\theta_e$ is the induced electromotive force in that specific phase that is orthogonal to each other and depend on the speed of the machine.

For further reduction of alternating behaviour of the voltage and current, to get that in dc like qualities, these variables can be transformed from *αβ to dq* variable. This transformation is referred as Park's transformation, in which the two-axis rotates at the speed of rotor, so called rotor reference frame. The transformation equation can be expressed as

$$v_d = v_\alpha \sin\theta_e + v_\beta \cos\theta_e \tag{2.7}$$

$$v_q = v_\alpha \cos\theta_e - v_\beta \sin\theta_e \tag{2.8}$$

And similarly

$$i_d = i_\alpha \sin\theta_e + i_\beta \cos\theta_e \tag{2.9}$$

$$i_q = i_\alpha \cos\theta_e - i_\beta \sin\theta_e \tag{2.10}$$

In *dq* axis the PMSM can be expresses as following expression

$$v_d = R i_d + L_d \frac{di_d}{dt} - \omega_e L_q i_q \tag{2.11}$$

$$v_q = R i_d + L_q \frac{di_q}{dt} + \omega_e L_d i_d + \omega_e \lambda_{PM} \tag{2.12}$$

$$\omega_e = \frac{d\theta_e}{dt} \tag{2.13}$$

The expression for the electromagnetic torque can be written as,

$$T_e = \left(\frac{3}{2}\right)\left(\frac{P}{2}\right)(\lambda_{PM} i_q - (L_q - L_d) i_q i_d) \tag{2.14}$$

and the equation for the motor dynamics is

$$T_e = T_L + B\omega_e + J\frac{d\omega_e}{dt} \tag{2.15}$$

In the above equations P is total number of poles, T_e is electromagnetic torque, T_L is load torque, B is damping coefficient and J is moment of inertia.

The inverter frequency is related to the rotor speed as

$$\omega_i = \left(\frac{P}{2}\right)\omega_e \tag{2.16}$$

The voltage and flux linkage equations suggest the equivalent circuits shown in Fig. 1.

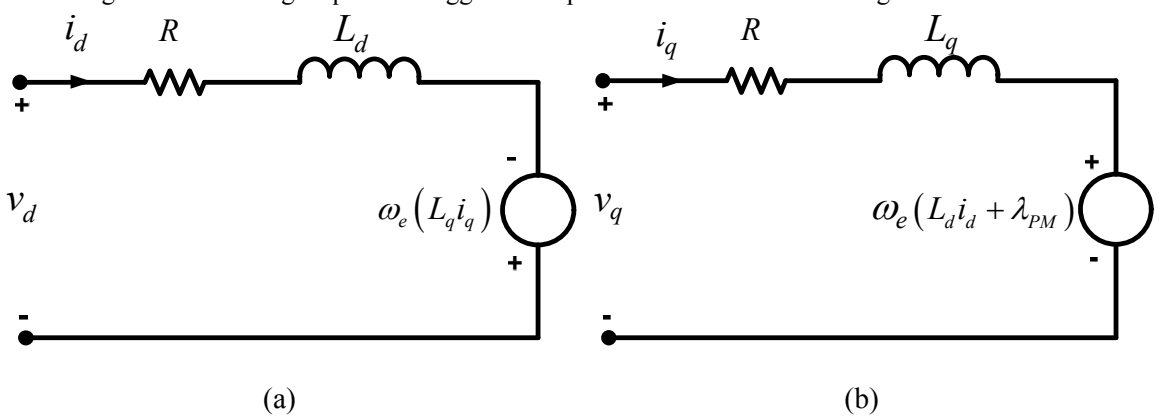


Fig. 1. Equivalent circuit of permanent magnet synchronous motor in rotor reference frame: Park's equation

III. CONTROL STRATEGY OF PMSM DRIVE WITH SPEED AND CURRENT CONTROLLER

Basically, PMSM drive is considered as four main sections: a power converter, a PMSM, measuring and sensing equipment and control algorithm as shown in Fig. 2. The power electronics converters transfer power from input source which is a dc source into to ac with corresponding frequency based on the firing pulses are given to this converter. The sensing equipment senses different signals like position, speed, current and voltage. Normally, speed sensors, requires in PMSM drive, is encoder or resolver type to measure exact position of rotor flux, this is required to align the stator flux orthogonal to rotor flux in field oriented control to achieve minimum amount of current to produce the same torque. Current sensors are usually two and by mathematical expression the third current can be calculated if there is no neutral connection. For sensing voltage, sometime control algorithm voltage can be considered as feedback voltage to avoid the noise in feedback signal and to reduce the cost of transducers. Overall, based on

commanded speed/position, current and voltage of the machine is inputted to drive the machine to produce desired torque with minimum amount of current and better dynamic response.

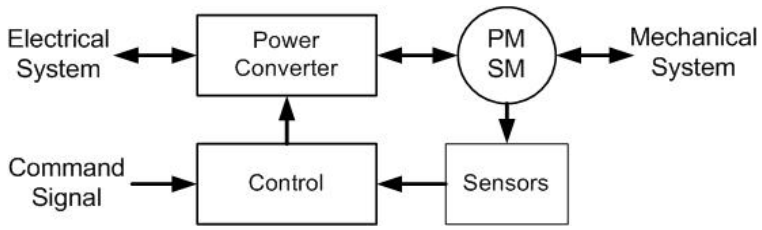


Fig. 2. PMSM Drive (12)

To achieve field oriented control, the *abc* current is transformed in the alpha-beta transformation which is Clerk's transformation. With this transformation, the stator current is now transformed to stationary reference frame where it is two current displaced each other by 90^0. These quantities are ac which need to transform in dc i.e. in *q-d* -axis. This can be achieved by Park's transformation with rotor flux position alignment. These two currents are called *q*-axis and *d*-axis current which is also called currents in rotor reference frame.

For speed control of PMSM, the reference speed is compared with actual speed and an error signal is generated. This error signal is processed in any given controller, which produces the torque command. In PMSM, especially in surface mounted magnet type of motor, only the q-axis current is torque producing current. There is no d-axis current as there is no reluctance torque (L_d and L_q are same in surface mounted PMSM) and no flux weakening is considered in the given study. For wide range of speed control, the *d*-axis current is need to generate, which can be produced using look-up-table or mathematical formulation with given boundary condition of stator current and dc voltage. Now, required *q*-axis current and *d*-axis current (zero) is compared with actual currents which produces current error signal. These errors are processed through PI controllers which give required *q*-axis and *d*-axis voltage vectors. These voltage can be transformed in *abc* using given position of rotor flux which is the reference or required output voltage. Using pulse width modulation (PWM) technique, through the converter the reference voltage is produced from dc to ac which is given to the terminals of machine. This drives the machine at the given the speed and conceptual diagram for complete scheme is shown in Fig. 3.

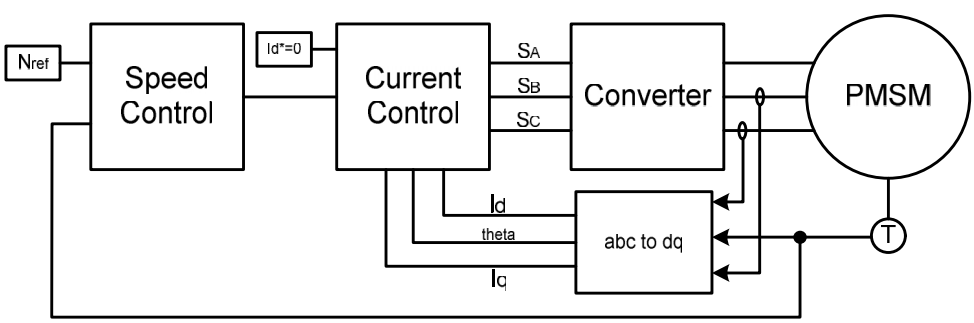


Fig. 3. PMSM Drive with speed and current control loop

IV. SIMULATION OF PMSM WITH ANFIS SPEED CONTROLLER

The simulation of the scheme is performed in MATLAB and Simulink platform. The Fig. 4(a) shows overview of complete simulation. The upper block PMSM_Inverter (blue colour) consists of dc source, power converter unit and PMSM, the details of these system is shown in Fig. 4(b). The firing pulse and load torque are two inputs for this block. The measurement block gives different signal like position, speed, voltage and current. Some signals are used as feedback and some are used to refer the condition and behaviour of the drive operation. The block with Control_Algorithm (green colour) consists of the complete scheme of speed control of PMSM using FOC with ANFIS. The basic inputs are given to achieve these are, reference speed, actual speed, rotor flux position and stator current of the machine.

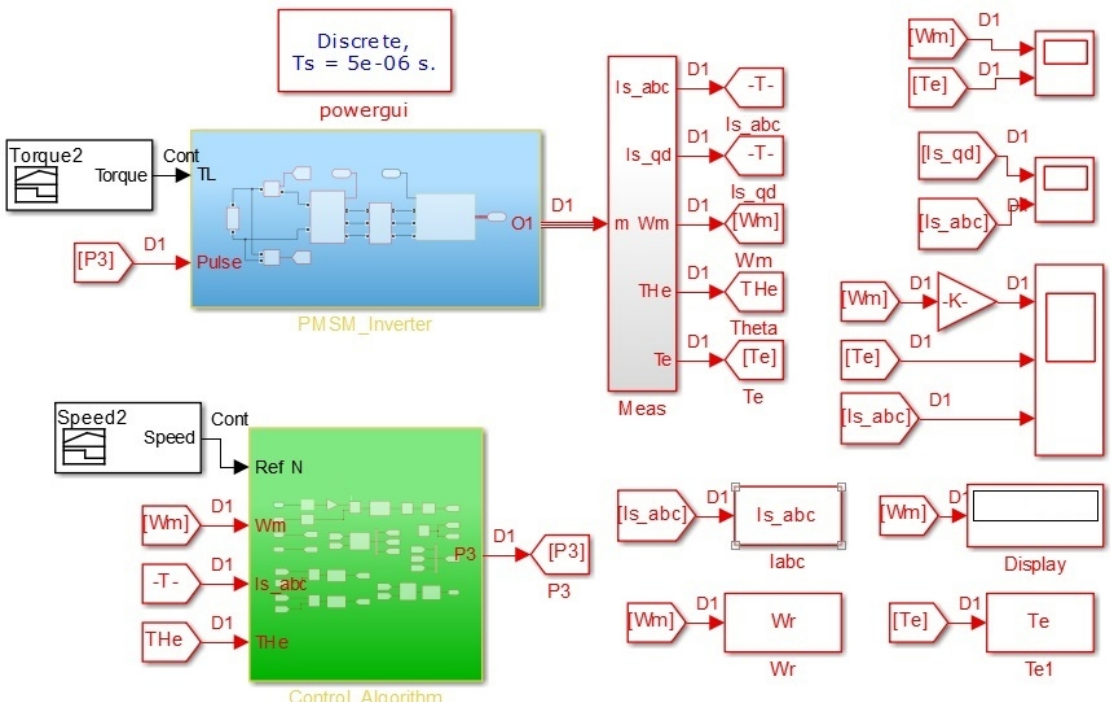


Fig. 4(a). Simulation Diagram of PMSM Drive

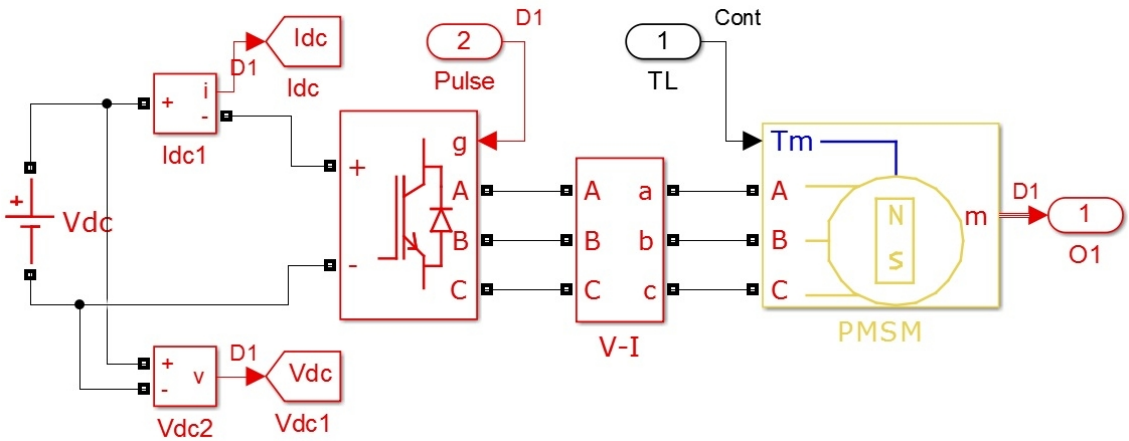


Fig. 4(b). Power Blocks of PMSM Drive

The speed control and current control algorithm system in detail is shown in Fig. 5(a). As mentioned, the reference speed is compared with the given speed and the error is processed in ANFIS controller as shown in Fig. 5(b). The data variable E, DE and OP is generated to train the network and these are used when system is running with normal fuzzy logic controller or PI controller. Once the network is trained and ANFIS is configured, these signals are not used afterword. The output of speed controller is limited by limiting block to limit the stator current at specified or rated value. The output of speed controller of ANFIS is used as reference current for *q*-axis and *d*-axis reference current is taken as zero.

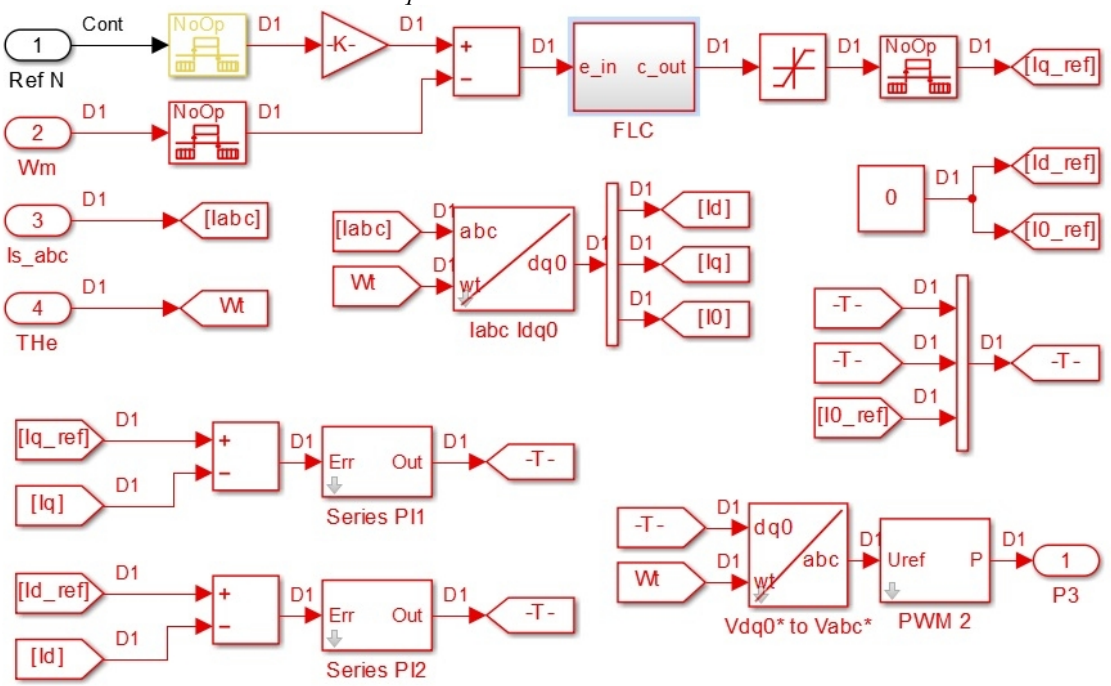


Fig. 5(a). Speed and Current Control Blocks of PMSM

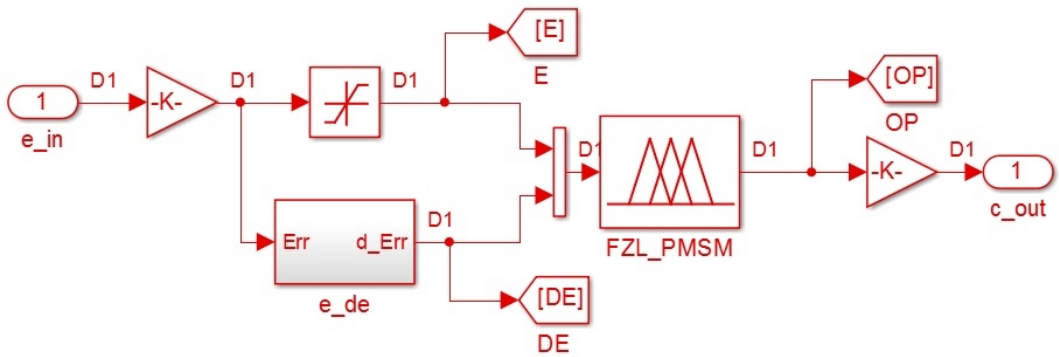


Fig. 5(b). Fuzzy logic control

The outputs from current regulators can be termed as *q*-axis and *d*-axis reference voltage, which is converted to *abc* reference voltage. With PWM technique, the pulses are generated which are given to converter system.

V. TRAINING OF ADAPTIVE NEURO-FUZZY INFERENCE SYSTEM

Adaptive Neuro-Fuzzy Inference System (ANFIS) is a feature of fuzzy logic toolbox available in MATLAB. ANFIS uses hybrid learning algorithm to identify the membership function parameters of single-output and this is applicable to sugeno type fuzzy inference system. ANFIS tunes the FIS with the given input/output data of a defined nonlinear system.

Sugeno type FIS is modelled as shown in Fig. 6(a). Two inputs, error and change in error is given as input and output is generated based on the rule base. The 7 membership functions for each input and output as shown in Fig. 6(b) are used to define total 49 rules as given in Table 1 and Fig. 6(c). The complete FIS with two inputs and one output can be viewed from the surface viewer as shown in Fig. 6(d).

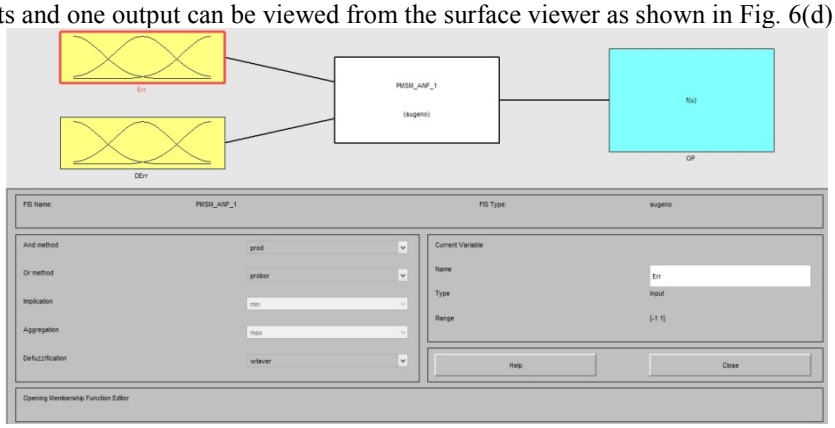


Fig. 6(a). Fuzzy Logic input and output variable

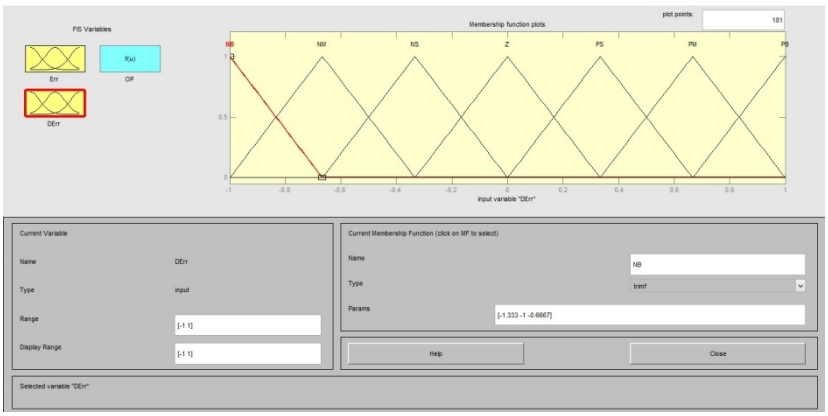


Fig. 6(b). Fuzzy Logic membership function

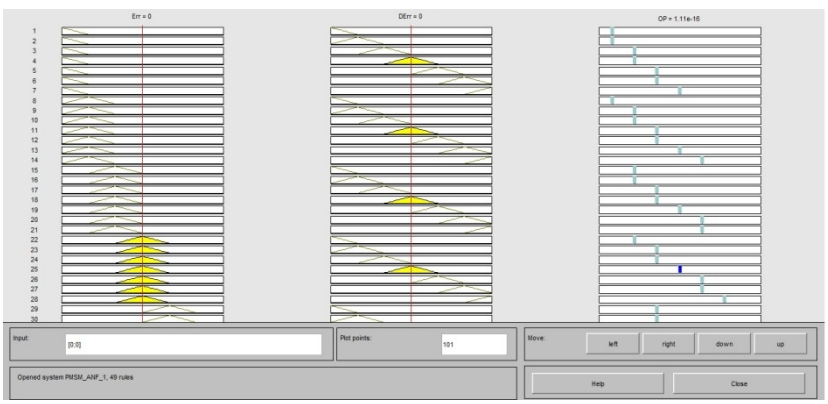


Fig. 6(c). Rule base

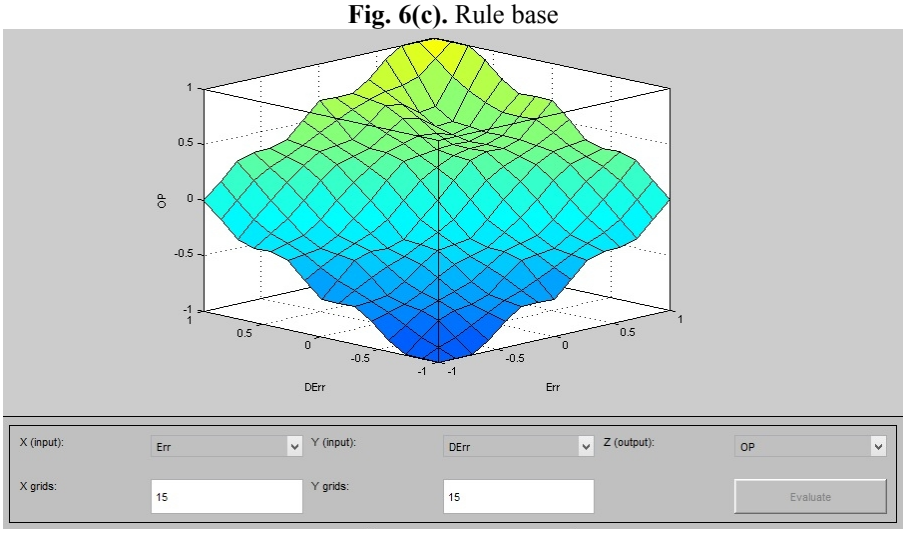


Fig. 6(d). Surface viewer

Table 1. Rule base for input and output

$e \downarrow de \rightarrow$	**NB**	**NM**	**NS**	**Z**	**PS**	**PM**	**PB**
NB	NB	NB	NM	NM	NS	NS	Z
NM	NB	NM	NM	NS	NS	Z	PS
NS	NM	NM	NS	NS	Z	PS	PS
Z	NM	NS	NS	Z	PS	PS	PM
PS	NS	NS	Z	PS	PS	PM	PM
PM	NS	Z	PS	PS	PM	PM	PB
PB	Z	PS	PS	PM	PM	PB	PB

Sugeno FIS is implemented in given drive system and input variables E, DE and output variable OP is recorded. From the large database of the system inputs and output, function used to select specific number of data for each case as testing and checking inputs to train the network. These databases are used to train the ANFIS as shown in Fig. 7(a). With the sampled training, testing and checking database, the ANFIS is trained with hybrid optimization method. The system structure in ANFIS is shown in Fig. 7(b) with two input and one output.

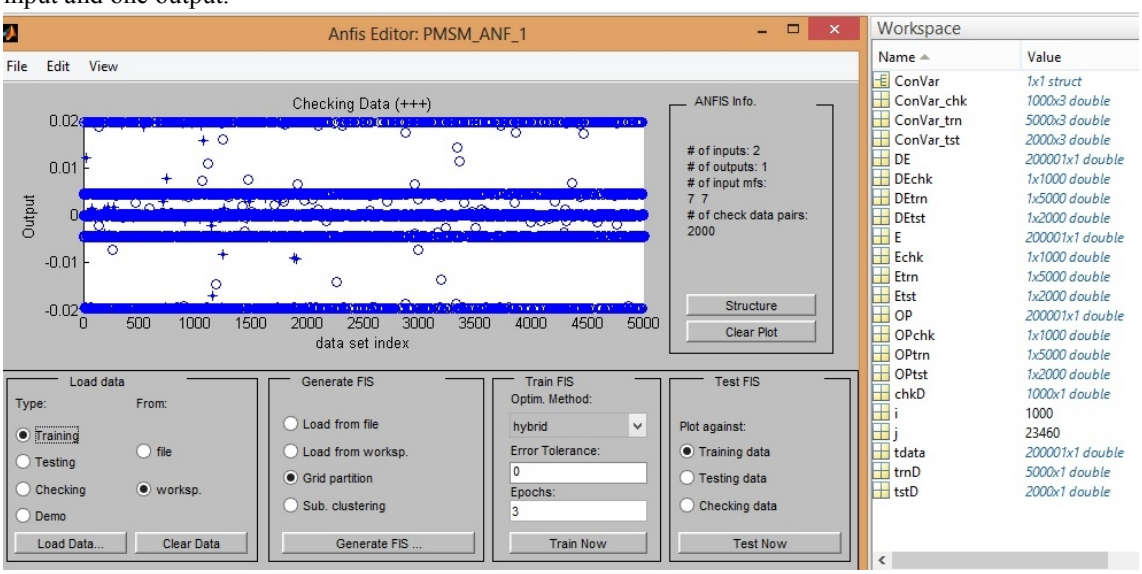


Fig. 7(a). Data inputting to train ANFIS

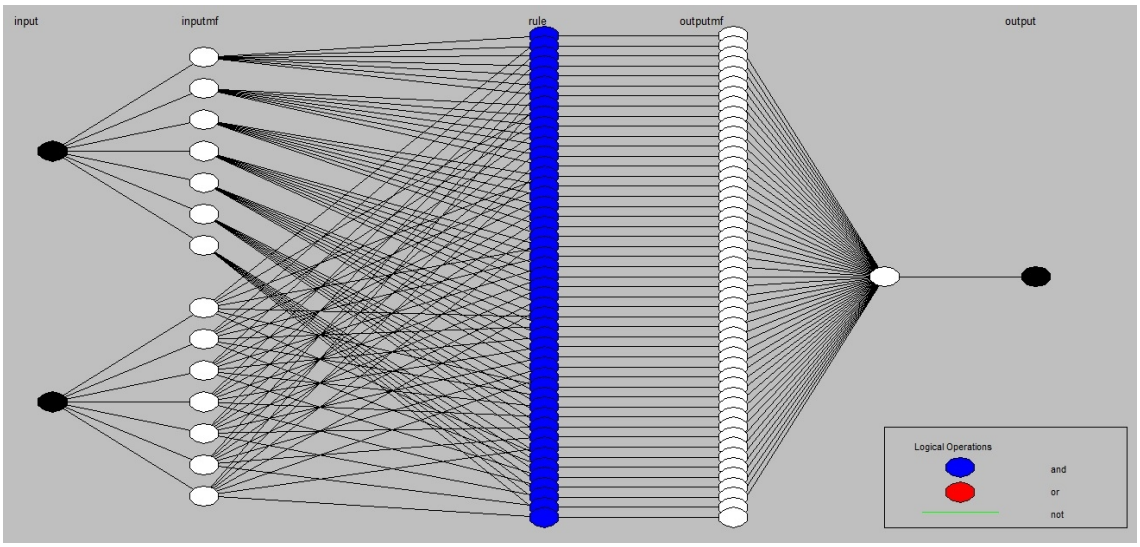


Fig. 7(b). Structure of ANFIS

VI. RESULT DISCUSSION

The dynamic behaviour of proposed algorithm is simulated with different variation in speed and load torque. Three different sets of reference speed and load torque are applied to confirm the behaviour of the trained ANFIS network. Fig. 8(a) and Fig. 8(b) is the first set of commanded input and output. In this case speed is ramped from 0 to 1500rpm in 0.1s. Speed command is kept constant from 0.1s to 0.4s. At this constant speed, the maximum load torque is applied as 10Nm from 0.2s to 0.3s. From 0.4s to 0.6s, the speed is ramped down from 1500rpm to -1500rpm. That is reversal of direction. During reverse motoring operation, the load torque is applied for the same span of time. The simulation results of speed, developed torque and stator currents of the motor displays the motor closely follows the commanded input as speed, and develops the electromagnetic torque as required by the load dynamics.

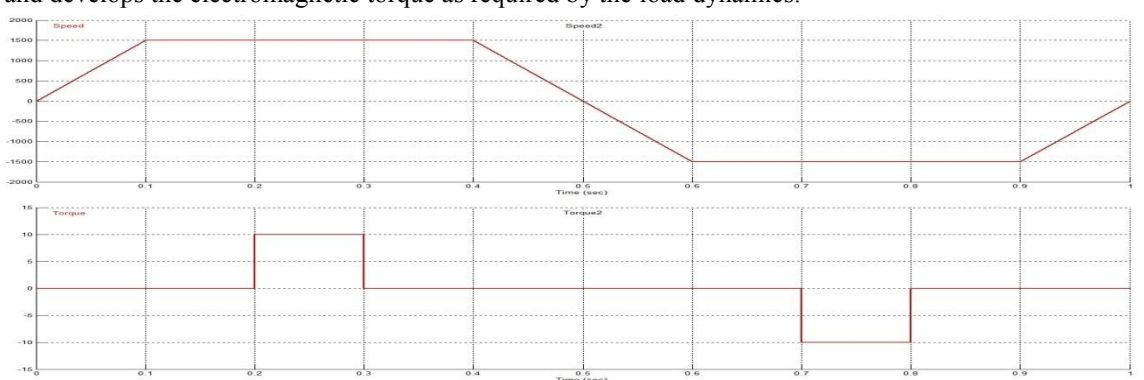


Fig. 8(a). Input speed and load torque Scale is not readable

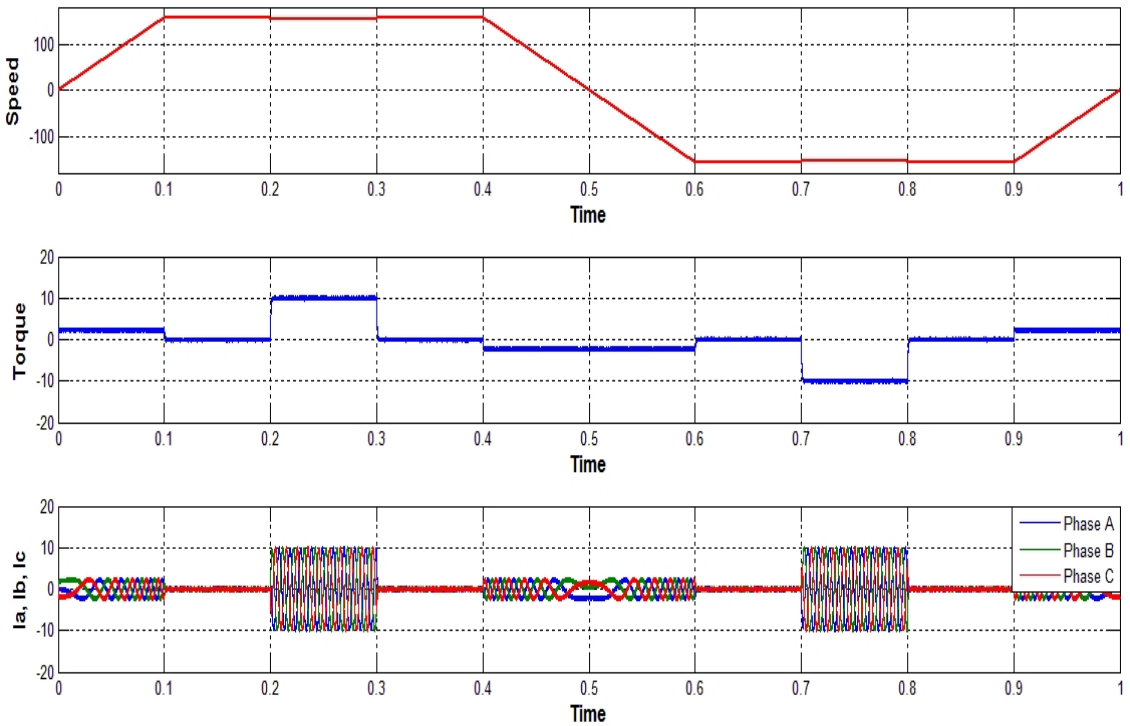


Fig. 8(b). Output speed, torque and stator currents

Fig. 9(a) displays another set of commanded speed and load torque and Fig. 9(b) show simulation results of the system. In this case, the reference speed is applied as a step input. Forward high and low, reverse high and low speed is given as speed input and corresponding the load torque also changed in both the motoring region. The output result shows that motor accelerates at maximum torque limited by torque limiter at 20Nm to achieve the reference step input. The output speed remains constant once it reached to the reference. The stator current increases as the load torque. The step down speed command leads to generate the deceleration torque (-20Nm) command to reduce the speed at faster rate. The simulation result of the output closely follows the reference step input with different given conditions.

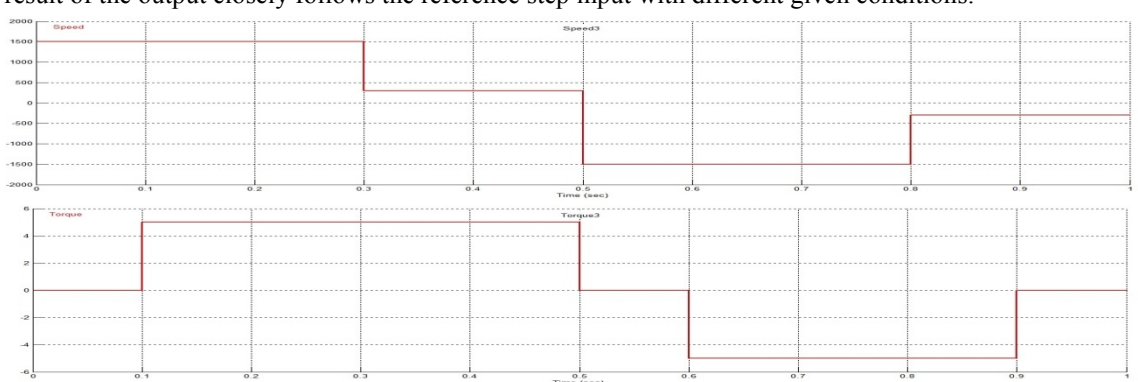


Fig. 9(a). Input speed and load torque

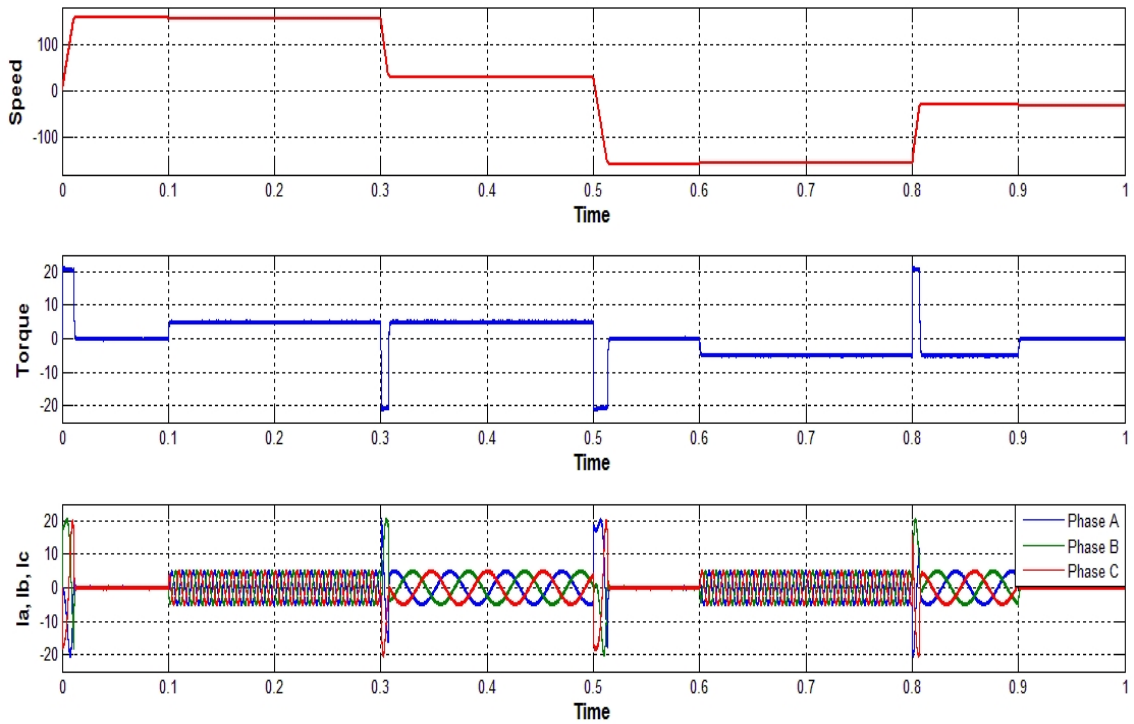


Fig. 9(b). Output speed, torque and stator currents

The following Fig. 10(a) and Fig. 10(b) is third set of commanded inputs and corresponding outputs. In this case, the step and ramp speed is given as input. This case specially considers to view the behaviour of the system for wide speed range; as low, medium and high as input to confirm the behaviour for control algorithm. The simulation result follows the commanded input and achieves the output speed, electromagnetic torque as desired.

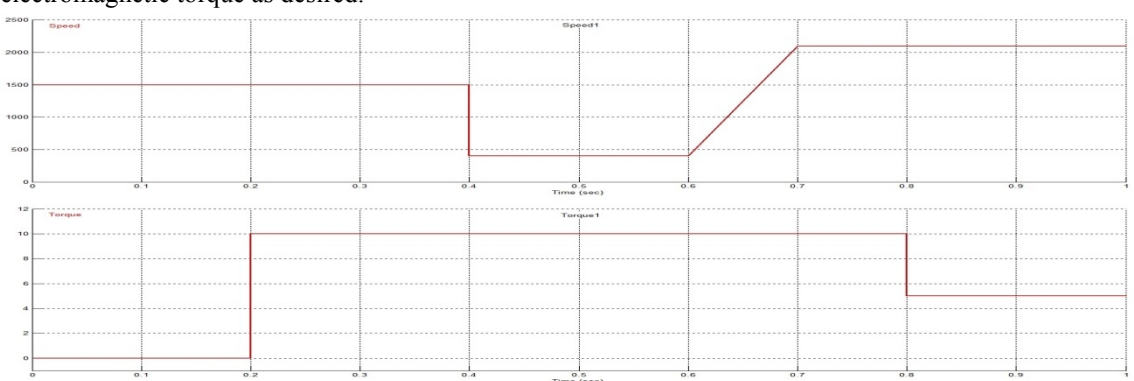


Fig. 10(a). Input speed and load torque

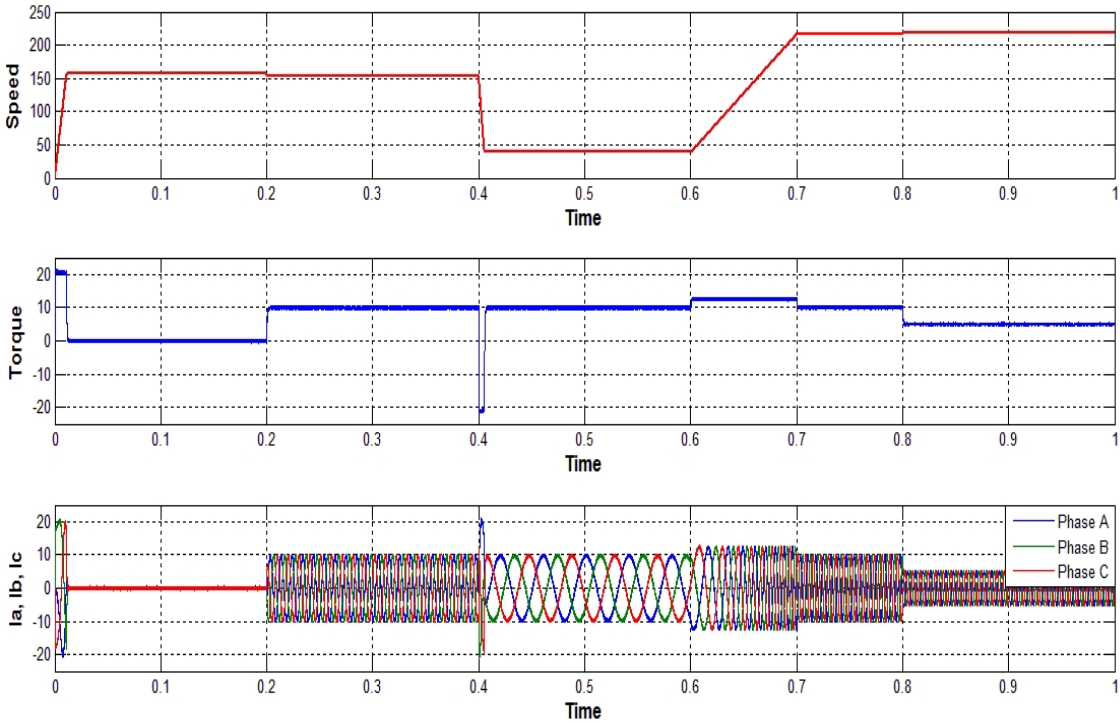


Fig. 10(b). Output speed, torque and stator currents

VII. CONCLUSION

The PMSM drive with field oriented control is simulated in MATLAB and Simulink. For speed control, ANFIS is trained and replaced by PI or FLC. FLC is simulated and database is generated to input ANFIS. For current control, the series PI regulators are used. The behaviour of complete close loop control is tested for three different sets of input. The output result of speed, developed torque and stator current closely follows the input command. It shows that once ANFIS is trained, it works for any dynamic behaviour and converges at faster rate which reduces the computational time. For nonlinear behaviour system such as PMSM drive, ANFIS can be more useful to improve the dynamics of the system.

APPENDIX – I

Parameter of PMSM
Stator resistance = 0.4578 Ω
Armature Inductance = 3.34 mH
Flux link by magnet = 0.171 V.s
Number of Poles = 8
Moment of Inertia = 0.001469 kg.m^2
Viscous damping = 0.0003035 N.m.s

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Performance Measurement of Face and Fingerprint Recognition Systems

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ABSTRACT

In the digital era of computers, identity management is one of the important aspects. Biometric technologies are becoming more and more popular now a days. Out of all biometric technologies, fingerprint and face recognition methods are older mature. In this paper, the researcher has tried to evaluate both the Biometric traits by using database of 30 persons. FAR and FRR have been calculated to evaluate the performance of fingerprint and face recognition methods.

SUMMARY

The researcher has implemented face and fingerprint recognition system to evaluate the performance of unimodal biometric systems.

Keywords: ***Biometrics, Face recognition, Fingerprint recognition, FAR, FRR***

INTRODUCTION

Biometric authentication systems are one of the most popular and interesting area for research in today's digital world. Biometric authentication methods can be divided with two categories:

- Methods based on physical traits of human
- Methods based on behavioral traits of human

Physical biometrics methods include fingerprint, face, hand geometry, ear shape, palmprint, iris and retina scanning. While behavioral biometrics methods include voice, gait, keystroke recognition etc. *(1)*

Fingerprint and Face recognition methods can be implemented with two different ways: Identification and Verification. Application of biometrics at Airports used for surveillance is an example of identification. While applying biometrics at border of the country is an example of verification. Fingerprint and Face recognition can be used on both the ways. As fingerprint and face recognition are among mature technologies, we cannot doubt on their applicability *(2)*.

Different face recognition techniques

Zhao et al. in their survey categorized face recognition techniques in three ways:

- Appearance based techniques
- Feature based techniques
- Hybrid techniques

Appearance base technique uses holistic texture features and they are applied to either whole face or specific regions of the face image. Feature based technique uses geometric facial features like mouth, eyes, cheeks etc. and geometric relationship between these features. As human being, we are used to for matching face as a whole with holistic approach as well as with the help of features of the faces, which can be considered as hybrid techniques *(2)*.

In the appearance base approach, one of the widely used methods is techniques based on Principal Component Analysis (PCA). First successful experiment of face recognition was made by Turk and Pentland in 1991 *(3)*. Moghaddam and Pentland in 1997 *(4)* extended this method to Bayesian approach. Face recognition system using Linear Discriminant Analysis (LDA) has been successful. LDA training is performed with Scatter Matrix Analysis. Another technique was suggested by Lin et al. in 1997 *(5)*. They proposed system based on Probabilistic Decision Based Neural Network (PDBNN).

The earlier methods of face recognition were working with structural matching. These methods were based on geometry of local features. They were using the features like width of the head, distance between eyes, and the features of mouth, nose and chins *(6)*. Hidden Markov Model (HMM) based methods use lines of pixels covering eye, nose, mouth, chins etc *(7)*. Modular Eigenface approach uses global Eigenfaces and local eigenfeatures both *(8)*.

Different fingerprint recognition techniques

Comparison of two fingerprints can be done with various methods. Almost all the methods use mathematical approach for the said purpose. All matching techniques compute degree of similarity and returns some score. Depending on this score, the system decides whether there is matching or non-matching. Many algorithms have been proposed till this date for fingerprint matching. Out of which, the popular techniques are:

- Correlation based method
- Minutiae based method
- Ridge feature based method

With correlation based method, the fingerprints are aligned and correlation is computed for each corresponding pixel. It is necessary to apply correlation for all possible alignments as displacement and rotation are unknown. Disadvantage of this method is its computational complexity.

This is the most popular and effective method for fingerprint comparison. With minutiae based method, align the minutiae of train sample and test sample and then find matched minutiae points. Correct alignment of the minutiae is important to have good performance of matching.

With ridge feature based method, computation of the difference of two fingerprint vectors (train and test) is done. Here also, proper alignment of fingerprints is important. This method uses circular fingercodes, considering center as the core point*(9)*.

In our experiment, we have used minutiae based method for matching fingerprints.

FACE RECOGNITION USING PCA WITH EIGENFACE METHOD

Transformation of set of data obtained from correlated variables in to set of values of uncorrelated variables called principal components is done by PCA.

It is required to find the principal components; which can be considered here as eigenvectors of the covariance matrix of facial images *(9)*.

(1) Step - 1

Here, it is required to outline a training data set. It is possible to represent two dimensional image in to one dimensional vector with concatenation of rows. Image will be transformed in a vector with length N=m*n.

$$I = \begin{bmatrix} x_{11} & x_{12} & \cdots & x_{1n} \\ x_{21} & x_{22} & \cdots & x_{2n} \\ \vdots & \vdots & \ddots & \vdots \\ x_{m1} & x_{m2} & \cdots & x_{mn} \end{bmatrix}_{mxn} \xrightarrow{CONCATENATION} \begin{bmatrix} x_{11} \\ \vdots \\ x_{1n} \\ \vdots \\ x_{2n} \\ \vdots \\ x_{mn} \end{bmatrix}_{1xN} = x.$$

Then there will be creation of a matrix of learning images X with M vectors of length N. Then the matrix is centered. The next step is to determine vector of mean values and subtracting that vector from each image vector.

Average vectors will be arranged to form a new training vector with size (NxM).

(2) Step - 2

In the second step, it is required to calculate covariance matrix C, and finding its eigenvectors and eigenvalues. Covariance vector C will be having dimension NxN. From this matrix, itis possible to get N eigenvectors and eigenvalues. Ranking of covariance matrix is limited by number of images in learning set. Eigenvector, which is associated with highest eigenvalue, replicates the highest variance and one associated with the lowest eigenvalue, replicates the smallest variance.

The vectors must be sorted on the basis of eigenvalues so that first vector matchto the highest eigenvalue. Then these vectors should be normalized. It will create a new matrix in which each vector will be a

column vector. The dimension of matrix now should be NxD (D represent preferred number of eigenvectors). Every original image now should be reconstructed by adding mean image to weighted sum of all vectors.

(3) Step - 3

Last step is recognition of faces. Image of the person which is required to find in training dataset is transformed into vector P, reduced by the mean value and projected with a matrix of eigenvectors.

Classification is done by identifying distance between person's eigenvector and each vector of matrix Y. Euclidean distance is one of the most common method to calculate distance. Other methods can also be applied.

If the minimum distance between test face and training faces is higher than threshold value, then person will be unknown otherwise it will be known one.

FINGERPRINT REGOCNITION USING MINUTIAE BASED METHOD

The whole process of minutiae matching is divided in three stages namely:

- Preprocessing
- Feature extraction
- Matching

Figure 1 show above process.

The whole method has been explained clearly in *(10)*. We will see here few important aspects of processing.

Asmost of the fingerprint matching algorithms depend on minutiae matching, minutiae information is identified as most significant features for AFRS. The main methods of minutiae feature extraction require either the gray-scale image to be converted to a binary image, or can work directly on a raw or enhanced gray-scale image.
In the binary image based method, the binarization of the gray-scale image is the first step. It requires each gray-scale pixel intensity value to be converted to a binary intensity of black (0) or white (1).

After this, the binary image is applied for a morphological thinning operation. Here ridge structures are reduced to 1-pixel thickness, which is referred as skeleton, which is used for minutiae detection. The final binary thinned image has each pixel, p, analyzed to find minutiae location.

General process of fingerprint matching is shown in figure-1. In minutiae-based matching, minutiae are represented as minutiae structures called minutiae triplets. This minutiae-based matching concentrates on performing a one-to-one mapping or pairing of minutiae points from a test image minutiae set to a train image minutiae set.

But, we cannot proceed without doing some preprocessing because of the reasons like, difference in orientation and offset. To overcome this problem, global registration required. Global registration concerns the alignment and overlay of the train and test fingerprints so that respective regions of the fingerprints will have minimum geometric distance from each other. Registration can be achieved

geometrically by applying a heuristically guided affine transform to the test or train fingerprint minutiae set.

After registration process, it is possible to produce geometric constraints to find out minutiae matching pairs. After global registration, a local search can be performed to match minutiae in the δ-neighbourhood to meet the constraints. After producing genuine minutiae pairs, a metric of similarity score, will be calculated. The similarity score describessimilarity of two fingerprintsaccurately, by considering all the related information collected from earlier stages, like number of genuine minutiae pairs and their similarity.

EXPERIMENTS AND RESULTS

As described in section 2 and 3, we have used PCA based Eigenface method for face recognition and Minutiae based method for fingerprint recognition.

With this experiment, we have taken database of 30 persons (students + faculty members) of Department of Computer Science, Saurashtra University. We have taken 10 samples of each person for both the traits i.e. face and fingerprint (Total 300 samples of face and 300 samples of fingerprint).

Calculating FRR for face recognition

Consider a database of 60 samples for training and 240 samples for testing. Here training set for each person with 2 samples and other 8 samples of the same person compared by taking different cases.

Case 1: minimum distance from all 8 test samples with 2 train samples
Case 2: maximum distance from all 8 test samples with 2 train samples
Case 3: average distance of all 8 test samples with 2 train samples

Table 1 show GAR and FRR for above mentioned three cases and figure 2 shows graphical representation of results.

Calculating FAR for face recognition

Consider a database of 60 samples for training. Here training set for each person with 2 samples and other 290 samples of the other persons compared by taking different cases.

Case 1: minimum distance from all 8 test samples with 2 train samples
Case 2: maximum distance from all 8 test samples with 2 train samples
Case 3: average distance of all 8 test samples with 2 train samples

Table 2 show FAR and GRR for above mentioned three cases and figure 3 shows graphical representation of results.

Calculating FRR for fingerprint recognition

Consider a database of 30 samples for training and 270 samples for testing. Here training set for each person with 1 sample and other 9 samples of the same person compared by taking different cases.

Table 3 show GAR and FRR for fingerprint recognition. Figure 4 shows graphical representation of results.

Calculating FAR for fingerprint recognition

Consider a database of 30 samples for training and 90 samples for testing. Here training set for each person with 1 sample and other 3 samples of the different persons compared.

Table 4 show GRR and FAR for fingerprint recognition system. Figure 5 shows graphical representation of results.

CONCLUSION

From the experimental results, it is clear that fingerprint recognition is more mature than face recognition as Unimodal Biometric trait. But at the same time we are able to conclude that none of the method gives 100% accuracy for identification or verification. These experiments have been performed only for 30 people. But for large population, these results may be even poorer. So, we can also conclude that there is a requirement of multimodal biometric system in the situation where any one of the trait get failed, then another trait can be used for identification of verification purpose.

FIGURES

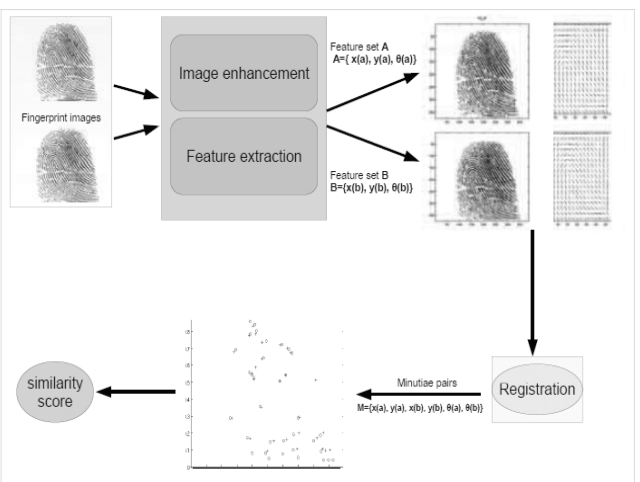


Fig 1**. General process of minutiae based fingerprint matching** ***(10)***

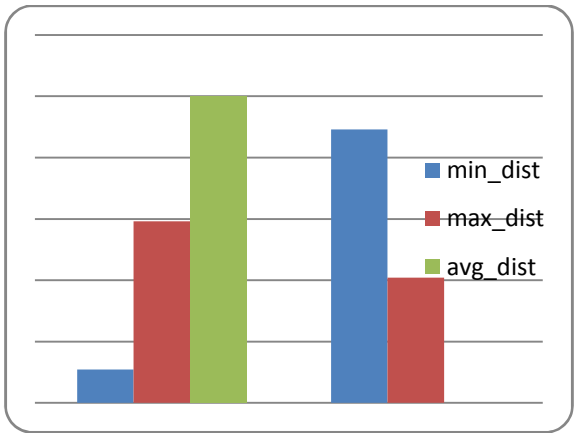


Fig 2. FRR for face recognition

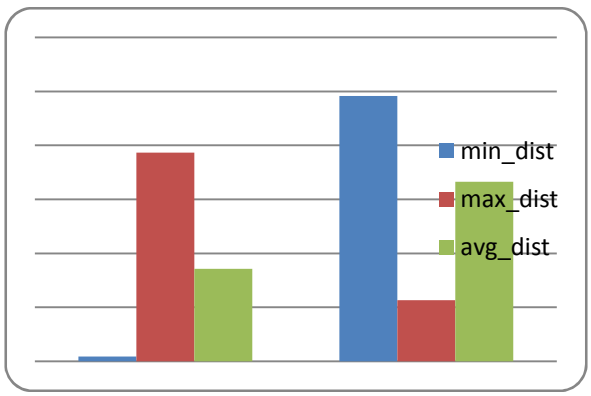


Fig 3. FAR for face recognition

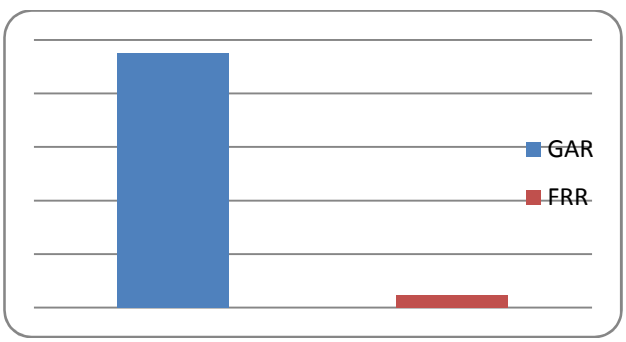


Fig 4. FRR for fingerprint recognition

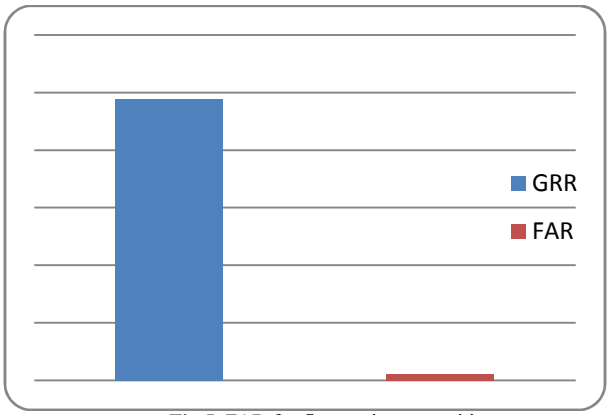


Fig 5. FAR for fingerprint recognition

TABLES

Case	Success	Failure	GAR	FRR
min_dist	26	214	10.83333	89.16667
max_dist	142	98	59.16667	40.83333
avg_dist	240	0	100	0

Table 1. FRR for face recognition

Case	False acceptance	Genuine Rejection	FAR	GRR
min_dist	153	8547	1.758621	98.24138
max_dist	6727	1973	77.32184	22.67816
avg_dist	2981	5791	34.26437	66.56322

Table 2. FAR for face recognition

Case	Success	Failure	GAR	FRR
1	257	13	95.185	4.815

Table 3. FRR for fingerprint recognition

Case	Success	Failure	GRR	FAR
1	88	2	97.77	2.23

Table 4. FAR for fingerprint recognition

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Design and Manufacturing of Single sided expanding collet for Rotary VMC Fixture

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ABSTRACT

Expanding Collets are extensively used to hold the workpieces with circular features. Due to the shape and narrow tolerances, designs of such collets are always challenging. The paper represents process of dimensional design of a single sided expanding collet. This collet is for rotary VMC fixture. The collet not only just designed but also manufactured. The paper shows implementation of technological concepts for a live industry component which will really help a collet designer to design such kind of collets. The design is carried out using PTC Creo Parametric 3.0 and AutoCAD 2016. Finite element Analysis is carried out with Creo Simulation.

SUMMARY

Design and manufacturing of expanding workholding collet for a rotary VMC fixture to be used in the industry.

Keywords: Expanding collet, Mandrel, Fixture

INTRODUCTION

Collets is one of the best clamping device used to fix circular workpiece. Collets hold the workpiece by expanding externally or internally. When the workpiece is fixed by clamping its outer surface, it is called external collet, otherwise it is internally expanding collet. Internal expanding collets are further classified according to its side of operation. As shown in the Fig. ***1***, single sided collets have open slots to one end only.

These kind of collets are easy to manufacture and hence is cheaper than double sided collets, but used for comparatively low clamping force. This paper shows a dimensional design process to design single sided expanding collet for a rotary CNC VMC fixture.

BACKGROUND

Collets are extensively used as fixture clamping elements. Shyr-long Jeng, Long-gwai Chen And Wei-hua Chieng*(1)* have found minimum clamping force for the stability of the workpiece while machining for different fixturing conditions. Excessive clamping force may deform the workpiece and less clamping force may not withstand the cutting load. Fixture clamping sequence is also has a major role to achieve the required accuracy. Anand Raghu, Shreyes N. Melkote*(2)* expressed that poor part location error leads to part location errors. The authors also refer that "In addition to typical error sources, such as fixture geometric error and elastic deformation of the fixture and part due to clamping forces, the clamping sequence used can also influence part position and orientation.". They have found influence of clamping sequence on part location errors by analytical model and experiments. J.H. Yehand F.W. Liou*(3)* have found identification of insufficient clamping force by analytical FEA model. Prediction of workpiece deformation helps the fixture designer to decide various parameters. Y. Wang, X. Chen and N. Gindy*(4)* have done deformation analysis between complicatedly shaped components and fixture elements. The verified FE analysis is used to predict surface error arising from deformations, and to evaluate the deformation distributions from fixture elements and work piece. K. Siva Kumar, and G. Paulraj*(5)* present the optimization of the locations of active (clamp) and passive (locator/support) elements in the workpiece-fixture system using genetic algorithm (GA) with ANSYS parametric design language (APDL) of finite element analysis. These authors have successfully developed a systematic procedure to optimise the locators and clamping positions to minimise the workpiece deformation using GA and FEA. Shane P. Siebenaler, Shreyes N. Melkote*(6)* studied various factors influencing the prediction of workpiece deformation with FEA. Prediction of deformations and locator reaction forces are also checked by experimental setup.

Traian Lucian SEVERIN and Vasile RATA*(7)* have studied on clenching mechanism for chuck model. The authors have expressed a mathematical model for chuck collet mechanism using theory of friction. Validation is also carried out by experimental setup. Stress model given by the authors can be used in the design of collet.

Thus, many researchers have worked for the analysis and design of fixtures, some on collets as well. Their findings individually have really helped the fixture design process and hence collet design process. But there are very less researchers who have designed and manufactured actual fixtures with collets which are to be used for industry purpose. The present paper gives design of expanding collet which is really to be used in the industry.

WORKING OF EXPANDING COLLET

Consider **Error! Reference source not found.**. The workpiece is kept in such a way, so that it touches the resting block. Taper surface of Mandrel and collet should match. Mandrel along with pull rod when joined using threads behaves like a single rigid bar. Puller rod when pulled downward, will push the taper contact surface of collet to expand outward and thus the workpiece is firmly clamped. To declamp the workpiece, the puller rod is again put to the original place.

STATEMENT OF PROBLEM

To design single sided expanding collet to hold workpiece for machining on VMC with 4^{th} axis(Rotary table with face plate support).

Material of the workpiece is Aluminum A360 Die Casting Alloy. The operations to be carried out are drilling and tapping. The fixture is rotary (4 axis) which is to be used with VMC machine. Fig. 3 shows orthographic views of the workpiece. As shown in the **Error! Reference source not found.**, the reference dimension of the internal hole is 23.875 mm with symmetric tolerance of 0.125 mm. This diameter is to be used to hold the workpiece using expanding collet. Thus the collet should hold the workpiece using this hole with consideration of thetolerance.Operations to be carried out are drilling and tapping on two sides of the workpiece as shown in **Error! Reference source not found.** and **Error! Reference source not found.**.

As the fixture is rotary in nature, each side will be machined by rotating respectively. At zero degree (Fig. 5), two tapping holes of M2.5 is to be done. At 90 degree rotation (Fig. 4), 3 holes of M3.5 and two tapping of M5 is to be done. Maximum cutting force is expected to be 1.25 kN. The clamping force should be about 1.5 times against the cutting force to avoid any undue situations. Workpiece material is A360 and has comparatively small depth of cut, single angle collet will be a good choice as it has less manufacturing cost. Clamping and declamping is suggested as hydraulic.

COLLET DESIGN PROCESS

Error! Reference source not found. shows concept design for collet. As shown here, the collet will remain vertical which will hold the workpiece from the inside diameter. This diameter is to be taken as reference. While designing collets, the reference diameter has greater importance. This diameter should be having higher surface finish and dimensional tolerance. Here the reference diameter has a surface roughness of 1.6 Ra and a symmetric tolerance of 0.125 mm which allows this diameter to be used as reference diameter using which the collet will hold the workpiece.

As shown in **Error! Reference source not found.**, the reference diameter is not even throughout the length but is has surface irregularities in term of steps. So, the collet cannot use the entire length of the reference diameter but it can use maximum of (5.7 + 2.3)8 mm for clamping.

The reference diameter has a symmetric tolerance of ±0.125 mm. Considering the worst condition, the minimum diameter of the workpiece can be 23.875 – 0.125 = 23.750 mm. The collet should be able to hold the workpiece for the entire range of the workpiece diameter. Clearance between the workpiece and normal diameter of the collet (without expansion) is taken as 0.020 mm on diameter. So, the collet

diameter should be 23.730 mm. And it should expand to clamp the workpiece with maximum diameter of 23.875 + 0.125 = 24.000. The collet should be able to expand by 0.020 + 0.125 = 0.125 = 0.270 mm.
Consider **Error! Reference source not found.**. As per an empirical relation, the collet should be 2 mm above at the initial state. As earlier stated, the collet diameter is to be taken as 23.750 which is closer to 24 by randomizing. To take 2 mm of collet thickness, internal diameter of the collet should be 20 mm. Considering taper angle to be 5^0 and extending the taper surface almost at the middle of the reference-diameter length, the taper surface should end at 24 mm from top surface. Then a step is provided to weaken the strength and thus to increase the stress concentration. This phenomenon helps the collet to expand evenly with respect to free end. The width of this section should be same as collet minimum thickness and the length should be according to length of the workpiece reference-diameter plus some clearance which is taken here as 5 mm. Workpiece resting is of 20 mm and collet flange is taken as 14 mm. As per these empirical relations remaining length of the collet is 39 mm. Collet thus designed is shown in **Error! Reference source not found.** by putting all other required dimensions as per need and experience. This collet will be firmly bolted with main fixture plate using 3 M6 bolts. **Error! Reference source not found.**shows complete collet assembly along with hydraulic cylinder.

FORCE CALCULATION

Depending upon the prevailing cutting condition, maximum cutting force will be 1.25 kN. This cutting force is given by the vendor, so here calculation for the cutting force is not necessary.

As the clamping force should be 1.5 times the cutting force,

Clamping force = Cutting Force X 1.5

$\therefore$ Clamping force = 1.25 X 1.5 = 1.875 kN which is randomize as 2 kN.

This force is to be generated by applying force on the entire taper surface of the collet by taper Mandrel. This Mandrel is pulled by puller rod and puller rod is connected with hydraulic actuating cylinder. Hydraulic cylinder has reciprocating piston which in term pulls the push rod. There should be enough amount of pressure inside the cylinder. To find intensity of the cylinder pressure, following procedure is carried out.

To achieve clamping force of 2 kN, pulling force required should be around 5.5 kN. Hydraulic cylinder is to be taken as standard element. Here, KOSMEK brand hydraulic cylinder is selected *(10)*. Looking to the specifications given, for various cylinders, LLR0400 will be a suitable choice. Fig. 11 shows specification of Hydraulic Compact Cylinder. The workpiece is clamped here when there is a pulling action in the cylinder. As shown in Fig. 11,

Pulling force or Cylinder force = P X 0.28

Assuming cylinder pressure P to be 20 MPa.
$\therefore$ Pulling force = 20 X 0.28 = 5.6 kN which is closer to pulling force required (5.5 kN).

FINITE ELEMENT ANALYSIS

The above design is a dimensional design and carried out using knowledge base and empirical relations. Dimensions thus achieved must be checked using FEA software. As most critical part in the entire assembly is collet, FEA is carried out for this component as below. Creo Simulation 3.0 is used to carry out FEA. Here assembly of mandrel and collet is considered and contact stress analysis has been carried out. The assembly is restricted at the either end of taper end. Pulling force is applied at the end of puller rod. Mesh size is restricted to maximum size of 5 mm.

The result (Fig. 11) shows that maximum stress produced is 290 MPa. The collet is made from EN24 for which tensile and yield stress is 850 N/mm^2 and 650 N/mm^2*(8)* respectively. Taper Mandrel is made of EN31 which has Tensile Strength as 750 N/mm² and Yield Stress 450 N/mm²*(9)*.

FEA result shows that the collet and mandrel are well within allowable stresses.

MANUFACTURING

The collet thus designed is manufactured also using the achieved dimensions. Table 1 shows manufacturing process plan for manufacturing of collet. Fig. 12 shows manufactured collet without component. Fig. 13 shows manufactured collet with component.

CONCLUSION

The paper presents a dimensional design of single sided expanding collet for fixture. This design achieves all the requirements needed for actual working. Manufacturing of the component has also been carried out. The component thus manufactured has been checked and is found within specified tolerances.

Many parameters are assumed here from past experiences. This design can be over safe and may use higher dimensions than what really are required. To get optimized dimension, the design should also be checked analytically.

FIGURES

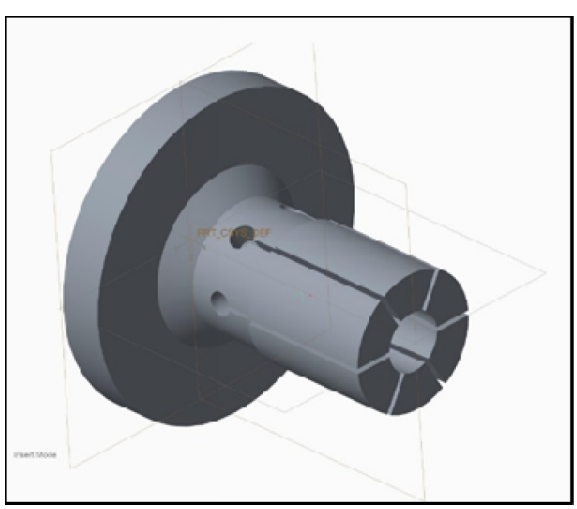


Fig. 1. Single Sided Expanding Collet

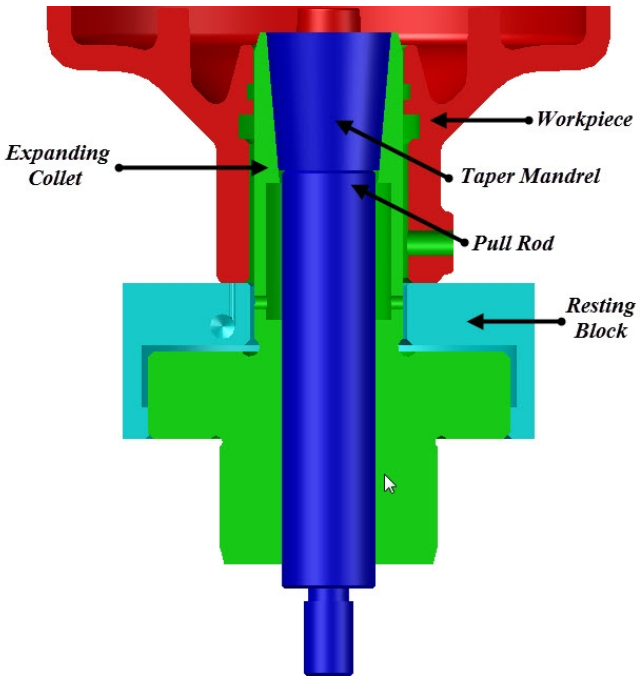
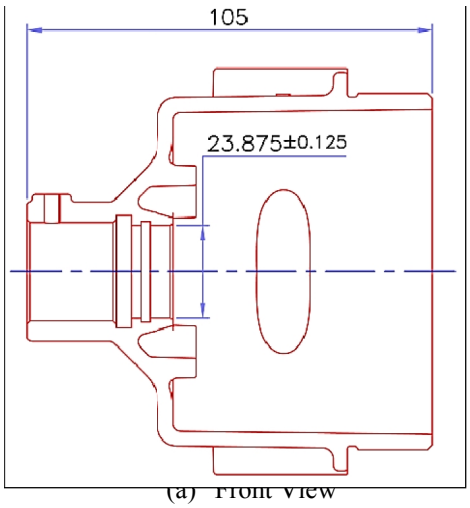
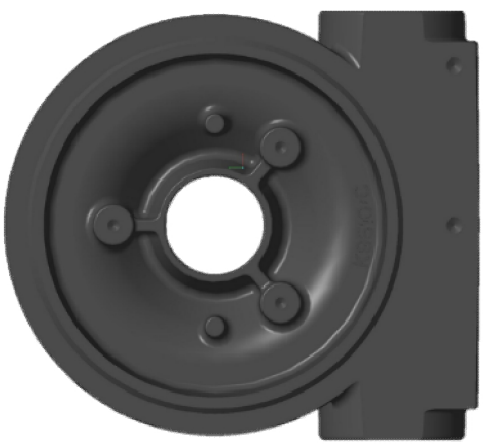


Fig. 2. Working of expanding Collet



(a) Front View



(b) Side View - Shaded



(c) Side View - Shaded

Fig. 3. Orthographic Views of Workpiece

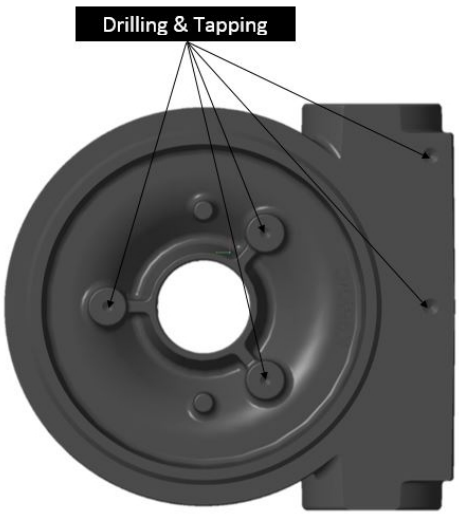


Fig. 4. Operations at 90^0



Fig. 5. Operations at 0^0

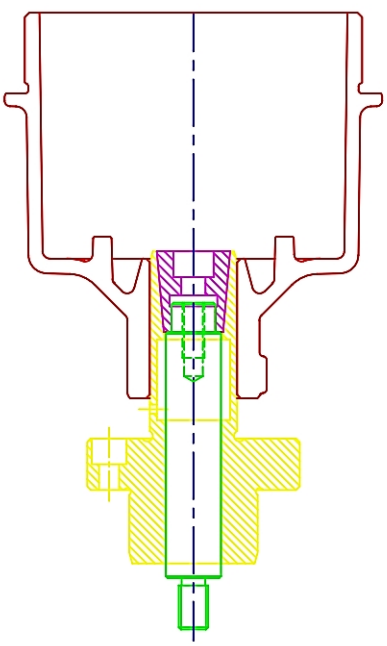


Fig. 6. Concept Design

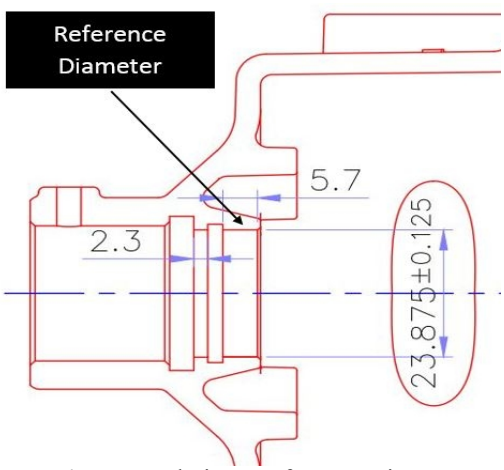


Fig. 7. Workpiece Reference Diameter

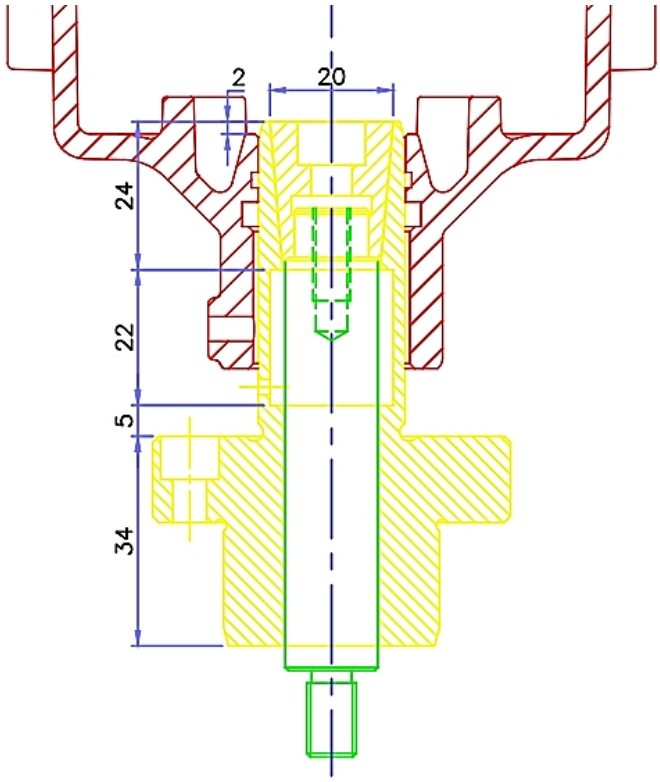


Fig. 8. Collet Design

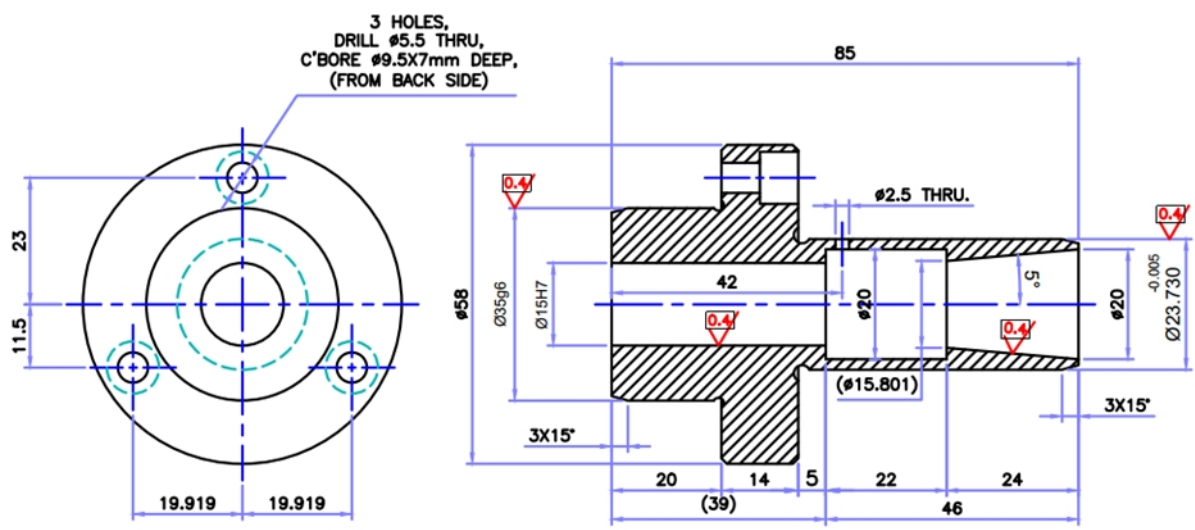


Fig. 9. Collet Drawing



Fig. 10. Exploded Collet-Assembly with Hydraulic cylinder

Specifications

Model No.		LLR0360-□□	LLR0400-□□
Full Stroke Y mm		Y : 1～50	
Cylinder Area cm^2	Push Side	4.5	5.3
	Retract (Pull) Side	2.5	2.8
Cylinder force (Calculation Formula) kN	Push Side	P×0.45	P×0.53
	Retract (Pull) Side	P×0.25	P×0.28
Cylinder Capacity (Calculation Formula) cm^3	Push Side	Y×0.45	Y×0.53
	Retract (Pull) Side	Y×0.25	Y×0.28
Cylinder Inside Diameter mm		ϕ24	ϕ26
Rod Diameter mm		ϕ16	ϕ18

Fig. 11. Specification of Hydraulic Compact Cylinder*(10)*

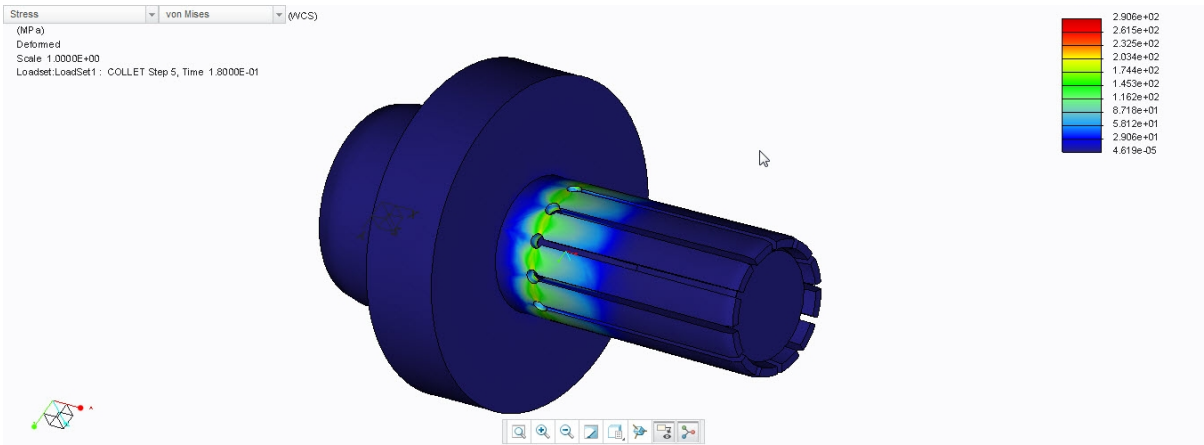


Fig. 12. FEA Stress Analysis Result

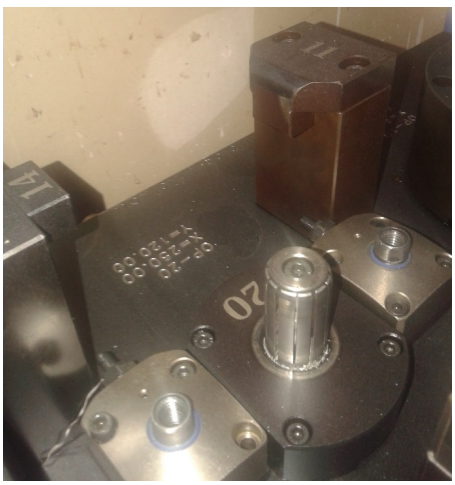


Fig. 13. Collet without Component

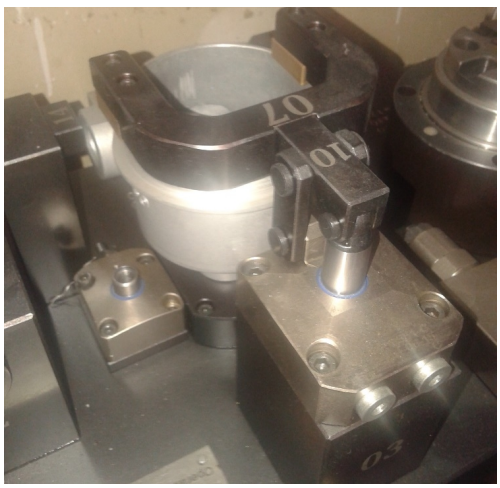


Fig. 14. Collet with Component

TABLES

Table 1. Manufacturing Process Sequence

Sr.NO.	Name of Operation	Machine
1.	Turning(Including taper surface)	CNC turning centre
2.	Hole on PCD	Jig Boring Machine
3.	Relieving Hole for slit cutting	Jig Boring Machine
4.	Heat Treatment	-
5.	I.D. and Face Grinder	Universal Grinding Machine
6.	O.D. Grinding	Universal Grinding Machine
7.	Slit Cutting	Wire Cut Machine

ACKNOWLEDGEMENT

The authors wish to acknowledge very kind support of Mr. Sudhir Thakar and Mr. Pradip Thanki, Supra Technology, Rajkot, Gujarat, India for carrying out this research work.

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9. Mechanical Property of EN31: http://saajsteel.com/?page_id=1098
10. Compact Cylider Specification: www.kosmek.co.jp/data/pdf/en/LL_R00_2014KW_GB.pdf, page 672

Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th& Jan. 6th, 2016)

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RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

Preparation of biodiesel from Karanja oil

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ABSTRACT

Biodiesel was produced from karanja oil using homogeneous catalyst by transestrification process. Due to high acid value(5.2 mg KOH/gm) of karanja oil two step process was used, first acid esterification using H_2SO_4 as a acid catalyst followed by transesterification using KOH as base catalyst. In both process methanol to oil molar ratio was kept 6:1. 90% yield was obtained by this process. Viscosity of karanja oil was reduced from 45.4 Cst to 4.5 Cst @ 40^0c and final acid value was 0.5 mg KOH/gm which was within limit of biodiesel standard.

SUMMARY

Non edible oil (karanja) is attractive feed stock for biodiesel production for India.

Keywords: karanja oil, biodiesel, catalyst

INTRODUCTION

Biodiesel is a fuel which can be produced from vegetable oils and animal fats. Both edible and non-edible oils are suitable to produce the biodiesel (*1)*. In India edible oils are very costly. India consumes 16.6 MT of edible oil in every year. In India consumption of edible oils is increasing at the rate of 5% per year. India imported more than 50 % of edible oils of its total consumption from other countries (*2)*. So for India non edible oils like jatropha, karanja, neem etc are only option for biodiesel production. Karanja oil is a non-edible oil of Indian origin. Karanja trees are found almost all states of India. Karanja is also called pongamia pinnata. It is a medium size tree; easy to grow becomes adult in four to five years. It is a monotypic genus and grows abundantly along coasts, riverbanks and reclaims marginal lands. karanja fruits has viability period of one year, seeds number are varies between 810-1410 per kilogram. The oil yield of karanja is 2.0-4.0 tones/hector/year. Its seeds contain oil and high percentage of poly unsaturated acid. The main fatty acid present in karanja oil is oleic acid. Parts of the karanja tree plants are used as a

crude drug for the various human treatment, soap making, fuel for lamp, in leather industries for finishing and tanning, veterinary medicines etc. the wood part of karanja is used for brushing the teeth. karanja seeds contain 25 to 40% oil which attract researchers to produced biodiesel from it(*3-7)*. The viscosity of pure karanja oil is 10 to 15 times higher than the diesel which prevents its direct use in diesel engine. High viscosity of karanja oil creates a problem in fuel injection system. So it is necessary to improve its viscosity before using in diesel engine (*8)*. Transesterification is a process which reduced the viscosity of vegetable oil (*9)*. This process does not affect the calorific value of oil. In this process oil is react with methanol with stirring and moderate heating, reaction rate will increase in present of catalyst.

MATERIALS AND METHODS

Karanja oil is obtained from D K Pharmaceuticals, Bhavnagar. Sulphuric acid (97%), potassium hydroxide (97%) and methanol (99%) of Rankem are procured from modern chemical, Bhavnagar. All procured chemicals were used without any further treatments**.**

Experimental set up

The experimental set up was consisting of hot water bath with temperature controller in which three necks round bottom flask was heated as shown in Fig-1. Mechanical stirrer was used for sterling of reaction mixtures. Condenser was attached with one neck to prevent any loss of evaporated methanol.

Esterification

The acid value of karanja oil was measured by titration method and found to be 5.2 mg KOH/gm. The kinematic viscosity of karanja oil was found to be 45.5 mm^2/s. The acid value of oil should be less than 4 mg KOH/gm for better yield in Transesterification of oil. So first esterification of 100 ml karanja oil was carried out using H_2SO_4 (0.6 vol % of oil) at 45^0c for 1hr using 6:1 molar ratio of methanol to oil which reduced acid value of karanja oil to 2.4 mg KOH/gm. After completion of the reaction the product was transfer to separating funnel where methanol with impurities moved to the top and was removed.

Transesterification

This is a second step for the production of biodiesel in which the product of esterification was transferred to reaction flask where first it was heated to 55^0c. KOH (1% vol of oil) was dissolved in methanol with 6:1(methanol to oil molar ratio). Product was heated and stirred for 90 Min than it was transferred to separating funnel where it was allowed to settle for 8 hr. Karanja oil methyl ester was moved on the top surface and glycerol with impurities was settle at the bottom surface (Fig-2). The separated karanja oil methyl ester was washed by water for 4 times to remove soap and catalyst. The yield obtained by this method was 90% and viscosity was reduced to 4.5 mm^2/s. The acid value of KOME was measured by titration and found to be 0.5 mg KOH/gm and density was reduced to 0.88 kg/lit which were meets Indian biodiesel standards.

RESULTS AND DISCUSSION

Crude karanja oil is transesterified by KOH and viscosity of oil is reduced to 4.5 mm2/s and 90% yield has been obtained by two step process. Non edible oils like karanja and jatropha are promising feed stock for biodiesel production in India. Due to high acid value of karanja oil (above 4 mg KOH/gm) two step process is required for KOH/NaOH to be used as a catalyst. Heterogeneous catalyst can be tried to produce biodiesel from karanja oil in single step process.

CONCLUSION

As edible oil is very costly for a country like India, biodiesel from non edible oil like karanja is a promising feed stock as an alternative fuel for diesel engine. Karanja oil methyl ester can be blend with diesel and used in conventional diesel engine without any engine modification.

FIGURES

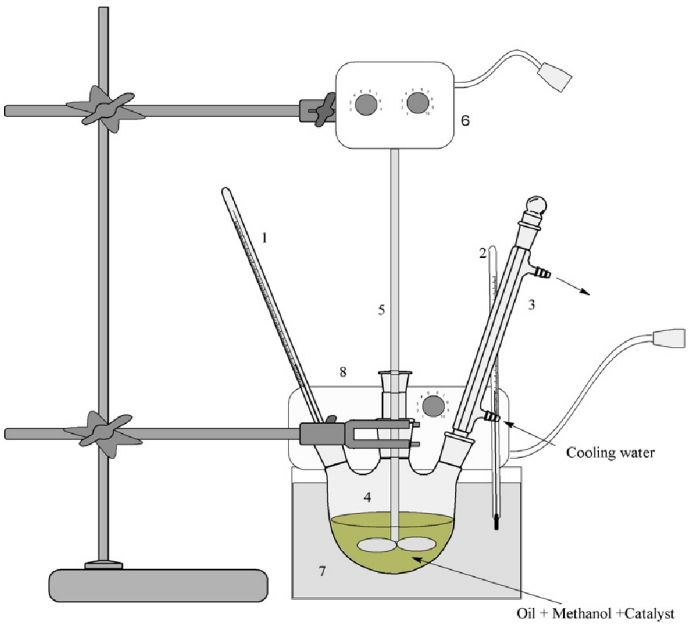


Fig-1 Experimental setup for biodiesel production. 1 and 2 thermometer, 3- condenser, 4- glass reactor, 5- Teflon stirrer, 6- agitation motor, 7- hot water bath, 8- temperature controller.



Fig-2 separation of glycerol and impurities and KOME

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An approach for Image Segmentation using SVM- a machine learning methodology

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ABSTRACT

In Computer vision system, image segmentation is fundamental task. Many techniques for image segmentation like Edge detection, Region based etc. are available but all these techniques not provide automatic segmentation. Machine learning method also use for image segmentation. Machine learning methods reduce the work for user because only first time training is needed after that all process performed automatically. This paper presents general procedure of SVM (Support Vector Machine) to segment the image. Segmentation of image can be done by selecting any of image feature like color, texture etc. In this paper it is done using intensity feature in gray images. SVM method classify image pixels into two groups, first group consist of pixels which belongs to a region of interest and second group consist of pixels which does not belong to it.

SUMMARY

Support Vector Machine is classification method which is used to classify data into two different class. This concept applied in this paper to classify image segments.

Keywords: Image segmentation, Machine learning, SVM, classification, Region based, Edge detection, intensity based segmentation.

INTRODUCTION

Image segmentation subdivides an image into its constituent regions or objects, for further analysis. Image segmentation can also define as the process of divide the image into regions with pixels having something in common. They may have similar brightness, texture or color [5]. These properties are used to define different regions. In segmentation process the image pixels are classified into two or more classes. Each of the class represents some region of image. Image segmentation is a prior process of image analysis and pattern recognition, but it is difficult to perform with improper segmentation.

In past years many segmentation techniques are used for image processing and these techniques are classifies into following five [1][2][3].

a. Region based
b. Edge based
c. Threshold based
d. Feature based clustering
e. Graph based

a. Region based[1][2][3]

This technique assigns pixels into a group based on similarity characteristic. Due to this Region based segmentation also known as "Similarity Based Segmentation". The area formed for segmentation should close then only consider as region. All pixels are considered in some region, no area remaining in this segmentation. Region based methods are: local techniques, global techniques, splitting and merging. After detecting the boundary, the edge is generated for further process. This technique is used for only simple images.

b. Edge based[1][2][3]

This is mostly used and simple technique. In this technique boundary pixels are identified and the mark as edge of objects. This is discontinuous base segmentation. For developing edge around region pixels are compared with their neighbor pixels value. Edge detection can perform by: Canny, Sobel, Laplacian, Gradient or Robert edge detector. These algorithms are work with simple and noise free images. So, noise removal is first step for detecting edge of image. With noise in image the edge detection not done properly

c. Threshold based [1][2][3]

This is easiest way of segmentation. In this technique the threshold value is obtain from histogram of edges of original image. Thresholding process converts multilevel image into binary image by assigning image pixels into background and foreground (using 0 and 1) based on any threshold value T(intensity or color value). Threshold value can be global or local for regions. Main drawback of this method is it not suitable with complex images or images which have target area very small compare to background.

d. Feature based clustering [1][2][3]

Clustering means putting together pixels into a cluster using their feature like colour, texture etc. Clustering in colored image is done by unsupervised Fuzzy C. if image is noisy then fragmentation is done. Another cluster technique K means is used for texture base image segmentation. Both are iterative techniques. Base on initial set of cluster the quality of solution can be define. Over segmentation is one problem in this method.

e. Graph based methods [3]

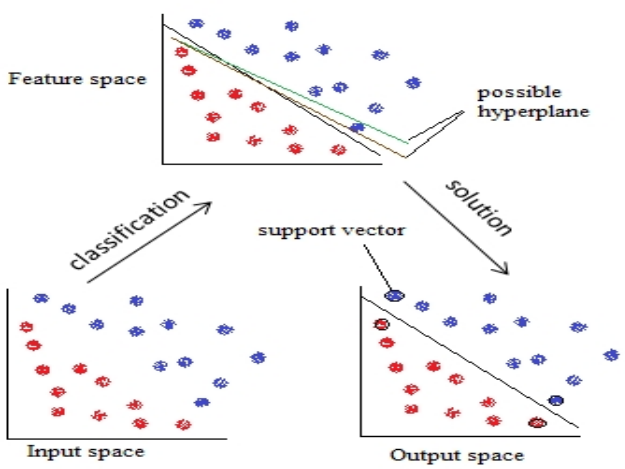
In this method, graph are created using nodes and arcs, where node represents any pixels and arcs represents the links with neighbor pixels. For segmentation, requirement is to minimize the weight that cut a graph into sub-graph. But it has disadvantage of high complex computation.

But none of this approach can apply in general conditions. And all this are manual process which require time constrain and each time lots of work to be done, so, requirement of automatic learning methods are arise in which machine is learn by themselves and provide output.

Following section of this paper discuss support vector machine (SVM) method as a machine learning method for image segmentation.

SUPPORT VECTOR MACHINE

SVM(support vector machine) is suitable alternative of the traditional classification methods due to its good generalization performance, when the number of training samples is very small and the dimension of feature space is very high[4]. The principles of SVMs have been developed by Vapnik



[6].

Fig. 1. SVM Algorithm

Segmentation is process by which image divide into different regions based on their characteristics and apply any label. Segmentation also provides classification based on which further information of image can be done. SVM can classify pixels base on their classes.

In SVM literature, a predictor variable is called an attribute, and a transformed attribute which is used to define the hyper plane is called a feature. Feature selection is task of selecting the most suitable representation. A set of features that describes one case is called a vector. Main aim of SVM modelling is to find out optimal hyper plane which separates clusters of vector in both side of plan in such a way that cases with one category of the target variable are on one side of the plane and cases with the other category are on the other side of the plane. The vectors which are near the hyper plane are the support vectors [7]. As shown in figure 1.

SVM find this hyper plane so, easily classification done and error in test sample is minimized. SVM find linear plane for simple data and for nonlinear separable data it convert data such a way that linear hyper plane can be form [4].

If the margin of the hyper plane is wider, the more clear classification can be done and it classifies more easily the same data into required classes.

For linear separable two classes

$$f(x) = s_i \{\sum_{i=1}^{m} \propto_i y_i(x_i * x) + b\} \quad (1)$$

For nonlinear separable

$$f(x) = s_i \{\sum_{i=1}^{m} \propto_i y_i K(x, x_i) + b\} \quad (2)$$

Where K(x,xi)is kernel function [4]. In case of linear it is simple a dot product. The kernel function in the SVM classifier plays the important role of implicitly mapping the input vector into a high dimensional feature space [4].

Types of kernel functions as follow

•Linear kernel

$$K(x, x_i) = x \cdot x_i \quad (3)$$

•Polynomial kernel

$$K(x, x_i) = [(x \cdot x_i) + 1]^q \quad (4)$$

•Gaussian RBF kernels

$$K(x, x_i) = e: \{-|x - x_i|^2/\sigma^2\} \quad (5)$$

Generally RBF kernel is used. Linear kernel is special type of RBF kernel. In case of large number of features linear kernel used for better results, RBF kernel is suitable for it. The values of y which appear in equation (1) and (2) can be +1 for positive classification training vector and -1 for negative classification training vector, and for classification the trainingdata (x,y) require to find classification function. Values α of denotes Lagrange multiplier obtain in minimization process and value of m denotes number of training vector which are chosen to form hyper plane [5].

SEGMENTATION PROCESS

Segmentation using SVM have following steps

- Pixel level feature extraction
- Training sample selection
- Training procedure
- Pixel classification

The process of image segmentation using SVM can summarize in figure 2.

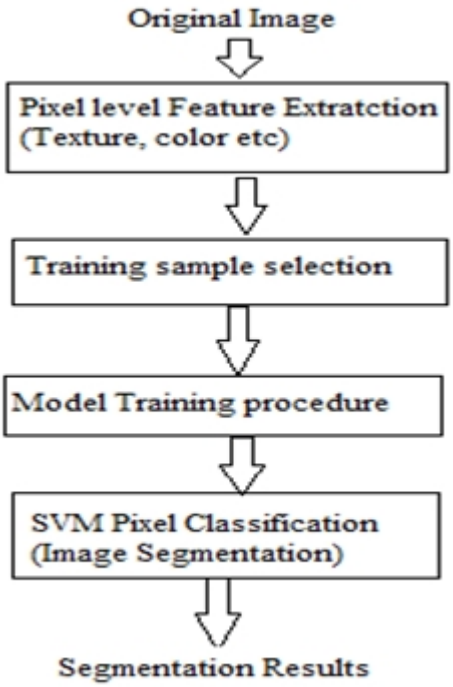


Fig. 2. Process of segmentation using SVM

A.Pixel level feature extraction

This is the first step of algorithm in which extraction of features on pixel levels perform. For segmentation features like color, texture or combination of both is used. If input image is color image then each pixel have RGB components. For only texture this image converted into grey image. All paragraphs must be indented. All paragraphs must be justified, i.e. both left-justified and right-justified.

B. Training Sample selection

Once pixel level features are extracted, next step is to select the sample images for training. The process of selection of image for training may be done on random manner or may take calculations. Image samples for training should have feature based on which hyper plane create. Images for training should be selected in manner to easily classify the classes.

C. Training procedure

Observer first defines +1 and -1 region manually for test sample in images. Then Observer provides label to images pieces accordingly according to feature selection. Fu Yan et al.[4] use wavelet energy feature as texture feature, and gray values of eight-neighbourhood of sample point as gray feature. H. GÓMEZ-MORENO et al [5] use color feature for label different pieces of image. Observer marks both regions which have to take in consideration and which not. All these information stored into vectors for training process of SVM.

Once training on sample complete, function is defined based on which the process of segmentation is done. Based on information gain in training period this function applies on images which are not in training set. The process of training is continuing iteratively until the desired result is produce.

D. Pixel Classification

This is simple process in which the vector of original image is provided, vector contain the feature information and based on function created in training procedure, test image pixels are classified into +1 or -1 classes. But it may happen some pixels are in between this two values then they are not taken in consideration. Value above +1 are consider as the +1 and values below -1 are consider are -1 all values between +1 and -1 are consider as the error values. So, this indicates that function should be choosing such that it minimizes error pixels.

H. GÓMEZ-MORENO et al [5] use color image for experiments and they get results and they conclude that the result is depend on the training procedure. Output segmentation results can be improved by improving the training. Fu Yan et al.[4] also conclude that the improvement occurs in classification with increase training samples.

EXPERIMENTAL RESULTS

This section presents the results of experiments which were carried out for explained algorithm. This implementation of the algorithm has been done using MATLAB R2012a. SVM has been implemented using one of the MATLAB library named LIBSVM which contains all the required functions, methods etc. for purpose of training and classification process.

As stated earlier in training phase intensity feature of image pixels are consider for classification. In training phase median intensity of image is taken for each image. Afterwards each image was divided into 4x4 pixel sub image and median intensity of sub image was compared with median intensity of actual image. Based on experiments, threshold value was derived. Compared results having less value then threshold, that region was considered as background. Other region having high value than threshold, considered as foreground.

Five real life images were taken as training and then on image tested based on model created in training. The input images shows in figure 3. The output images segment object from input images which shows in figure 4 and figure 5.

This algorithm implemented on images which have plain background and some small objects. In this experiment the results have been obtained using RBF kernel as Equation (5). The value for γ set to 0.058315 and for cost parameter C set to 0.46652 and 2-flod cross validation used.

This algorithm was tested on different sets of training images. Even with more number of training images this algorithm works efficiently. Thus, this algorithm works smooth irrelevant of number of training set images.



Fig. 3. Test images with one object.

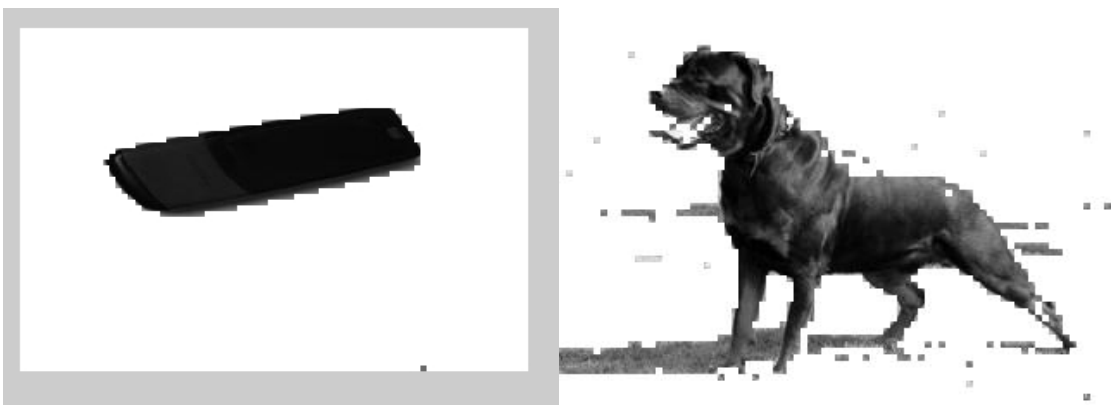


Fig. 4. Object identified using SVM.

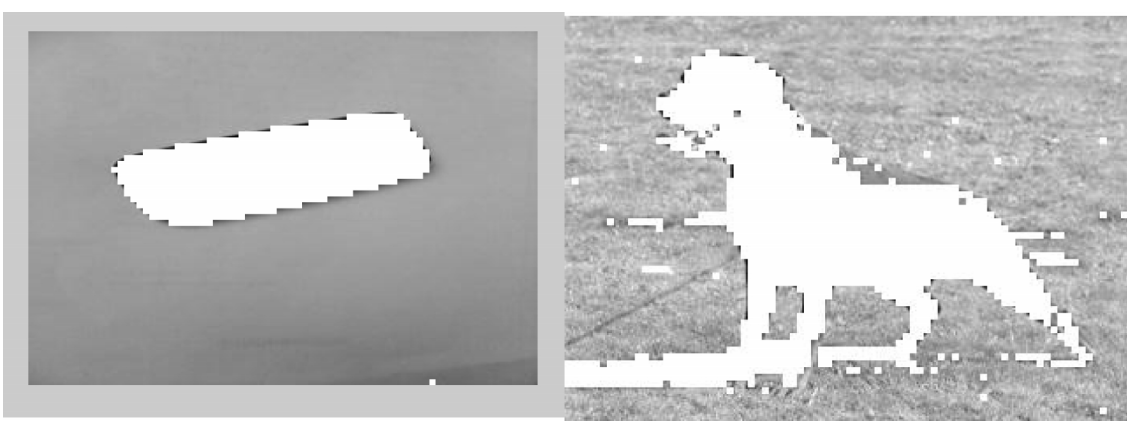


Fig. 5. Object cut from the original image. Background in image.

CONCLUSION

After the study of common methods used for image segmentation and process of segmentation of image using SVM. Hence the conclusion is that using SVM, segmentation process is performed automatically after providing first time training and with low training set even SVM provides more classification compared to other methods. SVM provides the general approach for segmentation. Thus, it is good alternative of traditional image segmentation methods which cannot use generally. As SVM provide automatic process that is easy to segment images and the procedure is iterative in training phase so, by improving the kernel function this can apply on different type of image.

ACKNOWLEDGEMENT

We heartily thankful to my Head of the Department, Dr. Amit M. Lathigara, for his continuous motivation and support to always look for innovations and to carry out research work. We also thankful to RK University for providing me great and precious infrastructure to carry out my research experiments. Last but not least, we appreciate the efforts provided by my colleagues, which helped me a lot to focus on my research experiments through their knowledge and experience sharing.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th& Jan. 6th, 2016)

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Energy and Power Conserving On-Demand Routing Strategy for Ad hoc Network

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ABSTRACT

Ad hoc network is having group of mobile nodes. Nodes can move at any time during the transmission. Routing of packets is the main task in ad hoc network. With existing on-demand routing protocols like AODV and DSR focused on shortest route for transmitting packets. Few decisive constraints in these protocols are energy and power consumption. Existing routing protocols have not considering energy and power into their account. Proposed routing strategy EPCP (Energy and Power Conserving Protocol) focused on energy and power conservation. In proposed routing strategy, routes are found not only considering number of hops, but residual energy also. Additionally, in this strategy transmission power is also conserved based on the distance of next hop available in the route. For the nearer nodes, it consumes less transmission power and also saves the residual energy of nodes. EPCP is tested using ns-2.34 simulator and also compared with existing routing protocols.

SUMMARY

Proposed routing strategy mainly focused on conserving transmission power during data packet forwarding. Hence it also conserves residual energy of nodes and provides better performance compared to other on-demand routing protocols like DSR and AODV.

Keywords:Ad hoc Network, AODV, DSR, EPCP

INTRODUCTION

Ad hoc network is a group of wireless nodes, which are mobile in nature. Nodes can roam anywhere in the network at any time. Ad hoc networks formed to serve some temporary purpose without having any established controller. As nodes are allowed to roam anywhere at any direction the structure of nodes change vibrantly. It is difficult to define node's position in ad hoc networks as nodes are mobile *(*13*)*. For provide communication between any two nodes, it must relays on multi-hop fashion. All nodes are equal without central controller, nodes are treated as routers. All nodes are cooperating with each other for forwarding data packets in hop by hop manner. For transmitting packets wireless radio channels are used. As all nodes are not within the range of each others, nodes are cooperating in gracious approach to forward packets ahead with store and forward method. The node in ad hoc network may be treated as an intermediate node, source, destination or router to facilitate other node's data forwarding. Ad hoc networks are highly utilized in emergency situations like normal tragedies, military operations, emergency medical circumstances, etc. Because of adaptable behavior and no need of central controller, ad hoc network is easy to set up. Even the main constrain in this network is forwarding of data between any two nodes *(*16*)*. Figure 1 represents example of node arrangements in ad hoc networks. Here, node S is source node it has some data for destination node D. For the same node S has to find out route towards destination. As both of the nodes S and D are out of transmission range, S must rely on intermediate nodes. The routes from the pair of S and D nodes is either through intermediate nodes C and E or A, F and G. Source node selects any of the routes like S-C-E-D and then begins transmission through it. Suppose during transmission of data packets any of the nodes either C or E may move from its original position. In such situations, source node will have to selects alternative route towards destination.

Routing is the main challenge in such networks. Even once route has decided from source and destination then also it is needed to route remains stable. Because nodes in ad hoc network work on battery so they have limited amount of energy. During various operations in networks, like forwarding packets, receiving packets, idle etc. nodes consume energy. Hence nodes have reduced their finite amount of energy. Even during transmission of packets, nodes used fixed transmission power irrespective of the distance of next node. It also consumes more energy and unnecessarily nodes reduced their life.

OVERVIEW OF EXISTING PROTOCOLS

For ad hoc networks, so many routing protocols are available for finding routes. Routing protocols mainly categorized based on their behavior in one of the three types:

1. Table - Driven routing protocols
2. On - Demand routing protocols
3. Hybrid routing protocols

In the first category protocols, nodes are having some predefined routes for every other node in the network. Nodes store such routes in routing table and when required to transmit data it utilize one of the available route. While in on - demand protocols, node find routes dynamically whenever required by using various strategies for discovering routes. With hybrid protocols they have mainly focused on zones.

For nearer distance nodes consider in one zone and for them they implement table - driven routing. While the nodes which are not in same zone, they have used on - demand routing and find the route to a particular destination when needed. For medium and large size networks on - demand routing protocols are provide better performance. Once route has been found between source and destination node, it works smoothly. Most of on-demand protocols have first process to find the route and then also provide mechanisms for maintaining the routes during transmission. On the traditional on-demand protocols, so many modifications have found and few of them related to this research is described in related work section.

RELATED WORK

Operations of ad hoc networks focused on battery life of nodes and accordingly lifetime of network is depended. Nodes are mobile so they can't be recharging their energy while continuously consuming during various operations. Suppose, in the selected route, nodes are having limited amount of remaining energy, nodes become fail after some time. Hence routes through those nodes will break and it also affect on overall life time of network. Various on-demand routing protocols for ad hoc networks like AODV *(*10*)*, DSR *(*1*)*, etc. have not considering energy level as a metric. Many routing protocols were proposed in recent years and they focused on energy level of node with on-demand routing mechanism.

The authors in *(*5*)* proposed routing protocol derived from DSR and developed energy efficient protocol. They focused on optimal route maintenance based on energy of nodes. After formation of routes, protocol verified energy level of active links. Protocols defined some minimum value of energy and if more than 75% of nodes from that link are having less value of energy then route error packet has been sent. So during forwarding process, source node initiates discovery and switch over transmission on alternate route. In *(*6*)* authors developed routing protocol with considering transmission power into account. For finding optimal route and maintain link quality, they calculate transmission power used at each node during route discovery process. At the end, source node calculates average transmission power for each route and selects route with highest transmission power to make route optimal. But in that, such route was also consume more energy i.e. route provides good quality of link with decreasing amount of residual energy. Recently authors in *(*8*)* proposed on demand routing protocol with adjusting transmission and receiving power based on the size of data packets. So for each packet the power level may change. For large size of packets protocol was consumed more power and for small size of packets it consumed less power.

MATERIALS AND METHODS

Proposed routing strategy EPCP is depended on on-demand routing protocols. After studying DSR and AODV as mostly used on-demand routing protocols. Here we have modified behavior of original protocols and found novel routing strategy. Proposed strategy provides source-initiated, energetic and multi-hop routing in ad hoc networks. When any node wants to send data packet, then first it will find route to that particular node using route discovery mechanism. Source node floods RREQ packet in the network. When RREQ packet received by other node that is either destination or intermediate node. If it

is destination node, then generate RREP and send RREP back to the source. If it is intermediate node then, it checks in cache whether route to a destination is available or not. If that route is present in cache and it is recently updated then generate RREP and sent back to the source. But if intermediate node has no recent entry for destination, then it further floods same RREQ towards destination by adding its own address in header. Finally if none of the node is having route to destination, RREQ packet has reached at destination node through multiple intermediate nodes. Destination node generates RREP and inserts its own residual energy into the header of RREP. After generating RREP, destination formed a reverse path for all from where RREQ has arrived. Now RREP has sent back towards source. Firstly RREP has reached at intermediate node present in the route. Each intermediate node first checks its own residual energy with predefined threshold value. If that node has higher residual energy then threshold then and then it allows in route formation process. If intermediate node is having less residual energy compared to threshold then it simple discards RREP and can't allow participation itself in route formation process. When it finds sufficient energy level of it, intermediate node inserts its own residual energy and forwards the same towards source. The same process is repeated by each intermediate node present in the route towards source during reverse path creation.

Additionally, each intermediate node computes the transmission power based on the distance from where it received RREP i.e. its previous node from destination direction. The transmission power can be computed according to the two ray propagation model *(4)*.

$$P_r = \frac{P_t G_t G_r h_t^2 h_r^2}{d^4 L}$$

In the above equation P_t and P_r are the transmission and receiving powers respectively. While d represents distance. The remaining fields like h_t and h_r are the heights of antenna, while G_t and G_r are the gain and L is the path loss. The all remaining fields are considered as constant and their computed values are 1, so they will be ignored. Above equation can be also written as:

$$P_r = k.\frac{P_t}{d^4}$$

Where k is 1 (constant value from all remaining fields mentioned above).

$$P_t^{'} = P_r k d^4$$

So finally we got the above equation to compute required transmission power during data forwarding process. It is computed based on the distance of next hop towards destination. The computed transmission power is stored by each intermediate node and stores in its cache.

The whole above process for finding route is represented by below algorithm:

Begin:

Step 1: Source broadcasts RREQ for finding path

Step 2: If intermediate node has route then

It sent back to source otherwise RREQ is again broadcast towards destination

Step 3: After receiving RREQ, destination generates RREP and inserts its own residual energy

Step 4: At Intermediate Node:

Checks: If residual energy < Threshold then

Generate RREP and send towards source

Else Discards RREP

Computes Transmission power and store it.

Step 5: Upon Receiving RREPs, source selects the route with max avg. energy

Step 6: Source initiates data forwarding

Step 7: Intermediate node adjusts Power and forward Packets towards destination

End

When RREP packet reached at source from multiple routes, source calculates average value of residual energy for each route. As residual energy is inserted by each intermediate node in the header of RREP. Source selects the route with highest value of average residual energy among all available routes and start transmission of data packet from it. Other routes are kept in cache by source. When data packet arrived at intermediate node, first that node adjust the transmission power already computed based on distance and then forward ahead towards destination. As transmission power is varied, less power is consumes for nearer nodes instead of fixed power, it also consumes residual energy of node during forwarding of packets.

.

RESULTS AND DISCUSSION

The existing protocols DSR & AODV and proposed routing strategy EPCP have been tested in NS – 2.34 *(14)* simulator for evaluating performance. With random way out mobility model simulation is performed. All modification have been done in the code of AODV and DSR protocols available with ns – 2. Various simulation environments and parameters are summarized in table – 1.

During simulation, DSR, AODV and proposed routing strategy EPCP has been evaluated and computes various results for various parameters like packet delivery ratio, routing overhead, average path length,

average remaining energy of nodes, delay etc. The simulation is performed for 200 nodes and compare above 3 routing protocols.

Figure 2 represents packet delivery ratio against various values of pause time. PDR is the ratio of total number of packets received by receiver and total packet sent. Form that it can be represented proposed routing strategy EPCP provides better performance in terms of packet delivery ratio. For all routing protocols PDR is increased as the value of pause time is increased. But proposed routing strategy provides very high PDR against all value of pause time compared to DSR and AODV.

Figure 3 shows average path length for all protocols against pause time. Path length to be considered as number of hops from the route and then average value will be taken for each time. As DSR focused on shortest path, DSR provides less average path length compared to EPCP. In EPCP it also verifies remaining energy of each path and then selects the route then also it provides good results compared to AODV protocol. Figure 4 represents average remaining energy for all nodes at the end of simulation against velocity. Once simulation is over, it computes remaining energy of each node and then calculated average value of them for each protocol like DSR, AODV and EPCP. In proposed routing strategy, it highly focused on remaining energy and also used only required transmission power, it conserves more energy for each node. From the figure 4 it can be said that, in EPCP the remaining value of energy is increased i.e. nodes lifetime is increased. This will direct effects on network life time and node remains more time with consuming less energy compared to DSR and AODV protocols. Remaining energy is computed against various values of velocity. As velocity is increased, nodes can move with more speed in network during simulation. That's why as increasing velocity, the remaining energy for all protocols are reduced.

Figure 5 represents end-to-end delay against pause time. In proposed routing strategy, it takes some more time during calculating remaining energy and computing transmission power. Because of the same, proposed strategy EPCP takes some more delay compared to DSR and AODV protocols.

CONCLUSION

In ad hoc network the main problem is routing. Most of the existing on - demand routing protocols focused to find shortest route. Nodes are mobile in ad hoc network, they have limited amount of resources. For conserving resource like energy, existing protocols have not provides mechanisms. In proposed routing strategy EPCP, mainly focused in conserving residual energy and transmission power. It also provides route for forwarding packets more stable with all nodes higher energy level. Proposed routing strategy provides better performance compared to DSR and AODV in terms of packet delivery ratio, average remaining energy etc. While in terms of average path length, EPCP has good performance than AODV but worse compared to DSR. During the computation of residual energy and transmission power, EPCP takes some more time and it results more end-to-end delay compared to DSR and AODV protocols.

FIGURES

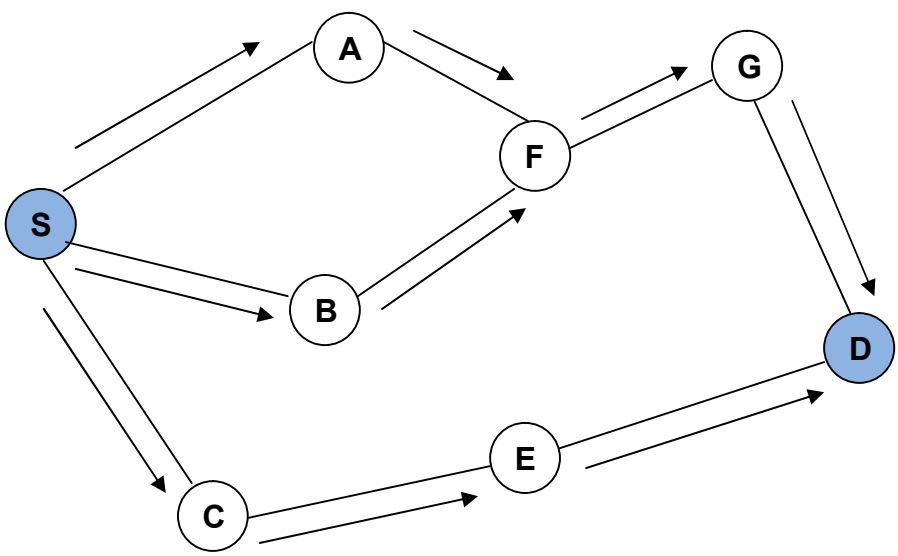


Fig.1. Topology of Ad hoc Networks

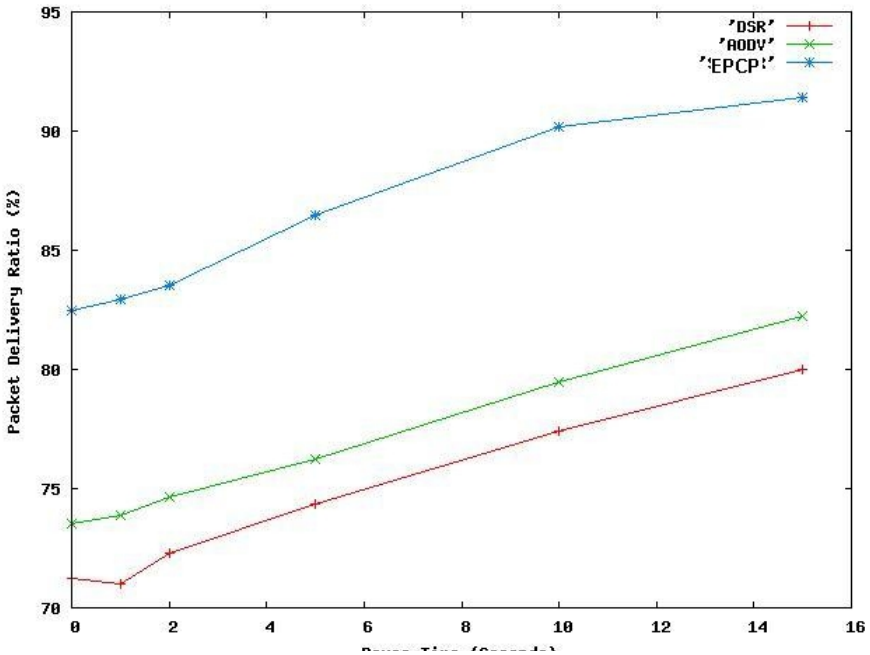


Fig. 2. Packet Delivery Ratio v/s Pause Time

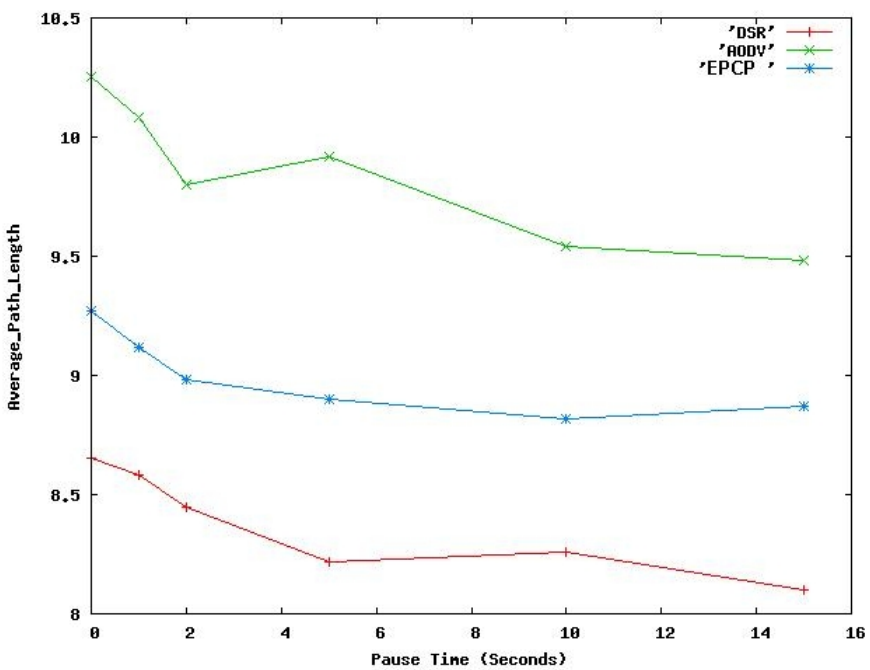


Fig. 3. Average Path Length v/s Pause Time

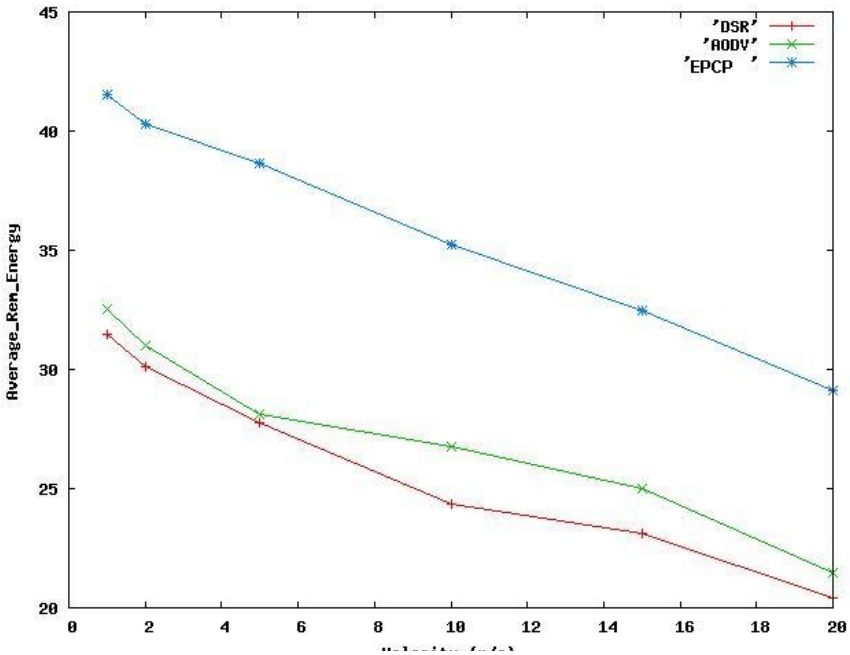


Fig. 4. Average Remaining Energy v/s Velocity

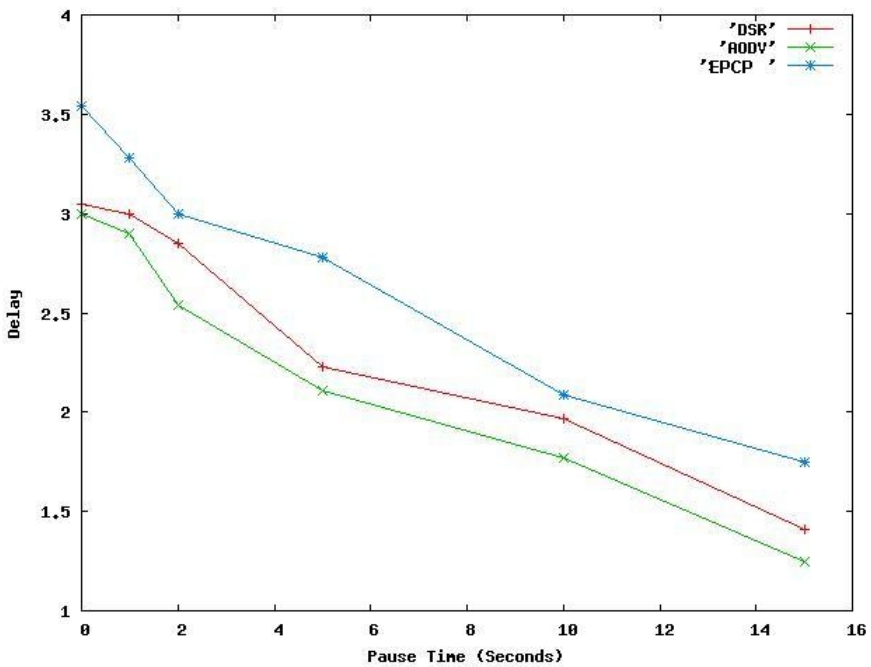


Fig. 5. Delay v/s Pause Time

TABLES

Table1. Simulation Parameter

Parameter	Value
Pause Time (sec)	0, 1, 2, 5, 10, 15
Velocity (m/sec)	1, 2, 5, 10, 15, 20
Number of Nodes	200
Bandwidth	2 Mbps
Packet Size	64 Bytes
Simulation time	1000 Sec
Initial Energy	100 J
TxPower (max)	1.5 W
RxPower (max)	1 W

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Comprehensive Study of FACTs Devices

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ABSTRACT

This paper is written with a view to get information regarding FACTs devices. FACTs devices are intended to solve the problems and issues occurring in power system operation. Because of modernization, the utilization of electrical power is increased. As a result, new problems and challenges have also cropped up in power system. This paper provides information regarding basics of FACTs devices, its types, benefits and applications. FACTs devices mainly are managed by impedance, phase angle and voltage level of the transmission line.

SUMMARY

To gain knowledge about types, benefits, applications and control of FACTs devices.

Keywords: FACTs ,STATCOM, SVC, SSSC, IPFC, TCSC, UPFC, GCSC

INTRODUCTION

A Flexible Alternating Current Transmission System (FACTs) is a system of static equipment used for the AC transmission of electrical energy. It is meant to enhance the controllability and increase power transfer capability of the network. It is generally power electronics based system. (*1,9*)



Fig.1Basic flow of electricity

Figure shows the flow of electricity. It reaches to the consumers after passing stages like generation, transmission and distribution. In modern era, the use of electrical power has increased manifold. To meet the demand, the generation of electrical power is also increased and interconnection of system is also working fully fledged. But it has raised many issues like

- Voltage stability limit
- Steady state power transfer limit
- Power system oscillation damping limit
- Short circuit current limit
- Transient stability limit
- Dynamic voltage limit
- Others.(*2*)

Every transmission may bear the issue/s listed above, constraining the entire system. Such problems can be addressed by FACTs devices.

FACTs stands for Flexible AC Transmission system. It has come in the recent years for better controllability in power systems by means of power electronic devices.

In most of applications the controllability is used to make it cost effective and regulate huge extension of power system for instance like advancement in substation and power lines. FACTs devices help to improve varying conditions and make the better use of existing infrastructure.

VARIABLES IN POWER SYSTEM:

Through following parameters, power system can be controlled directly (*2*).

- Impedance
- Phase angle
- Voltage

MATERIALS AND METHODS

1. CLASSIFICATION OF FACTS DEVICES:

There are mainly three types of FACTs devices as listed below.

1. Shunt connected controllers
2. Series connected controllers
3. Combined series and shunt connected controllers. (*1,7,10*)

1.1 SHUNT CONNECTED CONTROLLERS:

Shunt connected controllers covered in this paper are

1. STATCOM
2. SVC

1.1.1 STATCOM

The full form of STATCOM is Static Synchronous Compensators. To compensate static VAR, a static synchronous generator can be connected in parallel whose I_C and I_L can be operated without changing AC system voltage.

Among many controllers of FACTs, STATCOM is one of the important devices. It is founded on a voltage – sourced and current – sourced converter. (*1,5*)

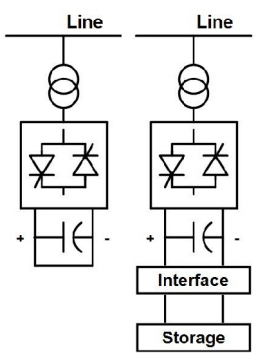


Fig.2 Line diagram of STATCOM

Figure 2 shows simple form of STATCOM. Voltage sourced converters are chosen often because of its cost effectiveness. AC output voltage is controlled by voltage sourced converter in a manner that required reactive current flow can be obtained. System harmonics can be nullified by voltage – sourced converter.

1.1.2 SVC

SVC stands for Static VAR Compensators. To interchange capacitive or inductive current, the output of static VAR absorber or generator is adjusted which is connected parallel to it. Through which particular variables of the electrical system can be managed.

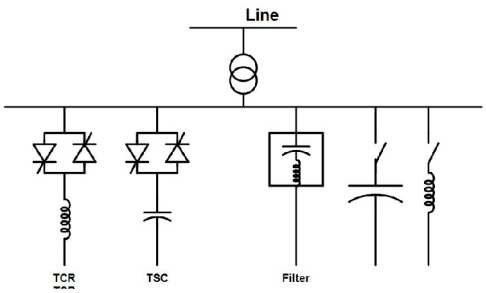


Fig.3 Line diagram of SVC

As shown in Figure 3 SVC includes TCR (Thyristor Controlled Reactor) or TSR (Thyristor Switched Reactor) and/or TSC (Thyristor Switched Capacitor) combination of all.

The base of SVC depends on thyristors without gate turn off capability. Different instrument is used for lagging and leading VARs. TCR or TSR for absorbing reactive power and TSC for supplying reactive power. SVC can be used as alternate to STATCOM because of it low cost. (*1*)

1.2 SERIES CONNECTED CONTROLLERS:

Series connected controllers covered in this paper are

1. SSSC
2. IPFC
3. TCSC

1.2.1 SSSC

SSSC abbreviation means Static Synchronous Series Compensator. As a series compensator, Static synchronous generator functions not using external energy. The output voltage of a static synchronous generator is at 90^{o} with line current, to regulate (lagging or leading) whole reactive voltage drop throughout line so it controls and transmits electric power.

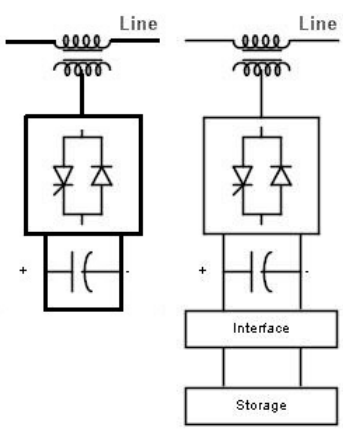


Fig.4 Line diagram of SSSC

Figure 4 shows simple diagram of SSSC. SVC is one type STATCOM not having AC output voltage in series with transmission line. It would be of current – sourced or voltage – sourced converter. The voltage which is given in the series is in little quantity compared to line voltage. (*1*)

1.2.2 IPFC

The full form of IPFC is inter-line power flow controller. This is newly added controller in FACTs devices which has no IEEE definition yet. But possible explanation can be, Static synchronous series compensators, in combination of two or more, which can be connected through a common dc link, to ease the bi-directional flow of real power between ac terminals of the SSSC. These are managed to allow independent reactive compensation for the adjustment of real power flow in each line and balance the required distribution of reactive power flow in lines. (*1*)

1.2.3 TCSC

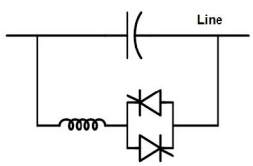


Fig.5 Line diagram of TCSC

Figure shows the line diagram of TCSC. TCSC stands for Thyristor controlled series capacitor. A capacitive reactance compensator is a combination of series capacitor bank and Thyristor controlled reactor which are connected in parallel, to provide smooth variable series capacitive reactance. Thyristor based TCSC is independent of gate turn-off capability. (*1*)

1.3 COMBINED SHUNT AND SERIES CONNECTED CONTROLLERS:

Combined shunt and series connected controllers covered in this paper are

1. UPFC
2. GCSC

1.3.1 UPFC

The full form of UPFC is Unified Power Flow Controller. STATCOM and SSSC are connected through common dc link (*6*) to have UPFC. It allows bi-directional flow of real power in SSSC and STATCOM. UPFC is made to control real time (*4*) dynamic compensation of ac transmission systems (*3*).

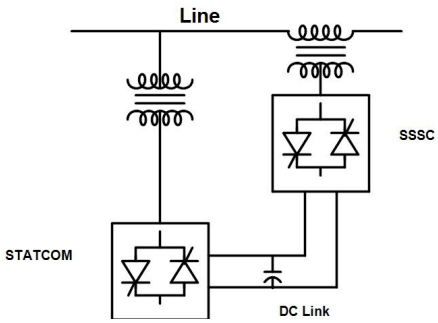


Fig.6 Line diagram of UPFC

Figure 6 shows the line diagram of UPFC. Power flow in the line is affected by variables like impedance, phase angle and voltage. These all variables are controlled by UPFC. This unique characteristic leads to keep its name as 'unified'.

1.3.2 GCSC

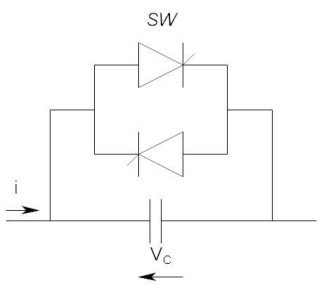


Fig.7 Line diagram of GCSC

Figure 7 shows the line diagram of GCSC. The abbreviation GCSC stands for Gate turn on thyristor Controlled Series Capacitor. A fixed capacitor and GTO Thyristor valve connected in parallel gives us GCSC which turns on and off in response intended.

To balance the ac output voltage across the capacitor is the main aim of GCSC and it can be done by controlling delay angle. (*1*)

2.OVERALL ADVANTAGES OF FACTS DEVICES:

- **Efficient dynamic and transient stability of the system**

In order to get high capacity of power transmission, FACTs devices are very useful. It reduces line tripping faults as huge transmission lines are interconnected to grids which are fulfilling the continuous changing demand and that is highly desired for faultless lines.

- **Better Accessibility and dependability of transmission system**

Many factors reduce accessibility and dependability of power supply which can be enhanced and corrected by FACTs devices.

- **Better Power quality for huge industries**

Desired frequency, constant power supply and constant voltage are essentials of good quality of electric power which is required by upcoming industries. If these above three factors of electric power are not controlled, industries have to sustain high economical loss. Here FACTs devices help to come the industries.

- **Better use of existing transmission system**

It is utmost important to transfer the power and regulate the load flow of the transmission system to fulfill the increased demand in the world. Number of load centers is installed to accomplish this control. To save the cost of more transmission line, FACTs devices are the key factor.

- **Environmental friendly**

The main benefit of the FACTs device is, it does not create any hazardous waste and so it is pollution free. By the use of FACTs devices, we can produce power more economically. As the devices use the available lines and we don't have to install new transmission lines.

3. RESTORATIVE ACTIONS OF VARIOUS FACTS DEVICES:

STATCOM & SVC	• Supply reactive power • Remove reactive power supply • Dampen oscillations • Post contingency voltage control
SSSC	• To correct voltage • Power factor correction • Power flow control • Reduces harmonic distortion
IPFC	• Increases transient stability • Reduces power oscillation damping
TCSC	• Reduce load • Limit line loading • Adjust series reactance/phase reactance • Limit short circuit current • Mitigate oscillations • Increase synchronizing torque • Dynamic load flow control • Reduce impact of contingency
UPFC	• Limit short circuit current • Increase synchronizing torque • Dampen oscillations • Dynamic voltage support and flow control • Reactive support • Network control action (*10*)
GCSC	• Reduces power oscillation damping • Better dynamic response • Controlled power flow (*8*)

4. PROPOSED WORK:

On the basis of FACTs devices, my proposed work would be to work on mitigation of SSR by sub-synchronous current injection with VSC HVDC. In proposed work, the work will be done to control the change in speed of the shaft of alternator when it is connected to load. Whenever the connected load changes, the variation in voltage will affect the alternator operation. This will be fulfilled by exciter connected to alternator. The frequency of the alternator connected with exciter and frequency of the alternator connected to turbine should not match when load is changed otherwise because of the change in speed of alternator shaft, as effect of that the shaft of an alternator will break which can affect the whole system. (*11*)

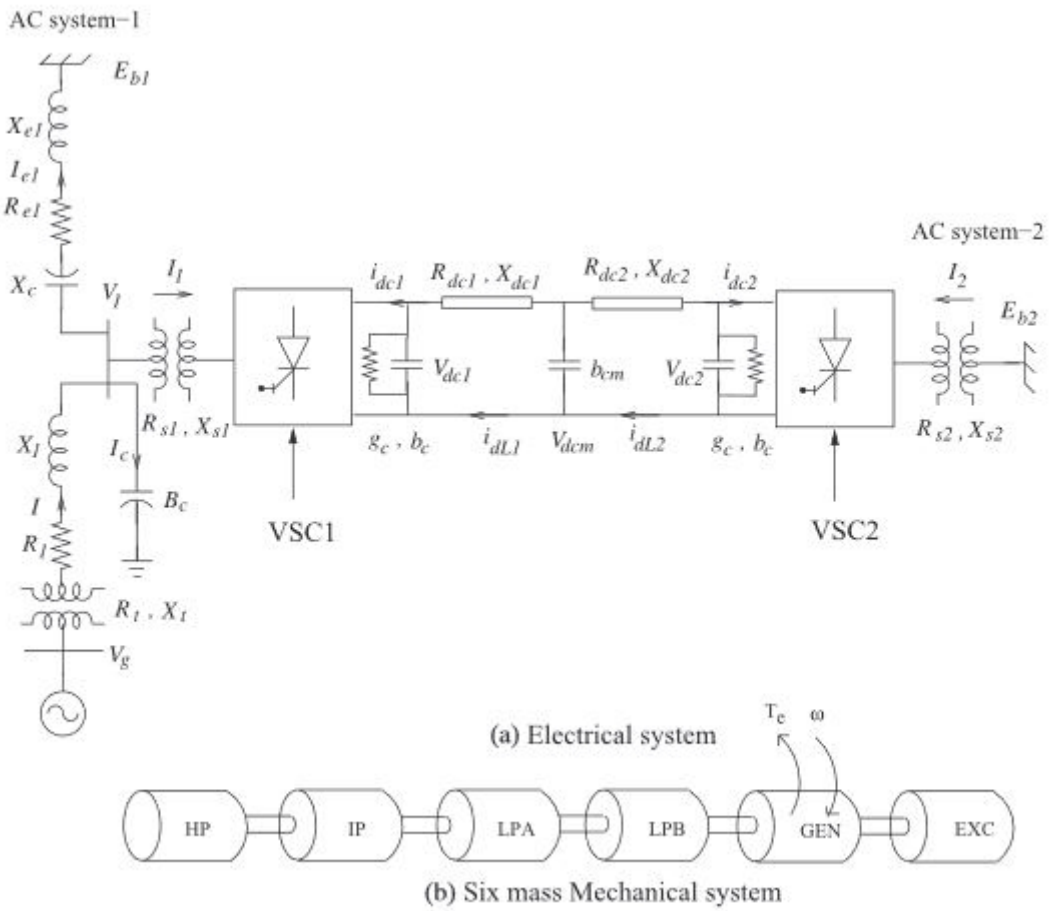


Fig.8 System diagram of VCS HVDC with AC line

CONCLUSION

The aim of this paper is to enumerate various FACTs devices and to understand technical and financial implications of such devices. FACTs devices are at present most essential for power sector. It gives us viable & sustainable solutions of various issues occurring in power system.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th& Jan. 6th, 2016)

(Proceedings available for download at rku.ac.in/icre)

EFFECT OF BLADE ANGLE AND NUMBER OF BLADES IN HEAD OF SINGLE STAGE OPEN WELL CENTRIFUGAL PUMP

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Abstract: Centrifugal pump has a wide variety of application in domestic, industrial and agricultural purpose. The performance of the centrifugal pump is very important parameter to study about. In this paper, the focus is kept on how the head is changed by changing blade inlet angle, Number of blade and blade exit angle. All the analysis has been carried out in ANSYS Workbench and the model is developed in Solid Works by cavity modeling. Present study is carried out for four and five number of vanes, for inlet blade angle 22, 24, 26 and 28, for exit blade angle it is 30, 35, 40 and 45. Through analysis it is found that maximum head when number of blades are 5, inlet angle is 26 degrees and outlet angle is 45 degree. Head generated is 19.64 meters at 2800 rpm.

Keywords: Centrifugal Pump, Number of Blades, Inlet Angle, Outlet Angle, Secondary Flow, Head.

Introduction:

Centrifugal pump has vast application in fields like irrigation, refineries as well as in power plants. The operating cost of centrifugal pump contributes 85% of its life cycle cost[1]. So it is quite essential that to optimize its performance operating cost is one of the major parameter.

Researchers have made efforts to improve its performance by way of increasing head [3][8] or discharge [4]or decrement in power consumption[15] so that overall efficiency of the pump is improved[16]. The flow inside the pump is very complex so many issues are there in which further investigation is possible. One of it is modification in impeller and casing [17][18]. The function of impeller is to impart energy to fluid by rotation. Impeller converts mechanical energy to kinetic energy of fluid [2,4]. It is quite essential that maximum mechanical energy should be converted into kinetic energy and this further converted into pressure energy which ultimately increases head of the pump [6]. Design and optimization of impeller shape and size is extremely important because by changing blade shape whirl component of velocity at inlet and outlet both will change which results in change in efficiency [9,19,22,24]. However most of the researcher focuses on blade exit angle which has a dominant effect on performance, blade inlet angle and number of blades will also have some effect in performance improvement [5,7,12,23].

In present paper different blade angles are developed in software and then do analysis in ANSYS workbench to find the head generated. This can be possible by doing an experiment but it is very expensive. Software analysis give result which has an error of 3 to 5% but it is acceptable.

Research Model:

Research model has been developed by cavity modeling in Solid Works and is analyzed in ANSYS CFX. The basic dimensions are taken from a working model and are implemented in a model developed in solid works.

Table1: Basic Parameters of single stage open well centrifugal pump Dimension

	DIMENSIONS
No. of impeller vanes	4
Diameter of impeller eye	55mm
Outer diameter of impeller	195mm
Width of impeller at intlet	6mm
Width of impeller at outlet	15mm
Blade thickness	5mm
Vane angle at inlet	10°
Vane angle at outlet	25°
Head	17.96 meter
Rated Discharge	5 kg/s
Rated Speed	2820rpm

Few assumptions were made during the simulation of Centrifugal Pump such as Flow is Steady state, Fluid is incompressible, Fluid properties are constant, No vapour is present in the water, i.e. single phase flow, There is no leakage in the whole pump, The surface of all the components is hydraulically smooth[10,11].

Table 2: Basic Parameters For Analysis

Parameters	Value
Number of Blades	4 and 5
Inlet Angle	22,24,26 and 28
Outlet Angle	30,35,40 and 45
Mass Flow Rate	5 Kg/sec.

Numerical Simulation and Performance Prediction:

For inner flow field ANSYS workbench was used. Most of the researchers have applied K-epsilon turbulence model. In current case Reynolds Average Navier Stokes Equations and Moving Reference Frame is applied instead of Static Reference Frame to get better results[13,14,20,21].

Table 3 Boundary Condition**:**

Boundary	Boundary 1
Type	Inlet
Location	Inlet

Mass Flow Rate	5 kg/sec.
Turbulence	Medium Intensity and Eddy Viscosity Ratio
Boundary	Boundary 2
Type	Wall
Mass And Momentum	No Slip Wall
Wall Roughness	Smooth Wall
Boundary	Boundary
Type	Outlet
Mass Flow Rate	5 kg/sec.

Modeling

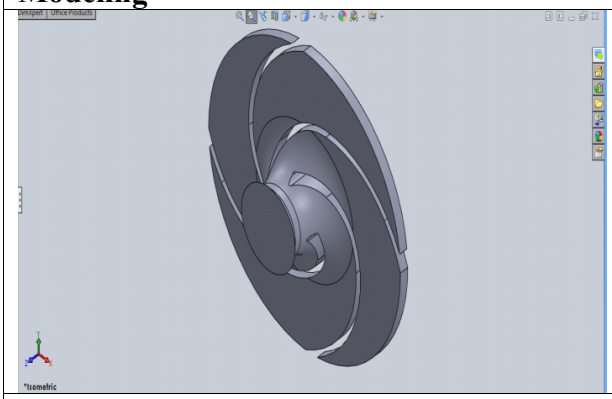


Fig 1 Impeller with four blades

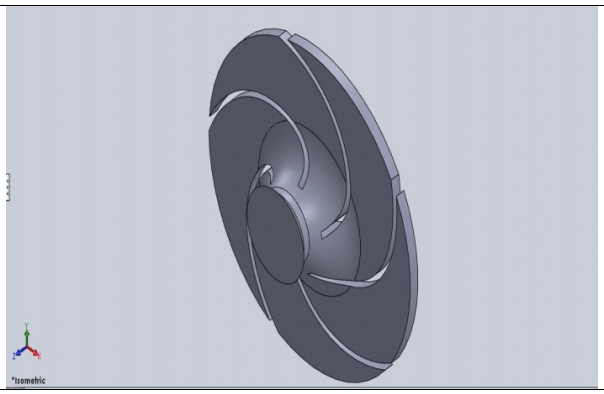


Fig 2 Impeller with five blades

Static pressure distribution

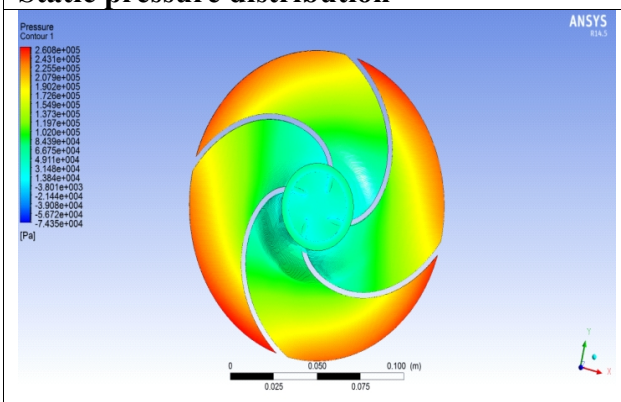


Fig 3 The static pressure distributions with four blades

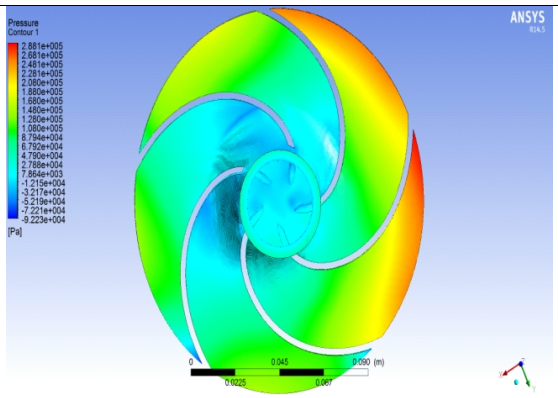


Fig 4 The static pressure distributions with five blades

From Figure it is evident that pressure increase from inlet to outlet and that is why most researchers think that effect of blade exit angle has more influence than blade inlet angle to increase pressure and ultimately head. However when the number of blade varies there is an increment in pressure rise.

When static pressure is divided by product of density and gravitational acceleration it shows net head.

Results and Discussions:

Experimental results of centrifugal pump shows a head of 17.96 m whereas by doing analysis shows it 19.04 m which is nearly 6% and this much error is because of assumptions made earlier. Once a model is

validated further analysis was carried out in ANSYS CFX workbench for different blade inlet and exit angle as well as for different number of blade. Result is as per table 4.

Fig 5 : Head for different outlet and inlet blade angle for four number of vanes.

Fig 6 : Head for different outlet and inlet blade angle for five number of vanes.

Result shows that with increase of blade numbers head also increases. Head is 19.64 m when number of blade is 5, Inlet blade angle and outlet blade angle is 24 and 35 degree respectively.

Conclusion:

In current paper model is developed in solid works and analysis was carried out in ANSYS CFX. Model is validated by experimental results with an error of 6%. Model was modified in solid works for four and five number of blades and different blade angles. Maximum head is produced when number of blade is 5 and Inlet blade angle and outlet blade angle is 24 and 35 degree respectively.

Further improvement is possible by considering secondary flow which exists between impeller blade because in current case it is considered to be negligible. In this research paper investigation of effect of impeller blade exit angle, inlet angle and number of blades on the head of the centrifugal pump has been done.

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An experimental investigation on natural frequency of un-damped free vibration on a spring mass system

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ABSTRACT

The natural frequency of 1-degree spring mass system can be calculated by using three well-known analytical methods (viz. Equilibrium method, Maximum energy method, and Rayleigh's method). The Equilibrium method is based the Newton's second law of motion and the D'Alembert's principle. The Maximum energy method and Rayleigh's method are based on the kinetic energy and potential energy of system. In this paper, the natural frequency of an un-damped spring mass system is calculated experimentally and compared it with that of the analytical value. The effect of mass and stiffness of system on the natural frequency is also discussed. The effect of experimental and exact (theoretical) natural frequencies is explained in detail.

SUMMARY

The article concerned with the comparison of experimental natural frequency and analytical natural frequency of a one degree spring mass system.

Keywords: *Stiffness, Time Period, Oscillation, universal vibration analyser.*

INTRODUCTION

All the objects have a tendency to vibrate at a particular frequency according to its loading conditions. The swinging of a pendulum is a very good example of simple vibration. Oscillatory motion of any system is known as vibration (*1, 3*). Uneven distribution of forces causes vibration. The study of vibrations is necessary to understand their effects on mechanical systems. Whenever, any system displaced from its mean position by means of external work it will always try to gain its mean equilibrium condition. This external work is stored into body in the form of strained energy. As the external forced is released, the body emits that strain energy in form of kinetic energy and potential energy and try to reach its mean position this phenomenon causes vibration. The conversion of physical problem into mathematical model and the method of obtaining equation of motion are explained in subsequent section (*9, 10*).Equilibrium method, Maximum energy method, and Rayleigh's method are also discussed in subsequent sections.

METHODS

The Equilibrium method

Equilibrium method is based on two methods. Namely, are (1) Newton's Second Low of Motion and (2) D'Alembert's principle. The equation of motion of a given system can be generated by using FBD.
Newton's Second Law of motion states that the accelerating force due to the mass should be equalled to the sum of external forces in the system (**Fig. 1.**).

$$m\ddot{x} = \sum E \qquad F \qquad \text{... (1)}$$

Where, m = Mass, and $\ddot{x}$ = Acceleration

When mass 'm' is attached to a free spring, its static deflection would be say 'Δst', due to the self-weight W=mg.

$$K = \frac{W}{\Delta s} = \frac{m}{\Delta s} \qquad \text{... (2)}$$

Where, W = self-weight, and Δst = static deflection

D'Alembert's principle(*4, 8*) converts a dynamic problem into a statically problem by adding the inertia force. As per D'Alembert's principle, a dynamic body can be converted into a static equilibrium condition

by means of considering an inertia force($F = m\ddot{x}$)passing through the centre of gravity of the body in the direction opposite to the acceleration and would have a magnitude equal to the product of the mass and the acceleration.

$$\sum I \quad F \quad + \sum E \quad F \quad = 0 \qquad \text{... (3)}$$

$$-m\ddot{x} + m \quad - K(x + \Delta s) = 0 \qquad \text{... (4)}$$

Where, x = Displacement

$$m\ddot{x} = m \quad - K \quad - K\Delta s \qquad \text{... (5)}$$

From equation (1)

$$m \quad = K\Delta s \qquad \text{... (6)}$$

$$m\ddot{x} = -K \qquad \text{... (7)}$$

$$\ddot{x} + \frac{K}{m}x = 0 \qquad \text{... (8)}$$

The Simple Harmonic Motion,

$$\ddot{x} + \omega^2 x = 0 \qquad \text{... (9)}$$

Now Comparing this with (8),

$$\omega^2 = \frac{K}{m} \qquad \text{... (10)}$$

Represent the natural frequency of the system,

$$\omega_n = \sqrt{\frac{K}{m}} \qquad \text{... (11)}$$

Where, $\omega_n = n \quad f \quad o \quad the s \quad m \quad s$

The time period,

$$T = \frac{2\pi}{\omega_n} = 2\pi\sqrt{\frac{m}{K}} \qquad \text{... (12)}$$

The frequency in hertz will be,

$$f_n = \frac{1}{T} = \frac{1}{2\pi}\sqrt{\frac{K}{m}} \qquad \text{... (13)}$$

Maximum energy method

The law of conservation of energy states that energy can neither be created nor destroyed, but it can only be converted from one form to another form. Using this method we can find the equation of motion (*4, 8*).

Let, U = Total Energy

PE = Potential Energy

KE = Kinetic Energy

$$U = P \quad + K \quad = C \qquad \text{... (14)}$$

As discussed earlier, for a simple spring mass system, the self-weight effect can be ignores as the static deflection accounts for it.

$$K \quad = \frac{1}{2} m\dot{x}^2 \qquad \text{... (15)}$$

Where, $\dot{x} = v$ o $the\ m$.

The potential energy is stored into the spring in form of strain energy. The strain energy is given by the area under the force against deflection graph of a spring as shown (**Fig. 2.**)

The equation of potential energy of system can be written as,

$$P \quad = \frac{1}{2} \times hi\, ht \times b$$

$$= \frac{1}{2} P$$

$$P = K$$

$$P \quad = \frac{1}{2} Kx^2 \qquad \text{... (16)}$$

From equation (14), (15) and (16),

$$U = K \quad + P$$

$$= \frac{1}{2} m\dot{x}^2 + \frac{1}{2} Kx^2 = c \qquad \text{... (17)}$$

Differentiating the Eq. (17) with respect to time, we get:

$$\frac{d}{d} = \frac{2}{2} m\dot{x}\ddot{x} + \frac{2}{2} K \ \dot{x} = 0 \qquad \text{... (18)}$$

$\dot{x} \neq 0\ a$ $the\ b$ ha $a\ d$ v ,

We get the equation of motion as

$$m\ddot{x} + K \quad = 0 \qquad \text{... (19)}$$

$$\ddot{x} + \frac{K}{m}x = 0$$

... (20)

Rayleigh's Method

The Rayleigh's method is based on the law of energy conservation. The method states that the maximum kinetic energy of the system is equal to the maximum potential energy (*7, 8*). The maximum kinetic energy occurs when the system has maximum velocity, and the maximum potential energy occurs when the system is at maximum displacement from the mean.

Let,

PE max = Maximum Potential Energy

KE max = Maximum Kinetic Energy

$$P_m = KE_m$$

... (21)

As discussed earlier, for a simple mass system, the self-weight effect can be ignored as the static deflection accounts for it. Considering SHM,

$$x = X \sin \omega$$

... (22)

When $\sin \omega = 0$ the displacement is maximum,

$$x_m = X$$ ... (23)

Differentiating,

$$\dot{x} = X \cos \omega$$

... (24)

When $\cos \omega = 1$ the velocity is maximum

$$\dot{x}_m = X$$

... (25)

From equation (16) and (23)

$$P_m = \frac{1}{2}Kx^2{}_m = \frac{1}{2}KX^2$$ ... (26)

Similarly from equations (15) and (25)

$$K_m = \frac{1}{2} m \dot{x}^2{}_m = \frac{1}{2} m X^2 \omega^2 \quad \text{... (27)}$$

From equations (21), (26) and (27)

$$\frac{1}{2} K X^2 = \frac{1}{2} m X^2 \omega^2 \quad \text{... (28)}$$

$$\omega_n = \omega = \sqrt{\frac{K}{m}} \quad \text{... (29)}$$

The SHM equation,

$$\ddot{x} + \omega^2 x = 0 \quad \text{... (30)}$$

Or

$$\ddot{x} + \frac{K}{m} x = 0 \quad \text{... (31)}$$

EXPERIMENTAL SETUP

First of all fix the spring to the stud and attach the weight holder to the bottom of spring and note the initial reading. Then, attach the different-different weight to the spring and note the deflection for particular weight and find out the spring stiffness 'K'. Repeats this experiment by using different springs and note the reading for free vibration and also not the time for the 10 oscillation (**Fig. 3.**). The analytical natural frequency and experimental natural frequency are calculated as below. The readings are shown in Observation Table (**Table 1.**).

- **Calculations:-**

1. m = 1 kg, δ = 0.014 m, t = 2.84 sec per 10 Oscillation, t = 0.284 sec / 1 Oscillation

$$W = m = 1 * 9.81 = 9.81\ N$$

$$K = \frac{W}{\delta} = \frac{9.81}{0.014} = 700.71\ N/m$$

$$f_{th} = \frac{1}{2\pi}\sqrt{\frac{K}{m}} = \frac{1}{2\pi}\sqrt{\frac{700.71}{1}} = 4.21\ H_z$$

$$f_e = \frac{1}{T_e} = \frac{1}{0.284} = 3.52\ H_z$$

$$D \qquad = 16.18\ \%$$

2. m = 1.5 kg, δ = 0.021 m, t = 3.52 sec per 10 Oscillation, t = 0.352 sec / 1 Oscillation

$$W = m = 1.5 * 9.81 = 14.71\ N$$

$$K = \frac{W}{\delta} = \frac{14.71}{0.021} = 700.71\ N/m$$

$$f_{th} = \frac{1}{2\pi}\sqrt{\frac{K}{m}} = \frac{1}{2\pi}\sqrt{\frac{700.71}{1.5}} = 3.44\ H_z$$

$$f_e = \frac{1}{T_e} = \frac{1}{0.352} = 2.84\ H_z$$

$$D \qquad = 17.44\ \%$$

3. m = 2 kg, δ = 0.028 m, t = 3.92 sec pre 10 Oscillation, t = 0.392 sec / 1 Oscillation

$$W = m = 2 * 9.81 = 19.62\ N$$

$$K = \frac{W}{\delta} = \frac{19.62}{0.028} = 700.71\ N/m$$

$$f_{th} = \frac{1}{2\pi}\sqrt{\frac{K}{m}} = \frac{1}{2\pi}\sqrt{\frac{700.71}{2}} = 2.92\ H_z$$

$$f_e = \frac{1}{T_e} = \frac{1}{0.392} = 2.55\ H_z$$

$$D \qquad = 14.43\ \%$$

RESULTS AND DISCUSSION

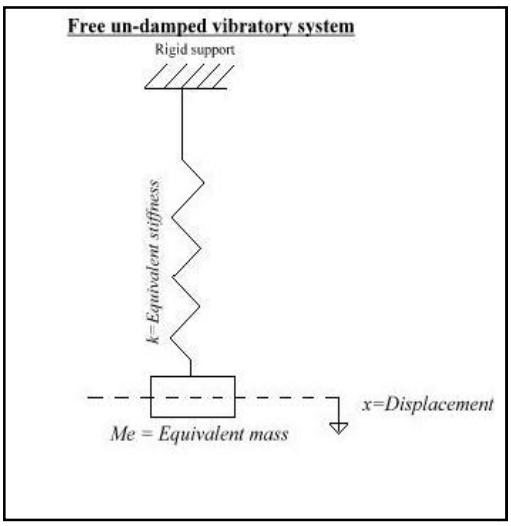
The natural frequency of spring mass system is analysed through experimental as well as analytical method. In the analytical method we are calculating stiffness 'K' of spring mass system by means of considering 10 independent runs as discussed earlier. Analytic natural frequency (f_{th}) and experimental natural frequency (f_e) can be calculated as per above calculation and they are compare with each other. The differences between both are: 17 %, 17.3 %, and 14.71 % for spring -1, for the given weight like, 1kg, 1.5kg, and 2kg respectively and the differences are 22.46 %, 22.38 %, and 7.21 % for a spring – 2

(**Table 2.**). These differences are because of damping resistance offered by air, and other mechanical and measurement error.

CONCLUSION

In this paper, the experimental investigation is discussed in order to calculate the natural frequency and it is compared with experimental data. The accuracy of the experimental natural frequency is investigated using 10 independent runs of this study with different-different springs and weights in air medium(Not space). A case study of experimental setup of a spring mass system shows that the importance of stiffness, the time for considering number of oscillation, and weight. The result of the case study revealed that the use of spring mass system without proper investigation on specific detail could result in misleading the theoretical natural frequency. Therefore, experiment setup should be free from any error and carried out appropriate natural frequency.

FIGURES



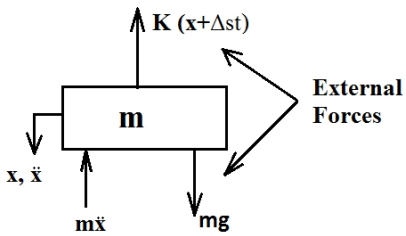


Fig.1. FBD of spring mass system

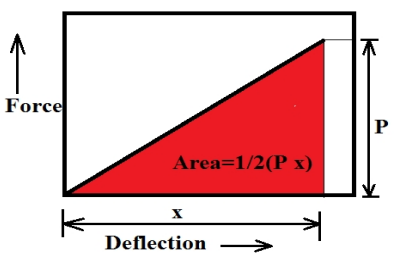


Fig. 2. Force versus Deflection of Linear Spring

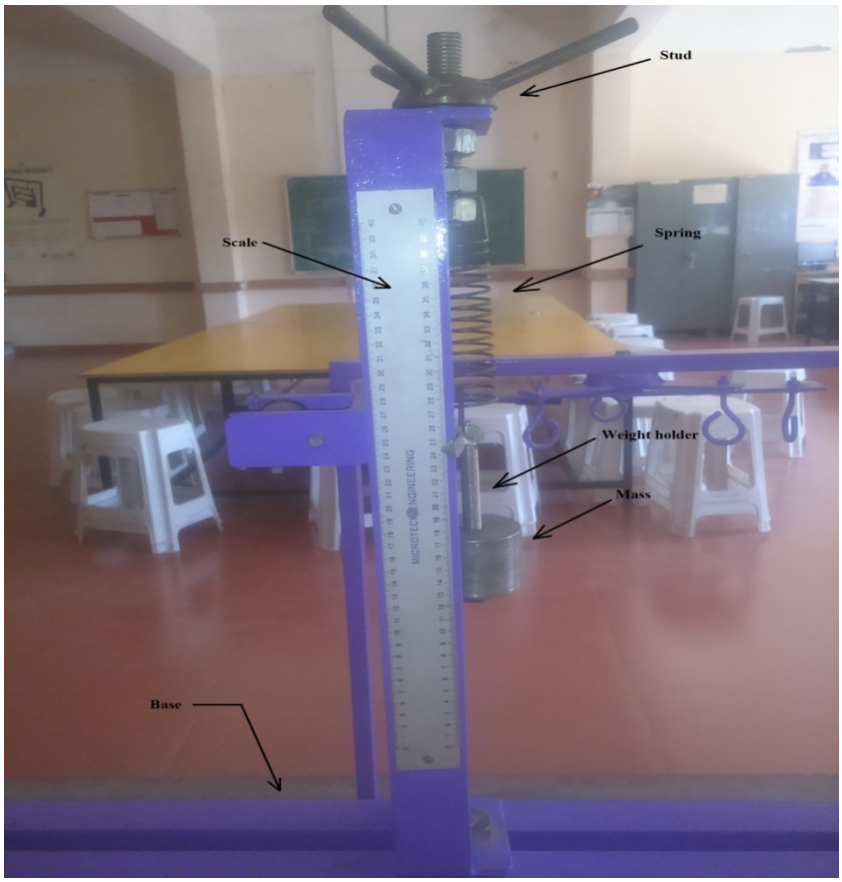




Fig.3. Experimental Setup

TABLES

Table: 1 Observation Table

Sr. No.	Spring	Attached mass m (kg)	Deflection (δ) (mm)	Time for 10 Oscillations, t (sec)
1	Spring - 1	1	25.2 - 23.8 = 1.4	1. 2.75
				2. 2.80
				3. 2.97
				Average Time: 2.84
		1.5	25.2 - 23.1 = 2.1	1. 3.48
				2. 3.56
				3. 3.52
				Average Time: 3.52
		2	25.2 - 22.4 = 2.8	1. 3.91
				2. 4.02
				3. 3.85
				Average Time: 3.92
2	Spring - 2	1	28.2 - 27.2 = 1.1	1. 2.56
				2. 2.60
				3. 2.62
				Average Time: 2.59
		1.5	28.2 - 26.6 = 1.6	1. 2.98
				2. 3.08
				3. 3.02
				Average Time: 3.02
		2	28.2 - 26.1 = 2.1	1. 3.13
				2. 3.10
				3. 3.17
				Average Time: 3.133

Table: 2 Result table

Sr. No.	Spring	Attached mass (m) (kg)	Natural Frequency (f_e)	Natural Frequency (f_{t_i})	Difference (%)
1	Spring - 1	1	3.52	4.21	17
		1.5	2.84	3.44	17.73
		2	2.55	2.99	14.71
2	Spring - 2	1	3.86	4.81	22.46
		1.5	3.05	3.94	22.38
		2	3.19	3.44	7.21

ACKNOWLEDGEMENT

We are thankful to the Department of Mechanical Engineering of RK-University, tramba- Rajkot (Gujarat), India and colleagues for providing very good facilities as well as very good support for the successful completion of this work.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th& Jan. 6th, 2016)

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Impact of Low Voltage and Scenarios of Low Voltage Ride-Through Capability Improvement in Different Types of Wind Farms

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ABSTRACT

With increased penetration of wind power and additional renewable sources, supplementary grid code requirements are announced by power system operators so as to maintain stability of emerging power system networks. Low voltage ride-through capability is the most challenging requirement that demands generators to stay connected during system disturbances in order to support the power system stability. This paper is about the impact of low voltage and the scenarios of low voltage ride-through capability improvement in different types of wind energy conversion systems with the analysis and effectiveness of alternative ways.

SUMMARY

Low voltage ride through capability of wind farm is an important concern of grid operators, so impact of low voltage and scenarios of low voltage ride through capability improvement in different types of wind energy conversion systemsare investigated.

Keywords: Low Voltage Ride-Through (LVRT), Squirrel Cage Induction Generator (SCIG), Permanent Magnet Synchronous Generator (PMSG), Doubly-Fed Induction Generator (DFIG), Wind Energy Conversion System (WECS)

INTRODUCTION

Performance characteristics of electrical generators significantly affect the operation of power system. In an ordinary power system, we have large-capacity synchronous generators of high moment of inertia. This aids the power system operation and control at the time of normal as well as abnormal condition. Now a day, penetration of wind energy generation is increasing due to convinced well known reasons. Wind energy generation undergone a remarkable development and now it is the world's best developing renewable energy generation (*1*). This gives rise to installation of different types of electric generators, whose operating characteristics are different from those of synchronous generators (*2*). As large numbers of wind farms are being connected to the power system, it leads to significant consequence on stability and power quality of the power system (*3-4*). Therefore, recently many power system operators have defined their new set of grid codes, i.e. connection necessities of their power grid; which involve the most challenging Low Voltage Ride-Through (LVRT) requirement for wind farms during the system disturbances (*5*). Since the German transmission operator, E-ON Netz has commenced a LVRT grid code; it is broadly acknowledged as a template for related needs (*6*). According to the E-ON Netz standards: "*The machine should remain connected to the grid, if the terminal voltage is higher than 0.15 per-units for approximately 0.6 seconds*" (*7*). Based on the dynamic characteristics of the concerned power system, LVRT requirements may differ (*8*). Hence, different countries have their own wind farm grid codes, which are more or less similar to each other. According to the US grid codes, set by Federal Energy Regulatory Commission (FERC): "*If the voltage does not fall below the minimum voltage indicated by the solid line in Figure 1 and returns to 90 percent of the nominal voltage within 3 seconds after the beginning of the voltage drop, the plant must stay online*" (*9*).

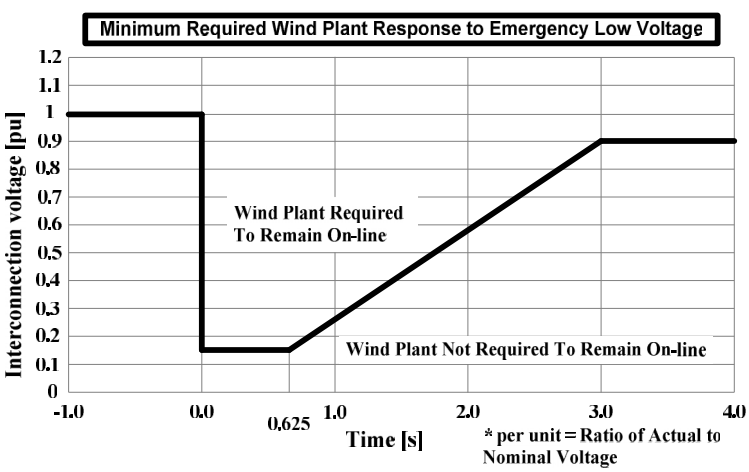


Fig. 1. Low voltage ride-through standard (*9*).

In continual endeavour to improve efficiency, increase reliability and reduce cost; mixtures of Wind Energy Conversion Systems (WECSs) have been developed. Broadly classifying, there are two types of WECSs, fixed speed WECSs and variable speed WECSs. The fixed speed WECSs employ Squirrel-Cage Induction Generator (SCIG) or Wound Rotor Induction Generator (WRIG) connected directly to the grid. Whereas, the variable speed WECSs employ Permanent Magnet Synchronous Generator (PMSG) or SCIG or Wound Rotor Synchronous Generator (WRSG) with full capacity power converters.

Furthermore, in variable speed WECSs; Doubly-Fed Induction Generator (DFIG) with reduced-capacity power converters is also being used. These variable speed WECSs have power converters which serves to interface the generator with the power grid; hence LVRT capability is far better compared to that of fixed speed WECSs.
Three different categories of WECSs are considered in following sections I, II and III to investigate the impact of low voltage and scenarios of LVRT capability improvement.

I. SCIG BASED FIXED SPED WECSs

SCIG based fixed speed WECS is extensively installed in wind farms due to some of its benefits like operational easiness, robust construction, low specific mass and cost efficiency (*10-11*). The stator side of SCIG is directly connected to the electrical power grid through a soft starter and a transformer as shown in Figure 2.

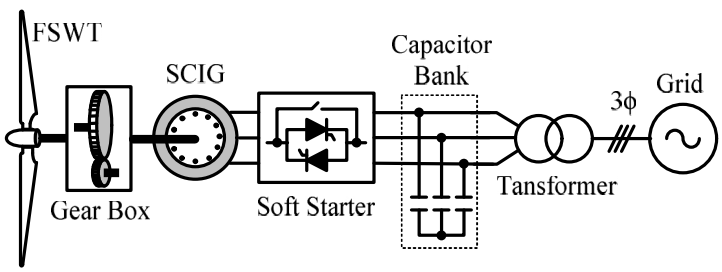


Fig. 2. Configuration of a typical SCIG based fixed speed WECS.

A. Impact of Low Voltage on SCIG based Fixed Speed WECSs

When a grid fault occurs, it drops the grid voltage; which immediately decreases the stator terminal voltage of the generator. Now, the electromagnetic torque of the generator is directly proportional to square of the stator terminal voltage. Therefore, the electromagnetic torque of the generator also drops immediately. But the mechanical torque provided by the wind turbine remains constant at that time. As a result, the rotor speed acceleration takes place due to this unbalance of the electromagnetic and the mechanical torques. Now, this increase in rotor speed ultimately leads to the more and more reactive power consumption by the generator. If adequate reactive power is not made available to electrical generator, it will not be in a position to operate normally after the grid disturbance. Finally, generator needs to trip off in order to protect it (*12*). But for the large scale wind power generation, the protective disconnection may result in the unacceptable transient stability issue. Hence there is a very strong need to improve LVTR capability in this type of wind farms.

B. Scenarios of LVRT Capability Improvement in Fixed Speed WECSs

1) *Pitch and active stall control:*These are the only inbuilt controls available in SCIG based fixed speed WECS. As mentioned in (*13*); pitch actuators can be operated to fully pitch the blades in very short time, but it requires huge dynamic forces. Therefore, pitch control is not reliable approach to improve LVRT capability. Another way to control the speed is to apply an active stall control. But on detection of the fault, the pitch is controlled near the stall position so as to regulate rotor speed. Hence an active stall control would no more be helpful to offer LVRT capabilities.

2) *Flexible AC Transmission System (FACTS) devices and energy storage systems:* Various FACTS devices like STATic synchronous COMpensator (STATCOM), Static Var Compensator (SVC), Solid State Transfer Switch (SSTS), Dynamic Voltage Restorer (DVR), Unified Power Flow Controller

(UPFC) and Superconducting Magnetic Energy Storage (SMES) have been proposed by different researchers, which became popular due to their advantages such as flexible power flow control, secure loading and damping oscillations of power system (*14-15*).

For reactive power compensation, SVC is proposed in (*16*). But in (*17-19*) it has been proved that STATCOM has rather better performance compared to SVC for reactive power compensation. However, STATCOM costs more than SVC, so STATCOM gets justified for large wind farms from the point of view of the cost. The SVC is cheaper for small to medium scale wind farms, but its reactive power capabilities decline with the drop in terminal voltage (*20*). In (*21-22*) concurrent control of pitch angle controller and STATCOM is proposed for enhancing the LVRT capability of the fixed speed WECS. In (*23*) a STATCOM is used under unbalanced grid voltage condition to reduce the torque ripple during the grid fault in addition to provide LVRT capability. As shown inFigure 3, STATCOM with Battery Energy Storage System (BESS) is proposed in (*24*), whichsupports active power in addition to the reactive power.

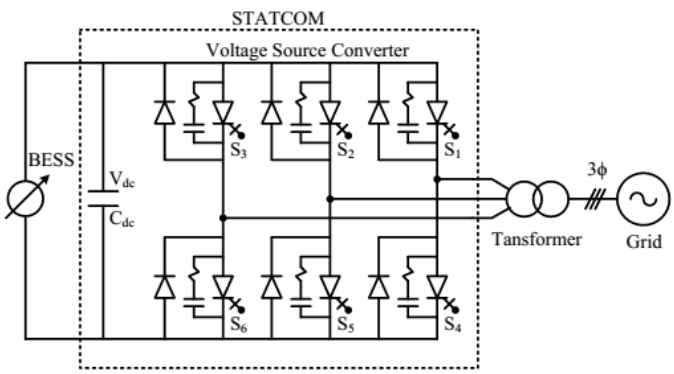


Fig. 3. Schematic diagram of STATCOM with BESS (*24*).

A STATCOM have a two-level voltage source converter, which controls only the reactive power output. So as to control active power in addition to reactive power, BESS is integrated with the STATCOM. But BESS have low response speed and short service life, which does not suit for wind power application (*24*). The controller of STATCOM with Energy Storage System (ESS) provides necessary commands to get desired system response in order to improving LVRT capability (*25*). In (*26*) it is reported that decentralized STATCOM with ESS control is very effective for improving the LVRT capability of fixed speed WECS. In (*27-29*) STATCOM, Energy Capacitor System (ECS) and SMES with power electronic devices have also been suggested in order to improve the LVRT capability of fixed speed WECSs.

In (*30*) Distribution-STATCOM (DSTATCOM) consists of series connected Insulate Gate Bipolar Transistors (IGBTs), which operates with Pulse Width Modulation (PWM) techniques for stabilization of fixed speed WECS. In (*31*) the combination of SVC and D-STATCOM is considered for the different fault cases, which revealed better performance compared to a single SVC placed in the same system. In (*6*) a novel DVR device is proposed to offer an effective LVRT capability. In (*32*) the analytical and simulation studies of the bridge type fault current limiter with a new control scheme is proposed for improving LVRT capability and it is also compared with the impact of an application of a Series Dynamic Braking Resistor (SDBR). In (*33*) the Unified Power Quality Conditioner (UPQC) is examined to improve LVTR capability of a fixed speed WECS. As a final point, all these FACTS devices and energy storage systems can more or less improve the LVTR capability of fixed speed WECSs but it leads to increased system investment cost.

3) *Combined installation of a variable speed and fixed speed WECS:*Recent scenario for enhancing LVTR capability of fixed speed WECS is the combined installation of variable speed WECS and fixed speed WECS in a wind farm located at a particular place. Variable speed WECSs are equipped with either fully or partially rated back to back power electronic converters, whose control scheme is designed to provide necessary LVRT capability to fixed speed WECS in addition to that of its own. This configuration seems to be the cost-effective solution to enhance LVRT capability of the existing fixed speed WECS by taking advantage of the flexibility in control scheme of variable speed WECS. Figure 4 shows such a typical configuration.

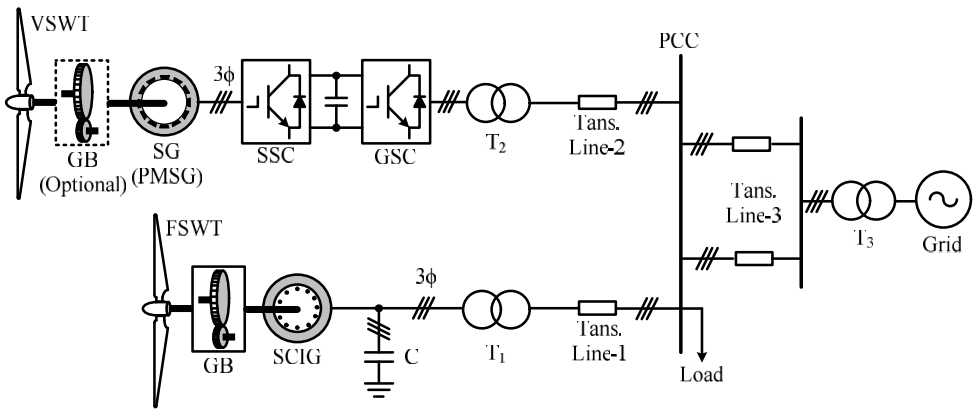


Fig.4. Combined installations of a typical VSWT-PMSG and FSWT-SCIG in a wind farm.

In (*5, 11, 34*) novel control strategy is proposed for the converter of PMSG based variable speed WECS located closely to the induction generator based fixed speed WECS in order to improve LVRT capability of induction generator based fixed speed WECS. In (*35-36*) the direct drive WECS with full capacity power converter is proposed whose control strategy enables it to inject necessary reactive power to stabilize the nearby fixed speed WECS during grid faults.

II. DFIG BASED VARIABLE SPEED WECSs

The generator of this WECS is known as doubly-fed induction generator, because its rotor and stator together feeds energy to the power grid. Figure 5 shows the system configuration of the DFIG based variable speed WECS.

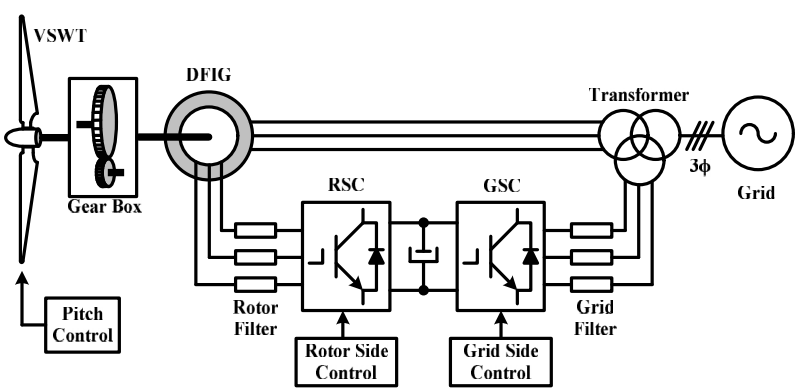


Fig. 5. System configuration of the DFIG based variable speed WECS.

Here, stator is directly connected with grid through a transformer and rotor is connected through the reduced capacity power converter. These power converters have to process only 30% of the rated power, which leads to the cost, weight and size reduction of the converter. In this converter, the torque or active power is controlled by the rotor-side converter (RSC), whereas the DC-link voltage and grid-side reactive power is controlled by the grid-side converter (GSC) (*37*).

A. Impact of Low Voltage on DFIG based Variable Speed WECS

As the stator of DFIG is directly connected to the grid, DFIG based WECSs are sensitive to the voltage dips (*38*). When grid fault occurs, voltage at PCC drops suddenly; which produces transients in stator current. As rotor and stator are magnetically coupled, this transients in stator current leads to large currents in the rotor converter (*39*). When this rotor side current or voltage crosses the power converter limit, it requires operating protection system. With the converter operating within its limits, the stator current may be highly unbalanced even with a small stator voltage imbalance. This leads to the torque pulsations; which may damage the gearbox, rotor shaft and blade assembly or at low levels, it produces acoustic noise (*40*).

B. Scenarios of LVRT Capability Improvement in DFIG based Variable Speed WECSs

1) Blade Pitch Angle (BPA) control: At the time of a grid voltage dip the wind power can be reduced by changing the BPA. This decreases the input mechanical power to generator, as a result it reduces the reactive power requirement due to decreased rotor speed.

2) Crowbar methods: As shown in Figure 6, the crowbar is a device installed at the rotor terminals to prevent the overvoltage induced at the time of voltage dips so as to protect the RSC (*41*).

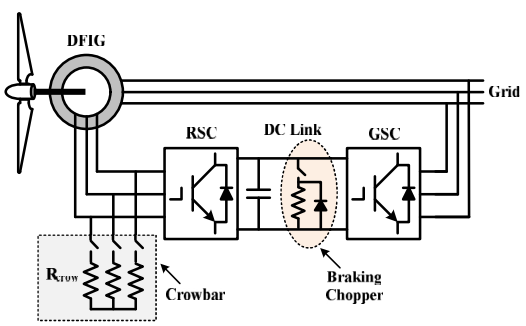


Fig.6.DFIG based system equipped with crowbar and braking chopper.

When crowbar is activated, it diverts the rotor current to the low impedance (R_{crow}) path and the rotor converter is deactivated. But it leads to circulate huge short circuit current through stator and rotor of generator. As shown in Figure 7 this short-circuit current can be avoided by using the active crowbar. It also allows deactivation to resumes normal operation.

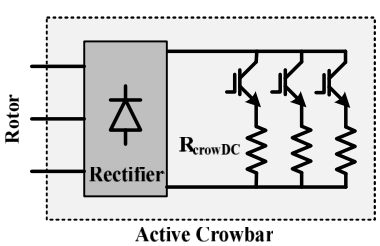


Fig. 7. Active crowbar.

The additional advantage of an active crowbar is that the crowbar resistance (R_{crowDC}) can be changed by connecting and disconnecting the resistors through PWM (*42*). In an active crowbar the selection of connection time is decisive; very long time may not fulfil the requirements of reactive current injection, whereas a very short time may not be enough to demagnetize the generator.

3) *Braking chopper:* As shown in Figure 6, braking chopper is a device used to prevent increase of the DC-link voltage beyond maximum specified level (*41*). It consists of a freewheeling diode to prevent overvoltages in the switch and a resistor which can be switched in and out. When the DC link voltage increase beyond maximum specified level (1.2 p.u.), the resistor is switched-in to dissipate the excess energy and it will be switched-out when DC link voltage drops below minimum specified level (1.1 p.u.).

4) *Energy Storage System (ESS):* ESS based methods can control the generator during voltage dip. For that the RSC should be designed in view of that so as to permit the flow of the fault currents through rotor circuit of DFIG. But it increase the cost and complexity, as it needs an additional energy storage devices.

5) *FACTS devices:* FACTS devices like DVR, SVC, STATCOM based methods provides general solution intended for the whole wind farm. The DVR provides series compensation by injecting capacitive voltage so as to increase the reactive power transfer from the grid. Whereas, SVC and STATCOM provides shunt compensation by injecting large inductive current. STATCOM consists of voltage source converter, which is connected in parallel with the wind farm.

6) *Optimised controller design:* Optimisation techniques such as Particle Swarm Optimization (PSO), Genetic Algorithm (GA), Fuzzy Logic, Bacterial Foraging (BF), etc. are being used to control the dynamic performance of the controller by adjusting the controller gain parameters to its optimal value in order to enhance LVRT capability of DFIG based variable speed WECS.

III. PMSG BASED VARIABLE SPEED WECSs

PMSG based variable speed WECS has become the leading type amongst various WECSs. In comparison with other types, it offers better performance in many aspects (*43*). The typical VSWT-PMSG system is shown in Figure 8. The GSC controls the reactive power supplied to the grid and DC-link voltage whereas SSC controls active power output through Maximum Power Point Tracking (MPPT).

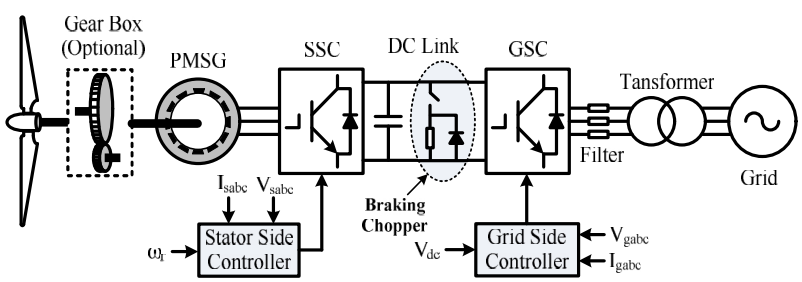


Fig.8. System configuration of the PMSG based variable speed WECS.

A. Impact of Low Voltage on PMSG based Variable Speed WECSs

Here, the full capacity back to back converter isolate generator from grid; therefore low voltage conditions at grid side do not have direct effect on the generator. However, there are certain consequences at the time of grid disturbances. When a grid voltage dip occurs; the power injected into the grid by GSC

reduces immediately, while the power output from the SSC does not change. This unbalance of power significantly increases the DC link voltage (V_{dc}), which may damage the power converter.

B. Scenarios of LVRT Capability Improvement in PMSG based Variable Speed WECSs

To improve LVTR capability of PMSG based variable speed WECS, researchers have proposed various control schemes for SSC and GSC. Some of them are highly promising for enhancing LVRT capability. Apart from these converter control schemes, following scenarios are there to improve LVTR capability of PMSG based variable speed WECSs.

1) *Blade Pitch Angle (BPA) control:* As already discussed in other types of WECS, BPA control have significant response limitations (*44*). Therefore it helps to enhance LVRT capability up to limited extent during grid voltage dips. In (*2*), pitch control is used to operate wind turbine at rated output power and to control the turbine speed so as to avoid over speed at high wind velocity.

2) *Braking chopper:* In (*34, 45*), the braking chopper is used for DC-link protection as shown in Figure 8. It protects the DC-link circuit by limiting increase in voltage across DC-link capacitor. The chopper circuit gets triggered while the DC-link voltage goes outside the control limit and consumes the excess energy in resistor (*10-11*).

3) *Capacitor sizing:* It is an arrangement to deal with surplus energy in DC-link at the time of voltage dip. As discussed in (*44*), the necessity of capacitor size depends on the duration and the severity level of the grid-voltage dip. With increase in voltage dip duration or severity level the required capacitor size will also increase. Therefore, in case of fully rated converters it increases the cost.

4) *Energy Storage System (ESS):* As shown in Figure 9, it is connected across DC link capacitor to reduce power unbalance at the time of grid voltage dip. In (*46*), ESS is suggested as one of the solution for improving LVRT capability of PMSG based variable speed WECS. Various batteries can be used as an ESS, which smooths the power injected to the grid by absorbing excess energy from the DC-link

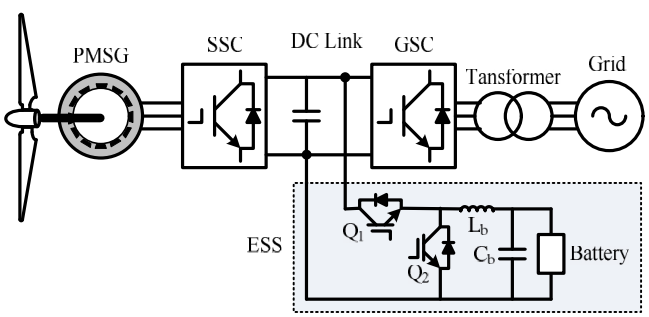


Fig.9.PMSG based variable speed WECS with ESS.

5) *FACTS devices:* These devices are connected at Point of Common Connection (PCC) to improve LVRT capability of PMSG based variable speed WECSs. FACTS devices like SVC, STATCOM are shunt converters whereas, DVR, SSSC, MERS are series converters. As shown in Figure 10, UPFC is a combination of STATCOM and SSSC coupled by a DC-link.

   The series converter (SSSC) injects voltage, which has two components; the q-axis component controls the variation in phase angle and the d-axis component controls voltage magnitude. The shunt converter (STATCOM) works as a reactive source by supplying necessary inductive or capacitive reactive power (*47*). UPFC permits the flow of active power in either direction between terminals of

series and shunt converters. This can effectively improve LVRT capability of WECS, but it increases the system cost.

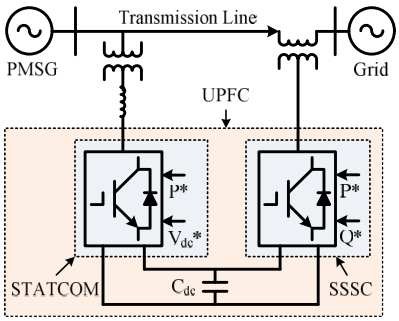


Fig.10. Schematic diagram of a UPFC.

CONCLUSION

LVRT capability is the most important and challenging requirement as per the new grid codes for grid integrated wind farms. This paper investigates the behaviour of some of the popular WECSs during grid voltage dip and scenarios of LVRT capability improvement are studied with their effectiveness, advantages and disadvantages. Requirement of LVRT capability improvement is of different level in various types of WECSs. SCIG based fixed speed WECSs are directly connected to the grid, so LVRT capability is very poor. DFIG based variable speed WECSs are employed with reduced-capacity power converters in rotor circuit with stator directly connected, so LVRT capability is far better compared to SCIG based fixed speed WECSs. And PMSG based variable speed WECSs are employed with full-capacity power converters, which fully isolates it from the grid, so LVRT capability is even better than that of DFIG based variable speed WECSs. Review on scenarios of LVRT capability improvement is carried out for different types of WECSs, wherein some of the solutions like pitch control of wind turbine, installation of FACTS devices and use of energy storage systems are applicable to all types of WECSs. Some other solutions like crowbar methods, braking chopper and optimized controller design are converter specific. Combined installation of variable speed WECS and fixed speed WECS in a wind farm is the cost effective and unique solution to improve LVRT capability of SCIG based fixed speed WECSs.

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Measuring the Degree of Leanness in Automobile Industry within Gujarat State: A Pilot Study

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ABSTRACT

Lean manufacturing system has the strong potential of developing and manufacturing products with effective value addition with minimal waste. Lean also offers a great throughput to automotive manufacturers to build their operating system. The Lean tools have established its excellent presence not only in Japan, but in many developed and emerging countries. The present pilot study investigates the underlying issue by exploring the scenery of implementation of Lean in Automobile Industry within Gujarat. Hence, by this research author has given his efforts to identify the types of barriers, critical success factors and possible performance improvement indicators for successful Lean implementation practices in Automobile industry. The research uses primary data; which is collected using a questionnaire developed for the study. Statistical Package for the Social Sciences (SPSS) software has been used for this pilot survey analysis.

SUMMARY

A pilot survey on widespread adoption of Lean Manufacturing Techniques and its outcome in Automobile Industry within Gujarat State.

Keywords: Lean Techniques, Barriers, Critical Success Factors, Performance Indicators, Survey

INTRODUCTION

Lean Manufacturing is a systemize methods which identifies and removes all types of non-value added activities; from the production, operations and service industry. This fundamental goal for manufacturing concepts given by Toyota Production System (TPS), its aim is to maintain the best trade-off between generated mudas and workflow (Vinodh *et al*., 2010). Recent trend turnaround the time matters much. Lean manufacturing helps to reduce production lead times significantly (Rahman *et al*., 2010). Lean principles are presently the state-of-the-art practice to survive in the global cut-throat competitive market scenario (Matt, 2008). Holweg (2007) has discussed the lean manufacturing genealogy. Lean thinking philosophy for improvements are emphasis on value creations and the removal of generating waste are the potential weapon (Muhammad S. Khan et al., 2011). Womack and Jones (1991) started the work and coin the term "Lean" in their publications which means "The machine that changed the world". Nicholas (1998) has discussed that wastes found everywhere at any time in many forms, Waste does not generate values consumes up resources. Lean Manufacturing System if studies in Indian context may provide a good opportunity for Indian manufacturers to fight against the competitive pressure. Generally, manufacturers across the world are keen to implement lean manufacturing practices (Matt, 2008; Anand and Kodali, 2008; Chen *et al.,* 2010) for the betterments of product delivery to the end customers (Brown *et al*., 2006). Lean system has been globally used for value creations and waste removals. Saurin *et al.* (2010) developed the framework oflean manufacturing practices formanufacturing cells used in automobile case. Achanga *et al.* (2006) suggested many useful critical success factors for implementing the concept of lean productions within SMEs. They also provided heuristic rules for lean implementationto help in decision-making.

This study can simply focus on measuring the degree of leanness in automobile industry. It will clearly indicate that lean strategies aimed to carry out production flow smoothly by the removal of wastes and by increase the value added. Lean manufacturing purely given values to the customers by the elimination non value added activities, which are considered as a waste. Any process which comprises resources, cost additions or without time creating values considered as the target for removal.

PILOT STUDY

This study explores the degree of leanness in automotive industries within Gujarat state. A survey questionnaire was used to measure the status of lean manufacturing practices. The objectives of the study has discussed below:

1. To identify the current status for implementation of Lean Manufacturing Techniques (LMT)
2. To identify the barrier affecting for implementation of Lean practices successfully
3. To identify the critical factors for successful implementation of Lean
4. To identify the performance improvement indicators lean techniques implementation

The survey was performed on 17 automobile industry. To carry out survey respondents were chosen from various region of Gujarat state, who are involved in the automobile parts manufacturer. The pilot analysis measures the status of lean within Gujarat's automobile industry. Hence, the factors which play a major role for delay the lean implementation process are also discussed. This study will useful for the manufacturer to identify the major issues for effective implementation of lean production practices. For pilot data samples are collected from automobile industry in different areas of Gujarat state.

DATA COLLECTION

A formal set of pilot questionnaire is prepared and data collected with details information of various respondents. The main purpose is to be collected primary data in sufficient quantity in standardize manner therefore consistent data achieved for analysis. A set of questionnaire is designed for educational level as a consideration of the respondent's experience. The user friendly language used for designing of the questions so respondentswere familiar. The most widely used Likert scale considered to obtain the response of the respondents for each of the statements. It was requested to respondents to select the any one of five response categories such as Not Important, Least Important, Can't Say, Very Important and Extremely Important as of their degree of agreement. The Likert scales are design such way so that it is easy administer and easy to understand by the respondent. Also,it is easy and suitable for personnel, mail and electronic interviews. The survey questionnaires are pre-tested by the involvement of the respondents in the proposed sample structure. The main intention of this research is to test the degree of understanding and difficulty faces by the respondents to understand the meaning of the questions, if the respondents understand the questions as per intention is to ascertain relevance of the questions. The purpose of this pretesting survey questionnaire is to get the realistic information for improving the questionnaires formats, sequences and related contents. Very often this kind of study leads to various amendments before the starting of survey. Therefore enough time has to give for this stage in the schedule time of the study. The pilot survey covers the different region of automobile industry within Gujarat state. The primary data has been collected from the industry, using the questionnaire developed for this purpose.

A pilot study was conducted with 17 Automobile industry of Gujarat State. Data was collected using an online and offline mode. This questionnaire covers 5 sections. Section-I contains basic information of company/industry and respondents, employee details, type of organization, production technologies, product types, operation types, etc. Section-II contains Lean Manufacturing approaches and awareness about the tools/techniques which are used for implementation of Lean practices in Automobile Industry of Gujarat. Section-III contains Barriers affecting for successful implementation of lean practices. Section-IV contains Critical Success Factors (input factors) and the major drivers; which are used for lean manufacturing practices in automobile industry of Gujarat. Section-V contains performance improvements indicators for lean manufacturing practices. This is the most important part. Respondents rate these factors on the basis of their perceptions about important performance improvement.

DATA ANALYSIS AND INTERPRETATION

In the current competitive scenario lean manufacturing system assumes a significant importance and demands for serious research attention. The study measures the present status and scope of LMT through an extensive survey conducted among the Automobile industry within Gujarat state. In this survey, opinion of automobile manufacturers across the Gujarat state has been assessed. SPSS software is used for the analysis work.

In this type of research, respondent's personal nature plays very important role in giving and expressing the responses about the questions. Hence, in this study, in Section-I a various results have been derived. In

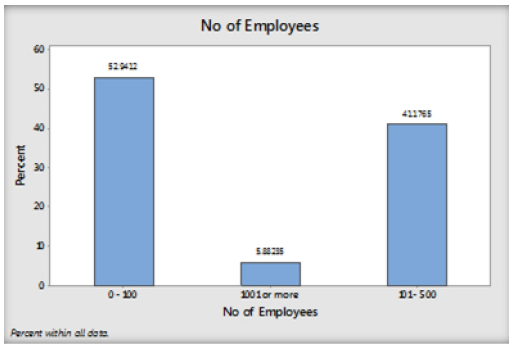


Fig. 1. No. of Employees

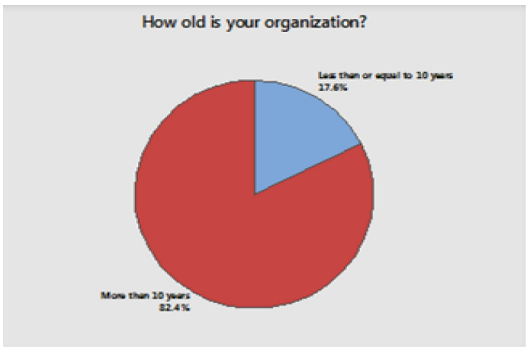


Fig. 2. About Organization

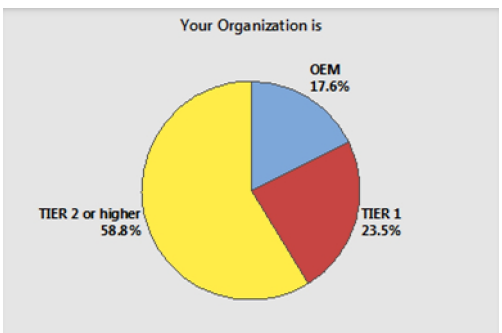


Fig. 3. Type of production

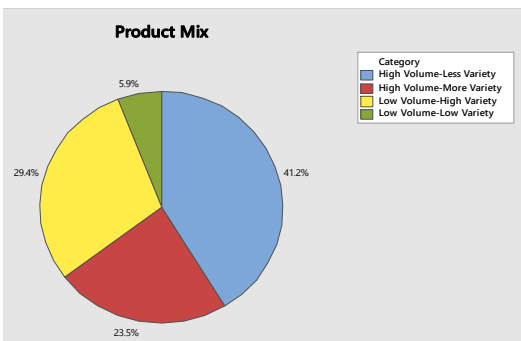


Fig. 4. Product Mix

these results, we observe that almost 53% of the companies were having employees up to 100 no, around 41% companies were found having number of employees in the range on 101-500 no and 6% companies were having more than 1000 employees (Refer Figure 1). About establishment, 17.6% industries was found less than or equal to 10 years old and 82.4% industries were found more than 10 years old (Refer Figure-2). 17.6% were OEM, 23.5% Tier-I and 58.8% companies observed were Tier-II or higher (Refer Figure-3). In the product mix category, High volume - less variety observed 41.2%, High Volume - More variety 23.5%, Low volume - Low variety 5.9%, Low Volume - High Variety 29.4% (Refer Figure-4). Type of operation observed make to order 47.1%, assemble to order 23.5%, and make to stock 23.5% and others observed 5.9% (Refer Figure-5).

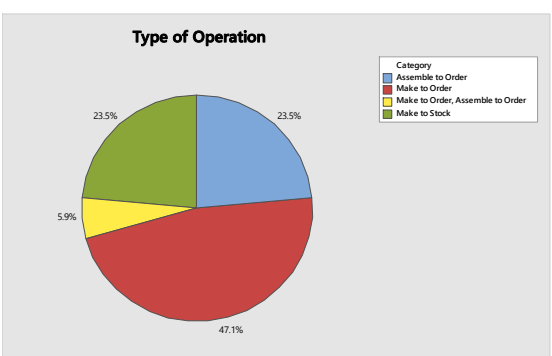


Fig. 5. Type of operation

In Section-II, we have observed 70.6% industries are aware with lean manufacturing practices (Refer Figure-6). From the survey, it is observed 33.3% source of awareness is due to top management involvement (Refer figure-7). It is also found 64.7% industry has started lean implementation (Refer Figure-8). The proportion of unawareness of lean found 29.4%. Among them, 42.9% did not know much about LMT and its implementation (Refer figure-9). Hence, 50% industries have started lean implementation since last year (Refer Figure-10). Figure-11 represents the benefits of using lean manufacturing practices.

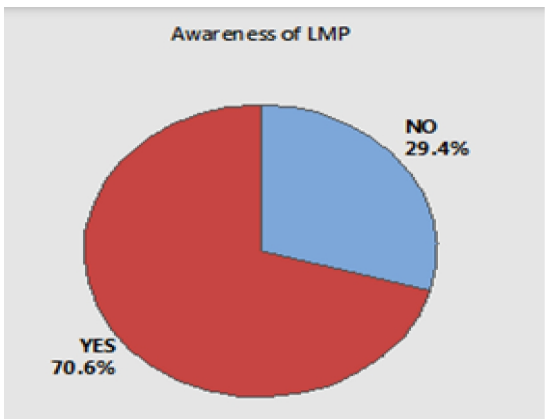


Fig. 6. Awareness of LMP

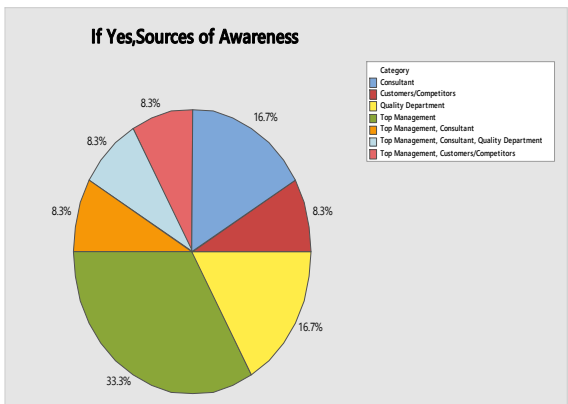


Fig. 7. Sources of awareness

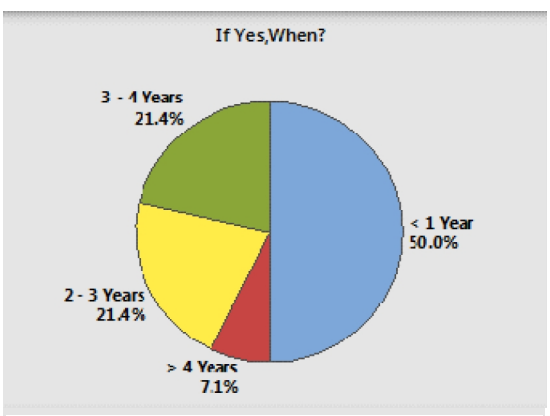


Fig. 10. Time of implementation of Lean

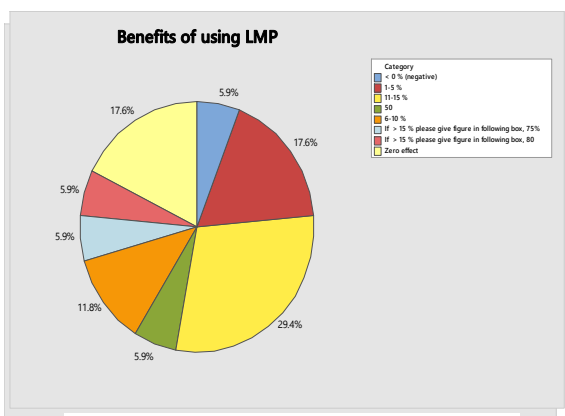


Fig. 11. Benefits of lean

In this survey, Cronbach's alpha is the important parameter to measure the internal consistency or reliability. It is widely used for multiple questions analysis of survey or questionnaire which forms a scale for determining the scale is reliable or not. Kuder & Richardson (1937) derived for scored data (0 or 1) and later presented by Cronbach (1951) to consider it in the account for any scoring method. The respondents have given responses of the survey questionnaire, which were analysed in SPSS software. Carefully ensured the accuracy of data file, checked the proof reading of data collected, also examined the statistics description and graphical representation of the given variables. Hence, in this study, reliability considered as a statistical measurement and it is mainly assessed in the forms of test-retest, alternation-formation and internally consistence.

Internally consistence of reliability is the mainly used for psychometric measurement to assess survey experiments and scales, it also indicates how effectively different items were measuring the same problems. Cronbach's alpha was used for measuring internal consistency. Nunnally and Bernstein stated that reliability measurements can be accepted with $\alpha \geq 0.6$, else $\alpha \geq 0.7$ should be threshold. If, $\alpha \geq 0.8$ should be very reliable. In this pilot study analysis all the measured parameters had a Cronbach's alpha is at least 0.6 or greater, so, as per the set of guidelines for new variables development results are well exceeding.

The results get by the SPSS analysis in the relations of these measurements are represented in Table 1. Cronbach's alpha considered to access the reliability of inter-item, values of alpha with 0.7 or higher indicates acceptable reliability for derived scales. All the alpha values exceeding threshold. Hence, overall Cronbach's alpha value found out to be 0.972 indicate the adequate reliability of the measurement scales. It is found that the mean ratings of lean manufacturing barriers in the range of very high practice to a very low practice level, as all the 25 no of items of barriers are rated from highest of 4.1176 to the lowest by 3.1176.

Table 1. Reliability Test for Barriers

Reliability Analysis : Barriers			
Name of Factor	**No. of Items**	**No. of Items Deleted**	**Cronbach's Alpha**
Barriers	25	None	0.972

Table 2 shows overall Cronbach's alpha value found out to be 0.965 indicate the adequate reliability of the measurement scales. It is found that the mean ratings of lean manufacturing critical success factors in the range of very high practice to a very low practice level, as all the 30 no of items of CSFs are rated from highest of 4.0588 to the lowest by 3.5882.

Table 2. Reliability Test for Critical Success Factors

Reliability Analysis : Critical Success Factors			
Name of Factor	**No of Items**	**No of Items Deleted**	**Cronbach's Alpha**
Critical Success Factor	30	None	0.965

Table-3 represents a reliability test for Performance Improvement Indicators. It is found that the Cronbach's Alpha for all the sub factors seems greater than 0.7. In case of sub-factors such as Financial and People, the mean ratings were found satisfactorily whereas in case of sub-factors like

Customers/Market measures, Process and Future, it was required to eliminate some variables to achieve the reliable Cronbach's Alpha (greater than 0.7).

Table 3. Reliability Test for Performance Improvements Indicators

Reliability Analysis : Performance Improvement Indicators				
Sr. No.	**Name of Factor**	**No of Items**	**No of Items Deleted**	**Cronbach's Alpha**
1	Financial	5	None	0.905
2	Customers/Market Measures	10	04	0.876
3	Process	12	02	0.922
4	People	5	None	0.857
5	Future	5	01	0.718

CONCLUSION

This research has covered a wide range of items with respect to previous studies, collated most of them. Hence, variables considered for barriers were 25 items, CSFs were 30 items and Performance Indicators has 5 sub factors includes total 37 items were constructed and tested. A widely used method for obtaining or confirming these parameters was the main contribution of this research work. The survey questionnaire was based on reviews of details literature, interviews of experts, it includes their opinion and most of the respondents were expecting the lean design at the moment. Initially, lean design was the part of the questionnaire, but here we have got various types of answers we analyzed that as a special investigation for the benefited in the future. In addition to determining the reliability of the survey, we also evaluated the validity of a measure.

Cronbach's co-efficient alpha was used to access the reliability test, the values alpha is 0.7 or more considered as acceptable reliability for deriving scales. Hence, overall Cronbach's alpha value found out to be 0.972 indicate the adequate reliability of the measurement scales. As it happens, the average scores of lean manufacturing barriers in the range of very high practical level very little practical, as all the 25 no of items of barriers are rated from highest of 4.1176 to the low of 3.1176. It is found that the mean ratings of lean manufacturing critical success factors in the range of very high practice to a very low practice level, as all the 30 no of items of CSFs are rated from highest of 4.0588 to the lowest by 3.5882. It is found that the Cronbach's Alpha for all the sub factors seems greater than 0.7. In case of sub-factors such as Financial and People, the mean ratings were found satisfactorily whereas in case of sub-factors like Customers/Market measures, Process and Future, it was required to eliminate some variables to achieve the reliable Cronbach's Alpha (greater than 0.7).

ACKNOWLEDGEMENT

It is a privilege for me to have been associated with Dr. M. N. Qureshi, College of Engineering, King Khalid University, Abha and Dr. T.N. Desai, Mechanical Engineering Department, SVNIT, Surat, during this research work. I have been greatly benefitted by their valuable suggestions and ideas. I express my deep sense of gratitude to them for their valuable guidance and constant encouragement throughout this work. I express my sincere thanks to the entire team of ICRE 2016 of R. K. University, Rajkot for arranging this event.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

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RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

Preparation and Properties of TiO_2:CuO:Cr_2O_3 Nanofluid for Solar Evacuated Water Heater Systems

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ABSTRACT

Nanoparticles have significant influence on thermo-physical and optical properties when dispersed in base fluid. This characteristic has great potential to capture solar energy. Solar radiation absorption enhancement leads towards the highest heat transfer rate. Which ultimately results in more and efficient heat transfer. This paper is an exploration about the idea of harvesting solar energy through the usage of nanofluid based evacuated tube solar collector. The synthesis of TiO_2:CuO:Cr_2O_3 based nanofluid made of equal proportions of nanoparticles, prepared by the ultrasonic probe method. Its stability has been checked for different time intervals of sonication. Several properties of nanofluid have been studied and observed remarkable improvement in thermal properties. The results derived from this study will lead towards the development of high efficient domestic solar water heating systems.

SUMMARY

This paper explores newly developed nanofluid for solar energy enhancement in evacuated tube collectors.

Keywords: Nanoparticles, Nanofluid, Solar Energy.

INTRODUCTION

This paper attempts to explain the synthesis, advantages and characteristics of nanofluid wheneve used as a heat transfer medium in solar water heating systems.

Nanoparticles dispersions in the basic fluid, i.e. nanofluids [1–3] have revealed the relevance of applications requiring effective and quick heat transfer. In conventional heat transfer fluids for photo-thermal applications like molten salts, water and ethylene glycol (EG), nanofluids are not transparent to the solar radiation; instead, they scatter significantly and absorb the solar radiation passing through them [4,5]. In this direction, by few researchers have proposed a non-concentrating solar collector by using nanofluids as the working fluid [6,7]. Similar kinds of attempts have been made to explore the viability & applicability of nanofluids in the case of high flux (heat) collectors [8,9]. A substantial rise of thermal conductivity in liquid, heat transfer co-efficient and liquid viscosity, are the unique characteristics of nanofluids. It is proved that almost metals have higher thermal conductivity in solid phase than base fluids [10]. The copper thermal conductivity at room temperature is nearly 680 to 700 times greater than water and 3000 times greater than engine oil. The thermal conductivity of metallic liquids is very much greater than that of non-metallic liquids. The liquids with suspended metal particles are subjected to thermal conductivity enhancement rather than pure liquids [11].

The heat transfer enhancement with Al_2O_3 nanofluid is possible up to 40% as compared to the base fluid. Regarding thermal conductivity of nanofluids of MWCNT (Multi Wall Carbon Nano Tubes) can be enhanced up to 150% [12]. For 2% volume concentration of Cupric-Water nanofluid, pumping power and overall heat transfer coefficient are more than base fluid [13]. Thermal conductivity, density and viscosity of the nanofluid increase with the increment of volume concentrations. The rise in the temperature, thermal conductivity & specific heat were observed to be intensified, and while the viscosity and density were decreased.

Advantages of nanofluid

Nanofluids have following advantages over base fluid:

(i) High degree of dispersion stability along with Brownian motion of particles,
(ii) Reduction in particle clogging with reference to conventional slurries, ultimately promoting system miniaturization,
(iii) Thermal conductivity & surface wetability can be enhanced by varying particle concentration, Low cost of production as compared to EG based fluid.

II. MATERIALS AND METHODS

Nanofluid Preparation

The nanoparticles in powder form dispersed in the base fluid, is called nanofluid. The enhancement of thermal conductivity of the nanofluid greatly depends on the preparation of nanofluid using nanoparticles. There are mainly two methods of synthesizing nanofluids.

1. Two-step method

2. One-step method

These methods are executed by chemical and physical routs to ensure that the liquid-solid mixture is quite stable to avoid clogging, agglomeration, probable erosion, poor thermal conductivity, additional flow resistance and poor heat transfer. It is noted from literature that nanofluids with oxide nanoparticles as well as carbon nanotubes can be produced well by two-step method.

2.1.2. Two-step technique

In a two-step method of nanofluid synthesizes, firstly the nanoparticles are produced separately and then these nanoparticles are dispersed in a base fluid for exactly measured quantity.

The advantage of two-step method is that nanofluid can be produced easily and economically. On the other hand, due to van der Waals effect force of cohesion between nanoparticles the fast agglomeration of individual particles before an achievement of complete dispersion takes place. This agglomeration is the biggest obstruction in achieving high heat transfer performance because the quick settling of nanoparticles out of the base fluids and becomes worse as the concentration volume increases. Agglomeration is a critical issue in all kinds of nanopowder technology, especially during nanoparticles transportation, drying and storage.

Several methods are used to eliminate agglomeration in a two-
step process exploration towards commercialization by facilitating nanofluids mass production. In Two-step methods like ultrasonic disrupter, stirrer, high-pressure homogenizer and ultrasonic bath are well-known methods of two-step technique. Rehena Nasrin et al. [14], S. Zeinali Heris et al. [15], Michael Saterlie et al. [16] prepared water based CuO nanofluid. Their ultimate intention was to find out agglomeration effect with a parametric study of volume concentration on thermal conductivity. The TiO_2–Water nanofluid was prepared with ultrasonic equipment and introduced by Murshed et al. [17]. Chakraborty et al. [18], Wong wises and Duangthongsuk [19] produced Al_2O_3 and TiO_2 nanofluids by dispersion of nanoparticles in distilled water by using ultrasonication for 30 minutes & ultrasonic stirring for nearly 20 minutes different two-step method. Sajadi and Kazemi [20] mixed the correct amount of TiO_2 nanoparticles with distilled water by mixing for 10 minutes. Then for dispersion, an ultrasonic cleaner was used for 30 minutes.

2.1.1. One-step technique

In the one-step technique, the nanoparticles are simultaneously prepared and dispersed directly into the base fluid. There are total fourteen various methods to produce nanoparticles [21]. This method is highly advisable to produce nanofluids of better thermal conductivity metals to avoid oxidation of particles and erosion. Agglomeration minimization is the biggest advantage of this method. This tendency increases equal dispersion in the hot liquids and the stability of the suspensions. The quantity limit of the production due to the gradual production process, low nanoparticles concentration & the high synthesis cost are the main disadvantages of one-step technique. Various methods are used evaporation, physical or chemical one-step methods have been used to reduce the cost & time.

2.2 Selection of Nanoparticles

Including nanofluid preparation, titanium dioxide is widely used in many areas. By production point of view TiO_2 nanoparticles can be easily obtained as they are readily produced on industrial scales. With respect to their physic-chemical profile, they have better stability when dispersed in a base fluid even without the addition of stabilizer[22]. The enhancement of thermal conductivity reported of TiO_2 nanofluids as compared to water based nanofluids, when Ethylene Glycol (EG), Propylene Glycol (PG) or paraffin oil used as base fluids. Investigation shows that for CuO nanofluid preparation the sonication time greatly affects the heat transfer performance & influenced by the nanoparticles concentration [23]. The thermal conductivity enhancement depends on the particle volume concentration and temperature of nanofluid. For the 1.2% volume concentration of CuO nanofluids, 10.8% to 43.2% enhancement of

nanofluid thermal conductivity of nanofluids is observed. Above mentioned characteristic is very much helpful in photo-thermal conversion kind of application for solar water heaters [24].

2.3 Selection of Base Liquid

The thermal conductivity of nanofluid is inversely proportional to that of base fluid [25]. Many host liquids are being used to produce nanofluids like organic and aqueous liquids (ethylene glycol, propylene glycol and oils) for thermal conductivity enhancement.

It is observed that thermal conductivities of TiO_2 and CuO kinds of nanofluids increases, which use paraffin oil, ethylene glycol or propylene glycol as base fluids seem to be remarkably lower than those based on water [26]. This is the only reason distilled water is preferable as a base fluid for nanofluid preparation.

2.4 Selection of Nanoparticles Size

The size of particles plays a decisive part in thermal conductivity as well as heat transfer enhancement of base fluids. Several suspension sizes of milli and micrometer particles in host liquid are used to enhance the thermal conductivity, at the same moment the particles agglomeration is quick and settled down in the liquid. The sedimentation time decreases to reach in few cases for more than a few days or weeks. Due to increase in surface area the thermal conductivity increases for the nanoparticles size in the range of 1–100 nm. Larger the relative surface area of nanoparticles, significant improvement in thermal conductivity capabilities and also rises in the stableness of the intermissions. In addition to that the size of nanoparticles is being reduced, the Brownian motion will be generated [27]. The nanoparticles size in the base liquid is quite important in research field of the present era.

In this experimental work of nanofluid preparation the, TiO_2 nanoparticles of size 25 ± 5 nm, CuO nanoparticles size up to 17 nm and nanoparticles of Cr_2O_3 are of size 35 nm as provided by Nano wings Pvt. Ltd, has been used.

2.5 Preparation of Nanofluid

In this experiment of nanofluid preparation, TiO2 nanoparticles of an average size of 25 nm with the purity of 99.9%, were used as purchased from the Nano wings Pvt. Ltd Company, Khamam, India. The size of the nanoparticles supplied from appropriate supplier and used in this experiment is 20 – 30 nm.

The base fluid is distilled water because boiling characteristics of the base fluids (water) are well known. The TiO_2 nanoparticles are commercially available. No surfactant or buffer was added in the nanofluids during the dispersion. The mixture of nanoparticles and the base fluid was sonicated with an ultrasonic probe for an hour to obtain nanofluid.

Cupric Acetate Monohydrate $(CH_3COO)_2Cu.H_2O$ powder in equal proportion is added in double distilled water for 0.6 molarities. For complete dispersion the correct proportion of water and Cupric Acetate is pre-requisite condition. Again stirring for 35 minutes and heating at 150°C for the complete dispersion of powder.

$C_{14}H_{23}Cr_3O_{16}$ (Chromium Acetate) powder for 0.4 molarities added in distilled water. Acetic acid (CH_3COOH) in the same quantity has been mixed up for nanofluid preparation. Complete mixture is stirred for complete dispersion of nanoparticles. Stirring with heating takes place at 170°C for 45 minutes. At the end of this task, complete dispersion of powder takes place and nanofluid is prepared.

All above individual nanofluids are mixed up with each other in 1:1:1 proportion to prepare a new one. Nanofluid prepared in this way for these processes remains stable for about a week and without any

agglomeration. The base fluid (distilled water) is taken for the dispersion of nanoparticles. This facility of Oscar ultrasonic machine PR- 1000 was availed by Saurashtra University, Rajkot .

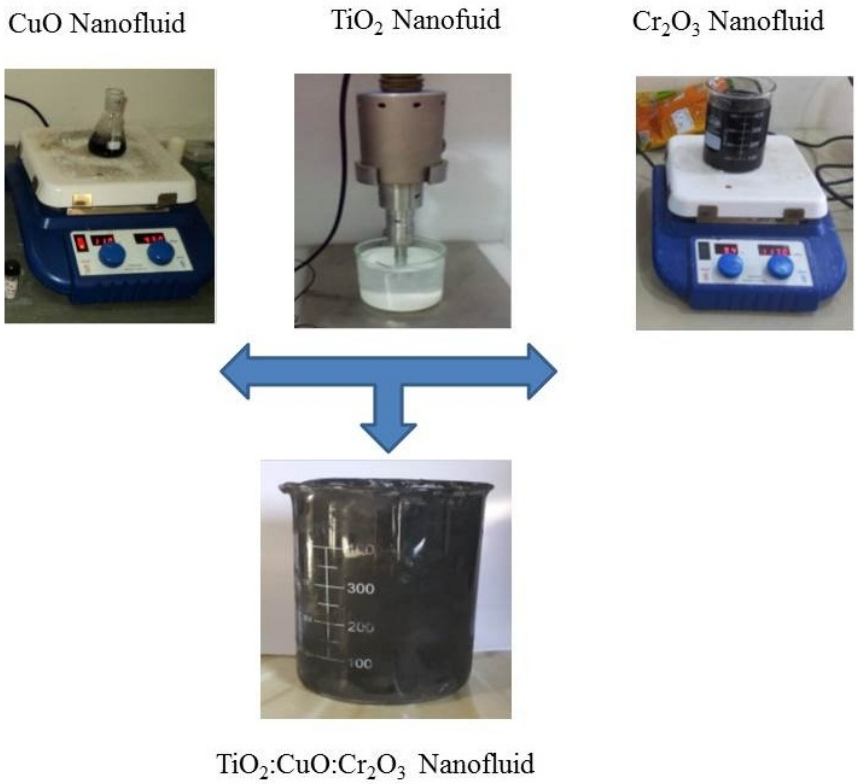


Figure 2.1: Preparation of nanofluid in ultrasonication.

The mixing of nanoparticles and base fluid were carried out in the Oscar horn type ultrasonic reactor. The horn is attached on the transducer that produces ultrasonic irradiation in the mixture. The frequency of Ultrasonic processor is in the range of 25 kHz to 30 kHz. Time ranges from 3 to 30 minutes along with the power supply from 100 W to 1000W in the steps of 100W. The transducer horn was submerged approximately 5 cm in the mixture of nanoparticles and base fluid.

III. RESULTS AND DISCUSSION

Tables 3.1 & 3.2 give all the required properties of the TiO_2, CuO, Cr_2O_3 nanoparticles and double distilled water as base fluid. The table 3.3 shows the properties of the nanofluids with different proportion of the nanoparticles.

Table: 3.1 Major Properties of TiO_2, CuO, Cr_2O_3 nanoparticles.

Nanoparticles (Powder phase)	Density g/cm^3	Specific Surface Area m^2/g	Thermal Conductivity W/mK	Specific Heat KJ/KgK
TiO2 (Anatase)	0.2-0.4	60	6	0.69
CuO (Monoclinic)	6.315	140	18	0.540
Cr Powder	0.13	80	42	0.46

Table: 3.2 Major Properties of Double Distilled Water as a base fluid.

Powder phase	Density Kg/l	Thermal Conductivity W/m K	Specific Heat KJ/Kg K	Viscosity CST
Pure Water	1	0.669	4.19	1.79

Table:3.3 Major Properties of Prepared nanofluid with water as a base fluid.

Nano particles	Proportion	Density g/cm^3	Kinematic Viscosity CST	Thermal Conductivity W/m K	Specific Heat KJ/Kg K	Boiling Point K
TiO_2	0.25%	0.9990	0.2820	0.67	4.164	377
	0.50%	0.9996	0.2929	0.70	4.154	381
CuO	0.25%	6.870	1.1650	1.01	16.1	250
	0.50%	6.920	1.2869	1.10	18.1	252
Cr_2O_3	0.25%	1.52	.2356	13.00	5.12	432
	0.50%	1.63	.2523	15.00	5.23	436
TiO_2+CuO + Cr_2O_3 nanofluid	0.25%	2.42	1.91	2.89	10.05	370
TiO_2+CuO + Cr_2O_3 nanofluid	0.5%	2.53	1.96	2.97	11.53	383

At a frequency of 25 kHz, 500 Watt power and mechanical stirrer method with 400 rpm. In table 3.4 & 3.5 the stability period of nanoparticles in the base fluid is mentioned.

Table: 3.4 Stability of nanofluids with Ultra sonic probe process.

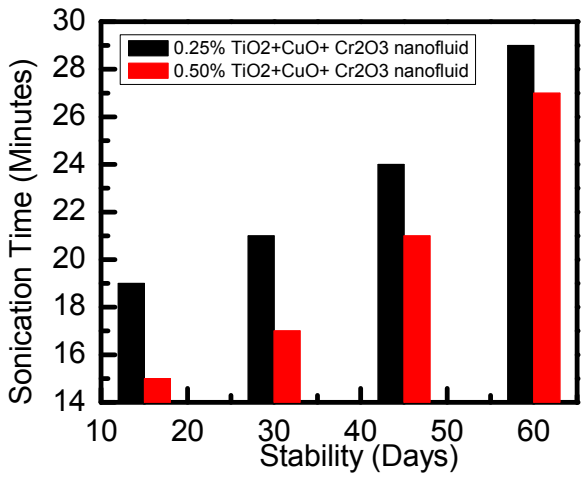
For 0.25% TiO_2+CuO+ Cr_2O_3 nanofluid			For 0.5% TiO_2+CuO+ Cr_2O_3 nanofluid		
Sr. No.	Time of sonication	Stability period	Sr. No.	Time of sonication	Stability period
1	15 min.	19 days	1	15 min.	15 days
2	30 min.	21 days	2	30 min.	17 days
3	45 min.	24 days	3	45 min.	21 days
4	60 min.	29 days	4	60 min.	27 days

Table: 3.5 Staybility of nanofluid with Mechanical stirrer process

For 0.25% TiO_2+CuO+ Cr_2O_3 nanofluid			For 0.5% TiO_2+CuO+ Cr_2O_3 nanofluid		
Sr. No.	Time of stirrer	Stability period	Sr. No.	Time of stirrer	Stability period
1	15 min.	2.1 hrs	1	15 min.	1.6hrs
2	30 min.	5.2 hrs	2	30 min.	3.7 hrs
3	45 min.	8.5 hrs	3	45 min.	6.3 hrs
4	60 min.	12.5hrs	4	60 min.	9.4 hrs

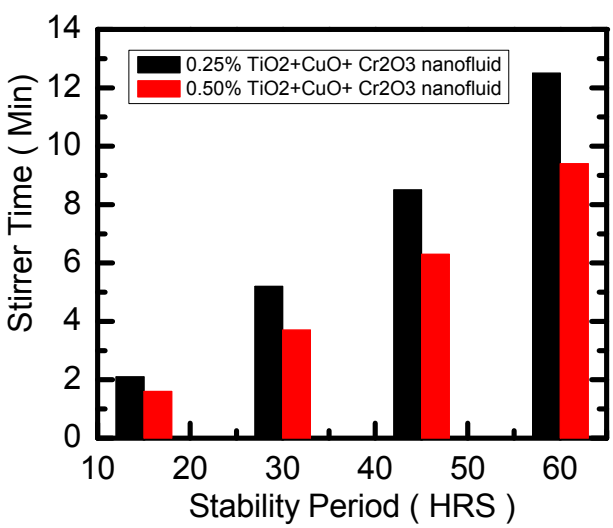
From above mentioned table it can be said that the time of sonication is increased for mixing of nanoparticles and base fluid, the stability period of nanoparticles increased. Similarly in mechanical stirrer method, as the stirring time increased the stability time also increased, but for the ultrasonic sonication method stability is much more than the mechanical stirrer method.

Fig.3.1 Sonication time effect on the stability of nanoparticles.



It has been from the above given results that in all methods the stability period increases with lesser % proportion of nanoparticles. Moreover as the % proportion of the nanoparticles increase separation of nanoparticles from base fluid occurs rapidly.

Fig.3.2 Effects of Mechanical stirring time on stability of nanoparticles



From above figures-3.1 and 3.2, it is can be judged that ultrasonic mixing of nanoparticles in base fluid shows greater stability than with mechanical stirrer method. Furthermore it is clear that stability of new prepared nanofluid is more as compared to all individual nanofluids.

Fig. 3.3 Effects of nanoparticles concentration on Mechanical Properties

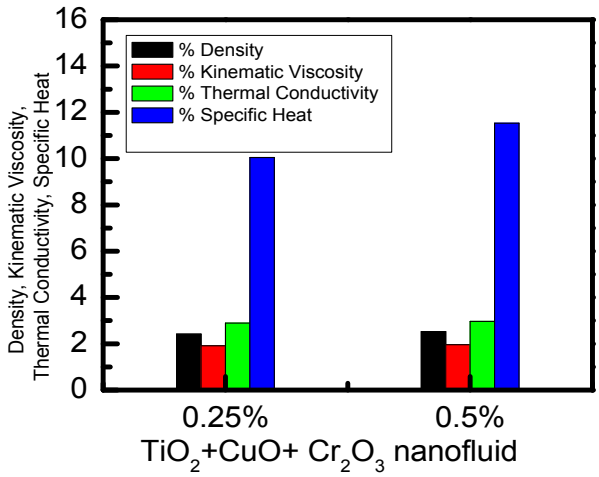


Table 3.3 shows the variation of different physical and thermal properties of nanofluid with the proportion of nanoparticles. At last it is concluded from the above results that thermal conductivity can be enhanced with increase of nanoparticles percentage. A little decrease in specific heat of nanofluid is observed with the rise in nanoparticles concentration The Density and Kinematic viscosity of nanofluid increases by normal value with the increase of % of nanoparticles. With the addition of nanoparticles in the base liquid the boiling temperature of nanofluid also rises.
The abstract should be up to 150 words long.

IV. CONCLUSION

This research article revealed some excellent parametric changes in new nanofluid to be used in solar evacuated tube for as a working fluid. It has highlighted the positive changes regarding stability; specific heat, thermal conductivity; viscosity and density of nanofluid prepared and compared with the base fluid.

V. ACKNOWLEDGEMENT

This work is supported by GUCOST (Gujarat Council On Science And Technology) – Grant No. GUJCOST/MRP/2014-15/2543.

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Design of Separable Reversible Data Hiding Technique With small distortion of Image

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ABSTRACT

This paper proposes a separable reversible data hiding technique based on lossless compression. One common drawback of virtually all lossless compression based techniques is that there is a tradeoff between payload capacity and robustness. Compared to previous approaches, the proposed method extend lossless compression based data hiding technique that reduce visual artifacts occurred due to hiding data within image. In addition to that, this paper also suggests the key distribution between sender and receiver.

SUMMARY

Develop a separable reversible data hiding technique that reduce distortion occur after embedding data within image.

Keywords: lossless compression, stego key, encryption key, Reversible Data Hiding, Separable Data Hiding

I. INTRODUCTION

In wide spread use of internet the use of data hiding techniques within the image are widely used. According to the recovery of image at receiver side the data hiding techniques are divided into two types 1.Reversible Techniques 2.Non-reversible Techniques.

In non-reversible data hiding techniques the receiver can retrieve secret data without any loss but, the image will recover with certain distortion that occurs due to embedding data whereas, in reversible data hiding techniques the receiver can retrieve both embedded data and original image without any loss. In certain applications like IPR (Intellectual Property Rights), medical images or any other images in which small distortion after decoding at receiver side is not acceptable.

Separable feature of data hiding technique allow separate recovery of image and data at receiver side according to the requirement and availability of keys.
A number of separable reversible data hiding techniques have been proposed, and they can be roughly classified into three types *(9)*:

1. Lossless compression based methods,
2. Difference expansion (DE) methods
3. Histogram modification (HM) methods.

Among all above mentioned reversible data hiding techniques hiding data in compressed code can reduce transmission cost and simultaneously make the transmission more secure.
In lossless compression based techniques (1-5) first the encrypted image is compressed using either lossless or lossy compression method and then using LSB technique secret data bits embedded into compressed area. Various lossless compression based method are currently in use for data hiding but still there is a scope of research for improving payload capacity and reduce the distortion.
Here in this paper we extend lossless compression based data hiding technique that reduce visualartifacts occur due to embedding data within image and also suggest the key distribution between sender and receiver.

Rest of the paper is organized as: Section II summarizes different data hiding algorithms based on lossless compression. Section III contains detail of step wise exposition of proposed algorithm. Section IV explains flow of proposed algorithm using chart. Further, Section V concludes and finally future scope is given in Section VI.

II. RELETED WORK

Lossless compression based data hiding techniques compress the cover media, such that the original content can be represented in a smaller size and the saved space can be used to embed messages.

V Agham et al*(1)* compressed LSB (Least Significant Bits) 5,6,7,8 of randomly selected pixels from an encrypted image and created extra space to embed additional data. Due to random selection of pixels for embedding data the distortion in image is more visible especially when the selected pixel belongs to smooth area. In this technique error rate rises as the payload capacity increase then 0.04bpp.

P. Kadam et al.*(2)* implemented lossy compression for embed additional data in encrypted image. Image is encrypted using AES algorithm. This technique improves execution time of image encryption and decryption but the improvement of payload capacity and PSNR value of stego image was not considered.

C. Rengarajaswamy*(3)* worked on improving payload capacity by selecting proper compression method. The researcher use lossy compression technique known as DCT(Discrete Cosine Transfer) for image compression. This technique improves payload capacity in comparison of lossless technique used in other reversible data hiding techniques but did not consider distortion occurring because of data being embedded into image.

Celiket al.*(4)* used G-LSB(Generalization Of Least Significant Bit) technique for embedding data into an image without loss. G-LSB offers more scalability and less distortion with high payload then conventional LSB-embedding techniques. However, the bit-plane correlation is usually weak so, the direct compression on LSB plane will create visual artifact.

Hong et al.*(5)* haven't directly embedded data into LSB of image pixels in sequence but data was embedded according to the smoothness of the image block ,so less visual artifact was generated.

Zhaoxia et al.*(6)*randomly selects two basic pixels in each block to estimate the block smoothness and additional data that are embedded according to the smoothness of the blocks using local histogram shifting.
Fridrichet al.*(7)* use JBIG(Joint Bi-level Image Experts Group) image compression method is a reversible data hiding technique. Here, using JBIG compression method, researcher find bit key plane whose compression provides enough space to hide 256 bit hash code. Key bit plane searching starts from 5^{th} LSB of each color channel of an image. This method is good when we require less payload capacity i.e. 256 bit hash code. If the image is noisy, then there is a need to select higher bit plane for embedding data that leads to more distortion which is easily visible.

III. PROPOSED ALGORITHM

The proposed algorithm of data hiding is different than existing compression based data hiding techniques in the way data gets embedded into the encrypted image. In proposed algorithm, more data is embedded into edge pixels in comparison of non-edge pixels because; edge pixels can tolerate more changes then non-edge pixels [8].

Keys used in Proposed Algorithm.

1. **Image encryption key**: Public key is used by RSA encryption method for encrypting image. That means sender will encrypt image using public key of receiver and receiver decrypt image using own private key.
2. **Stego key**: By using Stego key we can reduce the chances of attackers to find out the secret information. Thus, we make use of symmetric key (K1) as stego key for random selection of pixels from encrypted image which will be further utilized for embedding secret data.
3. **Data encryption key :**Before hiding data into the image, must be encrypted first. This requires usage of symmetric key (K2) for generating encrypted data stream.

Symmetric key Generation:

Using Diffie-Hellman Key exchange protocol, symmetric key (K1) is generated and used as stego key. The same key (K1) is used as session key to send data encryption key(K2) at receiver side.

Steps of proposed algorithm:

Sender Side Algorithm:

Following steps are followed by sender for sending stego image.

A: Image Encryption:

Before embedding data first the sender encrypts the original uncompressed image (I) using an encryption key IK1 to produce an encrypted image(I').

$$I'=E(Ik1,I)$$

B. Embedding data:

1. Using stego key (K1), select pixels for embedding data.
2. Encrypt the data before initializing the process of embedding to the encrypted image using shared key K2 D'=E(K2,D).
3. Selected pixels divided into two blocks, edge pixels and non edge pixels. The data-hider uses canny edge detection technique for finding edge pixels*(8)*.
4. Use 5, 6, 7, 8 LSB's of edge pixels and 6, 7, 8 LSB's for non-edge pixels for embedding data.

5. The bit stream of selected pixels in step-4 is compressed using lossless compression method and then secret data is embedded into spare space.

C. File Sending:

Send stego image X(I' + D') using internet connection.

Receiver Side Algorithm:

Receiver receives Stego image(X) with embedded data (D')

1. If receiver has only two keys **stego key(K1) and data encryption key(K2)**, then the receiver can extract the additional data even though the receiver does not have any idea about the original image content.
2. If the receiver at the destination has the **encryption key of the image(IK1) but not stego key(K1) and data encryption key(K2)** then, the receiver can decrypt the image similar to the original image. But receiver cannot extract the hidden data. The decrypted image is similar to the original image because the embedded data impact only to the LSBs' of image. Here edge pixels LSB's are used more for embedding secret data so that distortion rate remains low after embedding secret data.
3. If the receiver has **all three keys** i.e**stego key(K1), data en**cryptio**n key(K2) and image encryption key(IK1)** then, receiver can extract the additional data and recover the image i.e the original content of the image. After extracting secret data the receiver perform decompression to extract original image without any distortion.

IV. Flow of proposed Algorithm:

1. Sender Side Algorithm:

The flow chart in Fig.1 represents working of proposed algorithm at sender side. Sender will first encrypt image and then embed secret data in it.

2.Receiver Side Algorithm:

The flow chart in Fig.2 represents the working of proposed algorithm at receiver side. Receiver needs all three keys i.e. image encryption key, Stego key and Data encryption key to recover original image.

V. CONCLUSION

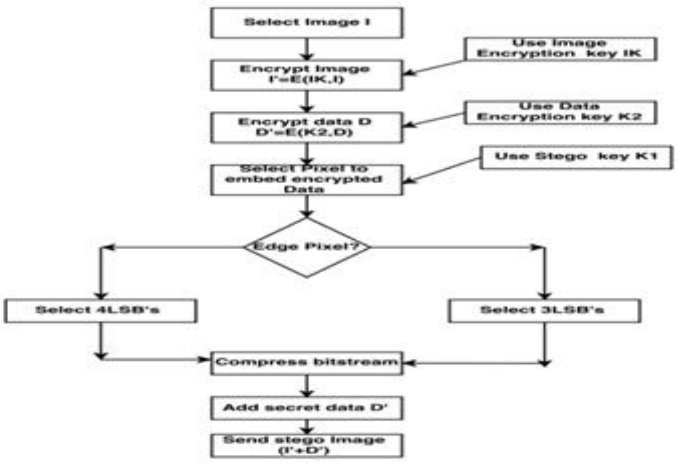
This paper gives design of a separable and reversible data-hiding technique that reduces visual artifacts occurring due to data embedding over the image. So the possibility of extracting data from stego image without knowing stego key will reduce to great extent. As we know that any changes that occur into the smooth area of image is more visible then changes that occurs into non-edge pixels. Using this concept, proposed method embed more data into edge pixels then non-edge pixels so that, any changes that occurs after embedding data into image is less visible. This paper also suggests usage of Diffie-Hellman Key exchange protocol for 1) stego key generation and 2) for encryption key distribution from sender to receiver.

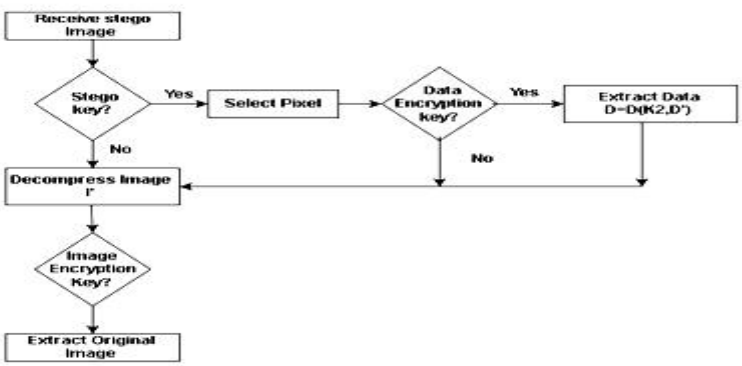
VI. Future Enhancement:

This proposed algorithm is designed considering only gray scale image. But, if the image is a colored image then there is a scope of more improvements in payload capacity. In colored image, data can be embedded into each separate color channel (RGB-Red Green Blue) of the image. This will increase embedding capacity about 3 times in color channels then the grayscale channels according to proposed algorithm.

FIGURES

Fig. 1. Flow chart of Sender side Algorithm





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Reduction in the Loading and Unloading Time of CNC machine by Automation

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ABSTRACT

Self-dictated systems, such as computers, industrial machinery and processes are known as "automation", which is replacing human operators. Typicalapplications of automationinvolve picking of the material, work piece, or a tool from one place and placing it at the desired place. This task can be done by pick and place robot. There are many gauges of performance for this task; including speed, accuracy, and reliability. The main constraint in the production is the loading and unloading of the material in CNC machines in which idle time is more. In the present study efforts are made to decrease loading and unloading time by the use of pick and place robotic arm by automation.

SUMMARY

Loading and unloading time can be decreased by the use of robotic arm and as a result productivity will be increased.

Keywords: *Robotics, Automation, Pick and place robot*

INTRODUCTION

Automation is a Greek word which means self-dictated, such that numerical control, programmable logic control or other industrial control systems in concern with computer applications or information technology to manipulate all the industrial machinery and processes, thus reducing the need of human involvement. For the growth and development of industries, automation is must and should supersede the mechanical growth.

There are three broad classes of industrial automation[1]:

- Fixed automation
- Programmable automation and
- Flexible automation

Among these three types, robotics coincides most closely with programmable automation. The origin of the word robot canbe traced to the Czech word "Robota", which means forced or compulsory labor *(1-3)*. The Robotics Industries Association (RIA) provided an official definition of an industrial robot as "An industrial robot is a reprogrammable, multifunctional manipulator designed to move materials, parts, tools, or special devices through variable programmed motions for performance of a variety of tasks"*(1,2).*

Pick and Place robot is used in a wide variety of materials. The main and basic application of its is the transfer of material from one place to another. For that the machine takes a product from one spot in the manufacturing process and places it into another location. The typical pick and place application requires high amounts of repetitive motion. These robots have high return on investment when consistent shaped parts or containers are handled. When the pick and place of the material carried out manually the loading and unloading time is more which can be reduced by using pick and place automation and hence productivity can be increased. Present study deals with the reduction in loading and unloading time by the use of robotic arm.

MATERIALS & METHOD

For the design of the robotic arm the present author has carried out experimental work on CNC machine available at Industrial Automation, Tirupatinagar-4 at Rajkot.

Standard data of CNC Pick and Place system*(4)*

1) Pneumatic Cylinder (Loading New Component into Loading Station) :- DNC 40 mm (Bore) × 20 mm (Stroke)
2) Pneumatic Cylinder(Loading Cylinder) :- DNC 100 mm (Bore) × 600 mm (Stroke)
3) Pusher Pneumatic Cylinder:- DNC 32 mm(Bore) × 40 mm (Stroke)
4) Unload Pneumatic Cylinder from CNC Turning Centre:- DNC 32 mm(Bore) × 40 mm (Stroke)
5) Catcher Pneumatic Cylinder :- DNC 40 mm (Bore) × 500 mm (Stroke)
6) Door Open and Close Pneumatic Cylinder:- DNC 63 mm (Bore) × 500 mm(Stroke)
7) M.S Frame
8) Two Jaw Mechanical Gripper
9) Teflon Spacers

   Machining time = 35 sec

Time required to load and unload the material manually was about 12 sec but with the help of pick and place robot the cycle time was calculated as discussed elaborately in results section.

RESULTS

Cycle Time Calculation

Velocity of Pneumatic Cylinder = 1 m/s

Pressure of Compressed air = 6 bar

Machining time = 35 sec

Loading and unloading time calculation

Time required to open the CNC door = 0.72 Sec

Time required to enter the catcher inside the CNC = 0.6 Sec

Time required to exit the catcher outside the CNC = 0.6 Sec

Total Cycle time for CNC Pick and Place Operation: (35 + 6.58) = 41.58 sec ≈ Approx 42 Sec

Accuracy Calculation

Fixture has a tolerance of 0.5 mm and hence one has to put the job with a accuracy of 0.5mm.This can be achieved by selecting proper pneumatic cylinder.

Fig. 1 shows the mechanism of automatic unloading of work-piece. When work-piece is held on chuck the spring will be compressed and when chuck is opened this spring force push the work-piece.

Calculation of the deflection of cylinder road

From the geometry of figure 2,

$$\Delta\theta = \frac{L}{\rho} = \frac{\Delta L}{y} \tag{1}$$

Where ρ is bending radius, L is the initial length of the beam, and y is the distance from neutral axis. Strain formula is

$$\varepsilon(y) = \frac{\Delta L}{L} \tag{2}$$

Combining (1) and (2) we get

$$\Delta\theta = \frac{\varepsilon(y)L}{y} \tag{3}$$

Now from the highlighted strip in fig. 2, differential moment applied to that strip is given by:

$$dM = y \times dF = y(\sigma \times dA) = y(\varepsilon(y)E \times dA)) \tag{4}$$

Combining equations (3) and (4) :

$$dM = (y(\varepsilon(y)E \times dA)) = \frac{y^2\Delta\theta}{L}EdA \tag{5}$$

Integrating equation (5) to find the applied moment, and solve for beam deviation angle Δθ:

$$M = \int \frac{y^2\Delta\theta}{L}EdA = \frac{E\Delta\theta}{L}\int y^2 dA = \frac{E\Delta\theta}{L}I \tag{6}$$

$$\Delta\theta = \frac{ML}{EI} \tag{7}$$

Where, I =Second moment of area.

Equation (7) represents the deflection of the beam with length L and applied moment M.

Fig.3 illustrates the cantilever beam with applied force. In this case one needs to integrate equation (7) with respect to the length of the beam. Applied moment becomes a function of length [6].

$$M(x) = F(L - x) \tag{8}$$

$$\Delta\theta = \int \frac{M}{EA} dx = \frac{F}{EA}\int (L - x)dx = \frac{FL}{EA}\left(x - \frac{x^2}{2L}\right) \tag{9}$$

Equations (7) and (9) represent beam deflections for two common load types. In general we need to derive Δθ term for each particular loading. Equations (10) – (12) show this dependence.

$$\frac{d^2 y}{dx^2} = \frac{d\theta}{dx} \tag{10}$$

$$\frac{dy}{dx} = \Delta\theta \tag{11}$$

$$y = \int_{x_1}^{x_2} \Delta\theta dx \tag{12}$$

Integrating Δθ in order to find beam deviation. In case of a cantilever beam,

$$y = \int_{x_1}^{x_2} \Delta\theta dx = \int_0^L \frac{FL}{EA}\left(x - \frac{x^2}{2L}\right)dx = \frac{FL^3}{3EI} \tag{13}$$

Rod is considered cantilever beam fixed at one end.

Further, deflection can be calculated by using below equation:-

$$\delta_m = \frac{F \times l^3}{3E} \tag{14}$$

E = 2×10^5 N/mm^2

L = 600 mm (Stroke)

Allowable Deflection: - 0.3 mm

$$I = (10 \times 600 \times 600 \times 600)/(3 \times 2 \times 10^5 \times 0.3)$$

= 12000 mm^4

$I = \frac{\pi}{6} \times d^4$

= (12000 × 64) / 3.14

d^4 = 244585.98

d = 22.23mm ≈ 22.5mm

Diameter of Cylinder Rod: - 22.5 mm

With the help of cylinder diameter (part catcher) the author has selected pneumatic cylinder of Festo Company which is shown in fig.4.

Part catcher will catch the object which is pushed by the spring when chuck is opened. Work holding device is fitted in chuck.

The side view of CNC pick and place system consists of four pneumatic cylinders is displayed in below fig. 5.

CONCLUSION

With the help of robotic arm the pick and place time of the material is reduced from 12 sec to 7 sec.

FIGURES

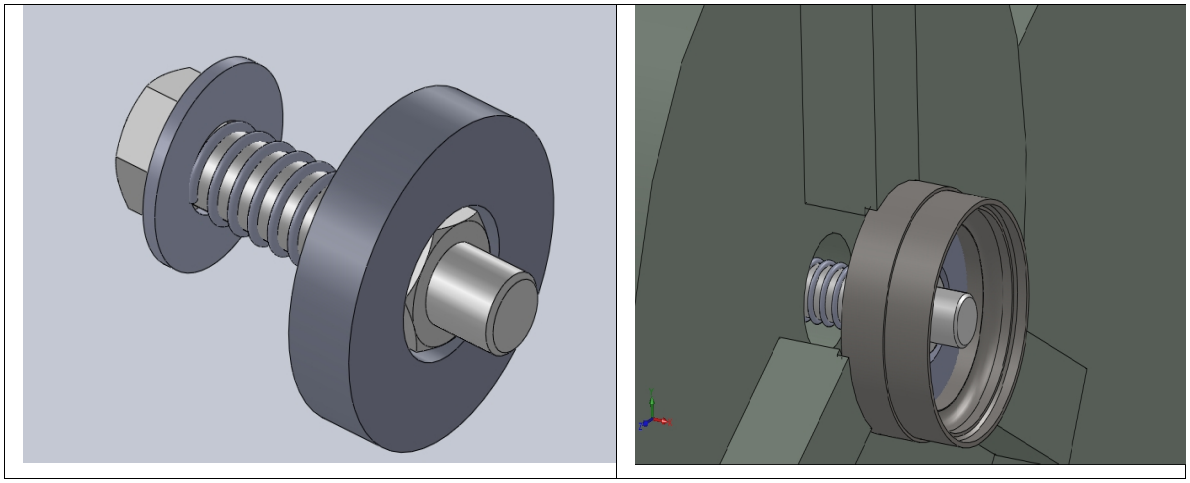


Fig. 1. 3D Model of (a) Mechanism for holding device (b) CNC turning centre with holding mechanism and work piece

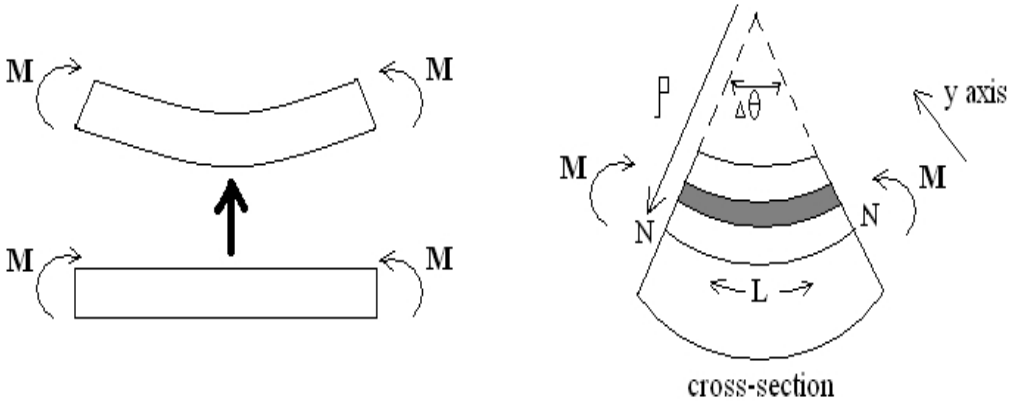


Fig. 2. Geometry of a beam undergoing pure bending by two applied moment*(5)*.

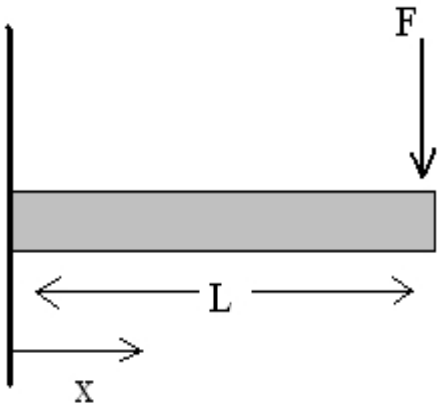


Fig.3. Cantilever beam with applied force

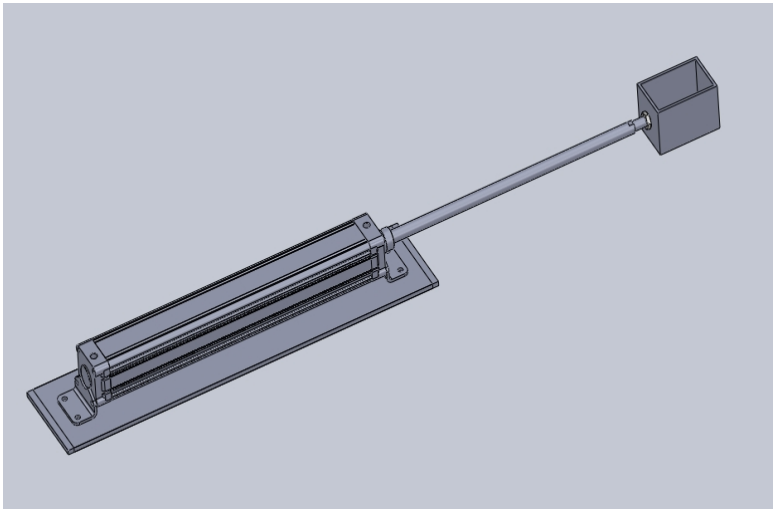


Fig. 4. Part catcher

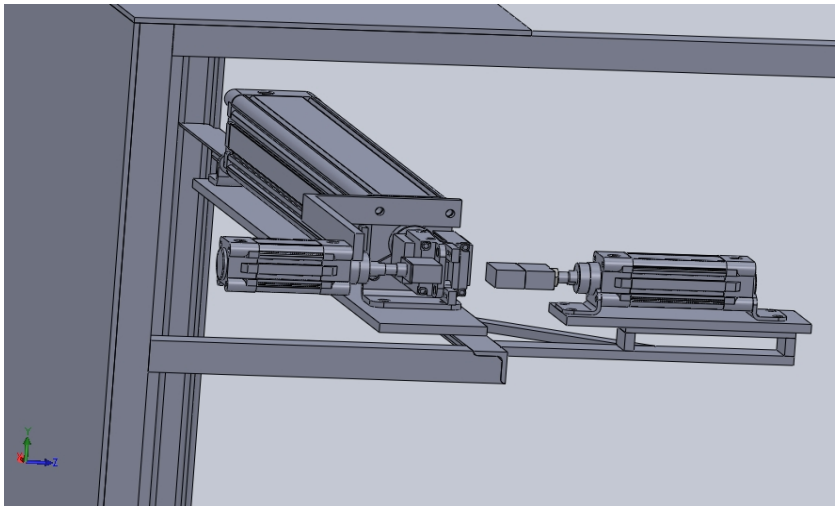


Fig. 5.CNC Pick and Place System

ACKNOWLEDGEMENTS

Authors are thankful to the owner of Industrial Automation, Rajkot for work permission, technical assistance and help. Further authors acknowledge the motivation provided by their parent institute authorities.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

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EXPERIMENTAL STUDIES OF OGEE WEIR AND PIANO KEY WEIR TO INCREASE THE DISCHARGE CAPACITY OF SPILLWAY

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ABSTRACT

Dams are an important tool to measure the infrastructural and technological development of a country. As on today, in India most of dams are approximately 30 to 40 years old. Rehabilitation has become necessary for safety purpose of older dam. Storage capacity of this dam is decreasing due to gradually increasing siltation. Most of the time spillway capacity has been increased in rehabilitation work. To investigate some pragmatic solution to increase the discharge capacity of a dam is the ultimate aim of this study. With a view to achieve that goal, Piano Key Weir has been selected. Hereunder, three different models of Piano Key Weir were selected. On comparing with the Ogee Weir it was found that Piano Key Weirs have more discharge capacity for a given working head.

SUMMARY

To investigate practical solution increases the discharge capacity of spillway.

Keywords: Piano Key Weir, Ogee Weir, Standing Wave Flume.

1. INTRODUCTION:

Most of existing free fall spillway have standardized shape (Ogee Weir) and are provided for concrete gravity dam structure. The main disadvantage is their low specific discharge capacity which is near to 2.2

$H^{1.5}$cu.m/sec/mt., where H is the head on crest in meters.So far as protection against probable maximum flood is concerned and to ensure safety of dam, rehabilitation work have been carried out to increase discharge capacity.

The Aim of this study is to carry out research for pragmatic solution to increase the Discharge Capacity of the Dam. In this respect Piano Key Weir was selected to increase the Discharge Capacity of Spillway. Downstream conditions were not taken in consideration because of our main aim is to increase the discharge capacityof spillway. A Piano key weir is most suitable for free fall weir.

1.1 GENERAL IMFORMATION FOR OGEE WEIR AND PIANO KEY WEIR:

1.1.1 OGEE WEIR:

The shape of Ogee weir profile follows the lower nappe of water jet of the Sharp crested weir. The crest resistance decrease the discharge capacity of weir at head lower than design head. The discharge at a head more than design head, the lower nappe of water jet go away from the profile of spillway which result in negative crests pressure. Cavitations are generated due to large negative pressure. The discharge capacity of ogee weir is given by,

$$Q=\frac{2}{3}C_d\sqrt{2g}\,L\,H_t^{3/2}$$

Where, L is crest width, Q is total discharge, H_t is total head (including velocity head) upstream from crest, g is gravitational constant, and C_d is Discharge co-efficient.

1.1.2 PIANO KEY WEIR:

A Labyrinth weir had been modified with a new concept similar to Piano Key viewed from top, this new concept was called Piano Key Weir (Ouamane and Lemperiere 2003). It was developed specifically for smaller structure footprint application. In Piano Key Weir inlet key and outlet key are over hanged on both sides. So it is structurally balanced.

This innovative Piano Key Weir has a considerably higher specific flow. A Piano Key Weir can increase storage and discharge capacity as well as the flood control efficiency of existing and new dams. The sloping floor of piano key weir provides passage for sediment from the reservoir area is anadditional benefit to decrease the siltation.

The Piano Key Weir crest length is 3 to 5 times more than that of Ogee Weir. The discharge capacity of Piano key weir differs with head and it is approximately 1.5 to 2.5 times more than that of Ogee weir.Piano key weir can also be used to increase the storage capacity by raising spillway crest without increasing submergence of U/S of weir. Piano key weir is the best solution where the spillway length is constrain or for the rehabilitation of existing dam. A Piano Key weir can pass same discharge at low head compare to Ogee weir.

For better understanding of the differences in discharge vs. Head relationships of Piano Key Weir and Ogee weir, the following study was undertaken.

2. EXPERIMENTAL SETUP:

For the experimental work, one ogee weir and three different Piano key weirs were selected. All models were prepared by using 1:35 scale.

2.1 TESTING FLUME DETAIL:

All Weirs were tested in 85 cm wide and 150 cm deep rectangular flume. Acrylic side wall provided for visual observation .Water enters the flume through calibrated standing wave flume (SWF) flow rate control by gate provided on channel.
A pointer gauge with least count 1 mm was used to measure head. Point gauge was mounted on top of flume. Flow rate measurement over standing (SWF) by using pointer gauge mounted on measuring tank near SWF. Measuring tank connected with SWF by pipe. So water level in SWF and measuring tank was at same level.

2.2 DESIGN OF MODEL:

All weir models were fabricated by using 1:35 scale and 8 mm thick acrylic and plywood sheeting. Geometric data of Piano Key weir isshownTable no 2.

2.3 TESTING PROCEDURE:

First of all, model was set in flume and leak test was carried out to check all joints were water tight. The model result was collected for discharge ranging from 8.41 $m^3/s/m$. to 69.30 $m^3/s/m$. Discharge were measured by using the calibreted rectangular SWF. Water level had been maintained to stabilise for a minimum five minute. To varify that stable flow condition had been achived, then readings were taken at various chainage on upstream and down stream weir. A spread sheet was used to calculate the total head and weir discharge co-efficient (Cd) at various flow rate.

3. RESULTS AND DISCUSSION

In first phase of experiment, A Ogee weir model study was carried out at various discharges and head on crest was measured at various chainage. In second phase of experiment, three Piano key weir models study was carried out at various discharges. The discharge capacity of Piano key weir depends on the ration of total crest length of Piano key weir to spillway width. Model PK1 M1 is more efficient for low discharge up to $20m^3/s/m$. At higher discharge, local submergence conditions at upstream side of outlet key decrease the capacity of outlet key to collect the flow from inlet key. The outlet key width influence on discharge capacity of Piano key weir.

4. CONCLUSION:

The efficiency of Piano key weir from various model studies had been discussed. From the model studies, conclusion can be made as under:

- The Piano key weir is very effective for low discharge. If we increasing hydraulic head on crest, the efficiency of Piano key weirdecreases.
- The most important parameter influencing the capacity of Piano key weir is the ratio of total Crest Length of Piano key weir to spillway length (L/B).

- Model PK1 M2 is more efficient up to 33.62 m^3/s/m discharge.
- Model PK1 M1 is more efficient from 33.62 to 46.23 m^3/s/m discharge.
- Model PK1 M3 is more efficient for 58.54 m^3/s/m discharge.
- By using the Piano Key Weir, we can decrease the head up to 30 to 50 %.
- Results show that discharge capacity of Piano Key Weir compared to Ogee weir is 1.5 to 2.5 times with same head.

5. FIGURES:

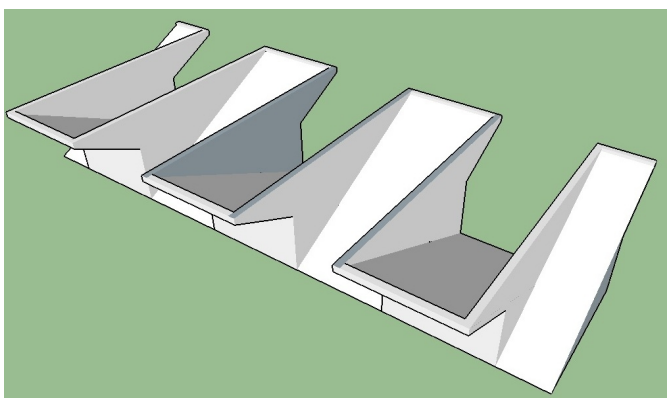


Fig. 1 PK Weir model

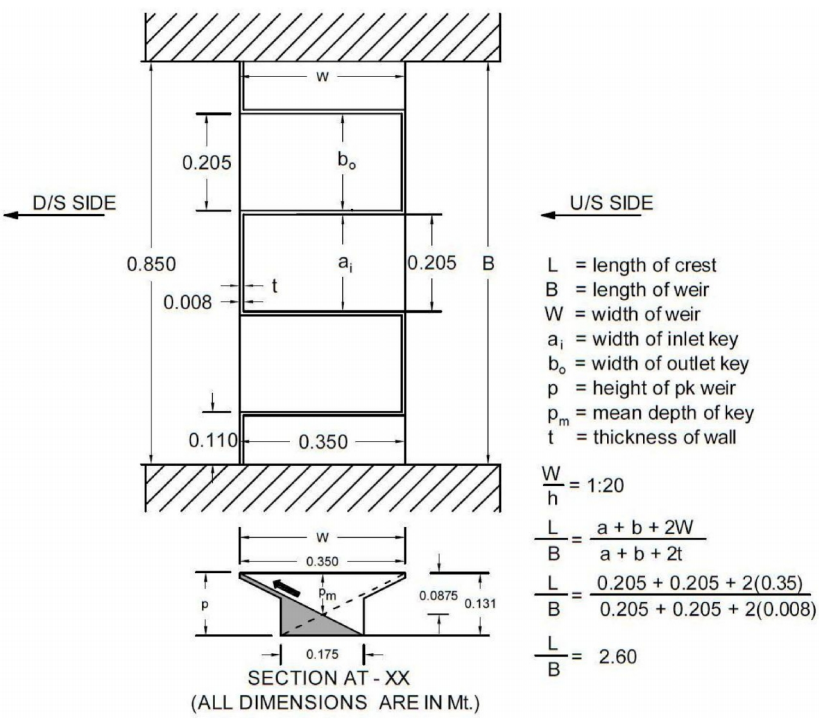


Fig. 2 PK1 M3 Model Weir detail

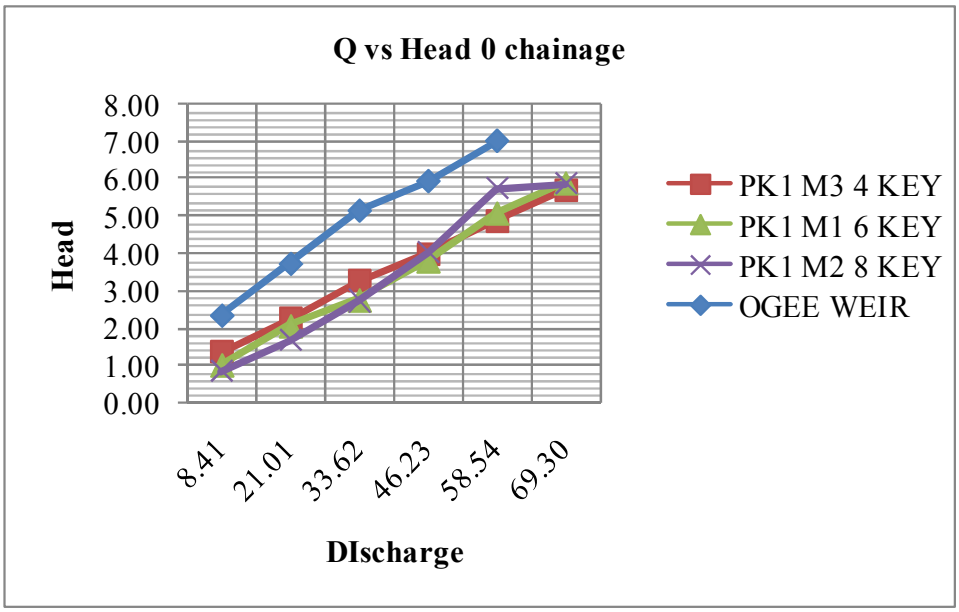


Fig. 3 Discharge vs. Head at 0 chainage for PK1 Model

6. TABLES:

Table 1. Head on crest at 0 Chainage.

Discharge in cu.m./s/m	Head on Crest						
	OGEE Weir	PK 1 M3 4 KEY Head in mt.	PK 1 M3 4 KEY Saving in Head (%)	PK 1 M1 6 KEY Head in mt.	PK 1 M1 6 KEY Saving in Head (%)	PK 1 M2 8 KEY Head in mt.	PK 1 M2 8 KEY Saving in Head (%)
8.41	2.31	1.33	42.42	1.01	56.28	0.83	64.07
21.01	3.71	2.21	40.43	2.06	44.47	1.64	55.80
33.62	5.15	3.26	36.70	2.76	46.41	2.72	47.18
46.23	5.95	3.99	32.94	3.81	35.97	4.02	32.44
58.54	7.04	4.86	30.97	5.10	27.56	5.77	18.04
69.30	-	5.71	-	5.91	-	5.88	-

Table 2. Geometrical Characteristics of the studied PK Weir (all dimension are in cm.)

Model No.	Scale	L/B	bo/ai	P	Pm	ai	bo	W	B	S in.	S out.	t
PK1 M3 4 KEY	0.07	2.60	1.00	13.10	8.75	20.50	20.50	35.00	85.00	0.50	0.50	0.80
PK1 M1 6 KEY	0.07	3.42	1.00	13.10	8.75	13.30	13.30	35.00	85.00	0.50	0.50	0.80
PK1 M2 8 KEY	0.07	4.23	1.00	13.10	8.75	9.80	9.80	35.00	85.00	0.50	0.50	0.80

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RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

Comparative Study for Pre and Post Reform Performance of Gujarat Power Distribution Sector

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ABSTRACT

In past three decades, the Electricity Industry throughout the world is undergoing restructuring and adopting the deregulated structure for better utilization of the resources and for providing choice and quality services to consumer at economical prices by improving the efficiency in the operation of the power system.

This paper examines the impact of governance reforms on efficiency, performance and service delivery in order to identifying the factors responsible for the success of reforms in the power sector in Gujarat. The study is aimed to know the gap between ARR & ACS and generation capacity for pre reform period, thrust for reform and to evaluate the business parameters of the distribution company like distribution losses, AT&C losses, collection efficiency, consumer mix, sales mix for the pre and post reform period.

SUMMARY

By the process of reform implementation, Gujarat power distribution sector has achieved remarkable improvement in performance.

Keywords: G.E.B., Distribution Reforms,Gujarat Power utility,Power distribution

INTRODUCTION

Along with the formation of Gujarat State in the year 1960, the Gujarat Electricity Board (GEB) was established under Section 5 of the Electricity (Supply) Act 1948. The generation capacity was 315MW and a consumer base was 1.40 million consumers and nearly 50000 employees working with the organisation. The GEB bearing investment of billions of rupees. In the first two decades after establishment the prime concern of the GEB was on electricity in the rural areas, resulting Gujarat became the first state to achieve the landmark of '100% Electrification of Villages'.

As per the 1991 Census, 17,940 out of 18,028 villages were electrified -which was notified as close to 100 %. *(1)*

Before the reformation i.e. during 1992 to 2000-01, GEB was facing huge financial losses and spoiling the image among the public due to unsatisfactory services. The first step for the reformation was initiated in the FY 2001-02 as a corrective measure like renegotiation of power purchase agreement, curtail the rate of interest on loans, detection of malpractice and mischief with power, reduction in T&D losses prior to unbundling the GEB. The government of Gujarat has implemented reformation process by unbundling the GEB in seven companies.

MATERIALS AND METHODS

For performing exploratory study, primary data was collected by experience survey and the secondary data was collected for the study from reports, published government documents, newspaper clippings, websites, books, journals and magazines. The supportive reports and presentations and meeting proceedings collected from holding company i.e. Gujarat Urja Vikas Nigam Ltd (GUVNL).The reports made available from GERC website like RIM and SOP as well as Annual reports of discoms' and GUVNL. The MYT submitted by DISCOMS' and Tariff order and Petitions from gerc website utilised for gathering the data.

RESULTS AND DISCUSSION

LITERATURE REVIEW

(Baijal, 1999) summarises the experience of many countries on power sector reforms. Further, he appeals to government to retain its role as a policy maker. As urged by him to set-up national and state level regulatory commission to supervise the working of the different players. As conclude by him, unbundling of SEBs would allow the formation of transmission, distribution and generation Companies.

(Morris, 2000) makes an appeal to address the various issues of power sector reforms such as enormous leakage of revenue of the SEBs, privatisation of distribution and generation of electricity business and changes in the institutional mechanism for the success of power sector reforms. He emphasizes on a need to consider a financial capacity of the state and SEBs before signing an agreement by IPPs. Further, he points out that hastily crafted structure of IPPs and fast track power generation projects have further increased the financial burden on SEBs.

Confederation of Indian Industry (CII) — *(A.T. Kearney Report, 2009)* "Sustaining Growth–

Future of Indian Power Sector" Message from R S Sharma Chairman - CII's National Committee on Power and Chairman and Managing Director NTPC Ltd. A robust and thriving Power sector is central to India's sustained economic growth India's power sector has responded strongly to the reform measures undertaken by the government with a wide spread participation across Public and Private sector, Indian and multinational companies

(Madhavan,2012) before 2001, GEB was passing through the huge financial loss facing dissatisfaction from the consumers, hated by farmers, full of mismanagement, bureaucracy, huge line losses, heavily accumulated debt, political interference in day to day working, difficulties in implementation due to wrong vertical structure- all these collectively brought GEB on the verge of bankruptcy.Year 2012 onwards an unbundled GEB – GUVNL and subsidiaries became a model public utility having clear vision, mission and core values. Excellent in services by adopting modern technology and IT enabled services. The GUVNL and subsidiaries won several global awards for competency, achieved award for best distribution utilities (PGVCL) and A^+ rating.

OBJECTIVE OF STUDY

The Gujarat power sector has achieved remarkable achievement after 2004-05, i.e. after reforms. How power sector could achieved this is always be a learning steps for other sector of the state.

The objective of the study is to evaluate the Power sector reforms process of Gujarat state vis a vis objective for reforms, early reform actions, comparative study of pre and post reforms business parameters affecting distribution utility like Distribution losses, AT & C losses, Collection efficiency, Consumer Mix and Sales (Revenue) mix

SCOPE OF THE STUDY

The scope of the study is to

1) Review of various performance parameter for pre reforms period of the Gujarat Electricity Board and to identify the reasons for reforms of Gujarat Electricity Board
2) The objectives (reason) for reforms of Gujarat power sector
3) Study & evaluation of implementation process of reforms in Gujarat
4) Impact of Distribution reforms in Gujarat
5) Comparative analysis of Gujarat Distribution sector for pre and post reforms period

1) Performance of Gujarat Electricity Board for pre reform period:

The prime concern and focus of the GEB (during pre-reforms) was to electrify the rural areas by erecting new transmission and distribution lines, supplying quality power to consumers by minimising interruptions and updating the generation capacity. The GEB was unfocused for effective revenue realisation and T&D loss levels. The heavy financial losses caused due to ignorance of revenue aspect and high subsidy level to agriculture consumers.*(4)*

A) Generation efficiency of Gujarat Electricity Board

The strength of the power sector of the state is always being measured by the generation capacity of the generating stations, plant load factor. As shown in *(Fig.1)* the plant load factor remained 60% to 65% on an average from year 1995 to 1999. It indicates the need of improvement in generating efficiency of the generating stations under Gujarat state.

B) Transmission Losses of Gujarat Electricity Board

As sown in *(Fig. 2)* the transmission loss level of GEB was reduced from 7.14% to 5.95% from year 1995 to 1999. High transmission loss indicates the lack of vigilance on high tension consumers, improper maintenance of high tension lines.

C) Distribution losses of Gujarat Electricity Board

The high level of distribution loss directly indicates the high revenue loss. The distribution loss mainly comprising of commercial losses, theft of power, non-metering, non-payments, unbilled cases and illegal use of electricity. Till 1999 the GEB was losing 7272MUs of electricity as a distribution loss as shown in *(Fig. 3.)*.

D) Financial performance of Gujarat Electricity Board

During 2000-01 a revenue realisation of Rs. 0.15/kwh only. This is because of extremely low tariff of 0.5 million agriculture consumers i.e. Rs. 350 per hp per year. To set right the financial deficit minimum RS.2 per unit subsidy was required for each unit power sale to agriculture consumers. The agriculture consumption was boosted from 16.7% of total units sold in the year 1970-71 to 43% in 1999-00. In the year 1999-00 the estimated loss of the GEB was Rs. 14 billion due to the low tariff power to agriculture consumers, lead to larger GAP between ARR and ACS *(3)*.

The gap between ARR and ACS is shown in *(Fig. 4.)***.** The larger gap leads to recurring financial deficits and not able to raise resources for investments. Average Revenue Realisation (ARR) of Rs.2.05/kwh and Average cost to serve (ACS) of Rs.2.49/kwh for pre reforms period was recorded as shown in *(Fig. 4.)*.

E) Power availability to consumer of Gujarat Electricity Board

In the year 1998-99 there was acute shortage of power due to insufficient generation capacity against demand of power, which lead to load shedding between 50 MW to 1,450 MW was experienced on 362 days of the year.*(4)*

F) Political Interference in day to day functioning of the Board

The organisational structure of GEB was overlapping between planning commission and state ministry of power. Due to that many a time interference of either body affected the performance of GEB. The

unpopular activity like detection of power theft and malpractices performed by GEB was directly affecting the public which indirectly invited the pressure from local leaders.

2) Reasons for reforms of Gujarat Electricity Board

Listed below are the main reasons for urgent reforms of the GEB.

1. In adequate generation capacity and poor generation efficiency
2. High level of Transmission and Distribution losses
3. Poor performance in distribution of energy
4. Recurring financial deficits
5. Political interference in day to day functioning of the board
6. Power sector reforms initiation in India

3) Process of Reforms implementation in Gujarat

As per the recognisation in Indian Electricity Act 2003, the electricity industry in gujarat needed to be reformed and establishing the Electricity Regulatory Commission in the state.

On the base of this, the organisational restructuring of the GEB took place by unbundling the vertically integrated structure into seven different companies. *(Fig.5.)*

One for generation and transmission each, four distribution companies (Discoms) and a holding company known as Gujarat Urja Vikas Nigam Limited (GUVNL).

All companies became fully operational from April'05 and started their business activities independently. The power distribution in the cities of Ahmedabad and Surat had been with private sector entity viz Torrent Power through it's subsidiaries as AEC and SEC.

Key features of reform implementation:

A) Full Support from staff

From the initial stage of reforms the extensive support of representatives of the unions and associations of the staff was a favourable feature in the process of reforms as it had been convinced that the GoG and GEB were not pursuing any hidden agenda. During entire process of transition from the GEB to companies no incident of strikes/protests took place from employee.

B) Transition support by the state government

The GoG took over the liability of debt payment of GEB, settled outstanding dues of Rs. 1627.72 crores payable to CPSUs up to September'01 and in lieu issued bonds to these CPSUs.

C) Financial support by the state government

At the time of reform there was liability of approx Rs.624 crore as loan, which had been converted in to equity shares in GUVNL by GoG. For remaining outstanding loan of Rs.842 crore moratorium periods of six years was allowed.*(1)*

Pre reform initiatives in Gujarat

Unlike the other state, Gujarat had started the process of reforms in early 2000 during the GEB days.

A brief of the measures undertaken by GEB is as below.*(5)*

A) Revenue enhancement measures
- Effective monitoring of the revenue situation
- Fixing the performance parameters for review
- Regular and systematic review of performance parameter and strict implementation there of
- Feeder level revenue monitoring by assigning the feeder manager

B) Efficiency enhancement measures

The GEB took massive actions to curb theft of power and dealt the theft and non paid bills cases sternly. The initiatives were like

- Setting up a vigilance department with 500 retired army personal headed by IPS officer to check power offenders
- Incentive scheme for power theft information from the public
- Formation of vigilance squad
- Creation of dedicated police stations at Surat, Baroda, Sabarmati, Rajkot and Bhavnagar which were created only to deal with cases of power and power property theft
- Sealing of Installation to stop the energy leakages

4) Impact of Distribution Reforms in Gujarat

The key areas of focus:

- ✓ Distribution loss reduction
- ✓ Reduction in commercial losses
- ✓ Revenues enhancement measures
- ✓ Enhancement in customer services and satisfaction

✓ DISTRIBUTION LOSS REDUCTION

The distribution loss includes Technical & Commercial loss. The discoms have focused on curbing of power theft, pilfering of installation, strengthening of network and fine tuning of processes and procedures to reduce the distribution loss level.

✓ REDUCTION IN COMMERCIAL LOSSES

Power theft was the only reason of high level of commercial loss. GEB had started actions for massive installation checking and same had been continued by the GUVNL and discoms in Gujarat. The actions were not acceptable and opposed by the people initially. Apart from installation checking other steps to avoid future power theft occurrence following steps were taken by discoms.

1. Replacement of old meters by new meters
2. Strengthening the cash collection services
3. Replacement of bare conductor by Insulated/Aerial bunch conductor to prevent hooking.

✓ REVENUE ENHANCEMENT MEASURES

Following revenue improvement measures were taken by the GUVNL to improve the financial position of Gujarat state's discoms:

1. Reduction in power purchase cost: In the FY 2003-04, savings of Rs 4.95 crore took place due to renegotiation of PPAs. In the subsequent negotiations in 2005-06, they managed to get a further reduction of Rs 64 crore.*(1)*
2. Centralized purchase cell: Formation of CPC under direct control of GUVNL leads to cost effective procurements of materials and inventory planning. This step lead to savings of almost Rs. 137.93crore over the period of 2002-06.*(1)*
3. Releasing new connections: Camps were arranged in poor areas and slums for on spot release of connection. A number of schemes, like TASP (Tribal Area Sub Plan), KutirJyoti, Zupadpatti, were started to provide connections to poor people.
4. Settlement of Old dues: By implementing Voluntary Disclosure Schemes (VDS) and one time settlement scheme were also started to clear old dues. One time settlement scheme was availed by 28793 consumers.*(6)*

✓ ENHANCEMENT IN CUSTOMER SERVICE AND SATISFACTION

At the time of unbundling of GEB, consumers were facing problems like delay in getting new connection, delay in resolving power complaints, inadequate bill details like address, name, etc. As a solution to all these problems Discoms in Gujarat took following measures:

1. Formation of Customer Care Centres (CCC) at all sub-divisions, divisions and circle offices as well as at corporate level for all Discom. These centers took care of all the customer queries related to
    a. Release of new connection
    b. Queries related to wrong billing
    c. Change of consumer master details like name, address, etc. related process
    d. Faulty/ fast meter and other technical parameter related queries
2. Central Trouble Call Management (TCM) centre: Over and above CCC at subdivision level, central Trouble call management center was setup to register and resolve power supply related

complaints through telephone.

3. Private cash collection agencies for bill collection: For providing better facilities to pay the energy bills by the consumers, discoms has started private cash collection centres by outsourcing the contracts, acceptance of payments at post office, Any Time Payment Machine (ATP) for 24Hrs acceptance of payment and online payment facility.
4. Geographical Information System (GIS): The mapping of distribution network and consumer database was done using GIS software. This lead to easy and accurate availability of consumer and network details for attending complains as well as system improvement works.
5. Jyoti Gram Yojana (JGY): The big issue faced by the rural consumers was non availability of power during load shedding hours of feeders. The consumers in rural areas hardly getting power supply for 8 to 10 hours in a day which lead to migration of people from rural to urban areas and non establishment of industries as well as no availability of proper education.

   By implementing JGY scheme the combine feeder catering power supply to villages and agriculture was made separate by installing huge HT/LT networks and transformers. The pilot for the scheme was done in 8 districts in Gujarat in September'03. In October/November'04 the scheme was extended to the entire state after successful completion of pilot.

5) Comparative analysis of Gujarat Distribution sector for pre and post reforms period

Majority of the parameters found improving after reforms took place in Gujarat. The Gujarat having adequate power generation capacity and qualitative services were availed to the consumers of discoms after reforms.

The parameter like generation capacity of electricity, distribution losses, cash collection, AT&C losses and consumer mix of all four discoms after reforms is tabulated below:

1. Generation of Electricity :

As shown in *(Table 1.),* the generating capacity of the Gujarat state found strengthening after reforms. The total Installed capacities of all Sources were 8761 MW at the time of reforms which reached to 19212 MW in the month of March-2015.

2. Distribution losses :

The distribution losses of GEB were 30.64 % *(Table 2.)* at the time of unbundling. After formation of Distribution companies' exhaustive loss reduction efforts were put up as elaborated above and resulting the overall distribution losses of GUVNL were 18.47 % *(Table 2.)* as on March-2015. This

clearly shows the improvement in loss level and realisation of more revenue after reforms. The graphical trend of reduction in distribution loss is shown in *(Fig. 6).*

The distribution losses of all four discoms' also found reducing in the span of time after reforms. *(Table 3.)* Shows the statics of distribution loss of the discoms.

3. Cash Collection :

The Distribution Company collecting the amount of energy bills from their consumers after issuing the Monthly/Bi Monthly bills for use of Electricity. The amount realised against the bills named as collection or cash collection. Non realisation will be booked under Arrears in the book of account. The Gap between Assessment and collection reflects poor collection efficiency which ultimately affects to the AT & C Loss picture of the company.

At the time of reforms the collection amount yearly was Rs.10204 crore (2004-05) which increase to Rs.34548 crore which shows the increase in business by the discoms. *(Table 4.)*
Considerable cash in flow increased in all discoms as shown in *(Table 5.)* and the graphical representation of overall cash inflow is represented in *(Fig.7)*

4. % AT & C Losses :

Aggregate Technical & Commercial loss of the Discom's indicates the overall performance of distribution utility (Billing as well as collection efficiency). The AT & C Loss level was 35.20 % in 2004-05 which reduce up to the level of 19.48 % in the year 2013-14 *(Table.6)* Still the loss level is high but distribution utility had put up all efforts in bringing down in last decade. The data for individual discom AT & C loss represented in *(Table.7).* The trend of reduction in AT & C loss clearly understood from *(Fig.8).*

5. Consumer Mix (Pre and Post reforms- GUVNL) :

The statics shows the 60 % rise in nos of consumers under all categories. *(Table. 8)* represents the consumers of GUVNL (All Discom's)

(Table. 9) represents the consumers of DGVCL, *(Table.10)* represents the consumer mix of MGVCL, *(Table.11)* represents the consumer mix of UGVCL and *(Table.12)* represents the consumer mix of PGVCL.

The category wise nos of consumers increase in all Discom's in last decade. It shows the increase in Business of Discom's after reforms.

RESULTS :

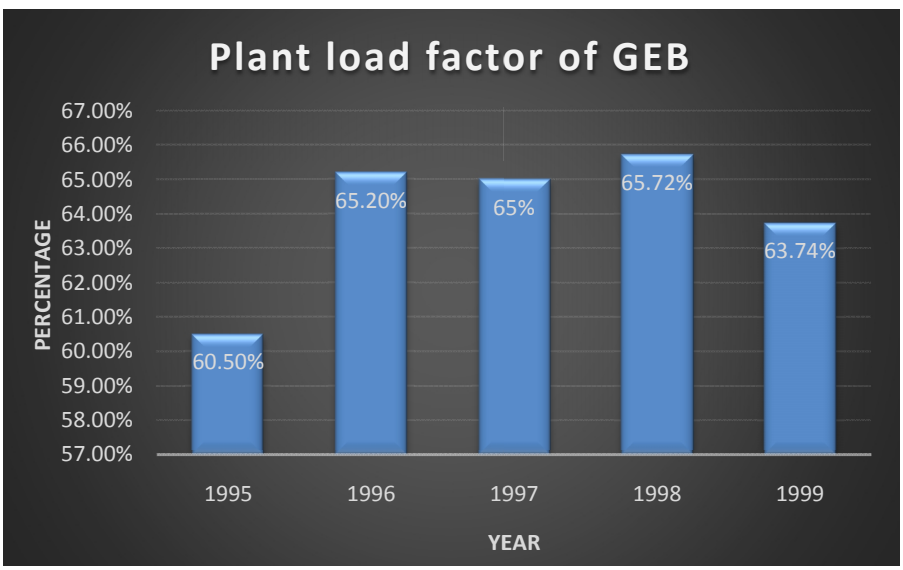
All the business parameters of post reform period indicate the improvement in performance of Power sector in Gujarat.

(Table.13) shows the comparison of parameters for pre and post reform periods. The parameter values reflects the total value for Gujarat (Sum of all discoms)

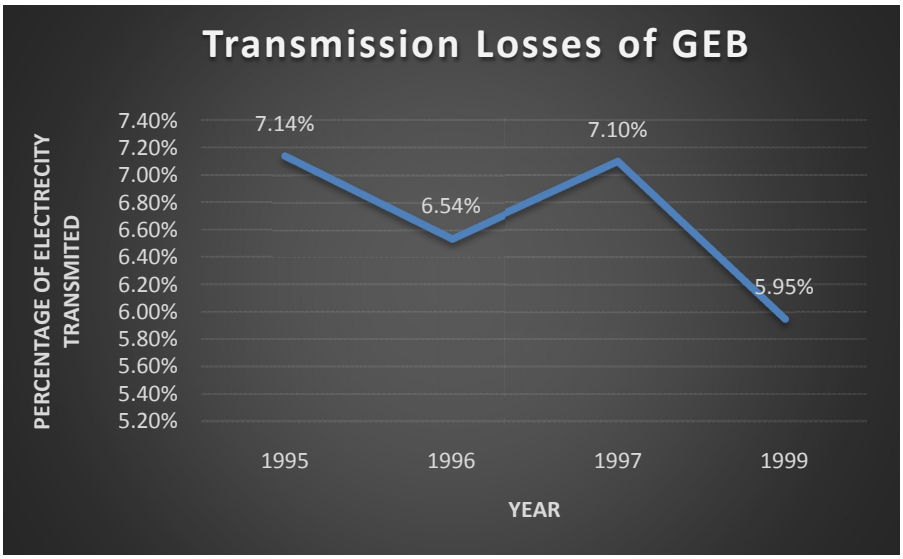
CONCLUSION:

By implementation of reform in power sector-Gujarat, the consumers get benefits in terms of quality power and prompt services. The achievements of all four companies are different because of geographical and other constraint. Still there are scopes for further improvements. On the base of historical data of business parameters, trend of performance of the discoms can be studied further and concrete actions in the direction of better results can be planned.

FIGURES



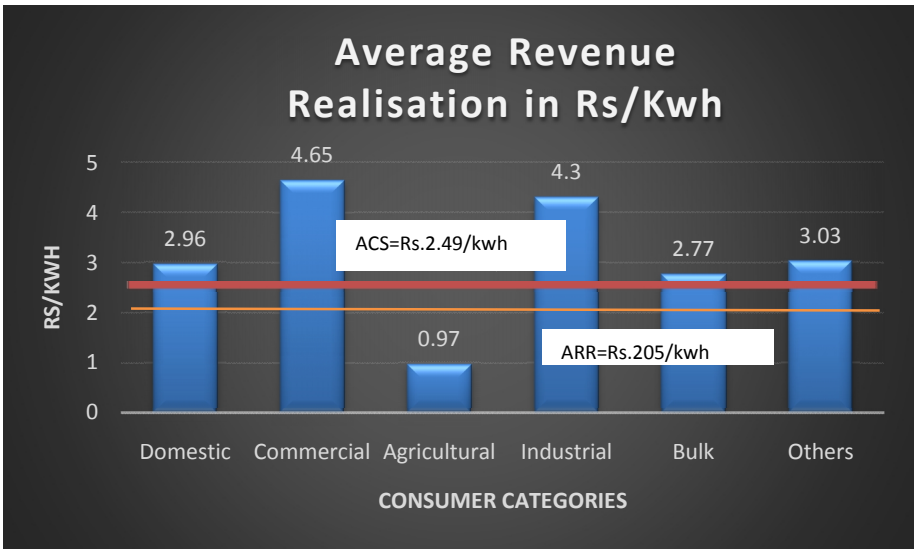
"Fig. 1. Plant Load Factor status of GEB before reformation"



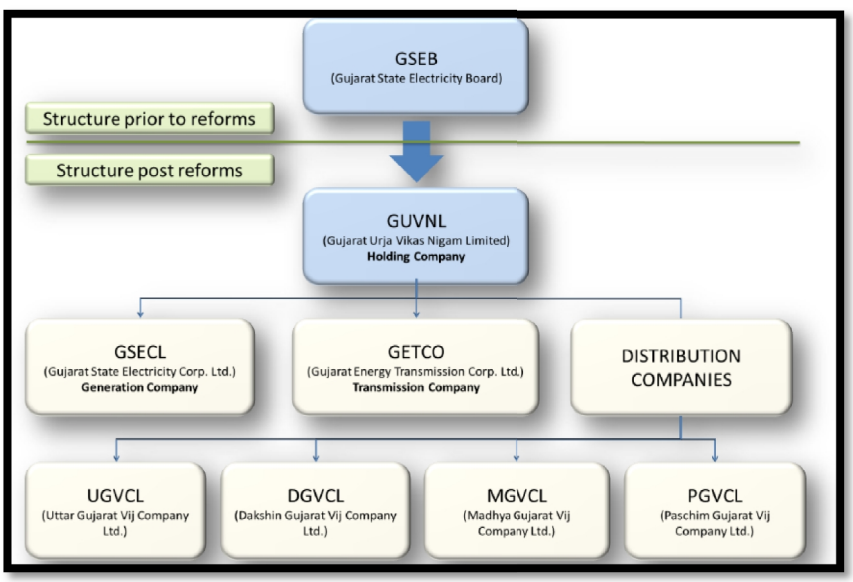
"Fig. 2. Transmission Losses of GEB"



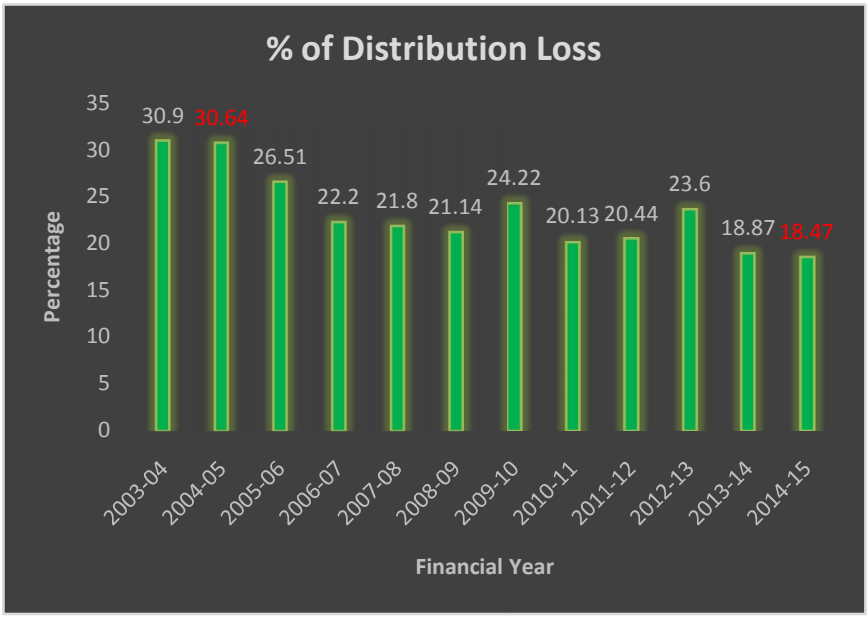
"Fig. 3. Distribution Losses (MUs) of GEB before reformation"



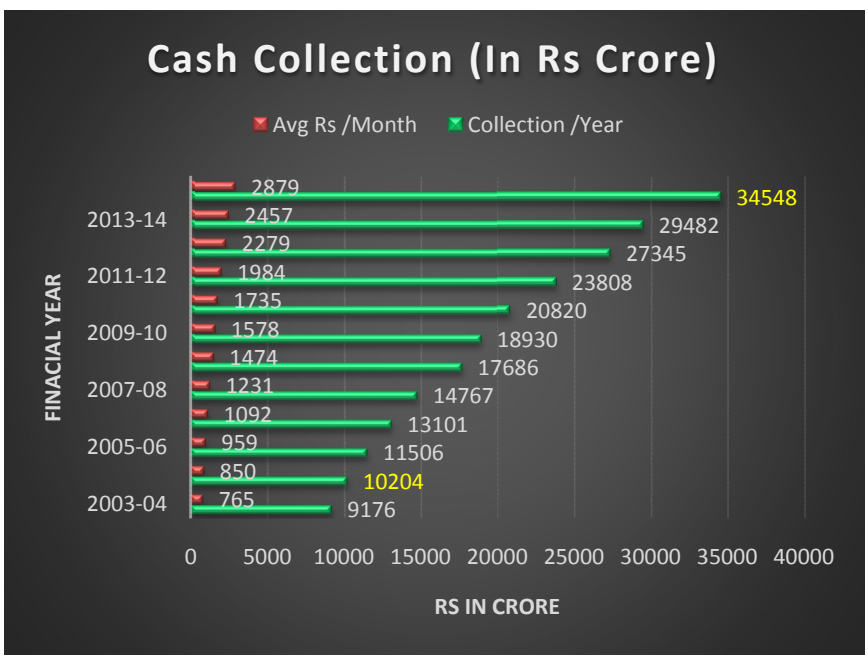
"Fig. 4. Gap between ARR & ACS of GEB before reformation"



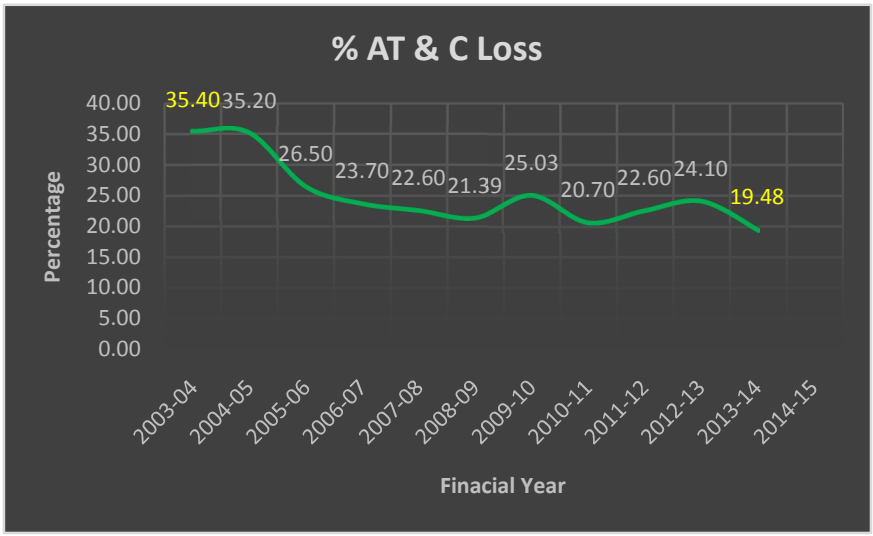
"Fig.5. Structure of GEB before and after reforms"



"Fig. 6. Representation of Distribution Losses Pre and Post Reforms"



"Fig. 7. Representation of Cash collection Pre and Post Reforms"



"Fig. 8. Representation of % AT & C losses Pre and Post Reforms"

TABLES

"Table 1.Sector wise Installed Capacity (MW)" (Source: GUVNL Annual report).*(7)*

Sectorwise Installed Capacity (MW)															
Particulars	**Pre Reforms**					**Post Reforms**									
	2000-01	**2001-02**	**2002-03**	**2003-04**	**2004-05**	**2005-06**	**2006-07**	**2007-08**	**2008-09**	**2009-10**	**2010-11**	**2011-12**	**2012-13**	**2013-14**	**2014-15**
GSECL (GEB+GSECL)	4960	4933	4888	4995	4995	4968	4968	4766	4766	5216	5216	4996	5496	5496	5496
IPPs (Public.Sect)	850	946	946	946	946	956	961	966	966	966	1216	1567	1567	1567	1567
IPPs (Pvt. Sect.)	1210	1210	1210	1210	1210	1210	1455	1455	1455	3102	4102	5563	7607	7607	8309
Central Sector	1568	1568	1538	1538	1610	1843	2177	2310	2677	2724	2820	3180	3600	3840	3840
TOTAL	**8588**	**8657**	**8582**	**8689**	**8761**	**8977**	**9561**	**9497**	**9864**	**12008**	**13354**	**15306**	**18270**	**18510**	**19212**

"Table. 2 GUVNL Dist. Losses" (Source: GUVNL Annual report).*(7)*

Year	% Dist Loss
2003-04	30.90
2004-05	**30.64**
2005-06	26.51
2006-07	22.2
2007-08	21.8
2008-09	21.14
2009-10	24.22
2010-11	20.13
2011-12	20.44
2012-13	23.6
2013-14	18.87
2014-15	**18.47**

"Table 3. DISCOM's Dist. Losses" (Source: GERC website)*(9)*

DISCOM	2005-06	2006-07	2007-08	2008-09	2009-10	2010-11	2011-12	2012-13	2013-14	2014-15
UGVCL	23.0	15.8	17.3	14.61	17.24	10.98	9.81	17.83	11.99	14.26
DGVCL	20.0	16.5	15.5	14.74	15.2	15.95	10.24	15.84	13.83	13.69
MGVCL	20.2	15.1	15.9	13.86	13.84	18.58	12.18	17.59	17.56	17.09
PGVCL	38.7	32.5	32.8	30.68	31.88	29.61	27.87	33.61	27.02	28.66

"Table 4. Cash collection (Rs.Crore)" (Source: GUVNL Annual report).*(7)*

Year	Collection /Year	AvgRs /Month
2003-04	9176	765
2004-05	**10204**	**850**
2005-06	11506	959
2006-07	13101	1092
2007-08	14767	1231
2008-09	17686	1474
2009-10	18930	1578
2010-11	20820	1735
2011-12	23808	1984
2012-13	27345	2279
2013-14	29482	2457
2014-15	**34548**	**2879**

"Table 5. DISCOM's Cash collection (Rs.Crore)" (Source: GERC website)*(9)*

DISCOM	2005-06	2006-07	2007-08	2008-09	2009-10	2010-11	2011-12	2012-13	2013-14	2014-15
UGVCL	2110	2494	2804	3814	4176	6064	6064	7383	8107	8415
DGVCL	3338	3566	3745	4574	4923	6637	6637	7632	8591	11546
MGVCL	1683	1924	2109	2768	2910	4092	4092	4621	4565	5428
PGVCL	2890	3671	4158	5376	5698	8319	8319	10130	10572	11804

"Table 6. % AT & C Losses" (Source: GUVNL Annual report).*(7)*

Year	% AT & C Loss
2003-04	35.40
2004-05	**35.20**
2005-06	26.50
2006-07	23.70
2007-08	22.60
2008-09	21.39
2009-10	25.03
2010-11	20.70
2011-12	22.60
2012-13	24.10
2013-14	19.48

"Table 7. DISCOM's % AT & C Losses" (Source: GERC website)*(9)*

DISCOM	2005-06	2006-07	2007-08	2008-09	2009-10	2010-11	2011-12	2012-13	2013-14	2014-15
UGVCL	23.0	15.8	17.3	14.61	17.24	10.98	9.81	17.83	11.99	14.26
DGVCL	20.0	16.5	15.5	14.74	15.2	15.95	10.24	15.84	13.83	13.69
MGVCL	20.2	15.1	15.9	13.86	13.84	18.58	12.18	17.59	17.56	17.09
PGVCL	38.7	32.5	32.8	30.68	31.88	29.61	27.87	33.61	27.02	28.66

"Table 8. Pre & post Reform Consumer Mix of GUVNL" (Source: GUVNL Annual report). *(7)*

Consumer Category	Pre reforms	Post reforms
	2004-05	**2014-15**
Residential	6400602	10315525
Commercial	947530	79630
LT Industrial	157691	1497713
HT Industrial	5194	12032
Agriculture	664059	1184303
Water works	31793	66581
Street light	18583	30388
Other	43	13
Grand Total	**8225494**	**13186185**

"Table 9. Pre and Post reform Consumer Mix of DGVCL" (Source: GERC website) *(8,9)*

Historical Trend in Category wise Consumers DISCOM : DGVCL											
Category	**2004-05**	**2005-06**	**2006-07**	**2007-08**	**2008-09**	**2009-10**	**2010-11**	**2011-12**	**2012-13**	**2013-14**	**2014-15**
Residential	1239716	1307843	1371201	1472747	1564107	1664164	1795059	1907301	2013974	2121964	2228197
Commercial	173605	189917	201337	212551	223121	233585	246472	252034	11928	12844	13586
LT Industrial	43790	44967	46471	48215	49667	51624	54043	56655	300389	308832	329371
HT Industrial	1799	1773	1827	1932	2060	2256	2419	2608	2803	2970	3156
Agricultural	74917	77184	79101	81279	84317	89080	92135	95177	104646	116541	131941
Waterworks	5780	6329	6759	7373	8315	9879	11438	13056	14813	17081	20008
Street Light	4398	3306	3463	3701	3976	4897	4564	4930	5314	5870	6511
Railway	4	5	5	5	5	5	5	6	6	6	6
Military	0	0	0	0	0	0	0	0	0	0	0
Licencee	1	1	1	1	1	1	1	0	0	0	0
GRAND TOTAL	**1544010**	**1631325**	**1710165**	**1827804**	**1935569**	**2055491**	**2206136**	**2331767**	**2453873**	**2586108**	**2732776**

"Table 10. Pre and Post reform Consumer Mix of MGVCL" (Source: GERC website) *(8,9)*

Historical Trend in Category wise Consumers							DISCOM : MGVCL				
Category	**2004-05**	**2005-06**	**2006-07**	**2007-08**	**2008-09**	**2009-10**	**2010-11**	**2011-12**	**2012-13**	**2013-14**	**2014-15**
Residential	1525144	1575838	1642434	1754484	1955446	2064337	2158484	2254446	2323621	2378126	2408189
Commercial	199725	205057	209188	213892	219412	232418	243560	268520	19364	271387	20905
LT Industrial	21466	21853	22973	23751	24638	25714	26724	6679	262273	7145	273644
HT Industrial	862	872	940	994	1123	1186	1293	1439	1595	1684	1741
Agricultural	56229	57332	58738	60147	62585	67320	69859	75533	82289	94682	110267
Waterworks	6013	6304	6711	7110	7330	8112	9114	10363	11622	13178	15133
Street Light	4416	4469	5501	5733	5878	6049	6472	5856	6133	6424	6662
Railway	6	6	6	6	6	6	8	6	6	7	6
Military	0	0	0	0	0	0	0	0	0	0	0
Licencee	0	0	0	0	0	0	0	0	0	0	0
GRAND TOTAL	**1813861**	**1871731**	**1946491**	**2066117**	**2276418**	**2405142**	**2515514**	**2622842**	**2706903**	**2773871**	**2836547**

"Table 11. Pre and Post reform Consumer Mix of UGVCL" (Source: GERC website) *(8,9)*

Historical Trend in Category wise Consumers							DISCOM : UGVCL				
Category	**2004-05**	**2005-06**	**2006-07**	**2007-08**	**2008-09**	**2009-10**	**2010-11**	**2011-12**	**2012-13**	**2013-14**	**2014-15**
Residential	1450169	1510239	1574925	1727765	1870033	1998494	2134121	2226817	2298488	2368434	2432337
Commercial	179842	188812	198606	210030	221980	236621	253486	264090	19209	19410	20483
LT Industrial	24684	26068	27366	29317	30350	32004	36029	28742	267930	281019	295632
HT Industrial	1212	1292	1445	1599	1734	1867	2082	2268	2551	2754	3001
Agricultural	201974	205478	208184	213639	217668	221836	225754	233396	245079	261281	283395
Waterworks	9466	9957	10455	11037	11703	12282	12994	13650	14511	15412	16540
Street Light	5844	6194	6514	6998	7455	7944	8449	8992	9693	10274	10923
Railway	1	1	1	1	1	1	1	1	1	1	1
Military	19	4	19	19	19	20	20	5	0	4	0
Licencee	4	4	4	4	4	0	0	0	0	0	0
GRAND TOTAL	**1873215**	**1948049**	**2027519**	**2200409**	**2360947**	**2511069**	**2672936**	**2777961**	**2857462**	**2958589**	**3062312**

"Table 12. Pre and Post reform Consumer Mix of PGVCL" (Source: GERC website) *(8,9)*

Historical Trend in Category wise Consumers							DISCOM : PGVCL				
Category	**2004-05**	**2005-06**	**2006-07**	**2007-08**	**2008-09**	**2009-10**	**2010-11**	**2011-12**	**2012-13**	**2013-14**	**2014-15**
Residential	2185573	2251332	2448989	2425826	2566565	2773828	2896718	2987617	3072070	3169624	3246802
Commercial	394358	409830	446593	449142	468382	501466	518134	460768	22552	23340	24656
LT Industrial	67751	70029	75916	75600	77980	80650	83773	158906	568821	583364	599066
HT Industrial	1321	1540	1814	2014	2224	2492	2741	3064	3417	3756	4134
Agricultural	330939	349799	364427	394131	411590	451394	457992	504221	559171	601456	658700
Waterworks	10534	10536	11759	10703	11053	12068	12339	12718	13342	14326	14900
Street Light	3925	3985	4306	4308	4539	4976	5096	5269	5647	6008	6292
Railway	0	0	0	3	6	6	6	10	0	0	0
Military	5	5	6	7	8	8	8	7	0	5	0
Licencee	2	2	1	1	1	1	1	1	0	1	0
GRAND TOTAL	**2994408**	**3097058**	**3353811**	**3361735**	**3542348**	**3826889**	**3976808**	**4132581**	**4245020**	**4401880**	**4554550**

Parameter	Unit	Pre reforms	Post reforms
		2004-05	2014-15
Generation capacity (Installed capacity)	MW	8761	19212
Distribution loss	%	30.64	18.47
Collection Amount	Rs.Crore/Annum	10204	34548
Aggregate Technical & Commercial loss (AT & C)	%	35.2	19.48 (2013-14)
Total nos of Consumer	Nos	822494	13186158

ACKNOWLEDGEMENT

This research paper is made possible through the help and supports from everyone, including family, friends, guide, colleagues, seniors, in essence all sentient beings. The product of this research paper would not be possible without all of them.

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A STUDY TO EVALUATE THE EFFICACY OF BODY'S OWN WEIGHT TRAINING PROGRAM ON LOWER LIMB STRENGTH & FUNCTION IN ELDERLY INDIVIDUALS

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ABSTRACT

BACKGROUND:

Ageing has been affecting all countries unprecedently developing countries. There is remarkable increase in life expectancy of individuals, but it brings alongwith several complications of ageing. The best way to fight this is regular physical exercise which would maintain health & well being.

AIM:

To evaluate the effect of body's own weight training exercise in lower limb strength & functions in older adults.

METHODOLOGY:

60 elderly individuals randomly selected, assigned in 2 groups, Group A (body‘s own weight training), Group B(Non-training group), Evaluation of quadriceps & calf strength & Lower extremity function initially and after 2 months of training program.

RESULTS:

Quadriceps & calf strength analysed using paired t –test for within group & unpaired t test between group. LEFS analysed using Wilcoxon test within group & between group analysis using Mann Whitney U test. Within group analysis showed statistically significant difference, between group analysis were statistically insignificant.

SUMMARY

Own body weight training program effective in improving strength and LEFS score, thus can be recommended to be included in the daily routine of elderly healthy individuals to maintain a healthy living.

Keywords: Keyword 1, Sarcopenia, 2, LEFS, 3, Geriatrics

INTRODUCTION

Global rise in number of older people, particularly developing countries, there seems to be that the older population is increasing much faster than expected, there is rise in human longetivity. Human lifespan is slowly increasing and medical sciences are making many trials to further prolong longetivity. However ageing brings with it physical dependency and poor quality of life.

World Health Organization(WHO) recommends 50 years of age and older as the general definition of an older person, however an elderly can be defined as per three criterias, that is their chronological age, changes in their work style and lastly changes in their capabilities and also their physical features.Ageing is universal phenomenon India is the second largest country in the world, with highest number of people above 60 years.

Geriatric population as the year passes underwent a lot of changes in terms of physical & functional characteristics. It affects almost all the systems of the body, degenerating them, the causes of which are partly understood.

Out of all the system, problems of musculoskeletal system ranks first, that lead to a serious complication of high chances of fall in geriatric population. The reasons behind these are well documented with development of conditions wherein there is great loss of bone & muscle mass. Even very small change in muscle size can make a big difference in strength, thereby balance, postural control & thus impaired function. There is much higher level of loss of strength in lower extremity compared to upper extremity and just little loss of muscle mass is much to a person who has already reduced levels. This leading to frequent episodes of fall in elderly.

However this weak muscle responds very well to the training & thus improving person's ability, independence & functional mobility. Lots of exercises are available to improve strength including aerobic training, resistive training, tai chi etc. Thus there inevitable need of introducing some program to fight against this age related changes.

NEED OF THE STUDY

Resisted Exercises focuses of strength training using external resistance like dumbbell, weight, therabands etc. basing it on 1 RM, but this can aggravate some pre-existing condition like hernia, or some muscle pain. Moreover this type of training many times proves to be expensive, needs a well established set up and also needs detailed assessment & supervision.

Similarly many such exercises has more of disadvantages over and above advantages, increasing risk of problems to geriatric. However Body's own weight training is one of the exercise that would be cheap, safe and doesn't need supervision. They are very easy to perform and has almost nil risk. Exercises can be progressed by working in end ranges, or by doing exercises without support. Moreover they require more flexibility as well as balance, thus it improves this both over and above strength. Since this exercises can be progressed as well as regressed, it can be prescribed to all level of

fitness individuals. These exercises very effectively strengthen core muscles and even alteration in terms of speed or surface quality can be done.

There are very few studies, analyzing efficacy of body's own weight training on lower limb muscle strength & function in elderly population. So, to analyze the effectiveness of this Safest, Inexpensive, and Easiest program in elderly population this study was designed.

AIM & OBJECTIVES

AIM:

To evaluate the efficacy of a training program using one's own body weight on lower limb muscle strength and function in elderly population.

OBJECTIVES:

- To find out the effect on lower limb muscle strength and function in the Training group of elderly population.
- To find out the effect on lower limb muscle strength and function in the Non-Training group of elderly population.
- To compare the effect on lower limb muscle strength and function between the two groups of elderly population.

MATERIALS



"**Fig. 4.1** Materials used in the study"

METHOD

- Study Design: Experimental Study
- Study Setting: Old Age Homes, Geriatrics Health Centers and Ashrams
- Sampling Technique: Simple Random Sampling
- Sample Size: 60 individuals (30 in each group)
- Study Population: Healthy Elderly Individuals (BMI = 18 – 24.9 kg/m^2)
- Study Duration: Training duration – 8 weeks, 3 days per week
- Total Study Duration – 9 weeks

INCLUSION CRITERIA:

- Healthy Elderly Individuals with age of 55 to 70 yrs.
- Individuals from both the genders were included.
- Individuals who were independent in activities of daily living.
- Individuals who want to participate in the study.

EXCLUSION CRITERIA:

- Individuals having any neurological, orthopaedic and/or cardiorespiratory disorder.
- Individuals suffering with any kind of infectious diseases.
- Those with laborious job work.
- Individuals with diabetes and hypertension.
- Individuals with visual and hearing problems.
- Healthy individuals with reduced cognition.
- Subjects who weren't co-operative to the study.

Subject were selected according to the inclusion criteria, 60 individuals were divided into two groups, Group A (body's own weight training – 30 individuals) & Group B(Non-training group – 30 individuals), Evaluation of quadriceps & calf muscle strength & Lower Extremity Function with LEFS scale initially (pre) and after 2 months (post) of training program.

Group A - Training Program The individuals were trained for 8 – 12 repetitions per set, 4 sets per day, 3 times per week with 1 – 2 minute rest between each set.

QUADRICEPS AND CALF STRENGTHENING EXERCISE	PERIOD OF EXERCISE
Basic Chair Squat, **Heel Rising with holding the chair,** **Half Squatting**	**1 – 4 weeks**
• **Heel Rising without holding the chair**	**5 – 6 weeks**
• **Static Lunges,** • **Single Leg Standing, the heel will rise from the ground holding the chair.**	**7 – 8 weeks**

Table 4.1 –Own Body Weight Training Program

Group B: Non-Training Group -The individuals didn't receive exercise training, they were instructed to maintain their routine day today activities.

All the pre and post training measurements were collected and statistically analyzed.



Fig 4.2 –HALF SQUATTING



Fig 4.3 – SINGLE LEG HEEL RISE WITH CHAIR HOLD

RESULTS

Quadriceps & calf strength analysed using paired t –test for within group & unpaired t test between group. LEFS analysed using Wilcoxon test within group & between group analysis using Mann Whitney U test.

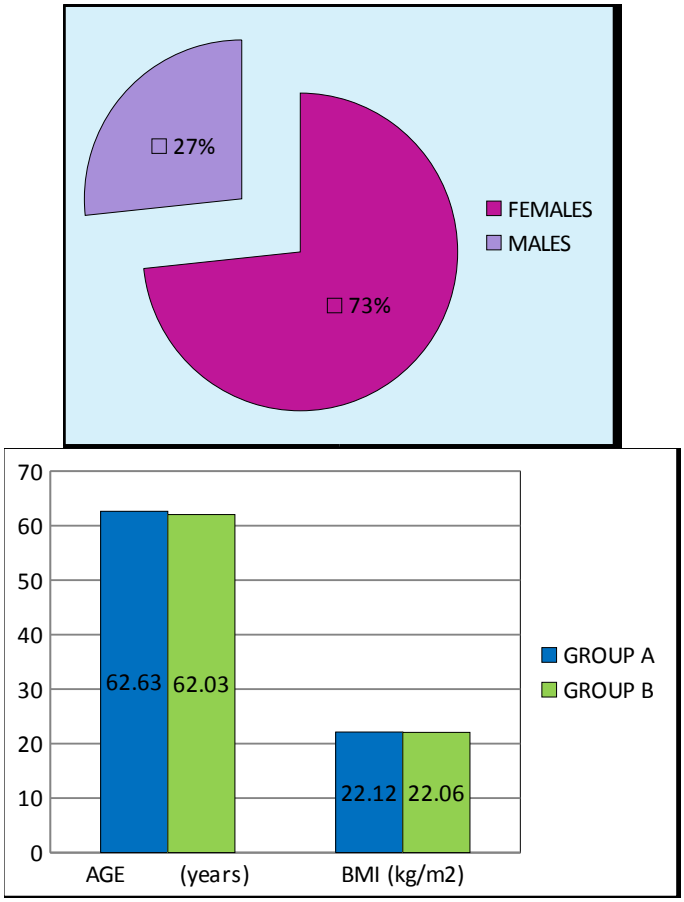


Fig 5.1 - AGE, BMI & GENDER DISTRIBUTION AMOUNG GROUPS

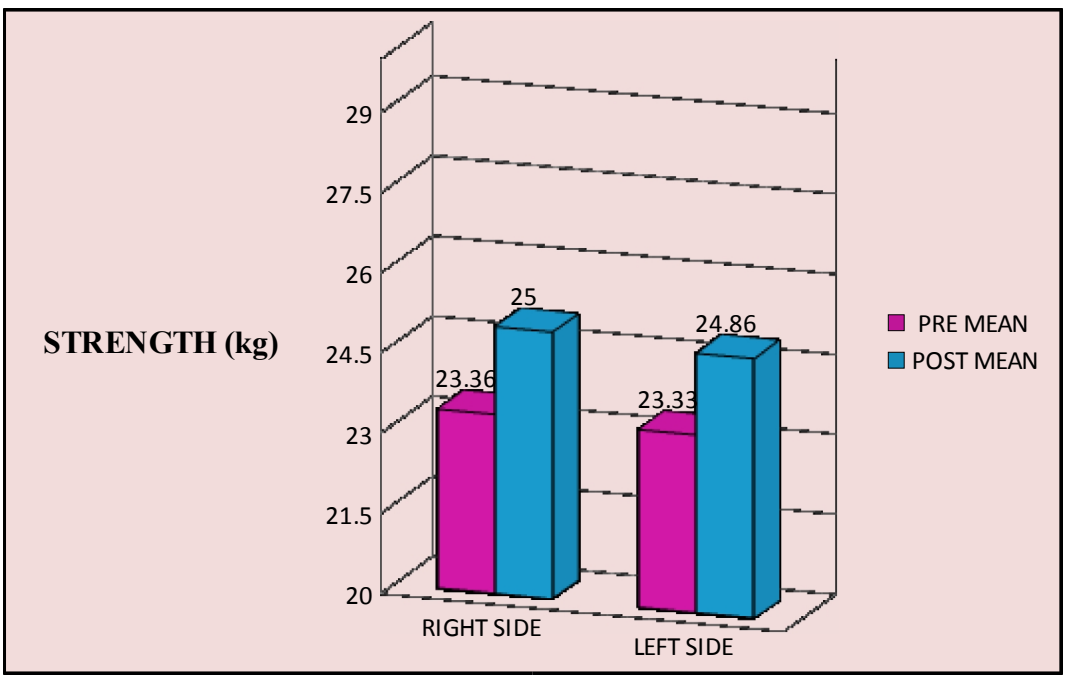
Table 5.1 ANALYSIS OF QUADRICEPS MUSCLE STRENGTH FOR GROUP A.

VARIANT	MEAN		SD		t VALUE	p VALUE	RESULT
STRENGTH	PRE (kg)	POST (kg)	PRE (kg)	POST (kg)			
RIGHT SIDE	23.36	25.00	9.7644	9.5339	9.642	< 0.05	HS
LEFT SIDE	23.33	24.86	9.9354	9.8950	9.761	< 0.05	HS

NS: not significant, S: significant, HS: highly significant

Interpretation:

The above table shows the comparison of pre and post quadriceps muscle strength values of Group A (training group) with pre strength mean = 23.36 ± 9.7644 (SD) and post strength mean = 25.00 ± 9.5339 (SD) for right side.Pre strength mean = 23.33 ± 9.9354 (SD) and post strength mean = 24.86 ± 9.8950 (SD) for left side.The results showed highly significant difference between pre and post quadriceps muscle strength with training group as t = 9.642 (right side) and t = 9.761 (left side) and p < 0.05.



GRAPH 5.2: GROUP A QUADRICEPS MUSCLE STRENGTH MEASURE

Table 5.2 ANALYSIS OF CALF MUSCLE STRENGTH FOR GROUP A.

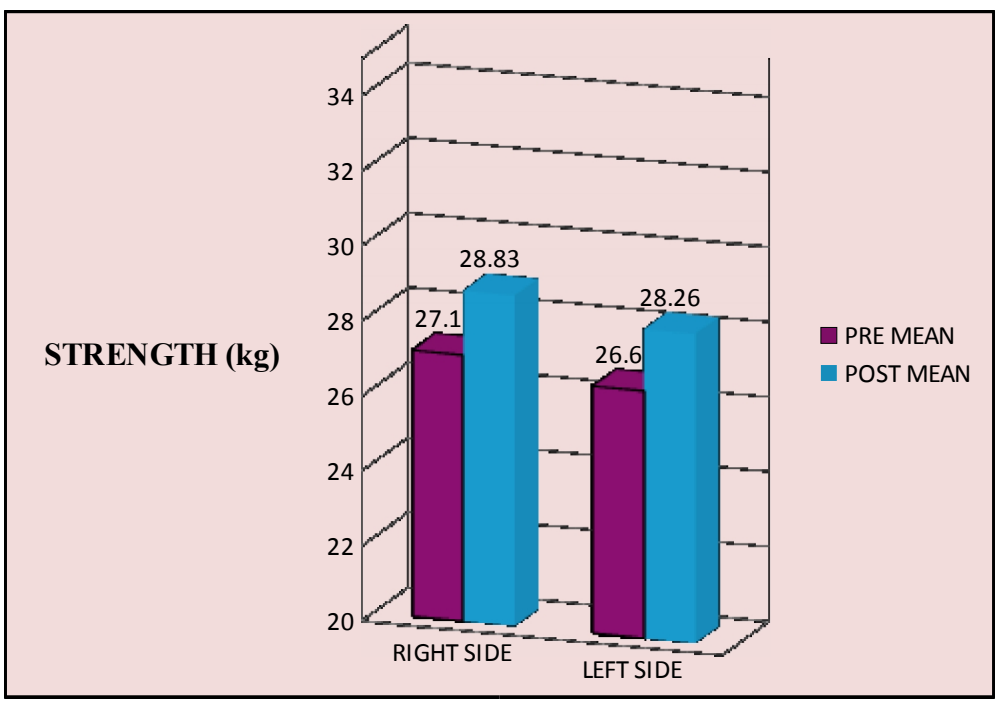
VARIANT	MEAN		SD		t VALUE	p VALUE	RESULT
STRENGTH	PRE (kg)	POST (kg)	PRE (kg)	POST (kg)			
RIGHT SIDE	27.10	28.83	8.6636	8.7022	10.933	< 0.05	HS

LEFT SIDE	26.60	28.26	9.2572	9.3879	10.326	< 0.05	HS

NS: not significant, S: significant, HS: highly significant

Interpretation:

The above table shows the comparison of pre and post calf muscle strength values of Group A (training group) with pre strength mean = 27.10 ± 8.6636 (SD) and post strength mean = 28.83 ± 8.7022 (SD) for right side. Pre strength mean = 26.60 ± 9.2572 (SD) and post strength mean = 28.26 ± 9.3879 (SD) for left side.The results showed highly significant difference between pre and post calf muscle strength with training group as t = 10.933 (right side) and t = 10.326 (left side) and p < 0.05.



GRAPH 5.3: GROUP A CALF MUSCLE STRENGTH MEASURE

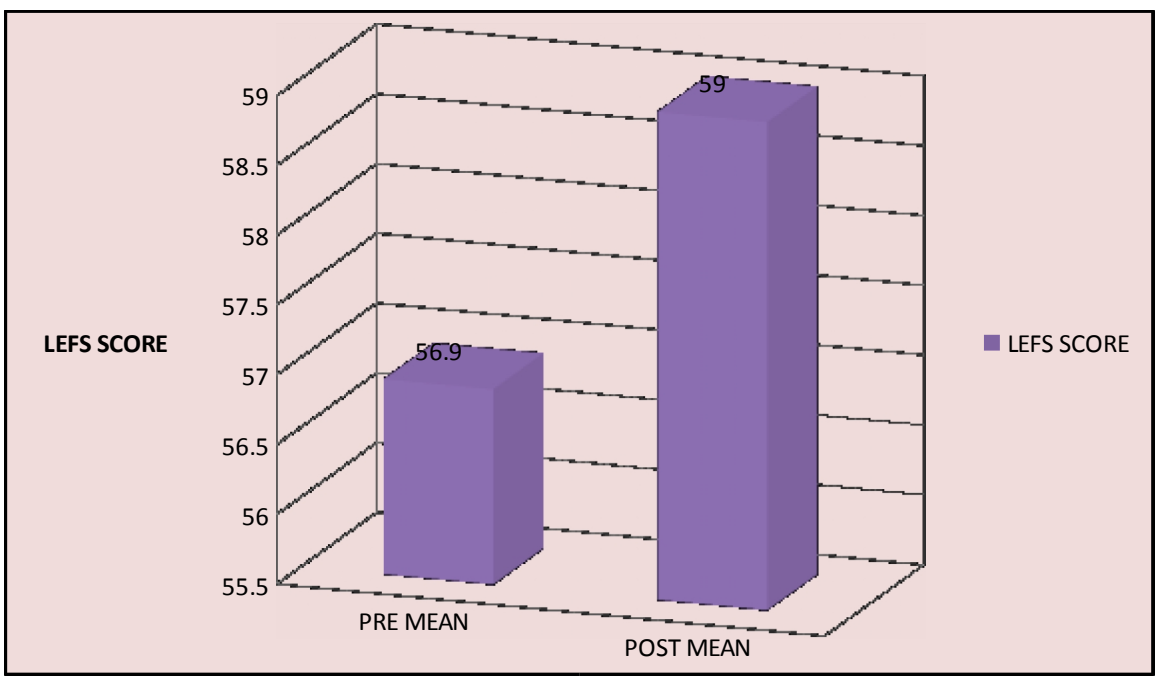
Table 5.3 ANALYSIS OF LEFS FOR GROUP A.

VARIANT	MEAN		SD		P VALUE	RESULT
LEFS	PRE	POST	PRE	POST		
	56.90	59.00	10.79	12.35	<0.05	HS

NS: not significant, S: significant, HS: highly significant

Interpretation:

The above table shows the comparison of pre and post LEFS score values of group pre mean=56.90,pre SD=10.79 and post LEFS mean=59.00 and post SD=12.35.



GRAPH 5.4: GROUP LEFS MEASURE

Table 5.4 ANALYSIS OF QUADRICEPS MUSCLE STRENGTH FOR GROUP B.

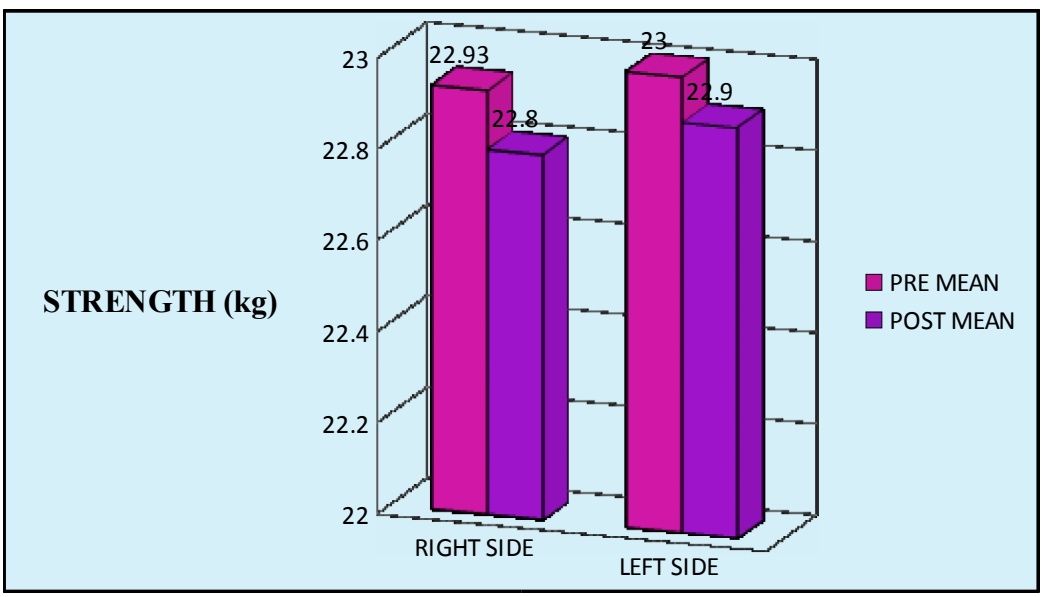
VARIANT	MEAN		SD		t VALUE	p VALUE	RESULT
STRENGTH	PRE (kg)	POST (kg)	PRE (kg)	POST (kg)			

RIGHT SIDE	22.93	22.80	5.3622	5.4355	2.112	< 0.05	HS
LEFT SIDE	23.00	22.90	4.8137	4.7368	1.795	< 0.05	HS

NS: not significant, S: significant, HS: highly significant

Interpretation:

The above table shows the comparison of pre and post quadriceps muscle strength values of Group B (non-training group) with pre strength mean = 22.93 ± 5.3622 (SD) and post strength mean =22.80±5.4355 (SD) for right side.Pre strength mean = 23.00 ± 4.8137 (SD) and post strength mean = 22.9 ± 4.7368 (SD) for left side.The results showed highly significant difference between pre and post quadriceps muscle strength with non-training group as t = 2.112 (right side) and t = 1.795 (left side) and p < 0.05.



GRAPH 5.5: GROUP B QUADRICEPS MUSCLE STRENGTH MEASURE

Table 5.5 ANALYSIS OF CALF MUSCLE STRENGTH FOR GROUP B.

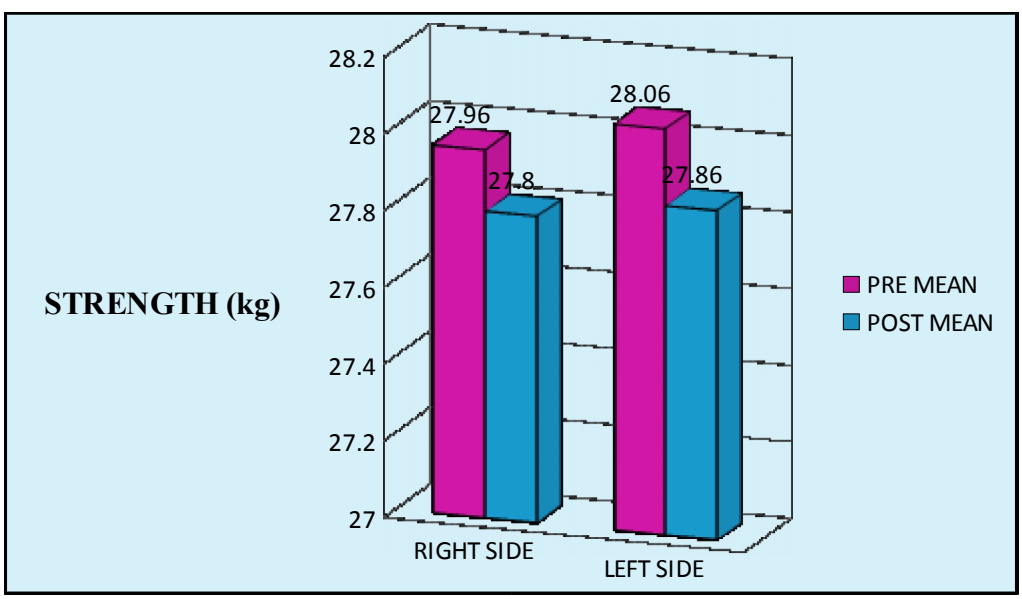
VARIANT	MEAN	SD	t VALUE	p VALUE	RESULT

STRENGTH	PRE (kg)	POST (kg)	PRE (kg)	POST (kg)			
RIGHT SIDE	27.96	27.80	6.5783	6.5516	2.408	< 0.05	HS
LEFT SIDE	28.06	27.86	6.2363	6.2352	2.693	< 0.05	HS

NS: not significant, S: significant, HS: highly significant

Interpretation:

The above table shows the comparison of pre and post calf muscle strength values of Group B (non-training group) with pre strength mean = 27.96 $\pm$ 6.5783 (SD) and post strength mean = 27.80 $\pm$ 6.5516 (SD) for right side.Pre strength mean = 28.06 $\pm$ 6.2363 (SD) and post strength mean = 27.86 $\pm$ 6.2352 (SD) for left side.The results showed highly significant difference between pre and post calf muscle strength with non-training group as t = 2.408 (right side) and t = 2.693 (left side) and p < 0.05.



GRAPH 5.6: GROUP B CALF MUSCLE STRENGTH MEASURE

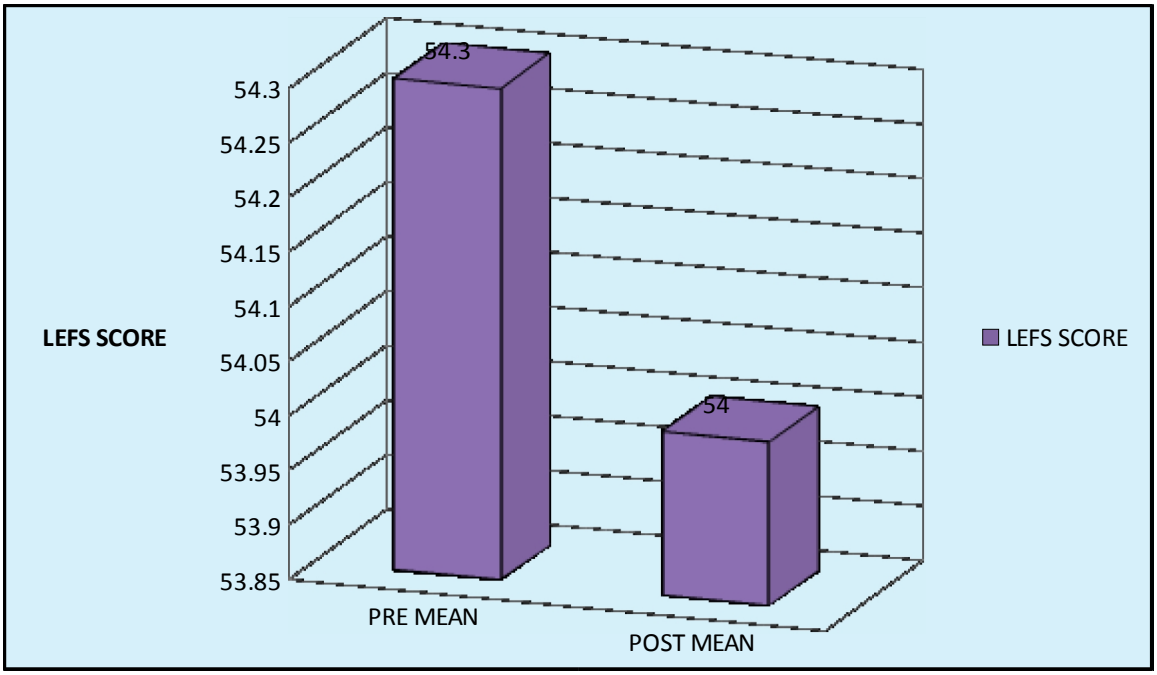
Table 5.6: ANALYSIS OF LEFS WITHIN GROUP B

VARIABLE	MEAN		S.D		P VALUE	RESULTS
LEFS	PRE	POST	PRE	POST		
	54.30	54.00	9.79	5.39	0.0244	HS

NS: not significant, S: significant, HS: highly significant

Interpretation:

The above table shows the comparison of pre and post LEFS score values of group B (own body weight exercise) with LEFS pre mean=54.30,pre SD=9.79 and post LEFS mean=54.00 and post SD=5.39.



GRAPH 5.7 : ANALYSIS OF LEFS WITHIN GROUP B

TABLE 5.7 INTER GROUP ANALYSIS OF QUADRICEPS STRENGTHMEASURE

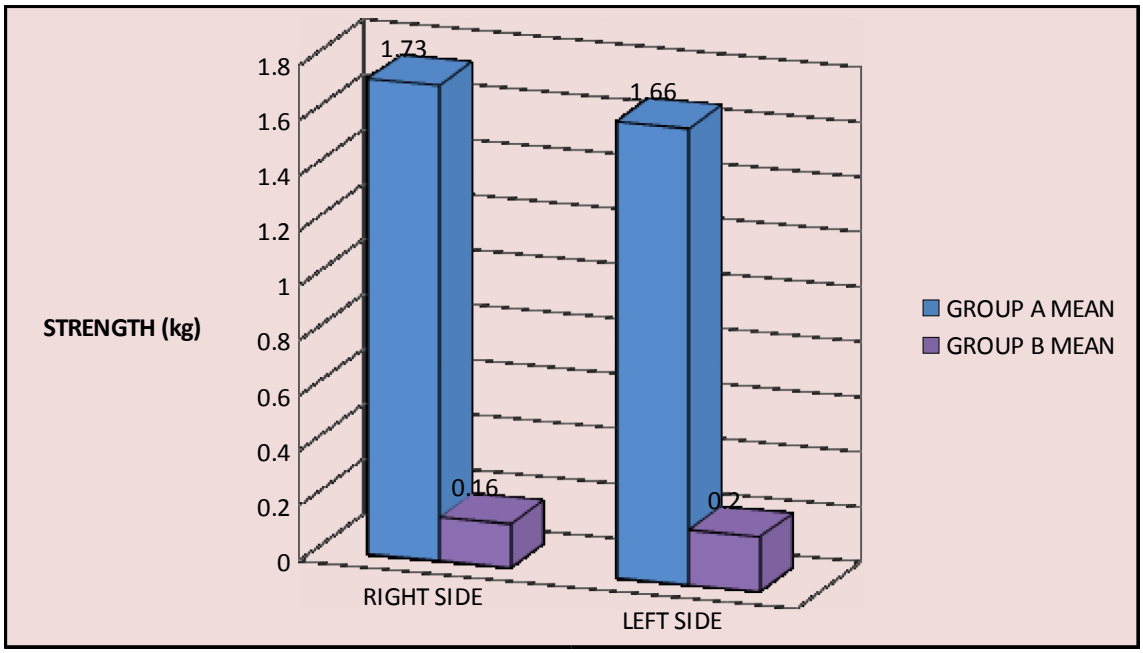
VARIANT	MEAN DIFFERENCE		SD		t VALUE	p VALU
	GROUPS		GROUPS			
STRENGTH	A (kg)	B (kg)	A (kg)	B (kg)		
RIGHT SIDE	1.6	0.13	0.8502	0.3457	8.951	< 0.0
LEFT SIDE	1.5	0.10	0.7303	0.3051	9.919	< 0.0

NS: not significant, S: significant, HS: highly significant

Interpretation:

The above table shows the mean of post test quadriceps muscle strength of Group A i.e. 1.6 ±0.8502 (right side) and 1.5±0.7303 (left side).The mean of post test quadriceps muscle strength of Group B i.e. 0.13±0.3457 (right side) and 0.10±0.3051 (left

side).The result showed significant difference for the quadriceps muscle strength between Group A and Group B as t = 8.951 (right side) and t = 9.919 (left side), and p < 0.05 for both the sides.



GRAPH 5.8: INTERGROUP COMPARISON OF QUADRICEPS MUSCLE STRENGTH MEASURE

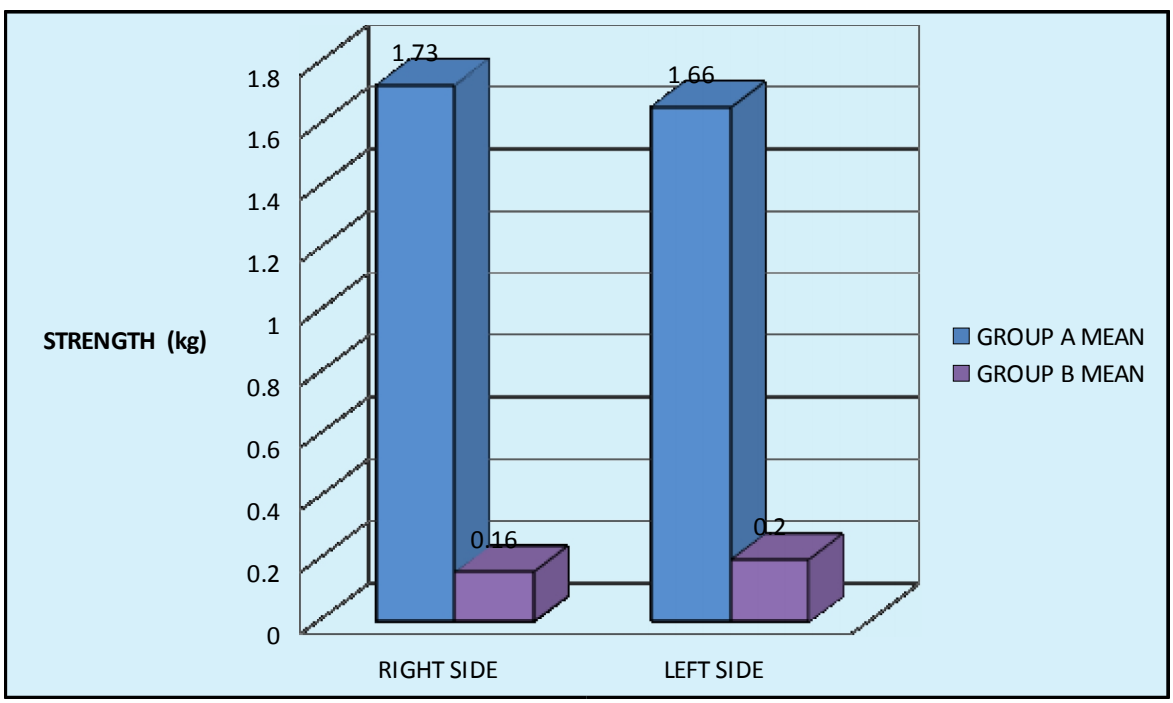
TABLE 5.8 INTER GROUP ANALYSIS OF CALF STRENGTH MEASURE

VARIANT	MEAN DIFFERENCE		SD		t VALUE	p VALU
	GROUPS		GROUPS			
STRENGTH	A (kg)	B (kg)	A (kg)	B (kg)		
RIGHT SIDE	1.73	0.16	0.7396	0.3190	10.324	< 0.05
LEFT SIDE	1.66	0.20	0.8441	0.4068	8.572	< 0.05

NS: not significant, S: significant, HS: highly significant, VHS: very highly significant

Interpretation:

The above table shows the mean of post test calf muscle strength of Group A i.e. 1.73 ±0.7396 (right side) and 1.66±0.8441 (left side).The mean of post test calf muscle strength mean of Group B i.e. 0.16±0.3190 (right side) and 0.20±0.4068 (left side).The result showed significant difference for the calf muscle strength between Group A and Group B as t = 10.324 (right side) and t = 8.572 (left side), and p < 0.05 for both the sides.



GRAPH 5.9: INTERGROUP COMPARISON OF CALF MUSCLE STRENGTH MEASURE

TABLE 5.9 INTER GROUP ANALYSIS OF LOWER EXTREMITY FUNCTIONAL MEASURE

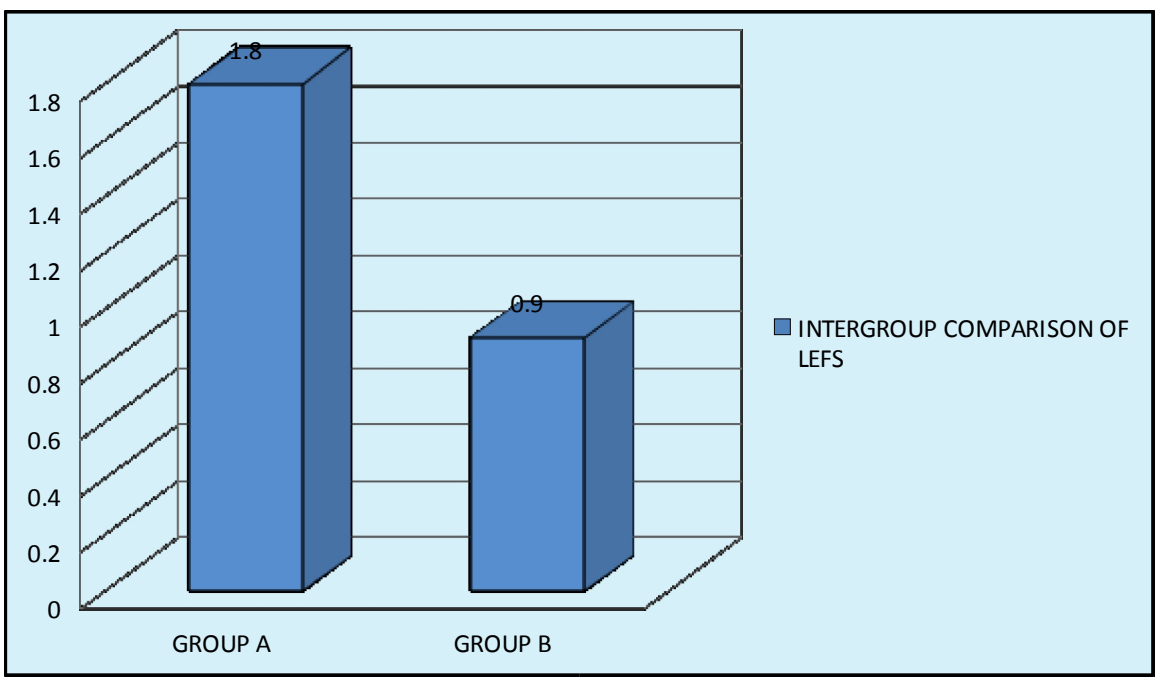
VARIANT	MEAN GROUPS	SD GROUPS	p VALUE	RESULT

LEFS	A (kg)	B (kg)	A (kg)	B (kg)		
	1.8	0.9	3.68	4.52	< 0.05	HS

NS: not significant, S: significant, HS: highly significant, VHS: very highly significant

Interpretation:

The above table shows the mean of post test of LEFS of Group A i.e. 1.8 ±3.68.The mean of post test LEFS score mean of Group B i.e. 0.9±4.52.The result showed significant difference for the LEFS between Group A and Group B as t = 0.289, and p < 0.05.



GRAPH 5.10: INTERGROUP COMPARISON OF LEFS MEASURE

RESULTS

Thus on using paired and unpaired t test, Wilcoxson & Mann Witney U test the results showed that there was improvement in quadriceps strength and calf strength & LEFS test measure in group A (interventional group) at the end of 8 weeks of body's own

weight training program. Whereas group B (control group) this improvement in strength and LEFS test measure was not found.

CONCLUSION

The efficacy of body's own weight training program in healthy elderly population was evaluated. The results showed that there was a significant improvement in Quadriceps and Calf muscle strength and lower extremity functions in 8 weeks training program which would be useful to fight against the loss of muscle and bone mass, and functional mobility impairements that accompanies ageing and thus aid healthy elderly living.

ACKNOWLEGMENT

We would like to thank our parentswho are my living Gods and & family membersfor their valuable support and encouragement, blessing and love which has always been a source of inspiration and strength in accomplishing this task.

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RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

Recovery and Purification of iodine from industrial Waste Liquid

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ABSTRACT

Natural resource of iodine is limited. Iodine is used as catalyst salt like potassium iodine, sodium iodine, trimethyl sulphasoxonium iodine in the synthesis of various drugs like fluconazole, Nebivolol etc. The waste liquor produced from such reaction contains soluble salt of used catalyst. The recovery of iodine is important to maintain the availability of iodine and also decreasing the synthesis cost of drug. Currently some methods are available but it is extremely hazardous. So, our aim is to develop a less hazardous and cost effective method for recovery of iodine from their industrial waste. We have conducted number of experiment by choosing different oxidizing agent alone and in combination. Combination of hydrogen peroxide and fewer amounts of sulphuric acid in cold condition produce optimum result among all experiments.

SUMMARY

This project includes purification of iodine from industrial waste.

Keywords: Iodine purification, recovery of iodine, industrial waste liquid

INTRODUCTION

Physical properties of Iodine:
Iodine is a non-metallic, nearly black solid at room temperature and has a glittering crystalline appearance.

Sources:*(1)*

Iodine is found in the form of iodides in sea-water and in the sea-weeds which absorb the compounds. It is used; in radiation exposure, as a sterilizing agent, as a expectorant, as a fibrocystic agent, diagnosis of hyperthyroidism, in goiter, grave disease etc. ***(2)***

MATERIALS AND METHODS

2.1. Method (sulphuric acid) *(3)*

KI (10 mL, 10 %w/v) + concentrated H_2SO_4 (13 mL) → black precipitate of Iodine

Procedure: 10 g Potassium Iodide was taken in 250 mL conical flask and 100 mL of distilled water was added. From this solution, 10 mL (10% w/v) was taken and drop by drop concentrated sulphuric acid was added till the color of solution changed from clear colorless to yellow and black precipitates of iodine observed.

Drawbacks

In this method concentrated sulphuric acid was used which is extremely corrosive and dangerous; also produce toxic fumes which contain hydrogen sulphide. So this process is more costly and hazardous to health and environment.

2.2. Method *(4)*

KI (30 mL, 10%) + H_2O_2 (10 mL) + concentrated H_2SO_4 (25.7 mL) → black precipitates of iodine

Procedure

10 g Potassium iodide was taken in 250 mL conical flask and 100 mL distilled water was added. From this, 30 mL (10% w/v) of solution was taken and 10 mL hydrogen peroxide was added in ice cold condition with continuous stirring on magnetic stirrer and drop by drop 25.7 mL of concentrated sulphuric acid was added. The colour of the solution was slowly changed from clear colourless to yellow and black precipitates of iodine were observed.

Drawbacks

In this method concentrated sulphuric acid was used which is extremely corrosive and dangerous; also produce toxic fumes which contain hydrogen sulphide. So this process is more costly and hazardous to health and environment.

2.3. Method

KI (30 mL, 10%) + H_2O_2 (15 mL) + concentrated H_2SO_4 (60 mL) → black precipitates of Iodine

Procedure

10 g potassium iodide was added in 250 mL conical flask and 100 mL of distilled water was added. From this solution 30 mL (10% w/v) was taken and 15 mL hydrogen peroxide was added at 10^0 C temp. (with continuous magnetic stirring) and drop by drop 25.7 mL of concentrated sulphuric acidic was added. Slowly colour of solution was changed from clear colourless to yellow and black precipitate of iodine was observed.

Drawbacks

In this method concentrated sulphuric acid was used which is extremely corrosive and dangerous; also produce toxic fumes which contain hydrogen sulphide. So this process is more costly and hazardous to health and environment.

2.4. Method

KI (30 mL, 10%) + H_2O_2 (15 mL) + concentrated HCl (80 mL) → No precipitates

Procedure

10 g Potassium iodide was taken in 250 mL conical flask and 100 mL of distilled water was added. 30 mL (10% w/v) of this solution was taken and 15 mL hydrogen peroxide was added in ice cold condition (with continuous magnetic stirring) and drop by drop 80 mL of concentrated hydrochloric acid was added. No colour change as well as no precipitates of iodine was observed.

Drawbacks
In this method more amount of sulphuric acid required which is costly, hazardous to health and not suitable for industrial purpose.

2.5. Method
KI (30 mL, 10%) evaporate up to 15 mL solution and cool + H_2O_2 (10 mL) + concentrated H_2SO_4 (28.5 mL) → black precipitate of iodine.
Procedure
10 g Potassium iodide was taken in 250 mL conical flask and added in 100 mL of distilled water. 30 mL (10% w/v) from this solution was taken and evaporated up to 15 mL using hot plate. To this solution, 10 mL hydrogen peroxide was added in ice cold condition with continuous stirring and drop by drop concentrated sulphuric acid was added. Slowly the color of solution was changed from clear colorless to yellow and black precipitate of iodine was observed.

Drawbacks
In this method more amount of sulphuric acid required which is costly, hazardous to health and not suitable for industrial purpose.

2.6. Method
KI (3 g) + water (10 mL) + concentrated H_2SO_4 (2 mL) + H_2O_2 (4 mL) → Black precipitate of iodine
Procedure
3 g Potassium iodide was taken in 250 mL conical flask and 10 mL of distilled water was added in ice cold condition with continuous stirring on magnetic stirrer. To this, drop by drop concentrated sulphuric acid and hydrogen peroxide were added. Slowly the colour of solution changed from clear colorless to yellow and black precipitate of iodine was observed.

Advantages
In this method less quantity of sulphuric acid is used, so it is economical and less hazardous method. Here hydrogen peroxide is used, which is less costly and hazardous than sulphuric acid. So it is less toxic for environment and human.

2.7. Method
KI (3 g) + H_2O (10 mL) + H_2SO_4 (1 mL) + H_2O_2 (3.3 mL) →iodine precipitate (black) in ice cold condition
Procedure
3 g Potassium iodide was taken in 250 mL of conical flask and 10 mL of distilled water was added in ice cold condition with continuous stirring and concentrated sulphuric acid and hydrogen peroxide was added drop by drop. Slowly the color of solution changed from clear colorless to yellow and black precipitate of iodine was observed.

Advantages
In this method less quantity of sulphuric acid is used, so it is economical and less hazardous method. In this method hydrogen peroxide is used which is less costly and hazardous than sulphuric acid. This method is less toxic for environment and human. Also this method is reproducible.
2.8. Method
Industrial waste liquid filtrate (10 mL) ice cold condition + H_2SO_4 (1 mL) + H_2O_2 (8 mL) → iodine precipitate (black)
Procedure
Industrial waste liquid was taken and filtered them. 10 mL filtrate was taken in 250 mL conical flask in ice cold condition with continuous stirring and concentrated sulphuric acid and hydrogen peroxide was added drop by drop till black precipitate of iodine observed.

Advantages
In this method less quantity of sulphuric acid is used, so it is economical and less hazardous method. In this method hydrogen peroxide is used which is less costly and hazardous than sulphuric acid. This method is less toxic for environment and human and also reproducible.

2.9. Method
Industrial waste liquid filtrate (10 mL) ice cold condition + H_2SO_4 (1.5 mL) + H_2O_2 (5.2 mL) → black precipitate iodine

Procedure
Industrial waste liquid was taken in 250 mL conical flask and filtered them. 10 mL filtrate was taken in ice cold condition with continuous stirring and concentrated sulphuric acid and hydrogen peroxide was added drop by drop till black precipitate of iodine observed.

Advantages
In this method very less quantity of sulphuric acid is used, so it is economical method and less hazardous.
.

RESULTS AND DISCUSSION

Methods	Condition	Hydrogen Peroxide	Sulphuric Acid	Observation
1	Normal	No	13 mL	Fume produce
2	Ice cold	10 mL	25.7 mL	Fume produce
3	10 ^{0}C	15 mL	60 mL	Fume produce
4	Ice cold	15 mL	No (but HCl 80 mL)	-
5	15 mL evaporated solution in ice cold condition	10 mL	28.5 mL	Fume produce
6	Ice cold	4 mL	2 mL	Optimize
7	Ice cold	3.3 mL	1mL	Optimize
8	Ice cold	8 mL	1 mL	Applied for industrial purpose
9	Ice cold	5.2 mL	1.5 mL	Applied for industrial purpose

CONCLUSION

First five methods are not useful for iodine purification, because in these methods more amount of sulphuric acid were required and it is very costly and hazardous to environment.
In method no. 6 and 7 very less amount of sulphuric acid is required, so it is useful for industrial purpose. Specially, method no. 9 was optimize for industrial iodine purification

FIGURE



Fig. 1. Iodine

TABLE

Table 1. Physical Properties of Iodine *(1, 5)*

Symbol	I
Atomic Number	53
Atomic Mass	127
Discovery	Bernard Courtois
Electronic Configuration	[Kr] $4d^{10}$ $5s^2$ $5p^5$
Word Origin	Greek Word – "iodes" = violet
Isotopes	23 isotopes of Iodine were known. Only 1 stable isotope was found in nature I^{127}
Boiling Point	184.35 ℃
Melting Point	114 ℃
Specific Gravity	4.93

ACKNOWLEDGEMENT

The authors thankful to management of School of Pharmacy, RK University, Rajkot for providing infrastructure and facilities to carried out this research work.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

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2D-QSAR study of indole derivatives for anti-microbial study

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ABSTRACT

A series of methyl 3-(2-amino-2-oxoacetyl)-6-chloro-1-methyl-1H-indole-5-carboxylate were screened for their anti-microbial activity against bacteria S.aureus. These compounds have showed moderate and very good antimicrobial activity. The Quantitative Structure Activity-Relationships (QSAR) study on the indole series was made using lipophilic, electronic and steric parameters. Several statistical expressions were developed and best models were validated. The studies confirm that the antimicrobial activity is dependent on selected lipophilic, electronic and steric parameters. The QSAR study provides important structural insights in designing of potent antimicrobial agents.

SUMMARY
This research helps to identify the lead compound with proper anti-microbial activity.

Keywords: QSAR, antimicrobial agents, indole derivatives, Multiple linear regression

INTRODUCTION

The development of new antibacterial agents has been a very important step for researchers. Most of the research programme efforts are directed toward the design of new drugs, because of the unsatisfactory status of present drugs side effects and the acquisition of resistance by the infecting organisms to present

drugs. The resistance of common pathogens to standard antibiotic therapy is rapidly becoming a major health problem throughout the world *(1, 2)*.

The investigation of the quantitative structure activity/property relationships (QSAR/QSPR) of substances is an important aspect of modern chemistry, biochemistry, medicinal chemistry and drug discovery. The data or results that are obtained from the QSAR study consist of mathematical equations which relate the chemical structure of compounds to a wide variety of their physical, chemical, electronic and biological properties. Once a correlation between structure and activity/property is found, any number of compounds, including those not synthesized yet, can readily be screened in silico for selection of structures with desired properties. Hence, it is possible to select the most promising compounds for synthesis and testing in the laboratory *(3,4)*.

A new approach called the Hansch approach is a new extra thermodynamic approach in the analysis of quantitative structure activity relationships (QSAR). It has been most widely and effectively used for theoretical drug design. This method works by assuming that the potency of a certain biological activity exerted by a series of congeneric compounds can be expressed in terms of a function of various physicochemical (electronic, steric and hydrophobic) effects.

This equation below helps to obtain relationships between functions and activity of compounds:

f (biological activity) =f (electronic) +f (steric) +f(hydrophobic) +[f (structural) +f (theoretical)]

If these functions could be formulated in an equation showing certain effects favorable for the activity, structural modifications that enhance such properties would be expected to generate potent active compounds *(5,6,7)*.

Anti bacterial agents:

Antimicrobial agents that can serve as replacements to conventional pharmaceutical antibiotics are disclosed. The antimicrobial agents comprise conjugatively transmissible plasmids that kill targeted pathogenic bacteria, but are not harmful to donor bacteria.

METHOD:

Firstly 20 indole deravatives were selected of wide diverse functional group substituents.

Using Chem Draw software all the structures were made, based on the structure physico-chemical properties were calculated in the same software. The results obtained were tabulated in the excel sheet and then multiple linear regression was performed and various QSAR models were generated. Based on model obtained the best model was selected. Here, 20 indole derivatives were selected for the QSAR study, out of which, 16 were chosen as training set compounds and4 were taken as test set compounds *(8)*.

RESULTS AND DISCUSSION:

Equation No. 1 was found as best equation amongst all generated equation because of high R^2 (0.875) value and low standard error (0.189). The generated model validated by test data set. The comparision of observed pMIC and predicted pMIC were found very close. The residual line is under limit. So generated model was valid model and it can be further used for prediction of anti-microbial activity of same series of compounds.

CONCLUSION

Classical QSAR approach was applied successfully to a 16 training set compounds from series of Methyl 6-chloro-3-[(N,N-dialkylamino)(oxo)acetyl 1-methyl-1H-indole-5-carboxylates with well expressed

antimicrobial activity. The genenerated best equation (No. 1) was validated with 4 test set compounds with same series. Quantitative structure–activity relationship studies revealed that the antimicrobial activities of these synthesized derivatives against the test microorganisms are mainly governed by the log P, index of reflection, polarizability and parachor parameters.

Among four selected parameter index of reflection and parachor produces negative effect on anti microbial activity while log P and polarizability produces positive effect on antimicrobial activity. Index of reflection has higher impact on antimicrobial activity because their coefficient is higher than rest of all. Thus a proper substitution of the group with lower index of reflection of aromatic ring probably improves the potency of these derivatives as antimicrobial agents.

FIGURES

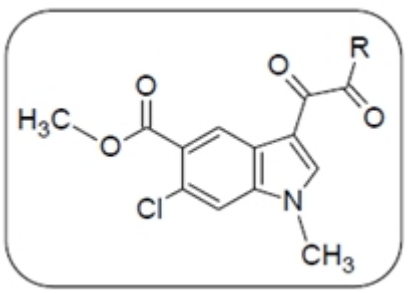


Fig.1. Methyl 6-chloro-3-[(N,N-dialkylamino)(oxo)acetyl 1-methyl-1H-indole-5-carboxylates

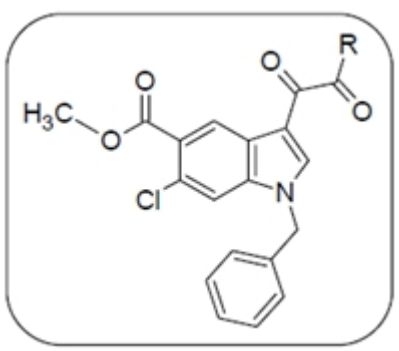


Fig.2. Methyl 1-benzyl-6-chloro-3-[(N,N-dialkylamin)(oxo)acetyl]-1H-indole-5-carboxylates



Fig.3. Chart of comparison between observed and predicted pMIC value for training set

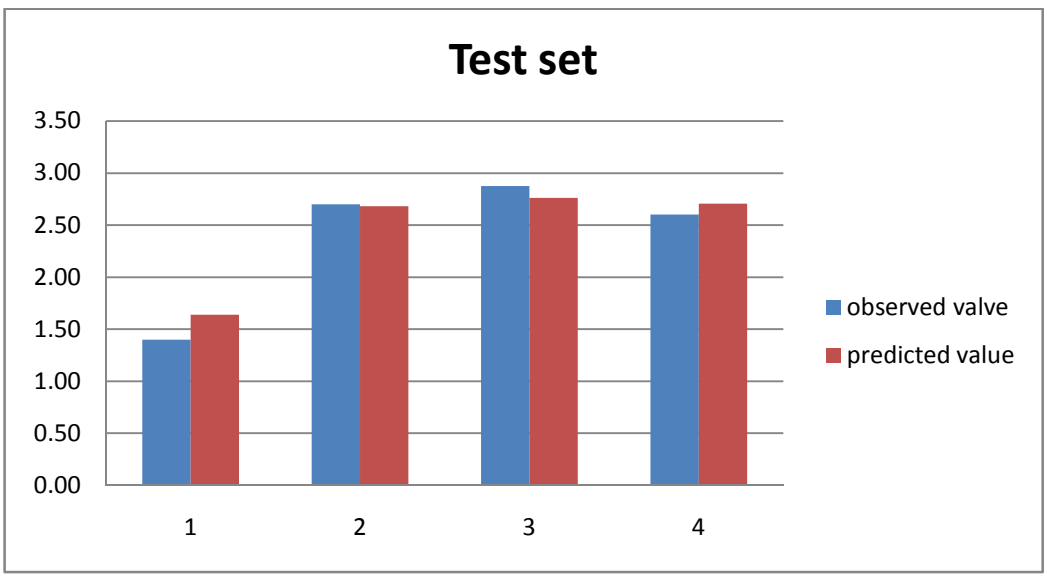


Fig.4. Chart of comparison between observed and predicted pMIC value for test set

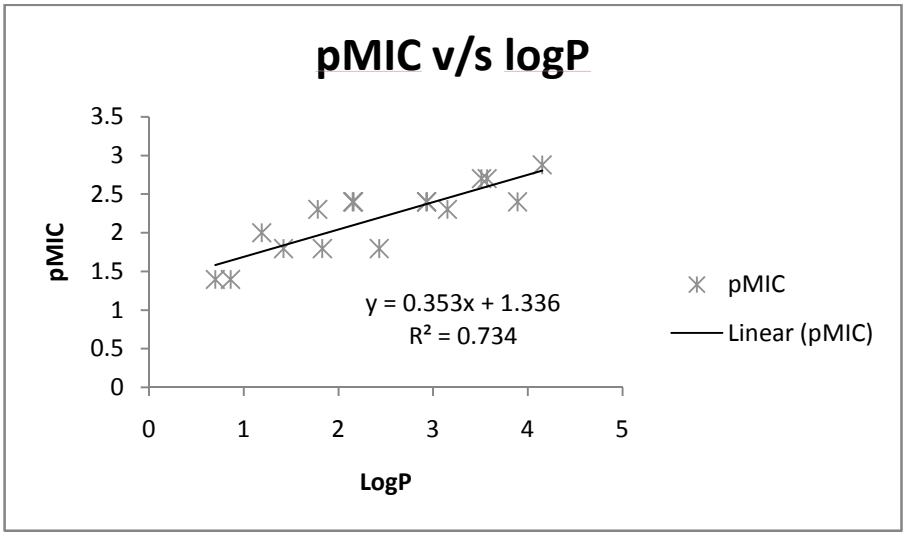


Fig.5. Chart of comparison between parameter (Log P) and pMIC values for training set

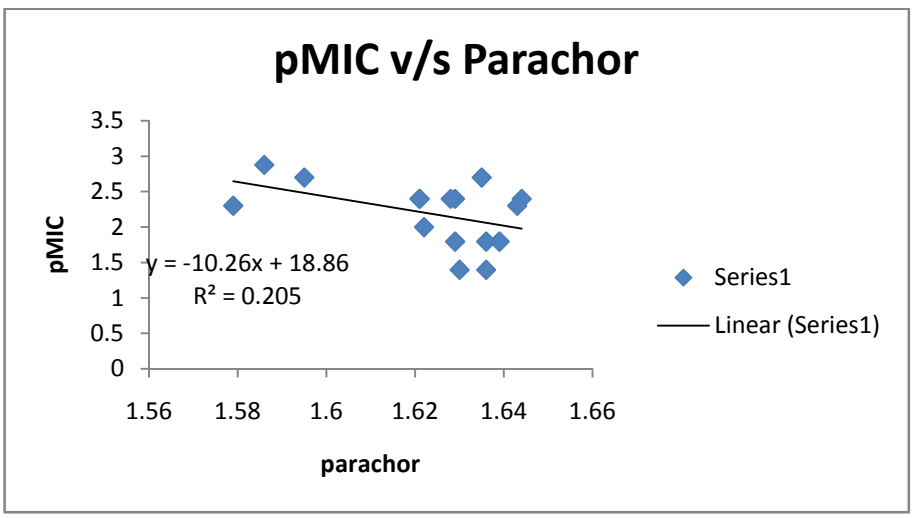


Fig.6. Chart of comparison between parameter (Parachor) and pMIC values for training set

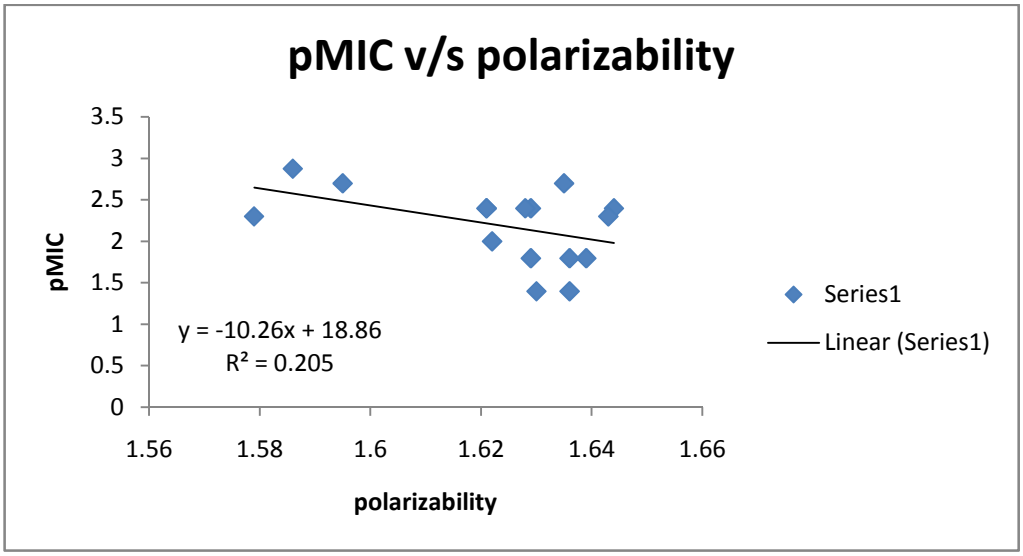


Fig.7. Chart of comparison between parameter (Polarizabilty) and pMIC values for training set

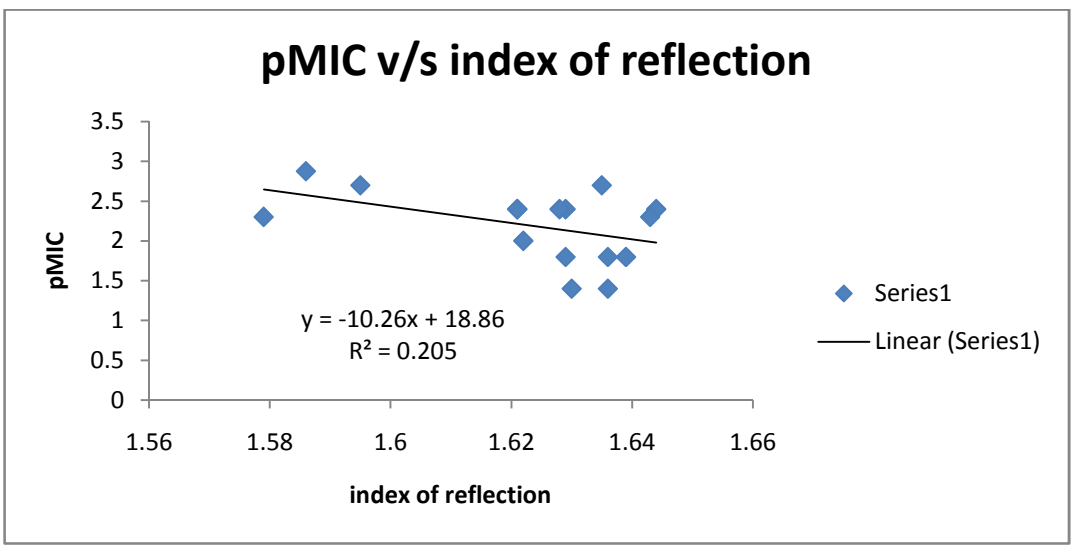


Fig.8. Chart of comparison between parameter (index of reflection) and pMIC values for training set

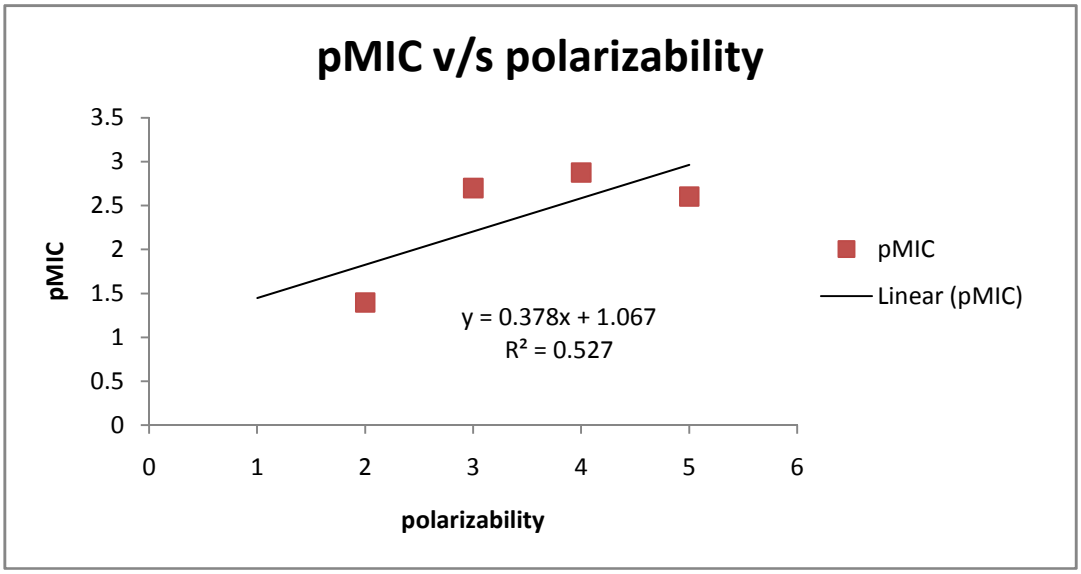


Fig.9. Chart of comparison between parameter (Polarizabilty) and pMIC values for test set

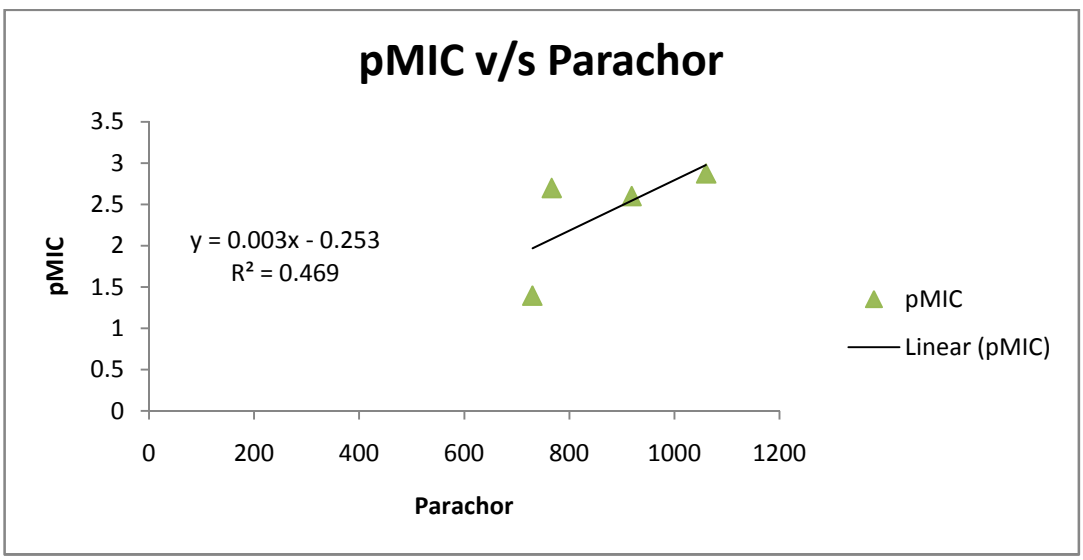


Fig.10. Chart of comparison between parameter (Parachor) and pMIC values for test set

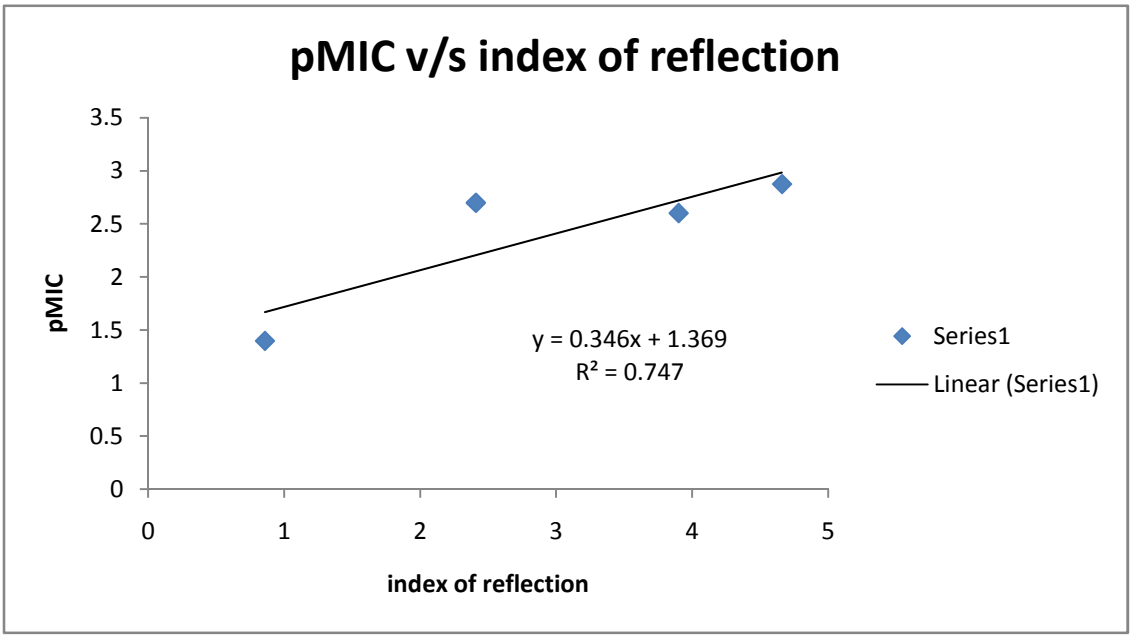


Fig.11. Chart of comparison between parameter (Index of reflection) and pMIC values for test set

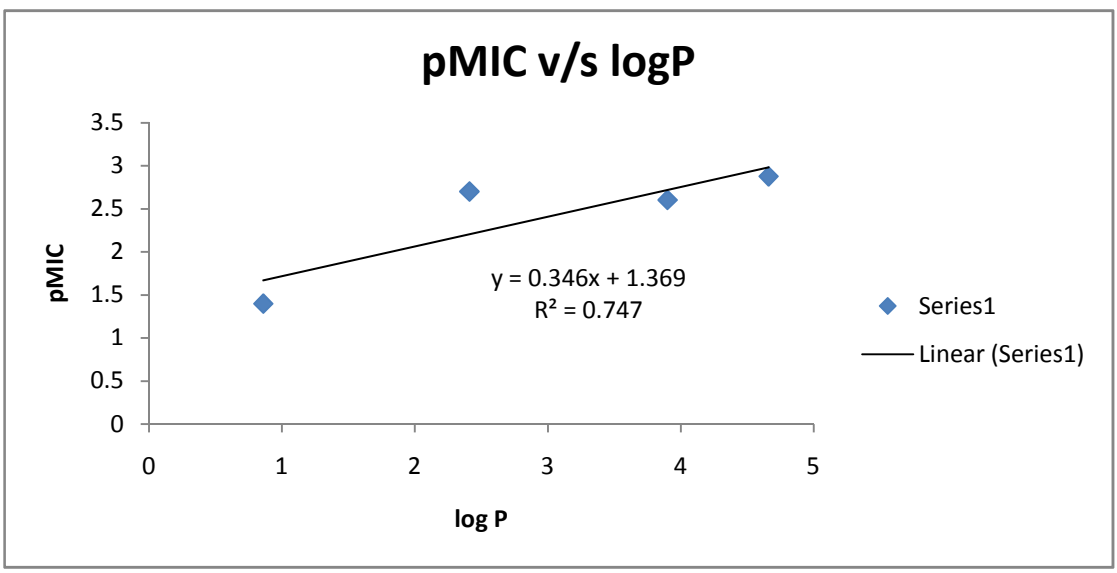


Fig.12. Chart of comparison between parameter (Log P) and pMIC values for test set

TABLES

Table 1. Compounds taken as training set

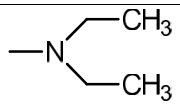
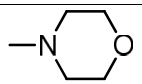
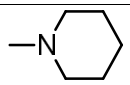
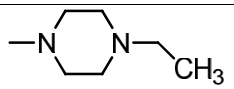
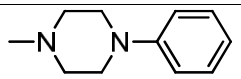
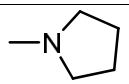
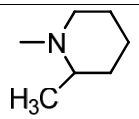
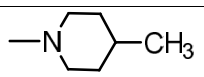
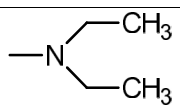
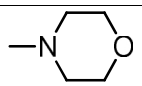
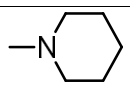
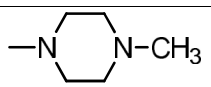
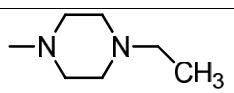
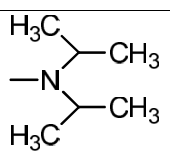
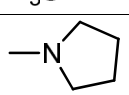
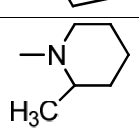
Sr. No.	R
1	
2	
3	
5	
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8	
9	
10	
11	
12	
13	
14	
15	
17	
18	
19	

Table 2. Table showing calculation of physicochemical properties done for training set compounds

Sr No	Compound	Log P	index of refraction	parachor	Polarizability (*10-24)	MIC	pMIC
						s. aureus	
1	1	1.78	1.579	704.3	36.27	200	2.30
2	2	0.7	1.63	683.7	35.96	25	1.40
3	3	1.83	1.629	703.1	37.24	62.5	1.80
4	5	1.19	1.622	768.3	40.56	100	2.00
5	6	2.93	1.644	875.5	46.93	250	2.40
6	8	1.42	1.639	664.5	35.41	62.5	1.80
7	9	2.15	1.621	734.2	38.99	250	2.40
8	10	2.16	1.621	734.2	38.99	250	2.40
9	11	3.51	1.595	888.7	46.3	500	2.70
10	12	2.43	1.636	868.1	45.99	62.5	1.80
11	13	3.57	1.635	887.5	47.26	500	2.70
12	14	0.86	1.636	914.1	48.76	25	1.40
13	15	2.93	1.629	952.7	50.58	250	2.40
14	17	4.15	1.586	950.9	49.81	750	2.88
15	18	3.15	1.643	848.9	45.43	200	2.30
16	19	3.89	1.628	918.6	49.02	250	2.40

Table 3. Compounds Taken As Test Set

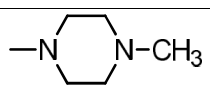
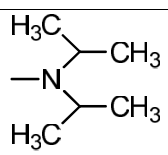
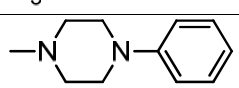
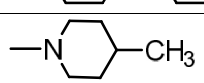
Sr. No.	R
4	
7	
16	
20	

Table 4. Table showing calculation of physicochemical properties done for test set compounds

Sr. No	Compound	Log P	index of refraction	parachor	Polarizability (*10-24)	MIC	pMIC

						s. aureus	
1	4	0.86	1.631	729.7	38.73	25	1.40
2	7	2.41	1.571	766.5	39.78	500	2.70
3	16	4.66	1.646	1059.9	56.96	750	2.88
4	20	3.9	1.628	918.6	49.02	400	2.60

Table 5. Comparison of predicted value and observed value for training set.

Sr No	Compound	pMIC	predicted value
1	1	2.30	2.29
2	2	1.40	1.48
3	3	1.80	1.99
4	5	2.00	1.75
5	6	2.40	2.24
6	8	1.80	1.83
7	9	2.40	2.25
8	10	2.40	2.26
9	11	2.70	2.58
10	12	1.80	1.94
11	13	2.70	2.45
12	14	1.40	1.50
13	15	2.40	2.19
14	17	2.88	2.99
15	18	2.30	2.31
16	19	2.40	2.70

Table 6. Comparison of predicted value and observed value for test set.

Sr. No	Compound	pMIC	predicted value
1	4	1.40	1.63
2	7	2.70	2.68
3	16	2.88	2.76

4	20	2.60	2.70

Table 7. Developed 2D-QSAR Models.

Model no.	Equation	Observations	R^2	Standard error	F
1	pMIC=35.764+(0.223*log p)+(0.018*MR)-(21.063*index of reflection)-(0.036*parachor)+(0.647*polarizability)	16	0.875	0.189	14.59
2	pMIC=12.087+(0.334*log p)+(0.008*MR)-(6.424*index of reflection)-(0.001*parachor)	16	0.828	0.209	14.22
3	pMIC=49.725+(0.041*MR)-(30.191*index of reflection)-(0.054*parachor)+(0.965*polarizability)	16	0.838	0.209	14.27
4	pMIC=28.227+(0.351*log p)-(16.065*index of reflection)-(0.026*parachor)+(0.476*polarizability)	16	0.859	0.195	16.78
5	pMIC=1.870+(0.403*log p)+(0.001*MR)+(0.008*parachor)-(0.182*polarizability)	16	0.803	0.231	11.21
6	pMIC=11.105+(0.343*log p)+(0.006*MR)-(5.807*index of reflection)-(0.024*polarizability)	16	0.834	0.211	13.87
7	pMIC=11.317+(0.386*log p)-(0.005*MR)-(5.848*index of reflection)	16	0.819	0.211	18.14
8	pMIC=11.985+(0.329*log P)-(6.523*index of reflection)	16	0.813	0.206	28.40

ACKNOWLEDGEMENT

The author thanks Dr. H. S. Joshi, Professor, Dept. of chemistry, Saurashtra University for providing compounds of indole derivatives to carry out this work. The author also thankful to management of School of Pharmacy, RK University, Rajkot for providing infrastructure and facilities for carried out this research work

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

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RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

Fabrication Of Transdermal Matrix Patch Of Lercanidipine Hydrochloride Using Natural Polymer And Essential Oil

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ABSTRACT

The goal of present research work was to develop and characterize transdermal matrix patch of Lercanidipine hydrochloride (LH) for controlled drug delivery using solvent evaporation method. To achieve controlled drug release, polymers such as psyllium and HPMC K15M were optimized. Moreover, the skin permeation effect of essential oils such as linseed oil, jojoba oil and pumpkin seed oil were investigated on wistar rat skin. A 3^2 full factorial design applied to optimize two formulation variables: concentration of essential oil as a permeation enhancer and polymer fixed-weight ratio. To study drug-excipients incompatibility fourier transform infrared spectroscopy (FTIR) had employed, which showed the absence of chemical interaction. All formulations evaluated for physico-chemical parameters, *ex-vivo* drug release study, *in-vivo* skin-irritation study on wistar rats and stability study. Developed matrix patch showed optimum physico-chemical properties with absence of skin irritation. *Ex-vivo* drug release study revealed that both formulation variables show effect on drug release from matrix patches. Effectiveness of

the oils as the permeation enhancer was found to be in the following descending order: Pumpkin seed oil > Linseed oil > Jojoba oil. Therefore, pumpkin seed oil selected as a permeation enhancer in the final formulation that shows highest flux (164.09±1.49 µg/cm^2/h) and desired drug release for transdermal administration. Stability study shows that the patch was stable up to 6 months at 40±2 ˚C and 75±5 % RH and 30±2 ˚C and 65±5 % RH.

Key words:

SUMMARY

The present investigation demonstrates that prepared matrix patch has a capacity to deliver therapeutically effective controlled release dose of lercanidipine hydrochloride (LH) via transdermal route using pumpkin seed oil as the permeation enhancer.

Keywords: Pumpkin seed oil, Psyllium, hypertension, Lercanidipine hydrochloride, propylene glycol, ex-vivo study, in-vivo skin irritation study

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INTRODUCTION

Transdermal devices in a recent time become more popular because it avoids hepatic first pass metabolism and maintains plasma concentration throughout the treatment, thereby decreasing the dosing frequency and reducing gastrointestinal irritation resulting in improved patient compliance. Easy removal of patch at any time from the target site will terminates the treatment preventing the chances of overdose and under dose (1-4). However, transport of compounds via skin is a considerable challenge due to the complex structure of skin. Therefore, suitable polymer matrix is required through which drug should be release at predetermined rate throughout the treatment (5, 6). Psyllium husk obtained from the plant of *Plantago ovata* is rich in polysaccharide and uronic acid contents, which renders it the property of making good thin patches (7-10). Hence, a polymeric mixture of psyllium husk and HPMC K15M was used as a controlled drug delivery component (11-16). The success of a transdermal matrix patch depends on the ability of the drug to penetrate into the skin in sufficient quantities to maintain required therapeutic levels (17, 18). Permeation enhancers are not drugs but they are molecules that reversibly alter the barrier nature of the *stratum corneum* and allow the drug to penetrate into the skin. The natural permeation enhancers available from literature review are essential oils, terpens, terpenoids, fatty acids, glycols and herbal extracts (19, 20). Essential oils gained more attention from the researchers because they are compatible with a huge range of hydrophilic and lipophilic drugs along with being non-toxic, non-allergic and clinically acceptable (21, 22). Pumpkin seed oil, Linseed oil, and Jojoba oil are the well-known essential oils have higher permeability because it contains unsaturated fatty acids which alleviate the lipid stratum

coneum by dekeratinization of corenocytes and increasing the permeation of molecules through the skin (23, 24). Propylene glycol, polyethylene glycol 400 and Dibutyl thealate are the commonly used plasticizers. Therefore, all three here optimized and PG was selected based on folding endurance study results. Selective drug candidate, Lercanidipine hydrochloride (LH) is a calcium channel blocker used in the treatment of hypertension and several other cardio-vascular disorders. It is administered orally with 10 mg daily dose having 30% bioavailability, so two times required in a day to maintain therapeutic level. The physico-chemical properties such as high lipophillicity (Log P value 6.42 at 20- 25 ^{o}C), low molecular weight (648.19g/mol), high melting point (197-201 ^{o}C) and high pKa (9.36) at 37^{o}C indicates its suitability for transdermal matrix patch. (22-27). A solvent evaporation method used for preparation of transdermal matrix patch due to its ease of manufacturing and the possibility of achieving a higher release and flux of the lipophilic drug loaded on the matrix as suggested by literature. (28-30). Hence, in this present research work hydrophilic polymeric matrix patches formulated using HPMC K15M and psyllium along with essential oils as permeation enhancers.

MATERIALS AND METHODS

2. 1 Materials

Lercanidipine hydrochloride (LH) got as a gift sample from Glenmark generics limited, Pune, India. Psyllium purchased from Shiv psyllium Industries, North Gujarat, India. All the investigated oils namely Linseed oil (LO), Jojoba oil(JO) and Pumpkin seed oil (PSO) purchased from Hamdard Laboratories, Ghaziabad, India, Hydroxyl Propyl Methyl Cellulose K-15M (HPMC K15M), and propylene glycol (PG) supplied by S.D. Fine Chemicals Ltd., Mumbai, India. All remaining chemicals and solvents were reagent grade. Double distilled water used throughout the study.

2.2 Animals

Wistar rats (180-200g, 6-8 week old) were supplied by Zydus Research center, (Village Moraiya, Near Nova Petrochem, Ahmadabad, Gujarat). The animals received after the study duly approved by the CPSCEA (committee for the purpose of control and supervision on experiments on animals of government of India) with protocol No. 984/06/2014-2-06. The experiments on animals' *in-vivo* skin-irritation study and *ex-vivo* permeation study using wistar-rat skin performed in accordance with the guidelines given by Animal Ethical Committee, CPCSEA.

2.3 Methodology

2.3.1 Dose calculation: Dose of LH for the transdermal patch calculated on the bases of value of targeted flux and transdermal flux. Targeted flux of LH is **166.46 µg/cm^2/hr (**calculated by equation: targeted flux Jss= Css×Clt×BW/A, where 3.3 µg/l and 3.37 ml/min/kg are Css and Clt respectively for LH). Oral dose of LH is 10 mg or 20 mg once daily and bioavailability is 40%. Therefore, orally available dose to

maintain plasma concentration is only 4 mg. To surplus the loss of drug in different layers of skin and for getting required flux here, double dose is required. Therefore, in present study dose of LH for the preparation of transdermal patch having 4-cm^2 area is 8mg (73 mg for total 6.8cm petriplate) and it gives flux 165 µg/hr/cm^2, which is very nearer to required flux (30, 31).

2.3.2 Method for preparation of transdermal matrix patch containing LH.

The transdermal matrix patches were prepared using different ratios of psyllium and HPMC K15 M. The polymers concentration was varied with 3%w/v, 4%w/v and 5%w/v by keeping the constant ratio (2:1) of HPMC K15M : psyllium and allowed to swell for 2 hrs in water. As per dose calculation, accurately weighed amount of LH dissolved in ethanol and this drug solution added into the polymeric solution with continuous stirring using magnetic stirrer. Then propylene glycol and essential oil incorporated as plasticizer and penetration enhancer respectively. Inverted funnel was kept over the petri plate for uniform evaporation, after complete drying biaxial oriented polyethylene, film glued as a backing membrane and a glossy paper having smooth surface used as a release liner. The dried films removed from the petri plate and cut into 4-cm^2 area, wrapped in an aluminum foil and stored in desiccators for further studies. (30-33).

2.3.3 Preliminary trial study for the optimization of matrix patch formulation

Preliminary trial batches were prepared and evaluated for the optimization of various concentrations of polymers, drug, plasticizer and permeation enhancers. Formulations L1toL3 were prepared with varying concentrations of LH 8mg, 10mg, and 12mg to study the effect of LH concentration on drug release and permeation. Batches L4 to L6 were prepared with varying concentration of polymers with 3%w/v, 4%w/v and 5%w/v, to study the effect of thickness of polymeric matrix on drug release and permeation. Batches PE1 to PE7 were prepared with varying concentrations of JO, LO and PSO for the optimization and selection of effective permeation enhancer. Formulations of all preliminary trial batches displayed in Table 1.

2.3.4 Statistical optimization of the formulation variables using experimental design approach

Preliminary trial study suggested that concentration of polymers and permeation enhancer mainly affect the release and permeation of LH from the patch. Therefore, further optimization of these two formulation variables performed using experimental designs by the fabrication of transdermal matrix patches having desire drug release and permeation flux. A 3^2 full factorial design, from Design Expert software 9.02 selected (A.M. Abdel Azim, et.al, 2014). This design involved three dependent variables (Y1, Y2, and Y3) and two independent variables (X1 and X2). The release response can be expressed as Y= f (X1, X2). The selected two independent variables for the present investigation were X1, polymer fixed weight ratio; and X2, essential oil concentration in the patches. All other formulation variables kept constant throughout the study. The dependent variables were Y1, drug release in 1^{st} hr (Q1), Y2, drug release at 16

hrs (Q16), and Y3, tensile strength of prepared patches. The composition of nine formulations based on this experimental design displayed in Table 2. After completion of statistical optimization experiments, polynomial equations and 3-dimentional plots generated to study the effect of X1 and X2 on Y1, Y2 and Y3 in order to identify the optimized LH loaded transdermal matrix patch. The final identified batch fabricated and subjected to validation of statistical optimization design.

2.4 Evaluation study of transdermal matrix patch containing LH.

2.4.1 FTIR study

FTIR spectroscopy used as analytical tool to find out compatibility between LH, HPMC K15M, psyllium, PSO, LO, JO and PG. FTIR spectra of pure drug and final formulation carried out using KBR disc method (31, 32).

2.4.2 Physicochemical evaluations of LH containing transdermal matrix patch.

The experimental design formulations, batches P1 to P9 evaluated for various physico-chemical evaluations such as thickness, folding endurance, moisture uptake and loss, tensile strength and drug content according to method given by, A.M. Abdel Azim, et.al, 2014 Rajesh Singh Patel and S.S. Poddar, 2009, Pichayakorn W., et.al, 2013.

2.4.3 *Ex-vivo* skin permeation study and preparation of rat skin

Wistar rats sacrificed with prolonged ether anesthesia and the abdominal skin of each rats were excised. Hairs on the skin of animal and subcutaneous tissues removed with sharp blade. The skin was washed with phosphate buffer saline, wrapped in aluminum foil and stored in a deep freezer at -20^0C until further use. At the time of *ex-vivo* permeation study, skin was brought to room temperature and hydrated in phosphate buffer solution for half an hour before the study and then placed over the receptor compartment of Franz diffusion cell with a diffusion area of 0.64 cm^2 and a receptor compartment capacity of 13 ml. The LH loaded transdermal matrix patch placed over the membrane by keeping the dermal side in contact with receptor medium. The receptor compartment filled with 13 ml of pH 6.8 buffer. The temperature of diffusion medium maintained at 32 ± 2^oC. This whole assembly kept on a magnetic stirrer and solution in the receiver compartment constantly and continuously stirred using magnetic bead. Samples were withdrawn (2 ml, each time) at different time interval and replaced with equal amount of pH 6.8 buffer. Sample analyzed at 357nm after suitable dilution using UV spectrophotometer. Amount of drug permeated per square centimeter at each time interval was calculated and plotted against time (34-40).

The *Ex-vivo* release data subjected to various kinetic equations to find out release mechanism and order of drug release. Transdermal flux was calculated using the value of slope of cumulative drug release curve that constructed by the steady state values of the cumulative amount of drug permeated (mg/cm^2) vs time. Permeation coefficients (cm/hr) calculated by dividing the flux with initial drug loading (mg/cm^2). Lag time calculated from back extrapolation. Diffusion coefficient (D/h^2) and permeability coefficient (Kp) also calculated from the data of *ex-vivo* studies using given equations, respectively ($D/h^2=1/6\times T_{lag}$, Jss =

(dq/dt).1/A, Kp = Jss/Cs). The regression analysis of steady state data and release rate was calculated. The experiment was performed in triplicate and mean results were recorded (27, 40-43).

2.4.4 Regression analysis of the optimization of formulation

The statistical analysis of factorial design batches performed using Design expert software 9.02. The results of dependent variables for the factorial design batches given in Table 3. To evaluate contribution of both the factors at three different levels on responses, two-way analysis of variance (ANOVA) performed using design expert software 9.02. To demonstrate graphically the influence of each factor on responses the response surface plots such as contour and 3D plots were generated using software. The response surface plots for dependent variables, tensile strength, % drug release in 1 hr(Q1) and % drug release in 16hrs (Q16) are shown in Figure 3,4 and 5 respectively. The value of $p<0.05$ was considered to be significant.

2.4.5 *In- vivo* skin irritation study

The study performed on wistar rats to determine irritation after single application of prepared transdermal matrix patch. Accurately cut 4 cm^2 size patch applied on the clean backside skin of rat and removed after 16 hrs. The exposed skin evaluated for formation of edema and erythema and any type of irritation. The rats were divided into 2 groups of 3 rats in each group (n=6), one group as control and another group as test (prepared matrix patch). Prior permission takes from the animal ethical committee for this study (44, 45).

2.*4.6* Stability studyThe final optimized batch subjected to stability study to evaluate any change in appearance and drug release when exposed to accelerated conditions of environment during storage, handling, transport and use. The study performed according to ICH guideline at 40°C and 75% RH and at 30±2˚C and 65±5 % RH in humidity chamber for a period of six months, and it was analyzed for physicochemical parameters at particular time intervals (43-45).

RESULTS AND DISCUSSION

3.1 FTIR study

Infrared spectra of LH pure drug (A) and LH loaded matrix patch final formulation (B), are shown in Figure10 and 11. Infrared absorption spectroscopy (IR) of LH show sharp band due to stretching vibration bands of OH, N-H and C=O, respectively. From the Figure 10 and 11, it observed that there were no changes in these main peaks in IR spectra of mixture of drug and polymers, which indicate physical compatibility between LH and all ingredients used in final formulation of transdermal matrix patch.

3.2 Preliminary trial study for the optimization of matrix patch formulation

The preliminary trial batches were prepared and evaluated to investigate the effect of formulation variables such as LH concentration, HPMC K15M and psyllium concentration, essential oils concentration as a permeation enhancer on permeation of LH from the transdermal matrix patch. Obtained results are outlined in Table-1; it revealed that as increase in LH concentration from 8mg, 10mg and 12mg, % cumulative drug release was also increased from 68.27±0.19 % (L1), 73.91±0.27% (L2), and 79.13±0.17% (L3), respectively. This increase in release based on fick's first low of diffusion, in which the drug release is directly proportional to the drug concentration gradient across the membrane, i.e higher amount of drug available for diffusion. The results of batches L4, L5, and L6 suggested that the rate of drug release decreased with an increase in the polymer concentration. Batch L4 contains 3%w/v of polymer concentrations shows highest drug release (68.27±0.19%) compare to L5 (57.79±0.21%) and L6 (50.32±0.19%) which contains 4%w/v and 5%w/v, respectively. This appears to be due to an increase in thickness of polymer matrix with increase in polymer concentration. Obtained results of batches PE1 to PE7 revealed that permeation increase with increase in concentration of essential oils. This is evident from the LH permeation at 16 hrs from formulations PE2 to PE7 containing LO, JO, and PSO with two different concentrations 10%w/w and 20%w/w. The results in Figure 1 also indicate that LO and JO was not sufficient to achieve desire permeation flux for controlled release of LH up to 16 hrs. On the other hand, 20% w/w PSO achieves the nearer targeted flux value 157.24 μg/cm^2/hr**,** it was sufficient for controlled release of LH up to 16 hrs and to maintain therapeutic plasma level. Thus, PSO's concentration selected as a one independent variable for further study. Briefly, the effect of above formulation variables on LH release in preliminary trial suggested that, the amount of LH released from the patch increase with increase in LH concentration, PSO concentration and decreased with an increase in polymer concentration.

3.3 Statistical optimization of the formulation variables

Based on the results of the preliminary studies further evaluations of formulation variables performed using experimental designs to optimize suitable combination of independent factors on the fabrication of transdermal matrix patch of LH having desire rate of drug release as well as permeation flux. A 3^2 full factorial designs nine batches summarized in Table 2. Drug release at 1hr (Q1), at 16th hr (Q16), and tensile strength selected as dependent variables to find out final formulation for the LH containing transdermal matrix patch. Prepared nine batches P1 to P9 further evaluated for the physico- chemical properties of Matrix patch. Cumulative drug release of LH from Matrix patch and its permeation through the rat skin shown in Figure 2, results of dependent variables listed in Table 3.

3.4 Physicochemical evaluations of LH containing transdermal matrix patch.

Transparent, flat, flexible and uniform transdermal diffusional matrix patch obtained using mixture of natural polymer psyllium and synthetic polymer HPMC K15M.The average weight of batches P1 to P9 range between 362±1.732 to 524±2.31 mg, which indicates that all the solid excipients uniformly

dispersed into the liquid and all batches were relatively in similar weights. The thickness of the patches measured by micrometer screw gauze, results found in between 0.81±0.04 to 0.88±0.13 mm. The results revealed that solution was uniformly casted on a previously lubricated petriplate and solvent uniformly evaporated from the petriplate. The drug content of the entire batches lie between 92.85 to 97.60 %, these results revealed that method select for the preparation of matrix patch was suitable and reproducible. The results of flatness study showed that all the batches have same length before & after cuts. Therefore, nearer to 100% flatness obtained and it indicates that all patch had a smooth surface. Tensile strength found in between 7.03±0.136 to 8.48±0.127 gm/cm^2, which revealed that the patch had sufficient mechanical strength to withstand during handling, transportation and administration. Same way results of folding endurance study revealed that the patch would not break & maintain their integrity with general skin folding applied. The results listed in Table 4.

3.5 *Ex-vivo* skin permeation study of LH

Permeation studies plot of cumulative amount of drug release versus time was generated and represented in Figure 2, from this plot permeation flux, permeability coefficient and enhancement ratio was calculated. The results listed in Table 5. The results revealed that batch P9 containing 30%w/w of PSO exhibited highest flux 164.09 ±0.14 µg/cm^2/hr and 89.93% drug release in 16 hrs. This higher release and permeation occur due to the presence of higher content of fatty acids of PSO. The results of e*x-vivo* release also suggested that the concentration of PSO and PG both had major influence on drug release because fatty acids of PSO increases the lipid fluidity and PG water fluidity. Data of e*x-vivo* release fit into different kinetic models to find out release mechanism, the release profiles of drug seemed to follow zero order and drug release mechanism was diffusion controlled so, it followed higuchi model. The correlation coefficient of R^2 values of batch P9 was $r^2 = 0.9976$ for zero order and $r^2 = 0.9733$ for higuchi model. Plot of kinetic studies represented in Figure 6 and 7.

3.6 Regression analysis of the optimization of formulation

Based on the values of dependent variables polynomial equations generated and listed with 3D and contour response surface plots, which indicates that both the formulation variables X1 and X2 played an important role in a controlled release of drug from the transdermal matrix patches. Tensile strength increases with optimum concentration of PG (20%w/w) and PSO (30%w/w). Obtained results revealed that the selected model was significant and drug release in a controlled manner for period of 16 hrs.

3.7 *In-vivo* skin irritation study

The results of *in-vivo* skin irritation study suggested that optimized batch P9 showed no irritation on rat skin after 16 hrs and photographs of skin irritation study represented in Figure 8 and 9.

3.8 Stability study

The optimized batch P9 exposed for stability studies as per ICH guidelines. The results listed in Table 6 and it revealed that, prepared patches stable and maintains its physical integrity throughout the study.

CONCLUSION

LH is the potent antihypertensive agent and very widely used in the treatment of hypertension but due to the first-pass hepatic metabolism drug bioavailability decreases. Therefore, in the present study the transdermal patch of LH was prepared which showed acceptable physicochemical and satisfactory *ex-vivo* controlled release after 16 hours could be helpful for the treatment of hypertension with improved patient compliance. Even though, extensive clinical studies are required to proved control release of LH from the transdermal matrix patch.

FIGURES

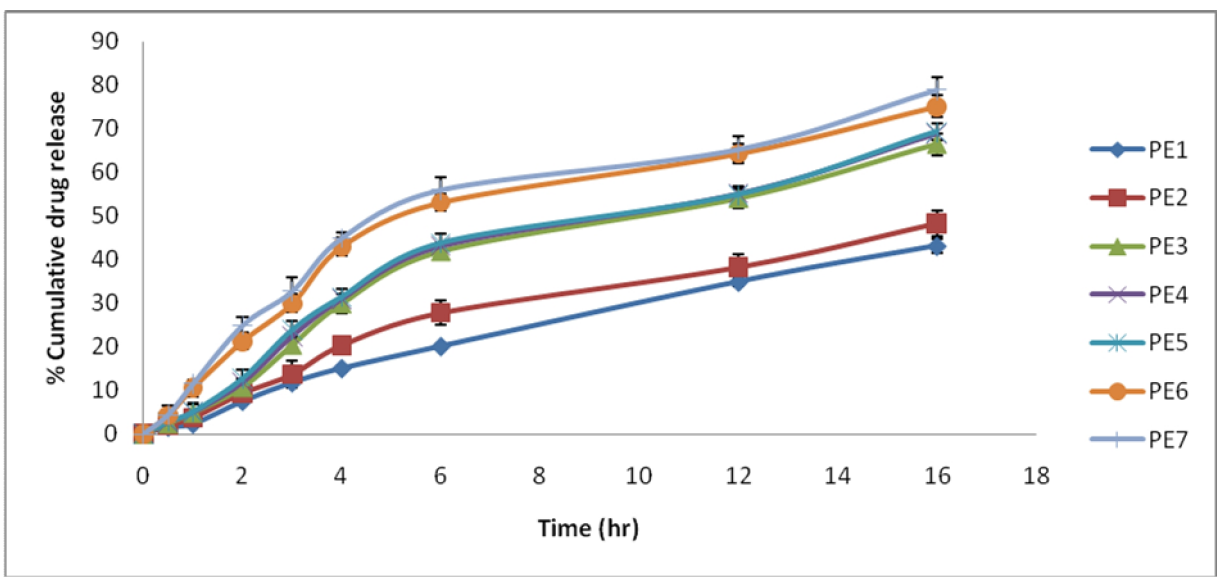


Fig.1. Comparative Drug Release profile of Batches PE1-PE7

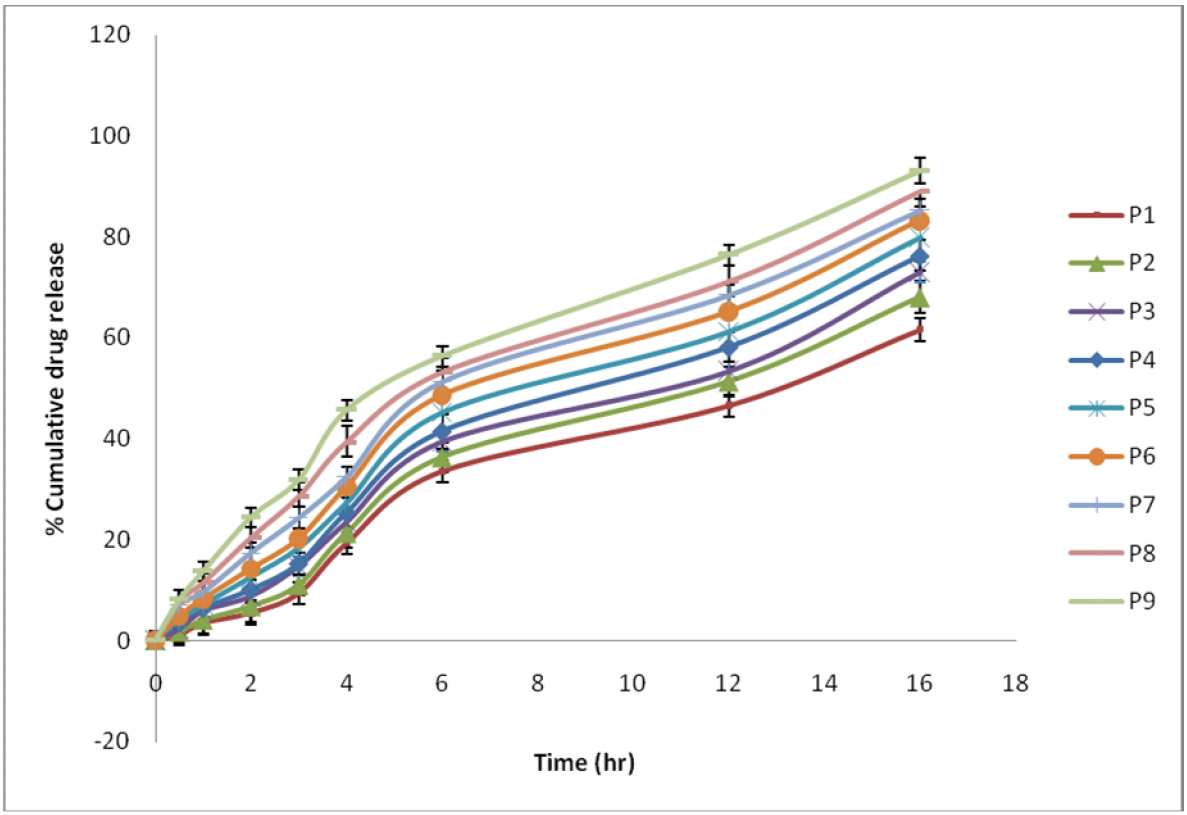


Fig.2. Comparative Drug Release Profile of Batches P1-P9

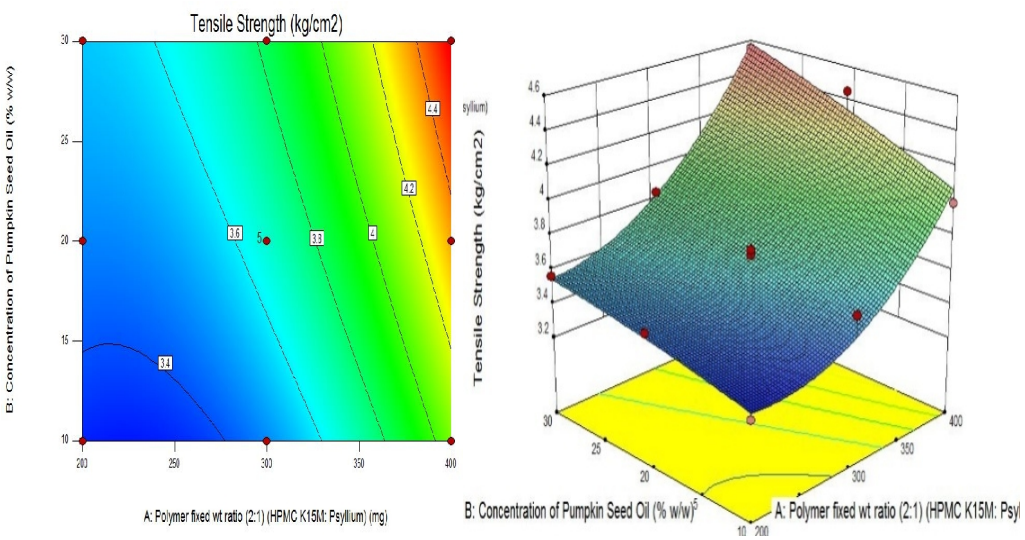


Fig.3. (a) counter plot and (b) response surface plot of effect of psyllium and pumpkin seed oil on tensile strength

Polynomial equation:

Tensile strength+3.67+0.44* A+0.18 * B+0.075* AB+0.23* A^2-0.020* B^2

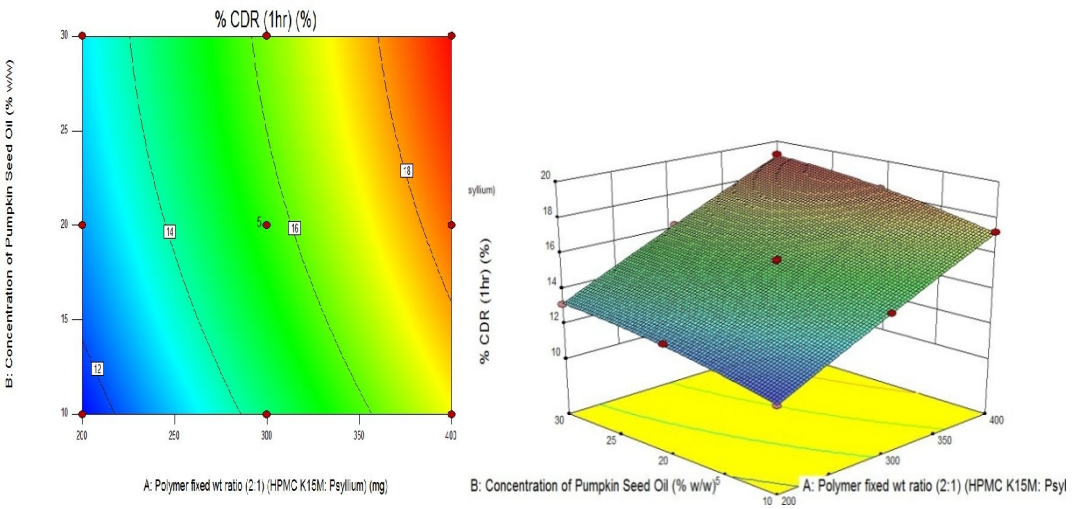


Fig.4. a) counter plot and (b) response surface plot of effect of psyllium and pumpkin seed oil on percentage drug release in 1 hr

Polynomial equation: % CDR (1hr) = + 15.62 +2.91*A+0.92*B+0.055*AB-0.078*A^2-0.29*B^2

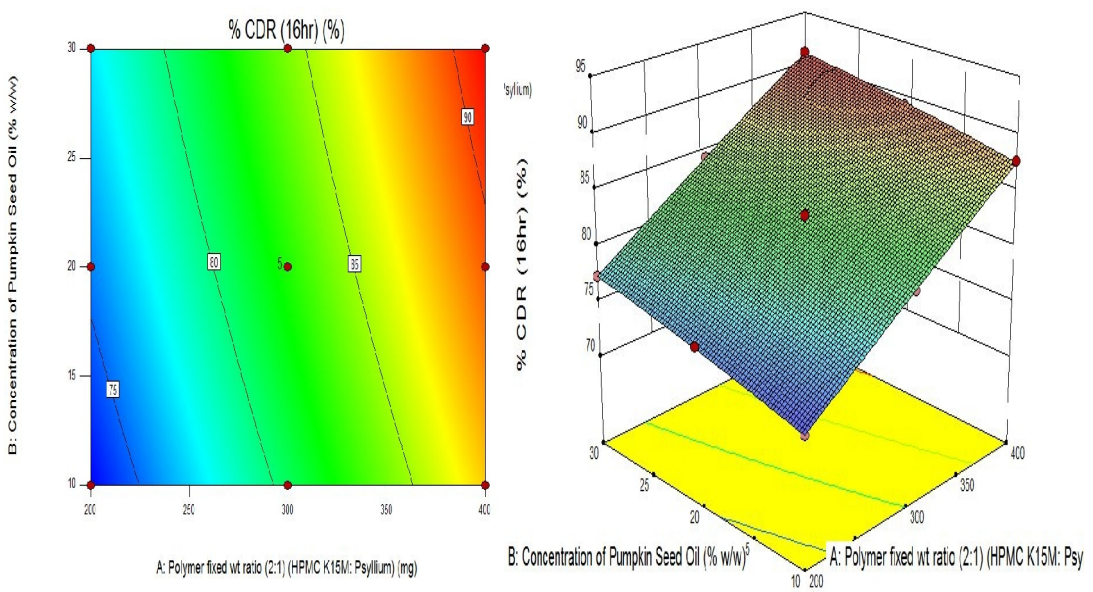


Fig. 5. a) counter plot and (b) response surface plot of effect of psyllium and pumpkin seed oil on percentage drug release in 16 hr

Polynomial equation:

% CDR (16hr) = +82.69+7.02*A+1.93*B-0.15*AB-0.19*A^2-0.23B^2

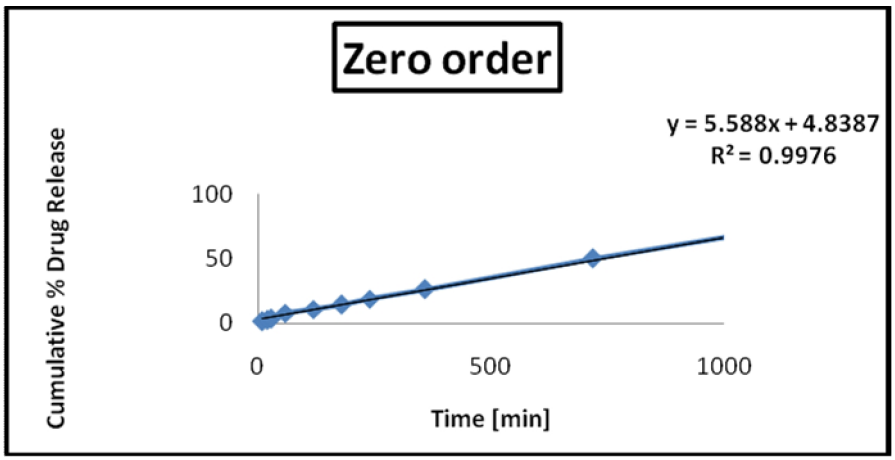


Fig. 6.Zero order plot for model release kinetic for batch P9

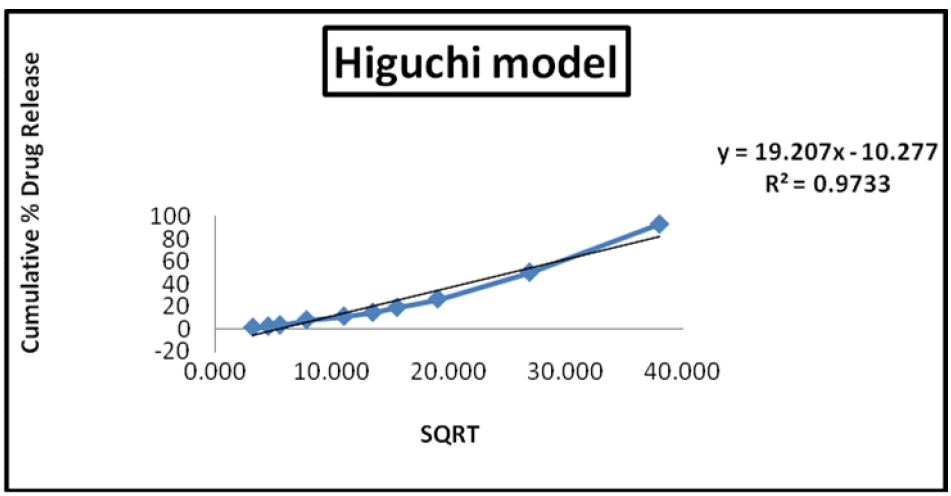


Fig. 7. higuchi plot for model release kinetic of batch P9



Fig. 8. Before skin irritation study



fig. 9. After skin irritation study

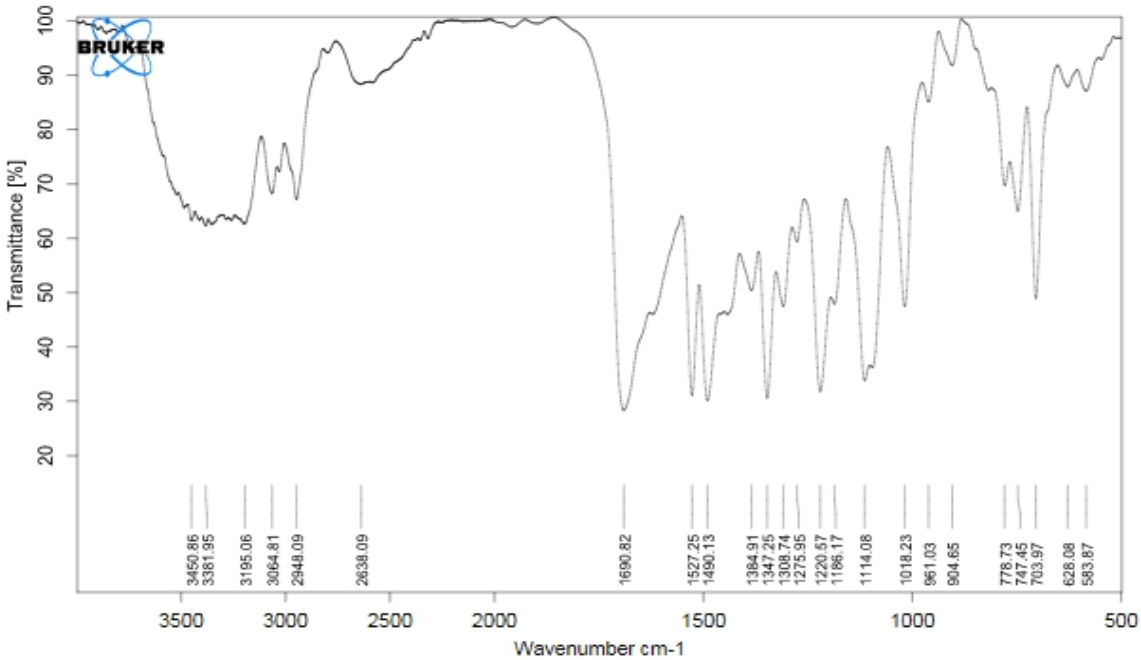


Fig. 10. Infrared spectra of Lercanidipine hydrochloride

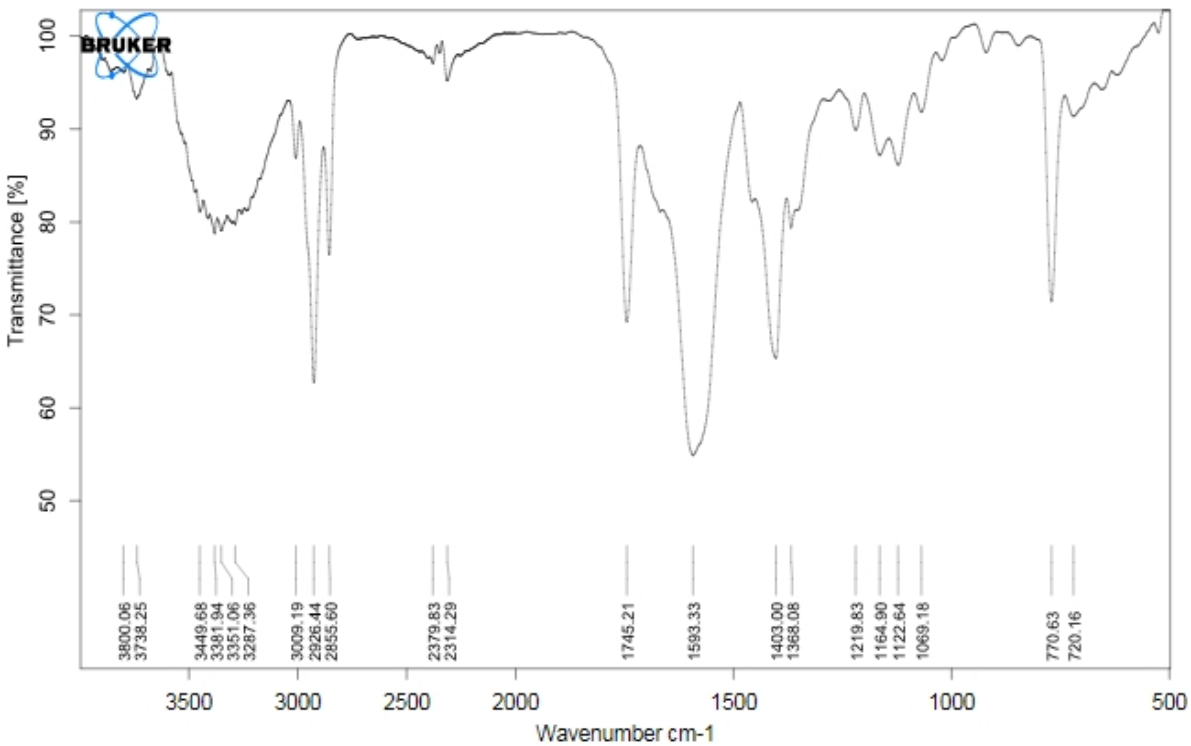


Fig. 11. Infrared spectra of final optimized formulatione.

TABLES

Table 1: Composition of preliminary trial batches and results of dependent variables (cumulative drug release at 1 hr(Q1), 16 hrs (Q16), and Tensile strength).

Batch code	*LCDH loading (mg)*	*HPMCK15M: Psyllium(2:1) (mg)*	*EO-Loading (%w/w total wt of polymer dry weight)*	*CDR at 1 hr(Q1)*	*CDR at 16hrs(Q16)*	**Tensile strength**
L1	**8**	**300**	**-**	8.22±0.23	68.27±0.19	3.27±0.03
L2	**10**	**300**	**-**	10.19±0.35	73.91±0.27	4.69±0.04
L3	**12**	**300**	**-**	11.11±0.31	79.13±0.17	4.72±0.03
L4	**8**	**300**	**-**	8.22±0.23	68.27±0.19	3.27±0.03
L5	**8**	**400**	**-**	7.18±0.16	57.79±0.21	3.45±0.04
L6	**8**	**500**	**-**	6.54±0.11	50.32±0.19	4.96±0.05
Effect of EO (essential oil) concentration as a permeation enhancer						

PE1-control	**8**	**300**	**Without EO**	8.22±0.23	61.27±0.19	3.27±0.03
PE2 - LO	**8**	**300**	**10% w/w**	9.19±0.15	64.76±0.06	4.19±0.02
PE3 -LO	**8**	**300**	**20% w/w**	10.41±0.13	66.42±0.19	4.22±0.053
PE4 - JO	**8**	**300**	**10% w/w**	9.12±0.14	68.89±0.11	4.17±0.03
PE5 - JO	**8**	**300**	**20% w/w**	10.08±0.19	69.32±0.17	4.48±0.02
PE6 – PSO	**8**	**300**	**10% w/w**	9.94±0.18	75.11±0.21	4.56±0.03
PE7 - PSO	**8**	**300**	**20% w/w**	11.57±0.13	78.81±0.13	4.67±0.04

Table 2: Composition of LH loading factorial design batches P1 to P9.

Batch code	***P1***	***P 2***	***P 3***	***P 4***	***P5***	***P 6***	***P 7***	***P 8***	***P 9***
Lercanidipine HCL(mg)	73	73	73	73	73	73	73	73	73
HPMC K15M(mg)	250	250	250	225	225	225	200	200	200
Psyllium(mg)	50	50	50	75	75	75	100	100	100
Water(ml)	12	12	12	12	12	12	12	12	12
Ethanol(ml)	8	8	8	8	8	8	8	8	8
Propylene glycol(%w/w of dry polymer wt)	20	20	20	20	20	20	20	20	20
Pumpkin seed oil(%w/w of dry polymer wt)	10	20	30	10	20	30	10	20	30

Table 3: results of dependent variables (cumulative drug release at 1 hr(Q1), 16 hrs (Q16), and Tensile strength

Batch Code	***Coded Value & Actual value***		***Dependent variables***		
	Polymer fixed wt ratio(2:1) (300 mg) X1	**PSO (% w/w) X2**	**% Drug released in 1 hr (%)Y1**	**% Drug released in 16 hrs (%)Y2**	**Tensile Strength (Kg/cm^2) Y3**
P1	-1	-1	11.42±0.24	73.16±0.52	3.30±0.01
P2	-1	0	12.72±0.45	75.54±0.81	3.49±0.02
P3	-1	1	13.16±0.41	77.28±0.13	3.56±0.03
P4	0	-1	14.41±0.43	80.41±0.16	3.59±0.03
P5	0	0	15.62±0.06	82.78±0.12	3.69±0.02
P6	0	1	16.24±0.31	84.36±0.16	3.84±0.02
P7	1	-1	17.22±0.16	87.64±0.06	3.99±0.03
P8	1	0	18.36±0.21	89.31±0.91	4.45±0.04
P9	1	1	19.18±0.07	87.14±0.14	4.56±0.03

Average of triplicate results. Mean ± SD standard deviation.

Note: X1 three levels: Polymer fixed wt ratio (2:1) (HPM K15M: Psyllium) (250:50, 225:75, 200:100), X2 three levels: 10%, 20% and 30%w/w of polymer dry weight

Table 4: Physicochemical evaluation of LH loading batches P1 to P9.

Batch Code	*Weight variation(mg)*	*Thickness (mm)*	*Folding endurance*	*Tensile strength (kg/cm²)*	*% Elongation*	*%moisture Uptake*	% moisture Loss
P1	362 ± 1.732	0.84±0.03	335±3.511	7.78±0.15	17.96±0.587	1.88 ± 0.07	2.84 ± 0.09
P2	408 ±2.516	0.86±0.08	348±3.7859	7.03±0.136	18.93±0.450	2.16 ± 0.20	2± 0.06
P3	457 ± 1.527	0.88±0.02	361±3.605	7.21±0.245	18.96±0.585	2.68± 0.06	2.23± 0.03
P4	387 ± 2.087	0.85±0.09	390±4.041	7.32±0.359	19.26±0.351	2.32± 0.08	2.72± 0.05
P5	433 ± 1.127	0.85±0.01	398±6.0277	8.16±0.245	19.50±0.684	2.89± 0.05	2.85± 0.05
P6	486 ± 1.527	0.88±0.07	397±4.00	7.17±0.125	20.46±0.493	2 ± 0.36	1.88± 0.08
P7	425 ± 2.00	0.86±0.11	373±3.605	7.91±0.183	22.50±0.458	1.98 ± 0.11	1.98± 0.09
P8	478 ± 1.527	0.81±0.04	378±2.00	7.76±0.15	20.98±0.602	2.83± 0.05	2.85± 0.06
P9	524 ± 2.51	0.88±0.013	384±3.21	8.48±0.127	21.84±0.335	2.50 ± 0.06	2.5± 0.13

Average of triplicate results. Mean ± SD standard deviation.

Table 5: Results of LH transdermal flux and lag time, permeability coefficient, diffusion coefficient and enhancement ratio of batches P1 to P9.

Batch Code	*Transdermal Flux Jss (µg/cm²/hr) ±SD*	*Lag time (hours)*	*Permeability Coefficient (Kp) (cm/hr) ±SD*	*Diffusion Coefficient (D) (cm/h×10-8) ±SD*	*Enhancement Ratio*
P1	97.30±0.11	1.25±0.11	1.21×10^{-3} ±0.21	0.01306±0.11	1.223±0.01
P2	99.17±0.12	1.30±0.12	1.29×10^{-3} ±0.22	0.01398±0.12	1.113±0.02
P3	119.6±0.12	1.34±0.12	1.48×10^{-3} ±0.22	0.01416±0.13	1.343±0.03
P4	117.6±0.13	1.35±0.13	1.46×10^{-3} ±0.23	0.0154±0.14	1.314±0.04
P5	127.4±0.14	1.38±0.14	1.58×10^{-3}± 0.24	0.0156±0.15	1.426±0.05
P6	129.6±0.15	1.29±0.01	1.61×10^{-3} ±0.25	0.022±0.16	1.449±0.06
P7	137.8±0.16	1.31±0.11	1.71×10^{-3} ±0.26	0.023±0.17	1.539±0.07
P8	151.2±0.17	1.30±0.12	1.78×10^{-3} ±0.27	0.026±0.18	1.606±0.08
P9	164.3±0.18	1.25±0.2	1.86×10^{-3} ±0.28	0.0345±0.19	1.674±0.09

Average of triplicate results. Mean ± SD standard deviation.

Table 6: stability studies results of optimized batch P9

Stability conditions	*Sampling time*	*Folding endurance*	*Drug content uniformity(%)*	*Ex-vivo drug release (%)*	*Visual appearance*

Room Storage (30±2˚C and 65±5% RH)	Initial (0 day)	398±1.52	98.39±0.65	93.14±0.85	Clear homogeneous appearance
	After 15 days	398±1.06	98.11±0.21	93.10±0.80	Clear homogeneous appearance
	After 30 days	398±2.64	98.34±0.65	93.12±0.09	Clear homogeneous appearance
	After 90 days	397±2.98	98.77±0.65	93.07±0.89	Clear homogeneous appearance
	After 180 days	397±3.65	98.12±0.12	92.94±0.54	Clear homogeneous appearance
Accelerated condition (40±2˚C and 75±5% RH)	Initial (0 day)	198±1.52	98.45±0.65	90.37±0.88	Clear homogeneous appearance
	After 15 days	218±1.98	98.89±0.35	82.12±0.42	Clear homogeneous appearance
	After 30 days	197±2.98	98.37±0.64	85.07±0.87	Clear homogeneous appearance
	After 90 days	193±3.65	97.19±0.17	86.34±0.54	Clear homogeneous appearance
	After 180 days	230±1.52	96.45±0.65	89.65±0.82	Clear homogeneous appearance

ACKNOWLEDGEMENT

The authors show their sincere thanks to Glenmark generics limited, Pune, India for providing the gift sample of Lercanidipine hydrochloride (LH). The authors are very thankful to Parul institute of pharmacy and research, Nootan College of Pharmacy and R.K.University for providing the necessary facilities to carry out the research work.

DECLARATION OF CONFLICT INTEREST

The authors declare that here no conflicts of interest. The authors alone are responsible for the content and writing of this article.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

(Proceedings available for download at rku.ac.in/icre)

RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

Development and Validation of a UV spectroscopic Method for the Estimation of Serratiopeptidase and Diclofenac potassium in Pharmaceutical Formulation

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ABSTRACT

A simple, rapid and accurate UV spectroscopic method has been developed for estimation of Serratiopeptidase (SER) and Diclofenac potassium (DCL) by spectrophotometry. The method developed is sensitive, rapid, accurate and economical. In this method, concentration of Diclofenac potassium was determined directly from calibration plot by measuring absorbance at 300 nm and concentration of Serratiopeptidase was determined after correction of absorbance of Diclofenac potassium at 300 nm. The Beer's law is obeyed in the concentration range of 1-40 µg/mL for Serratiopeptidase at 210 nm and 1-70 µg/mL for Diclofenac potassium at 300 nm and 210 nm. The regression coefficient (r^2) for SER and DCL were found to be 0.9996 and 0.9933 for DCL. Validation parameter was found statistically significant.

SUMMARY

Development of analytical method for simultaneous estimation of Serratiopeptidase and Diclofenac potassium by UV method.

Keywords: Serratiopeptidase, Diclofenac potassium, anti-inflammatory, absorbance correction method, spectrophhotometric

INTRODUCTION

Serratiopeptidase (SER), a proteolytic enzyme has anti-inflammatory activity *(1)*. The proteolytic enzyme which act as anti inflammatory binds to the alpha-2-macroglobulin in the blood, and helps masking antigenicity. This enzyme slowly moves towards area of inflammation. Oedema is caused due to Serratiopeptidase, hydrolyzebradykinin, histamine and serotoxin. This leads to reduction of swelling,

improving microcirculation as well as expectoration of sputum. Serratiopeptidase thus has anti-inflammatory, antioedemic and fibrinolytic activity. It acts quickly on localized inflammation *(2)*. Diclofenac potassium (DCL) is chemically, [2-[(2,6-Dichlorophenyl)Amino]Phenyl]Acetate *(3)*. Diclofenac potassium leads to inhibition of leukocyte migration and the cyclooxygenase (COX-1 and COX-2).

- Nonsteroidal anti-inflammatory drugs, also referred to as NSAIDs are the most common medicines that are prescribed for the treatment of illnesses such as arthritis *(5)*. The majority of the individuals is familiar with the non prescription NSAIDs including aspirin and ibuprofen *(6)*.
- Literature survey reveals that few HPLC and UV methods (7–14) are reported for determination of anti-inflammatory as single component in bulk, formulations as well as biological fluids. For SER and DCL, no UV and HPLC methods have been reported for simultaneous analysis. Some companies have made available the combination of 10mg of Serratiopeptidase and 50 mg of Diclofenac potassium in the market. So an attempt to estimate two drugs simultaneously by spectrophotometric analysis has been made. The primary objective is to develop and validate a method for the estimation of the combined dosage form by simultaneous UV spectroscopic method.

MATERIALS AND METHODS

Instrumentation:

The instrument used in the investigation was LABTRONICS double beam UV/Visible spectrophotometer (ModelLT-2900). Spectral bandwidth is of 1 nm. Weighing of all components was carried out on electronic balance (Shimadzu, Model BL – 220H).

Reagents and chemicals:

The pure analytical samples of SER and DCL were supplied by JC Biotech Private Limited, Hyderabad, India and Aarti Drugs Ltd. Mumbai, India respectively. The samples were analytically pure so were used directly. The dosage form used to estimate was Seraid tablet (Preet Remedies (p) Ltd.Baddi.). The film coated tablet contained 10 mg of Serratiopeptidase and 50 mg of Diclofenac potassium as labelled.

Absorbance correction method:

The standard stock solutions (100 µg/mL) of SER and DCL were prepared. This was done by dissolving 10 mg of drug in distilled water distinctively. SER had λ_{max} at 195 nm and DCL's λ_{max} was at 222 nm and 276 nm. To carry out simultaneous determination of SER and DCL, the two wavelengths selected should be such that at one wavelength the absorbance of one drug should be zero, whereas other drug shows significant absorbance value. At other wavelength, both the drugs should have significant absorbance value. So the two wavelength chosen were 210.0 nm and 300.0 nm. At 210.0 nm Serratiopeptidase and Diclofenac potassium both showed significant absorbance and at 300.0 nm Serratiopeptidase does not show any absorbance, but Diclofenac potassium showed significant absorbance.

The stability of the Serratiopeptidase in solvent (distilled water) was checked by measuring the absorbance of 10 µg/mL solution at 210.0 nm at specified time intervals. The stability was checked for four hours. The stability of the Diclofenac potassium in solvent (distilled water) was checked by measuring the absorbance of 50 µg/mL solution at 300.0 nm at specified time intervals. The stability was checked for four hours.

Samples of each drug were pipetted out from the standard stock solution (100 µg/mL). The samples were put into series of 10 mL volumetric flask. The volume was made upto the mark with distilled water. Sets of various concentration of solutions were obtained like 1, 2, 5, 10, 20, 30, 40 µg/mL for Serratiopeptidase and 1, 2, 5, 10, 20, 30, 40, 50, 60, 70 µg/mL for Diclofenac potassium. The absorbance

of all above solutions were measured at the specific wavelengths (i.e., 210 nm and 300 nm). Absorbances of each solution were plotted against respective concentrations. Drug concentration range which obeyed Beer's law was chosen. SER and DCL were linear with absorbances within range of 1-40 µg /mL and 1-70 µg/mL at their selected wavelengths respectively. Correlation coefficients obtained were 0.9996 for SER and 0.9933 for DCL. The optical characteristics and regression values for the calibration curve are presented in Table 1.

Solutions of 10µg /mL SER and 50µg/mL of DCL were prepared. This preparation was done by dilution of appropriate volumes of the standard stock solutions. The solutions of SER and DCL were scanned between 190 to 400 nm to obtain overlay spectra (Fig. 1). Absorbance and absorptivity of both standards was checked at selected wavelengths λ_1(300nm) and λ_2(210 nm). The mixed standard for pure drug was prepared from the stock solutions. Mixed standards that were prepared contained 10 µg/mL SER and 50 µg/mL DCL. The absorbances were recorded at selected wavelengths λ_1 and λ_2.

The concentration of C_{DCL} and C_{SER} is obtained by solving equation (1) and (2)

- $C_X = A_1 / ax_1$ ..... (1)
- $C_Y = [A_2 – (ax_2 \times C_X)] / ay_2$.....(2)

Concentration of DCL and SER in the powder mixture is found by using equation (3) and (4).

$C_{DCL} = A_1 / 20.14$ ..... (3)

$C_{SER} = [A_2 – (43.08 \times C_{DCL})]/14.28$ ..... (4)

- Where,
  - 20.14 and 43.08 are absorptivities of DCL at λ_1 and λ_2 respectively.
  - 0.00 and 14.28 are absorptivities of SER at λ_1 and λ_2 respectively.
  - A_1 and A_2 are absorbances of mixtures at λ_1 and λ_2 respectively.
  - C_{DCL} and C_{SER} are concentrations in gm/liter.

Twenty tablets of Serratiopeptidase and Diclofenac potassium in combination (brand name Seraid and manufactured by Preet Remedies (p) Ltd. Baddi.) were weighed. Average weight was determined. The tablets were converted to powder form with help of trituration. Tablet powder equivalent to 10.0 mg of SER and 50.0 mg of DCL was weighed. It was put in 100 mL volumetric flask. The content was dissolved in 50 mL distilled water followed by ultrasonication for 20 minutes. Final volume was made upto the mark with distilled water followed by filtration with the help of Whatman filter paper No.41.

From this tablet solution, 1 mL was pipetted out. Further dilution upto 10 mL with distilled water was done to obtain 10.0 µg/mL SER and 50.0 µg/mL DCL. The mixed sample solutions were scanned to obtain spectra and absorbance value at 210.0 nm and 300.0 nm were noted. The concentration of Serratiopeptidase and Diclofenac potassium were calculated from the equation (3) and (4). Here solution of Serratiopeptidase tablets were prepared and maintained in a dark room condition throughout experimental work.

RESULTS AND DISCUSSION

From the overlay spectra of two drugs in distilled water, the working wavelengths selected for absorbance correction method were 210 nm and 300 nm. At 300 nm only DCL shows significant absorbance, whereas SER shows zero absorbance. At other wavelength (i.e., 210 nm) SER and DCL both shows significant absorbances.

This is simple technique for simultaneous estimation of SER and DCL in combined sample solutions. Results of the analysis of pure drugs, analysis of tablet and recovery studies are reported in table No.2,4,6 and data for statistical validation are given in table No.3,5,7.
The relative standard deviation (%R.S.D.) is less than 2 % and standard error (S.E.) are very low, indicating high degree of precision of the method as per USP and ICH guidelines.

CONCLUSION

The developed method successfully estimated the amount of Serratiopeptidase and Diclofenac potassium in formulation containing 10 mg of Serratiopeptidase and 50 mg of Diclofenac potassium. The results obtained were similar to the labeled values. This method highly indicated non-interference of excipients in the estimation. This method was precise, accurate and sensitive. This method involves easy calculation. It can be effectively applied for routine analysis of Serratiopeptidase and Diclofenac potassium in tablet for assay and dissolution testing.

FIGURES

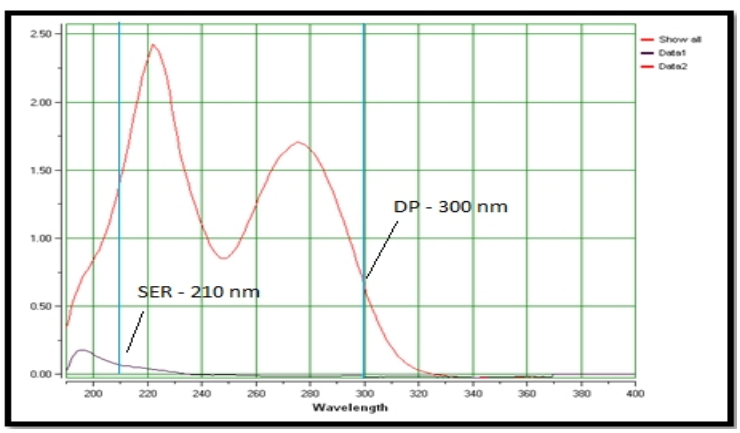


Fig. 1: Overlay Spectra of Diclofenac potassium and Serratiopeptidase

TABLES

Table 1: Regression and Optical Characteristics of Serratiopeptidase and Diclofenac potassium

Parameters	SER	DCL

Working λ (in distilled water)	210 nm	300 nm
Beer's Law range	1-40 µg/mL	1-70 µg/mL
Molar absorptivity (l / mole.cm)	1.5157×10^4	1.0780×10^4
Regression Values:		
1.Slope	0.0133	0.0221
2.Intercept	0.0050	-0.0313
3.Regression coefficient (r^2)	0.9996	0.9933

Table 2: Data for Analysis of Pure Drug

Sr. No.	Amount Present in µg/mL		Amount Found in µg/mL		Amount Found in %	
	SER	DCL	SER	DCL	SER	DCL
1.	10	50	O9.90	50.00	99.00	100.00
2.	10	50	09.87	50.20	98.70	100.40
3.	10	50	10.00	49.90	100.00	101.80
4.	10	50	09.90	50.20	99.00	100.40
5.	10	50	10.10	50.00	101.00	100.00
6.	10	50	09.87	50.10	98.70	100.20

Table 3: Statistical Validation of Pure Drugs

Drug	Mean* (%)	Standard Deviation*	Co-efficient of Variation* (% R.S.D.)	Standard Error*
SER	99.40	0.9186	0.9241	0.3773
DCL	100.46	0.6772	0.6740	0.2752

SER is Serratiopeptidase and DCL is Diclofenac potassium,* Here Mean is the average of (n=6) results.

Table 4: Data for Analysis of Tablet

Sr. No.	Label Claim (mg/tab)		Amount Found (mg/tab)		% of Label Claim	
	SER	DCL	SER	DCL	SER	DCL
1.	10	50	O9.70	49.40	97.00	98.80

2.	10	50	09.87	50.20	98.70	100.40
3.	10	50	10.10	49.90	101.00	101.80
4.	10	50	09.90	50.20	99.00	100.40
5.	10	50	10.10	49.50	101.00	99.00
6.	10	50	09.70	50.10	97.00	100.20

Table 5: Statistical Validation of Tablet

Drug	Mean* (%)	Standard Deviation*	Co-efficient of Variation* (% R.S.D.)	Standard Error*
SER	98.95	1.7930	1.8120	0.7399
DCL	100.10	1.0936	1.0925	0.4461

Table 6: Data for Analysis of Recovery Studies

Level of % Recovery	Amount Present (mg/tab)		Amount of Standard Added (mg)		Total Amount Recovered (mg)		%Recovery	
	SER	DCL	SER	DCL	SER	DCL	SER	DCL
80	10	50	8	40	17.80	90.10	98.88	100.10
80	10	50	8	40	17.80	89.80	98.88	99.77
80	10	50	8	40	18.10	90.10	100.50	100.10
100	10	50	10	50	20.10	99.50	100.50	99.50
100	10	50	10	50	19.80	100.20	99.00	100.20
100	10	50	10	50	20.10	99.50	100.50	99.50
120	10	50	12	60	21.95	109.80	99.77	99.81
120	10	50	12	60	21.95	109.70	99.77	99.72
120	10	50	12	60	22.10	109.80	100.40	99.81

Table 7: Statistical Validation of Recovery Studies

Level of % Recovery	% Mean Recovery*		Standard Deviation*		Co-efficient of Variation* (% R.S.D.)		Standard Error*	
	SER	DCL	SER	DCL	SER	DCL	SER	DCL
80	99.42	99.99	0.9353	0.1905	0.9407	0.1905	0.5431	0.1099
100	100.00	99.73	0.8660	0.4041	0.8660	0.4051	0.5000	0.2339
120	99.98	99.78	0.3637	0.0519	0.3637	0.0520	0.2100	0.0300

* Here % recovery is average of three results at each level.

ACKNOWLEDGEMENT

The authors thanks M/S JC Biotech Private Limited, Hyderabad and M/S Aarti Drugs Ltd. Mumbai for providing gift samples of Serratiopeptidase and Diclofenac potassium respectively.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

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Development and validation of an analytical method for the simultaneous estimation of Diclofenac Potassium, Paracetamol and Serratiopeptidase from bulk and multicomponent formulation by Spectrophotometric method

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ABSTRACT

Novel, simple, sensitive, rapid and accurate spectrophotometric method has been developed for simultaneous estimation of Serratiopeptidase (SER), Paracetamol (PCM) and Diclofenac potassium (DCL). In the method, concentration of Diclofenac potassium was determined directly from calibration plot by measuring absorbance at 309 nm and amount of Paracetamol was determined at 299 nm after correcting absorbance of Diclofenac potassium. The concentration of Serratiopeptidase was determined at 210 nm after correcting absorobance of Paracetamol and Diclofenac potassium at this wavelength. The Beer's law obeyed in the concentration range of 2-30 µg/mL for Diclofenac Potassium at 309 nm and 2-80 µg/mL for Paracetamol at 299 nm and 2-15 µg/mL for Serratiopeptidase at 210 nm. The regression coefficient (r^2) values were found to be 0.997 for DCL, 0.997 for PCM and 0.999 for SER. Result of the method was validated statistically and by recovery studies.

SUMMARY

Analytical method for simultaneous estimation of Serratiopeptidase (SER), Paracetamol (PCM) and Diclofenac potassium (DCL).

Keywords: Serratiopeptidase, Paracetamol, Diclofenac potassium, anti-inflammatory, absorbance correction method, spectrophotometric

INTRODUCTION

Non-steroidal anti-inflammatory drugs also referred to as analgesics. The non-steroidal anti-inflammatory medicines are a class of drugs that provide analgesic and antipyretic (fever-reducing) effects, and in higher doses it provides anti-inflammatory effects *(1)*.
NSAIDs inhibit the synthesis of prostaglandins and thromboxanes by inhibiting the activity of both cyclooxygenase-1 (COX-1) and cyclooxygenase-2 (COX-2) *(2)*.
Diclofenac potassium is a non selective cyclooxygenase-2 inhibitor with antipyretic, analgesic and anti inflammatory action *(3)*. It inhibits the leukocyte migration and the enzyme cyclooxygenase-1 and cyclooxygenase-2 *(4-7)*. Chemically it is 2-[(2,6-Dichloro phenyl) Amino] Phenyl] Acetate.

Paracetamol is a non selective COX-2 inhibitor with antipyretic, analgesic with poor anti-inflammatory action *(3)*. It inhibits both isoforms of cyclooxygenase, COX-1, COX-2, and COX-3 enzymes involved in prostaglandin (PG) synthesis *(8-11)*. Chemically it is 4-hydroxyacetanilide *(12)*.

Serratiopeptidase produces anti-inflammatory effect by hydrolysis of histamine, bradykinin and serotonin. Serratiopeptidase also increases plasmin activity by inhibiting the plasmin inactivators, thus produce fibrinolytic and proteolytic effects *(13)*.
Literature survey reveals that few UV methods and HPLC *(14–27)* have been reported for determination of anti-inflammatory as single component in formulations, in bulk and in biological fluids. The objective of the investigation is to develop and validate a method for the estimation of the combined dosage form by simultaneous UV spectroscopic method.

MATERIALS AND METHODS

Instruments Used

FTIR: Infra 3000A FTIR
Analytical Balance: Dhona
Ultrasonic Bath: Today – Tech

UV Visible Spectrophotometer (Double beam)
Make: LABTRONICS
Model: LT-2900
Specification: Wavelength range: 190 to 1100 nm
Spectral Bandwidth: 2 nm

Reagent and Materials
Paracetamol: Farmason Pharmaceutical Gujarat Pvt. Ltd.
Serratiopeptidase: JC Biotech private limited, Hyderabad
Diclofenac potassium: Tripada Pharmaceutical, Ahmedabad
Methanol: Molychem, Mumbai
Market Formulation: Seradic™-P (Marketed by: OBSURGE BIOTECH LTD. Laxmi Nagar, Delhi-110092)

Here, solution of Serratiopeptidase was prepared and maintained in a dark room condition throughout experimental work.

Absorbance Correction method
Selection of Analytical wavelength:

Standard stock solutions of 100 µg/mL of DCL, PCM and SER were prepared by dissolving separately 10 mg of Serratiopeptidase, 10 mg Diclofenac potassium and 20 mg Paracetamol in 50 mL of solvent. Mixture of methanol and distilled water in a ratio of 40:60 is used as the solvent.

Using appropriate dilution of standard stock solution, three solutions were scanned separately in order to get good results. By using overlay spectra of three drugs, working wavelengths selected were 210 nm, 299 nm and 309 nm. (Fig.3) Here, amount of Diclofenac potassium was determined at 309 nm using standard calibration curve (using equation y = mx+c), because at this wavelength both Serratiopeptidase and Paracetamol shows zero absorbance whereas Diclofenac potassium gives significant absorbance value. At 299 nm Serratiopeptidase does not shows absorbance, but Paracetamol and Diclofenac potassium gives significant absorbance value. So, amount of Paracetamol was determined at 299 nm after correcting absorbance of Diclofenac potassium. The concentration of Serratiopeptidase was determined at 210 nm after correcting aborobance of Paracetamol and Diclofenac potassium at this wavelength.

The stability of the Serratiopeptidase in solvent was checked by measuring the absorbance of 20 µg/mL solution at 210 nm for five hour. The stability of the Diclofenac potassium in solvent was checked by measuring the absorbance of 20 µg/mL solution at 210 nm, 299 nm and 309 nm for five hour. The stability of the Paracetamol in solvent was checked by measuring the absorbance of 20 µg/mL solution at 210 nm and 299 nm for five hour.

For each drug from the standard stock solution, appropriate aliquots were pipetted out into series of 10 mL volumetric flask. The volume was made upto the mark with solvent to get a set of solutions having the concentration of 2, 5, 10, 15, 20 µg/mL for Serratiopeptidase and 2, 5, 10, 20, 30 µg/mL for Diclofenac potassium and 2, 5, 10, 20, 30, 40, 50, 60 µg/mL for Paracetamol. The absorbance of Serratiopeptidase was measured at 210 nm. The absorbance of Diclofenac potassium was measured at 210 nm, 299 nm and 309 nm. The absorbance of various solution of Paracetamol was measured at 210 nm and 299 nm. The absorbance was plotted against concentration in each case. The concentration range was chosen over which the drugs obeyed Beer's law. The range was found to be 2 µg/mL to 20 µg/mL for Serratiopeptidase, 2 µg/mL to 30 µg/mL for Diclofenac potassium and 2 µg/mL to 60 µg/mL for Paracetamol.

The absorptivity of all three drugs was calculated at selected wavelength using data of calibration curve (Table 1). The absorptivities were used in forming equation for absorption correction method (equation 25, 26 and 27). The concentration of $C_{DCL,}$ C_{PCM} and C_{SER} can be obtained by solving this equation (1, 2 and 3).

$C_{DCL} = A_1 / 8.86$ … (Eq. 1)

$C_{PCM} = (A_2 - (23.93\ C_{DCL})/4.23$ … (Eq. 2)

$C_{SER} = (A_3 - (89.5\ C_{DCL} + 36.61\ C_{PCM}))/ 31.08$ … (Eq. 3)

Where,

$A_{1,}$ A_2 and A_3 = absorbances of sample solution at 309 nm, 229 nm and 210 nm respectively.

DCL

8.86 = absorptivity coefficients of DCL at 309 nm,

23.93 = absorptivity coefficients of DCL at 299 nm

89.5 = absorptivity coefficients of DCL at 210 nm

PCM

4.23 = absorptivity coefficients of PCM at 299 nm and

36.61 = absorptivity coefficients of PCM at 210 nm

SER

31.08 = absorptivity coefficient of SER at 210 nm.

Now if a mixture of Serratiopeptidase, Diclofenac potassium and Paracetamol were to be analysed a solution of suitable dilution should be prepared in solvent. The absorbance of the solution at 210 nm, 299 nm and 309 nm were measured. Then values were substituted in equation (1, 2 and 3) to get concentration of Serratiopeptidase, Diclofenac potassium and Paracetamol.

Twenty tablets of Serratiopeptidase, Diclofenac potassium and Paracetamol in combination were weighed. Their average weight was determined and tablets were crushed to make powder sample. Tablet average weight was found to be 466.3 mg. From the triturate, 7.173 mg tablet powder (equivalent to 0.23 mg of Serratiopeptidase, 0.7692 mg of Diclofenac potassium and 5 mg of Paracetamol) was weighed and then transferred into 50 mL volumetric flask and dissolved in solvent and the content was kept in ultrasonicator for 20 min. Finally the volume was made upto the mark with solvent. The solution was filtered through Whatman filter paper No.41. From this solution 32.50 mL was transferred into another 50 mL volumetric flask and 3.5 mL standard stock solution of Serratiopeptidase (100 µg/mL) was added. Volume was made upto the mark with solvent to make final concentration of 10 µg/mL for Serratiopeptidase, 10 µg/mL for Diclofenac potassium and 65 µg/mL for Paracetamol. Absorbances of this prepared solution was measured at 210 nm, 299 nm and 309 nm. Concentration of Serratiopeptidase, Diclofenac potassium and Paracetamol in tablet formulation was calculated by putting absorbance values into equation (1, 2 and 3).

RESULTS AND DISCUSSION

From examination of overlay spectra of drugs, the three wavelength chosen were 210 nm, 299 nm and 309 nm. Here, Serratiopeptidase showed absorbance at 210 nm, Paracetamol showed absorbance at 210 nm and 299 nm, whereas Diclofenac potassium showed absorbance at 210 nm, 299 nm and 309 nm. This method for simultaneous estimation of DCL, PCM and SER in combined sample solutions was found to be simple, reproducible and accurate. Table No.1 shows data for the optical characteristics. Results of the analysis of pure drugs, analysis of tablet, recovery studies, intra day precision and inter day precision are reported in table No.3,5,7,9,11 and statistical validation data are given in table No.4,6,8,10,12. When the equations are determined, analysis requires only the measurement of the absorbances of the sample solution at the two wavelengths selected, followed by a few simple calculations.

The SD, RSD and SE calculated are low which indicates high degree of precision of the method.

CONCLUSION

The spectrophotometric methods were developed, validated and successfully applied to multicomponent tablet dosage form for determination of amount of SER, PCM and DCL. The results of tablet analysis and recovery studies revealed that developed method can be successfully applied to the routine determination of these three drugs from dosage forms.

FIGURES:

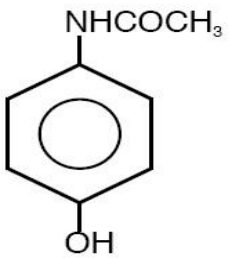


Fig. 1. Structure of Paracetamol and Serratiopeptidase

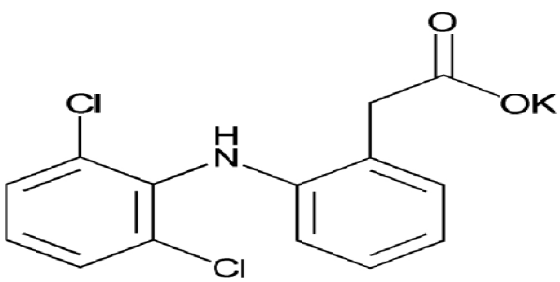


Fig. 2. Structure of Diclofenac potassium

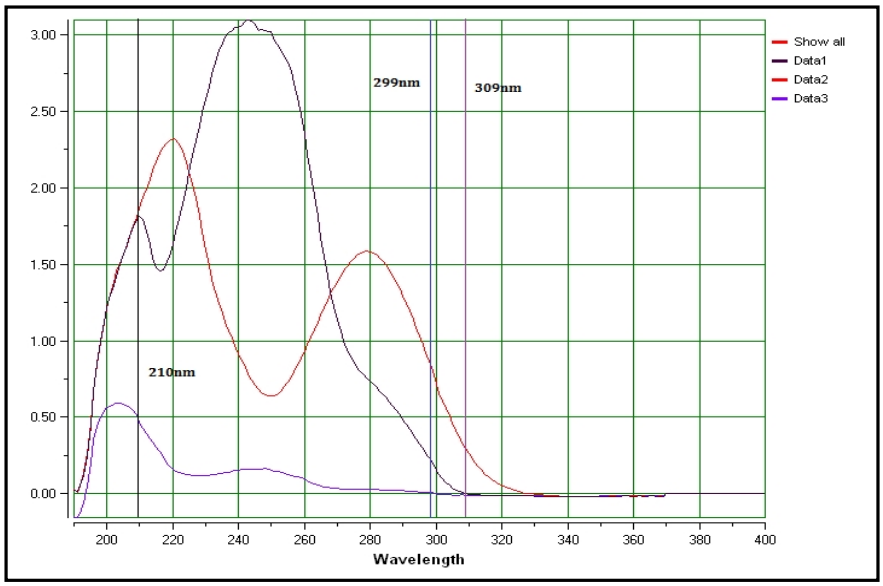


Fig. 3. Overlay spectra of Diclofenac potassium, Paracetamol and Serratiopeptidase

.

TABLES

Table 1. Absorptivity measurement

Absorptivity	SER	DCL	PCM
210 nm	31.08 (az_3)	89.5 (ax_3)	36.61 (ay_3)
299 nm	-	23.93 (ax_2)	4.23 (ay_2)
309 nm	-	8.86 (ax_1)	-

Table 2. Optical characteristics and regression of Diclofenac potassium, Paracetamol and Serratiopeptidase

Parameters	DCL			PCM		SER
Working λ	210	299	309	210	299	210
Beer's law range	2-30 µg/mL	2-30µg/mL	2-30 µg/mL	2-80 µg/mL	2-80µg/mL	2-15 µg/mL
Regression coefficient (r^2)	0.995	0.998	0.997	0.996	0.997	0.999

Table 3. Analysis of powder mixture

SR. No.	Amount present in (µg/mL)	Amount found (µg/mL)	Amount found in (%)

	SER	DCL	PCM	SER	DCL	PCM	SER	DCL	PCM
1	10	10	65	10.391	10	64.28	103.91	100	98.90
2	10	10	65	10.456	10.11	66.66	104.56	101.1	102.55
3	10	10	65	10.520	10.22	64.76	105.20	102.2	99.63
4	10	10	65	10.585	101.11	68.71	105.85	101.1	101.92
5	10	10	65	10.262	103.3	64.52	102.62	103.3	99.26
6	10	10	65	10.423	104.4	65	104.23	104.4	100

Table 4. Statistical validation for powder mixture

Drug	Mean* %	Standard Deviation*	Co-efficient of Variation*	Standard Error*
SER	104.395	1.114	1.067	0.454
DCL	102.016	1.619	1.587	0.661
PCM	100.376	1.498	1.492	0.611

***n=6**

The % Relative Standard Deviation is less than 2% as required by ICH guidelines and USP.

Table 5. Analysis of tablet formulation

Sr. No.	Label Claim (mg/tablet)			Amount of drug present in prepared tablet solution (µg/mL)			Amount Found (µg/mL)			% of Label Claim		
	SER	**DCL**	**PCM**	**SER**	**DCL**	**PCM**	**SER**	**DCL**	**PCM**	**SER**	**DCL**	**PCM**
1	15	50	325	10	10	65	10.391	10.00	64.28	103.91	100	98.90
2	15	50	325	10	10	65	10.456	10.11	64.76	104.56	101.11	99.63
3	15	50	325	10	10	65	10.423	10.44	65.71	104.23	104.44	101.92
4	15	50	325	10	10	65	10.262	10.22	64.52	102.62	102.22	99.26
5	15	50	325	10	10	65	10.585	10.33	65.95	105.85	103.33	101.46
6	15	50	325	10	10	65	10.649	10.00	66.66	106.49	100	102.55

Table 6. Statistical validation of tablet formulation

Drug	Mean* %	Standard Deviation*	Co-efficient of Variation*	Standard Error*
SER	104.61	1.390	1.328	0.567

DCL	101.85	1.812	1.779	0.739
PCM	100.62	1.543	1.533	0.630

***n=6**

The % Relative Standard Deviation is less than 2% as required by ICH guidelines and USP.

Table 7. Recovery studies

Level of % Recovery	Amount present (mg/tablet)			Amount of standard added (mg/tablet)			Amount of drug present in prepared tablet solution (µg/ml)			Amount Found (µg/ml)			%Recovery		
	SER	DCL	PCM	SER	DCL	PCM	SER	DCL	PCM	SER	DCL	PCM	SER	DCL	PCM
80	15	50	325	12	40	260	10	10	65	9.894	9.872	65.46	98.94	98.72	100.72
80	15	50	325	12	40	260	10	10	65	10.72	10.72	65.73	100.72	101.83	101.13
80	15	50	325	12	40	260	10	10	65	10.25	10.25	67.45	102.50	100.00	103.77
100	15	50	325	15	50	325	10	10	65	10.03	10.00	65.47	100.30	100.00	100.73
100	15	50	325	15	50	325	10	10	65	10.19	10.19	66.64	101.95	103.33	102.53
100	15	50	325	15	50	325	10	10	65	10.35	10.35	67.97	103.55	102.75	104.57
120	15	50	325	18	60	390	10	10	65	9.85	9.854	63.84	98.54	98.45	98.23
120	15	50	325	18	60	390	10	10	65	10.00	10.00	65.90	100.00	98.95	101.39
120	15	50	325	18	60	390	10	10	65	10.14	10.14	66.00	101.45	100.50	101.55

Table 8. Statistical validation for recovery studies

Level of % Recovery	% Mean Recovery*			Standard Deviation*			Co-efficient of Variation*			Standard Error*		
	SER	DCL	PCM	SER	DCL	PCM	SER	DCL	PCM	SER	DCL	PCM
80	100.72	100.18	101.87	1.780	1.563	1.655	1.767	1.560	1.624	0.726	0.638	0.675
100	101.93	102.02	102.61	1.625	1.778	1.921	1.594	1.742	1.872	0.663	0.726	0.784
120	99.99	99.30	100.39	1.45	1.068	1.872	1.450	1.070	1.864	0.562	0.436	0.764

***n=3**

The % Relative Standard Deviation is less than 2% as required by ICH guidelines and USP.

Table 9. Intra – day precision

Sr.	Amount present (µg/mL)	Amount found	% of Label Claim

No.				(µg/mL)					
	SER	DCL	PCM	SER	DCL	PCM	SER	DCL	PCM
1	10	10	65	10.39	10	64.28	103.90	100.0	98.89
2	10	10	65	10.45	10.22	64.76	104.50	102.2	99.63
3	10	10	65	10.26	10.11	65.23	102.60	101.1	100.35
4	10	10	65	10.52	10.22	65.00	105.20	102.2	100.00
5	10	10	65	10.48	10.33	64.52	104.80	103.3	99.26
6	10	10	65	10.71	10.00	66.42	107.10	100.0	102.18

Table 10. Statistical validation for intra – day precision

Drug	Mean* %	Standard Deviation*	% Relative Standard Deviation*	Standard Error*
SER	104.68	1.490	1.423	0.608
DCL	101.46	1.332	1.312	0.543
PCM	100.05	1.164	1.163	0.475

*** n=6**

Table 11. Inter – day precision

Sr. No.	Label claim (mg/tab)			Amount found (mg/tab)			Amount found in %		
	SER	DCL	PCM	SER	DCL	PCM	SER	DCL	PCM
1	10	10	65	10.45	9.888	64.52	104.5	98.88	99.26
2	10	10	65	10.52	10.00	64.76	105.2	100	99.63
3	10	10	65	10.39	10.11	65.23	103.9	101.1	100.35
4	10	10	65	10.06	10.22	65.71	100.6	102.2	101.09
5	10	10	65	10.35	10.33	66.19	103.5	103.3	101.83
6	10	10	65	10.42	10.00	66.66	104.2	100	102.55

Table 12. Statistical validation for inter – day precision

Drug	Mean* %	Standard Deviation*	% Relative Standard Deviation*	Standard Error*
SER	103.65	1.600	1.543	0.653
DCL	100.91	1.624	1.609	0.663
PCM	100.78	1.276	1.266	0.521

*** n=6**

ACKNOWLEDGEMENT

The authors thank JC Biotech Private Limited, Hyderabad, Tripada Pharmaceutical, Ahmedabad and Farmason Pharmaceutical, Gujarat Pvt. Ltd. for supplying gift samples of Serratiopeptidase, Diclofenac potassium and Paracetamol respectively to carry out this work.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

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RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

Preformulation Studies & Preparation Of Cyclophosphamide Loaded Solid Lipid Nano Particles

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ABSTRACT:

Cyclophosphamide is a chemotherapeutic drug belongs to nitrogen mustard alkylating agents. It is derived from oxazophorines group. It has good anti-cancer activity. Many injectable forms of cyclophosphamide are available, but all has poor bioavailability and nonspecific targeting problems. It was attempted to formulate cyclophosphamide in the form of cationic solid lipid nanoparticles. Solid lipid nanoparticles were obtained by adsorption of lipid dispersion in the organic phase and diffusion in the aqueous phase of the high-speed Homogenizer technique at 30000 RPM. Eventually, pre-formulation tests are carried out to escalate compatibility of drug and excipient. We performed FTIR, DSC, XRD studies. No significant changes observed in drug and solid lipid compatibility. Melting point, partition coefficient, UV, HPLC studies shows satisfactory results. After pre-formulation study cyclophosphamide loaded solid lipid nanoparticles was prepared. It is concluded that solid lipid nanoparticles of cyclophosphamide could be formulated with the selected lipids.

SUMMARY: Preformulation studies and preparation of cyclophosphamide loaded solid lipid nanoparticles.

Keywords: Cyclophosphamide, Solid lipid nanoparticles, Pre-formulation studies, DSC, FTIR, XRD.

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1. INTRODUCTION: Glioblastoma multiforme (GBM) one of the most devastating brain carcinoma, it has a habitual recurrence with narrow prognosis (***1***)**.** This physiological condition got recently much more medical attention, as the number of deaths increasing rapidly by brain carcinoma. For a considerable period of time chemotherapy is the last choice in GBM, but the systematic administration of chemotherapeutic drugs causes savior non-selective cytotoxicity on normal cells, secondary alopecia is also observed on the diseased. So specific targeting to the cancerous cell become a paramount importance in cancer treatment (***2***). Nanoparticular drug delivery systems emerging as an excellent approach to circumvent all those associated problems with chemotherapy, as nanomedicines are below 1µm and because of this property, easy endocytosis through the Blood Brain Barrier (BBB), enhance cell internalization and delivery of drugs to

the intracellular matrix are possible (***3***). Apart from it, conjugation with biodegradable polymers, anti - epidermal growth factor, folic acids, antibody along with core drugs, enhance cellular uptake and selectivity. With up gradation in nano delivery systems, Solid Lipid Nano Particles (SLNs) becoming an emerging nanocarrier for chemotherapeutic drugs because of its high cellular uptake and rapid clearance. SLNs prepared from lipids such as Triglycerides (Trilaurin™, Tristearin™, Dynasan122™), Acylglycerides (Glyceryl monostearate, Glyceryl Palmitostearate) Fatty acids (Stearic acid, Palmitic acid, Behenic acid, Waxes (Cetyl alcohol, Beeswax, cholesterol, Emulsifying wax NF), Hydrogenated soybean oil (Lipo ™ or Sterotex™) (***4***). Recent studies confirmed (Giulia Fulci *et al*) Cyclophosphamide a synthetically modified antineoplastic drug obtained from nitrogen muster, enhanced glioma virotherapy by inhibiting the inner immune response. In rat glioma model (***5***) cyclophosphamide enhance the natural killer cells, microglia associated with macrophages of CD68+ & CD163+, INF-ϒ. This research accelerates the need for extensive research on cyclophosphamide as a nanocarrier drug. In this research, we carried out our pre-formulation studies on the model drug as cyclophosphamide. We attempted to formulate Solid lipid nanoparticles of cyclophosphamide using a different ratio of Glyceryl monostearate and citric acid as a lipid, Soya lecithin as co-surfactant, Brij 78, Poloxamer 188, Tween 80 as a surfactant, HPLC water as an aqueous solvent, Dichloromethane, methanol (acetone free) as an organic solvent. The formulation was prepared by solvent diffusion and dispersion technique (***6***).

2. MATERIALS: Cyclophosphamide model drug was obtained as a gift sample from Emcure Pharmaceuticals LTD. Ahmadabad, India. Glycerol monostearate (product No: 17145) purchased from Astron chemicals, Ahmadabad, India. Soya lecithin (Product no: 88993) purchased from Astron chemical, Ahmadabad, India. Poloxamer 188 purchased from Balaji drugs Surat, India. Dichloromethane from Chem dyes corporation–Rajkot. Brij 78 obtained from Chem dyes corporation–Rajkot. Tween-80 procured from fine star industry. Carbon tetrachloride procured from Chem dyes corporation–Rajkot. Methanol (acetone free) was purchased from Chem dyes corporation–Rajkot. Polyvinyl alcohol and lactose monohydrate from Astron chemicals Ahmadabad, India. Ultipor N56 Nylon 6, 6 membrane filter-0.45mm, procured from Pall life science. Phosphate buffer from J&K Scientific LTD, 1L HPLC grade water from Fisher scientific Ltd.100ml Acetonitrile was purchased from Imperial Chemical. Ammonium molybdate, Ferric chloride solution, Stannous chloride are gift sample from RK University.

3. METHODS:

3.1. UV analysis:

3.1.1. Estimation of cyclophosphamide by UV spectroscopy (*7*):

3.1.2. Apparatus: Measurement was carried out by using SHIMADZU-1880UV-VIS Spectrophotometer at 722nm.

3.1.3. Sample: Cyclophosphamide model drug was obtained as a gift sample from Emcure Pharmaceuticals, Ahmadabad, India.

3.1.4. Materials and reagents for standard absorbance and linearity:

All the chemicals used were of analytical grade. All the solutions were prepared freshly and the deionized water is used throughout the experiment.

3.1.4.1. Ammonium molybdate (0.005M): Weighed exactly 0.61795g (± 0.0001g) of ammonium molybdate in 100 ml volumetric flask it was dissolved and diluted to the mark using deionised water.

3.1.4.2. Ferric chloride solution (0.01M): Weighed exactly 0.22560g (±0.0001g) of ferric chloride in 100 ml volumetric flask it was dissolved and then diluted to the mark using double distilled alcohol.

3.1.4.3. Stannous chloride (0.01M): Weighed exactly 0.16221g (±0.0001g) of stannous chloride in 100 ml volumetric flask it was dissolved and diluted to the mark with deionized water. Pharmaceutical grade Cyclophosphamide (CP) claimed to be 99.7 % pure was received from the Emcure Pharmaceuticals, Ahmadabad, India. Which is used as a reference standard for the analysis without any further purification?

3.1.5. Standard drug solution:

A stock solution of CP (1 mg/ml) was prepared by using 10 mg of the reference standard drug in a 10 ml volumetric flask, added 5 ml methanol to dissolve the content and brought up to the mark with methanol, mixed thoroughly for homogeneity. Working solution of lower concentration (100µg/ml) was prepared by further dilution of the above standard stock solution with methanol.

3.1.6. Linearity studies: For linearity studies we selected a different set of concentration, but the best curve fits between 20- 45 µg/ml of standard cyclophosphamide. At first six 10ml volumetric flask were taken & prepared 100µg/ml working standard first, from that 2ml,2.5ml,3ml,3.5ml,4ml,4.5ml were withdrawn and transferred into six 10 ml volumetric flask, then 1ml ferric chloride were added and all the content were kept in warm condition for 5 minutes, then contents were filtered and 2ml ammonium molybdate followed by 2ml of hot stannous chloride were added, slowly formation of blue colour phosphomolybdate complex were seen, then all the volumetric flasks were filled up to 10ml with double distilled water and kept at room temperature for cooling. The experiments were repeated for three times, and all eighteen volumetric flask content were measured at 722nm using reagent as a blank The average absorbances were selected for standard curve(figure1,table1).The calibration curve was plotted against absorbance(nm) versus concentration(µg/ml).

3.1.7. Determination of the absorption maxima (λmax): To determine the λmax of the coloured species, 1 ml of 100 µg/ml of the CP was added to a test tube and 1 ml of ferric chloride solution, heated the contents for 5 min. Filtered the contents of 10 ml standard volumetric flask, add 2 ml of ammonium molybdate solution followed by adding 2 ml of hot stones chloride solution resulting in the formation of a deep blue colored phosphomolybdate complex than the flasks are allowed to cool to room temperature and the solution made up to the mark with water. The coloured species were measured against reagent blank in the range of 400 NM at 800 NM. The λmax of the complex was found to be 722 mm. The absorption spectrum of the proposed method was shown in figure2. Under the experimental conditions, each reagent blank showed a negligible absorbance at the corresponding λmax.

3.2. HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY STUDIES ON CYCLOPHOSPHAMIDE:

3.2.1 Chromatography condition (*8*): A chromatographic system Dionex ultimate 300, consisting of a quaternary solvent delivery pump, and autoinjector, column oven and UV detector, A thermal scientific column ODS, a stationary phase with particle size 5µ, and pore size 100A was used. The column is end capped and carbon content of 11%, the flow rate was maintained 1.5ml/min (Table2).Blank and standard drug estimated by HPLC (figure 3&4, table 2, 3, 4, 5, 6)

3.2.2. Preparation of stock solution: 50mg standard cyclophosphamide API was accurately weighed and transferred into the 50 ml volumetric flask, later dissolved with diluent (HPLC water 50 ml: acetonitrile 50 ml % v/v) to produce 1000 µg/ml. Further, withdraw 1ml of above solution and transfer in the 100 ml volumetric flask. Add above-mentioned diluent to produce 100ml.This will give 10 µg/ml solution.

3.3. FOURIER TRANSFORM INFRARED SPECTROSCOPY: The pure and dryad cyclophosphamide were triturated with powdered potassium bromide in a proportion of 1:200.The triturated amount should be

taken such a way that, it would spread the area of the disk 5-15 g per 2 mm. By using high pressure or vacuum pressure a portion of the mixture was inserted. In suitable holder mount the resultant disk. Sometimes impurities in holder disk, inadequate or excessive grinding, and moisture in triturate causes unpleasable disks. After visual all inspection the disk to be placed. IR spectra run within 4000-400cm^{-1} range. Same way IR spectra of Drug with a physical mixture of Solid lipids; GSM, Stearic acids were performed. The experiments were carried out in SHIMADZU–FTIR-8400s model (figure 5, 6, 7 and table 7, 8).

3.4 DIFFERENTIAL SCANNING CALORIMETRY (DSC): The interaction between cyclophosphamide, GMS, stearic acid, soya lecithin has been checked in the solid state by SHIMADZU–DSC-60 Plus instrument. The chance of complex formation within the substances was marginal. By measuring the thermal behaviour of all substances with the drug showed in the phase diagram, indicates there was no interaction between the drug and excipients. The DSC curve of cyclophosphamide showed a melting endodermal at 51.20^{O}C.The normal melting point of cyclophosphamide as per monograph is in between 45-53 ^{O} C which is an incompatible range. The drug and physical mixture were prepared from GMS and Stearic acid, both the compound has a melting point between 50-61^{O}C. The drug and physical mixture endotherm show pick at 52.88^{O}C, and in 123.23^{O}C, 161.25^{O}C respectively, indicated no significant difference in melting endotherm with respect to melting point of an individual ingredient(figure 8,9).This proved the computability of the drug with other two ingredients(***9***).

3.5. MELTING POINT DETERMINATION: The fully dryad, free flowing powder of cyclophosphamide was loaded into a capillary tube, the capillary tube must be sealed in at one end before use. The powder must get settled in the bottom of the capillary tube against a hard surface. It was cross checked that the optimum 3-4mm of powder height in capillary could provide reproducible and accurate results. Finally, in Buchi ® B-540 melting point apparatus slot the sample capillary was inserted. The temperature at which cyclophosphamide state's meeting was recorded. It was found to be 53^{0}C.

3.6. CYCLOPHOSPHAMIDE POWDER X-RAY DIFFRACTION (PXRD):

Dataset Name:	STD drug-cyp
Filename:	D:\XRD Data\Demo\Sankha 7 12 15\Std drug.xrdml
Measurement Date / Time:	21-08-2012 06:34:35
Operator:	XPERT
Raw Data Origin:	XRD measurement (*.XRDML)
Scan Axis:	Gonio
Start Position [°2Th.]:	5.0042
End Position [°2Th.]:	79.9882
Step Size [°2Th.]:	0.0080
Scan Step Time [s]:	5.7150
Scan Type:	Continuous
PSD Mode:	Scanning
PSD Length [°2Th.]:	2.12
Divergence Slit Type:	Fixed
Divergence Slit Size [°]:	0.8709
Specimen Length [mm]:	10.00
Measurement Temperature [°C]:	25.00
Anode Material:	Cu

K-Alpha1 [Å]: 1.54060

Generator Settings: 40 mA, 45 kV

Goniometer Radius [mm]: 240.00

Incident Beam Monochromator: No

The diffraction pattern of the Plain drug showed characteristic high-intensity diffraction peaks at 6.66, 14.67, 17.47, 25.36, 28.88, 29.75, 33.60, and 35.31 of 2theta.This indicates that the pure drug of cyclophosphamide is in crystalline form (figure 10, table 9).

3.7. DETERMINATION OF PARTITION COEFFICIENT OF CYCLOPHOSPHAMIDE DRUG AND WITH GLYCERYL MONOSTEARATE (LIPID):

By taking known amount of drug in separating funnel, add an equal amount of aqueous phase (double distilled water) and organic phase as chloroform. Shake vigorously and releases pressure in the frequent interval, shake for 10 minutes and allow the layer to separate. Calculate the amount of drug in both layers using UV analysis.

3.7.1. Observation:

Pka = Drug concentration of organic layer / Drug concentration in aqueous = **0.63**

3.8. Partition coefficient of drug in Glyceryl monostearate (Lipid) and Phosphate buffer p H 7.4:

10 mg of Cyclophosphamide was added in a mixture of melted lipid, Glyceryl monostearate (Melting point >55ºC-1g) and 10ml of Phosphate buffer of 37ºC (Fisher Scientific) and shaken for 30 minutes in a mechanical shaker (Remi, Mumbai), using a moderate hot water bath maintained 10ºC above the melting point of the lipid. The aqueous phase of the above mixture was separated from the lipid by centrifugation at a speed of 6600 RPM for 20 minutes at -57ºC temperature using Refrigerator centrifuge (EMTEK Instrument India).The clear suspension was suitably diluted with p H 7.4 phosphate buffer and the cyclophosphamide content was quantified using UV-visible spectrophotometer (Elite) at 722 nm against a solvent blank.The partition coefficient of Cyclophosphamide in lipid/ p H 7.4 PB was calculated using equation.Partition coefficient = C_L/C_A . C_L is the amount of cyclophosphamide in lipid and C_A is the amount of cyclophosphamide in pH7.4 PB**.**

3.8.1. Observation: The partition coefficient in GSM was found to be **1.45**

3.9. PERMEABILITY COEFFICIENT: The permeability coefficient of drug was calculated by using Potts and Guy equation as mentioned bellow:

LogKp = -6.3 + 0.71X log Ko/w -0.0061 X Molecular weight

Where**,** **Log Kp** =Permeability coefficient

Ko/w = Partition coefficient.

Log Kp value of cyclophosphamide was found to be -8.144. Were Molecular weight of cyclophosphamide is 279.10 & partition coefficient is 0.63.

LogKp value of cyclophosphamide with GMS was found to be -10.086, where the molecular weight of cyclophosphamide and GMS combined; that is 637.7. Where Ko/w was 1.45.

LogP = In (Ko/w) =-0.46(Cyclophosphamide)

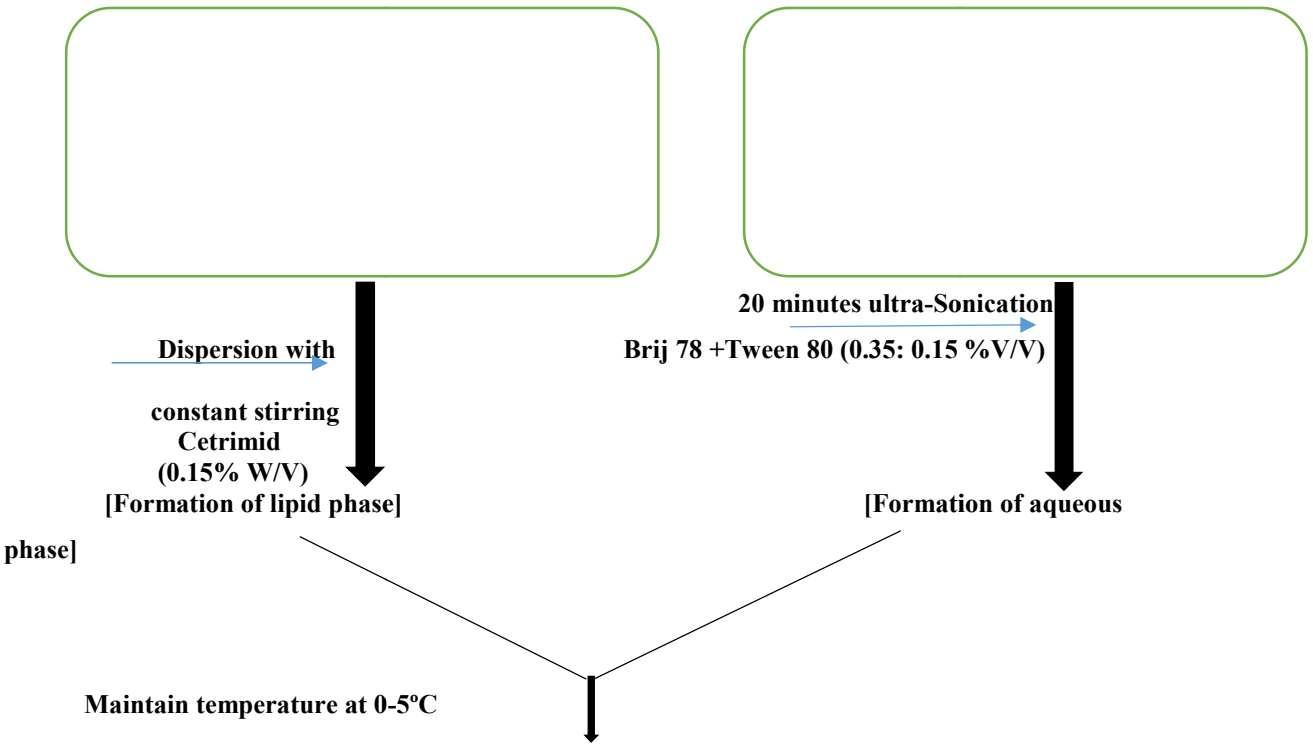
Log P = In (Ko/w) = 0.371 (Cyclophosphamide +GMS)

3.10. SOLUBILITY ANALYSIS: 10 mg of cyclophosphamide was dissolved in 10 ml of different solvent i.e., water, carbon tetra chloride, methanol (acetone free), dichloride methane, chloroform, glacial acetic acid, and ethanol. Solubility were tested as per physical appearance.

4. SCHEMATIC REPRESENTATIONS ON PREPARATIONS OF CATIONIC SOLID LIPID NANOPARTICLES OF CYCLOPHOSPHAMIDE

SLNs prepared by melt dispersion technique and ultrasonication method.

: Formulation of Cationic solid lipid Nanoparticles:



[Aqueous phase is dispersed in lipid phase with constant stirring using High-pressure homogenizer at 30,000rpm speed for 25minutes]

[Ultra-sonicate for 10 minutes]

[Colloidal SLNs light bluish dispersion formed]

[Filter in 0.22-micrometer membrane filter and lyophilize]

[Cationic–SLNs of cyclophosphamide]

5.RESULT AND DISCUSSION:

5.1. UV analysis of cyclophosphamide:

Preformulation usually performed to select a perfect drug and ingredients for the formulation.Cyclophosphamide produces a maximum absorbance at 722nm.

5.2. A standard curve of cyclophosphamide: Cyclophosphamide does not possess linearity bellow 20µg/ml concentration.So concentration was selected between 20-45 µg/ml.For linearity, we prepared 3 different sets of samples ranging from 20-45µg/ml.Absorbance was taken at 722nm after complete blue color phosphomolybdate complex formation in 10ml volumetric flux. Mean absorbance was selected for linearity.The slope was found to be 0.0344 and regression coefficient was found to be 0.9991.

5.3. High-Performance liquid chromatography: Reverse phase HPLC of cyclophosphamide was performed using a solvent as HPLC Water: Acetonitrile (50:50%v/v) and 0.05M Potassium dihydrogen orthophosphate (60): Acetonitrile (40) mixture as the mobile phase .The flow rate was maintained inclined at 15ml/min. The chromatogram was obtained for blank at 1.23 & for standard 2.12.

5.3.1. An important observation during operation:

Occasionally sample runs for 30 minutes, but during this period of this experiment, no characteristic picks were obtained. The sample run time was extended up to 50 minutes. The blank is showing some extra pick because the observational range is the only 200nm, due to narrow range, the possibility of showing other picks maximized apart from used solvent picks.

5.4. Fourier transform infrared spectroscopy: FTIR studies identified the drug, and also concluded that the drug has no interaction with a physical mixture including GMS, and Stearic acid. The principle picks were at 1100 cm-1, 2370cm-1, 1300 cm-1, 3100 cm-1, 1790 cm-1, 2750 cm-1, 3690cm-1, 1780 cm-1, 1250 cm-1.Each pick represents the functional group. It was concluded that there is no drug ingredient interaction and the drug is compatible with solid lipids.

5.5. Differential scanning calorimetry:

The interaction between cyclophosphamide, GMS, Stearic acid, Soya lecithin has been checked in the solid state by SHIMADZU-DSC-60 Plus instrument. Thermographs showing that there was no interaction between drug and excipients.

5.6. Melting point: Melting point of cyclophosphamide by capillary method was found to be 53 ^{o}C.

5.7. XRD study on cyclophosphamide API: The diffraction pattern of the Plain drug showed characteristic high-intensity diffraction peaks at 6.66, 14.67, 17.47, 25.36, 28.88, 29.75, 33.60, and 35.31 of 2theta.This indicates that the pure drug of cyclophosphamide is in crystalline form.

5.8. Determination of partition coefficient, permeability coefficient, and LogP value:

	$K_{o/w}$	**$LogK_p$**	LogP
Drug	**0.63**	**-8.144**	**-0.46**
Drug+GMS	**1.46**	**-10.086**	**0.371**

As per pharmacopeias standard if Log P value is below zero, then the substance has injectable property, and if 0-3 then substance can consider for oral route administration. The drug-lipid partition coefficient is within the standard limits.

5.9. Solubility analysis: The drug is highly soluble in methanol (acetone free), fairly soluble in water, rapidly soluble in acetic acid, and slightly insoluble in dichloromethane.

6.CONCLUSION: Cyclophosphamide[N,N-bi's(2-chroethyl)-1,3,2-oxazaphosphinan-2-amine]maximum absorbance determined at 722nm using UV-Visible spectrophotometry (figure 1, 2). In linearity curve, the regression coefficient was found to be R^2=0. 991 (figure 1, table 1). In Reverse phase, HPLC method retention time of the drug was tested, it was found to be 2.12 (Figure 3&4, Table 2,3,4,5,6). In FTIR studies, characteristic picks were observed at web number 1300,1985, 2370 cm^{-1}(figure 5,6,7 and table 7,8).The drug was found to be compatible with solid lipids (GMS & Stearic acid). In DSC study drug shows picks at 52.20,180.84 ^{O}C and drug and physical mixture shows picks at 52.88,122.23 ^{O} C, It was concluded that drug and physical mixture is compatible with each other (figure 8,9).In XRD studies characteristic high-intensity diffraction peaks at 6.66, 14.67, 17.47, 25.36, 28.88, 29.75, 33.60, and 35.31 of 2theta (figure 10).This indicates that pure drug of cyclophosphamide is in crystalline form. The partition coefficient of drug and drug with GMS was found to be 0.63, 1.45 respectively.The melting point was found to be 53 ^{O} C. The solubility study indicate that drug is mostly soluble in methanol(acetone free) and in acetic acid (table 10). The SEM analysis of prepared solid lipid nanoparticle showing below 1µm ranged particles (figure11,12). Pre-formulation studies signify that cyclophosphamide is compatible with solid lipids, it is showing good retention of 2.12 in HPLC method, XRD, DSC, FTIR showed satisfactory results, the melting point is also within the limit, the drug is showing good partitioning with principle lipid of GMS. After solubility study, it was concluded that methanol (acetone free) could be the best alternative for preparing solid lipid nanoparticular pre-emulsion. Based on the preliminary results an attempt was made to prepare cationic solid lipid nanoparticles, initials results showing good stability and nano ranged particle formation. **7.**

7.FIGURES:

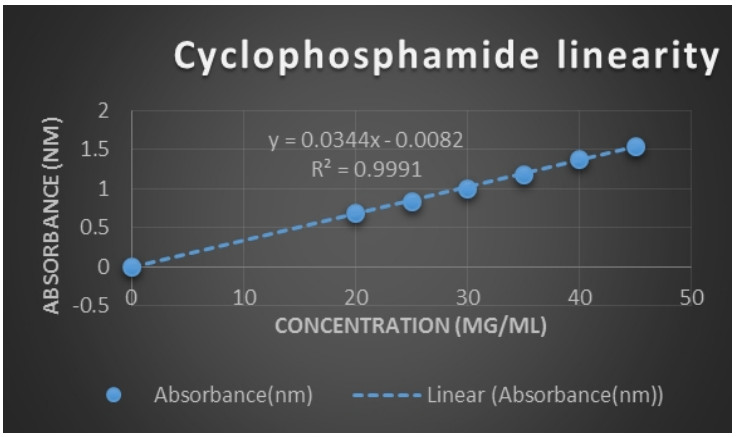


Figure 1: Cyclophosphamide linearity curve

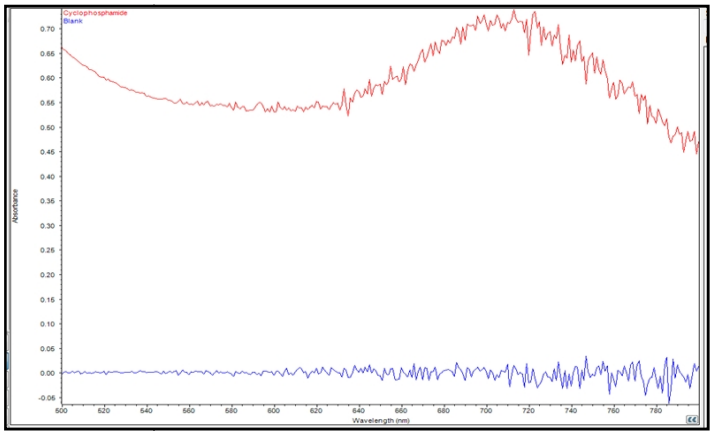


Figure 2: λmax of Cyclophosphamide observed at 722nm

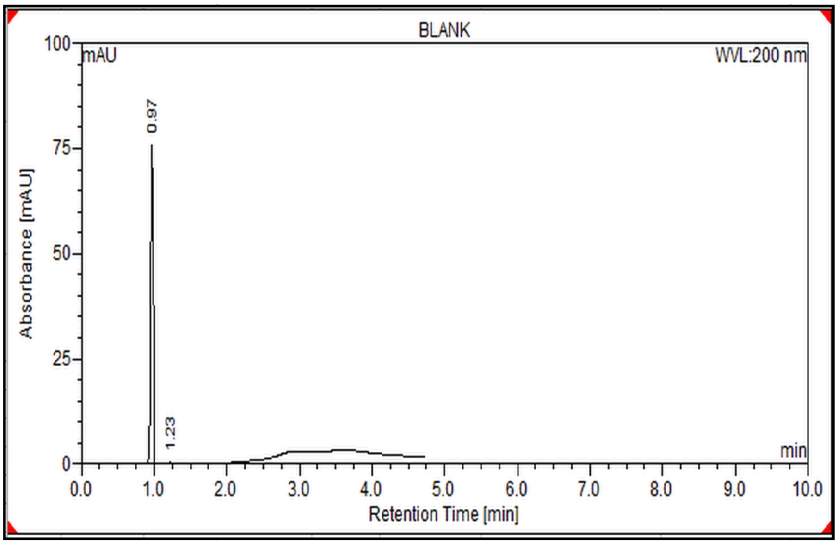


Figure 3: HPLC chromatograph of blank

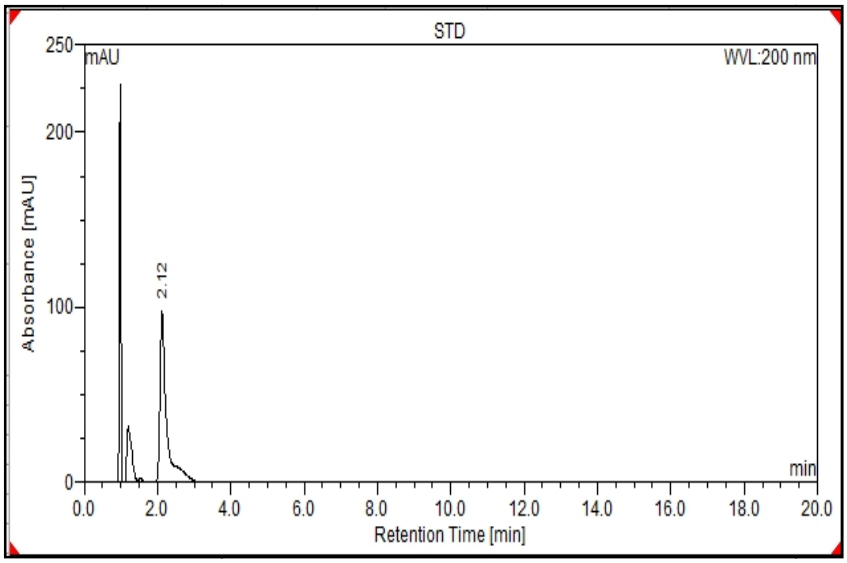


Figure 4: HPLC chromatograph of cyclophosphamide

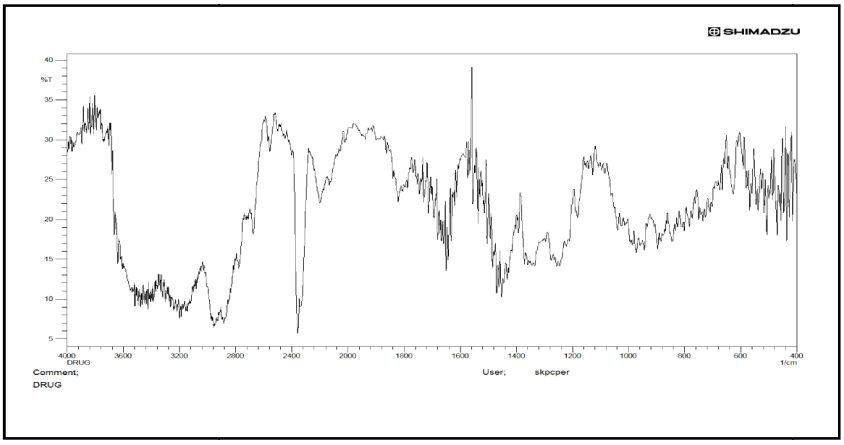


Figure 5: IR spectra of standard cyclophosphamide drug

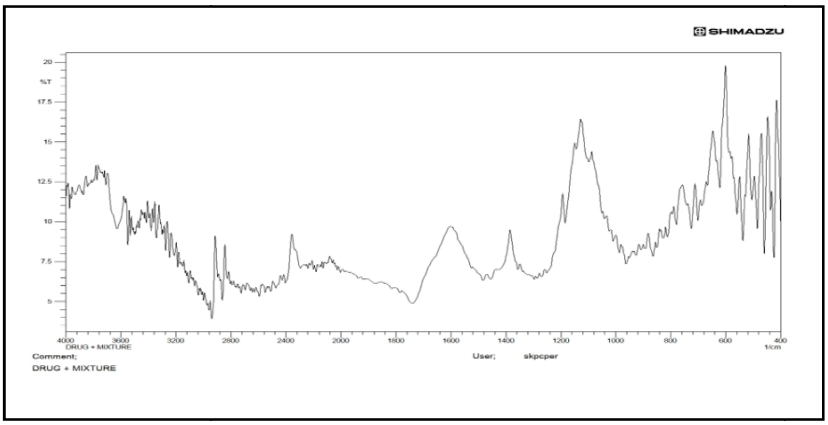


Figure 6: IR spectra of drug and physical mixture

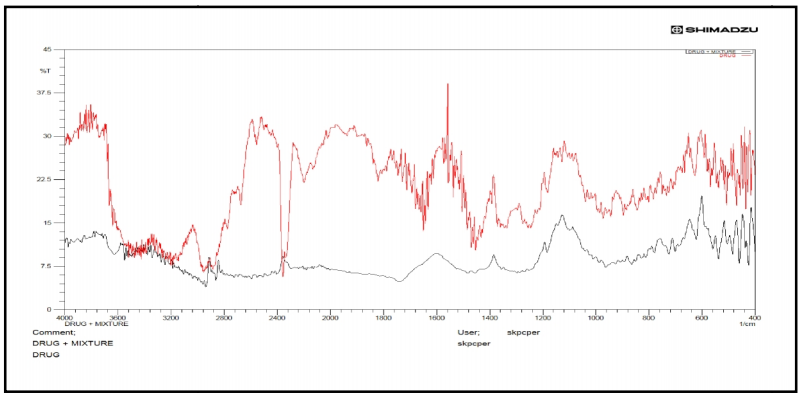


Figure 7: Compatibility of Cyclophosphamide and physical mixture

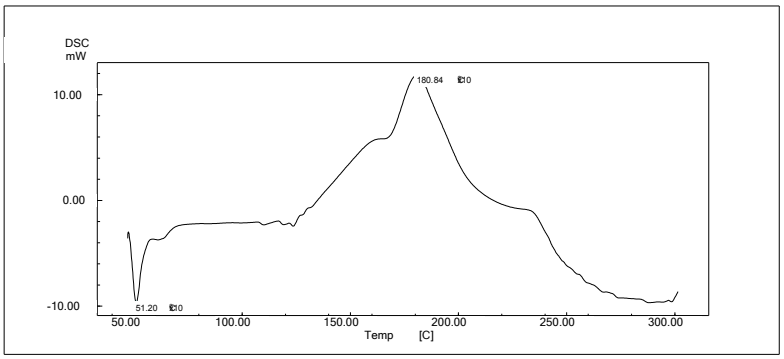


Figure 8: DSC of cyclophosphamide pure drug

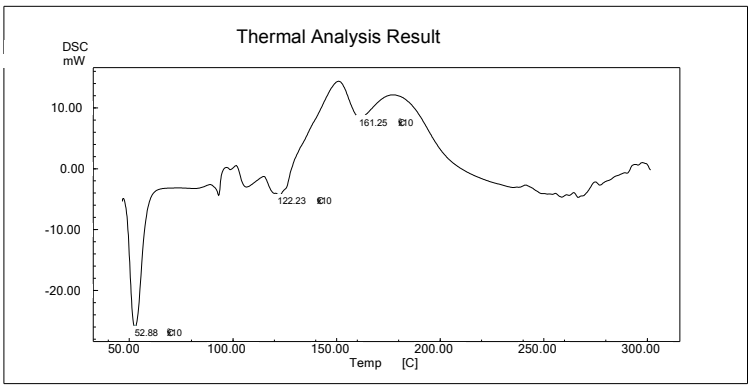


Figure 9: Cyclophosphamide +Physical mixture (GMS+stearic acid)

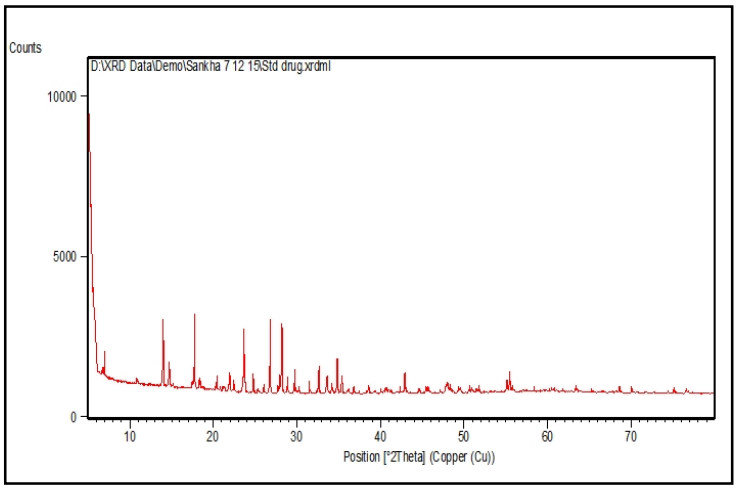


Figure 10: XRD graph of Cyclophosphamide

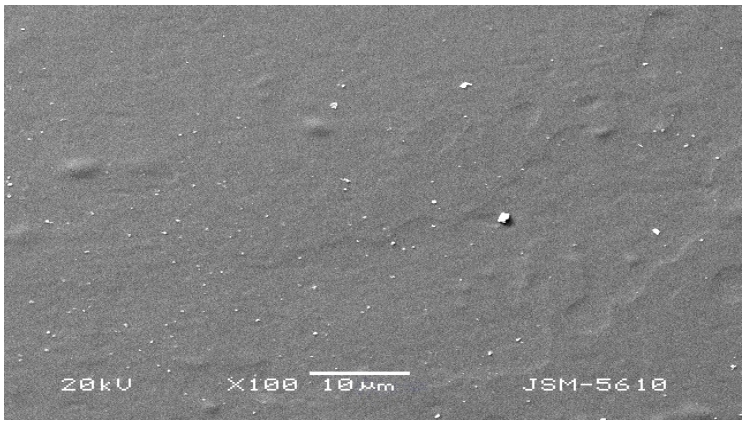


Figure 11: Scanning electron microscopy photographs for cyclophosphamide solid lipid nano particles .a field containing different sized particles in a scale of 10µm using 100× magnification power

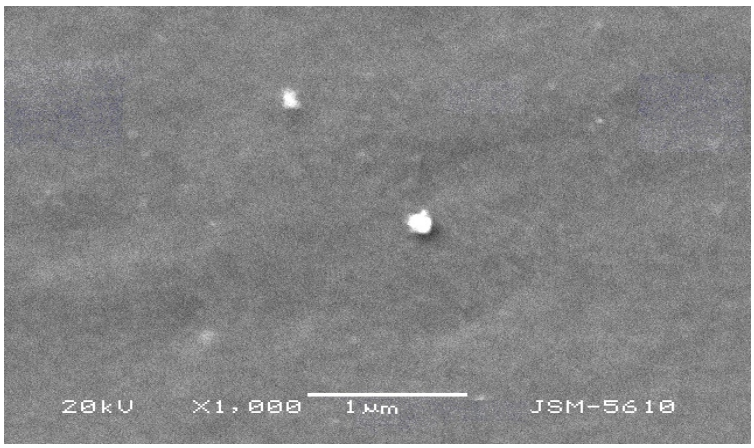


Figure 12: Scanning electron microscopy photographs for the cyclophosphamide solid lipid nanoparticles field containing different sized particles on a scale of 1µm using 1000× magnification power

8. TABLES:

Table1: Values of absorbance's observed at 722nm

Concentration (µg/ml)	Absorbance(nm)			Mean absorbance (n=3)
	I	II	III	
0	0	0	0	**0**
20	0.701	0.690	0.680	**0.690±0.003**
25	0.837	0.835	0.838	**0.836±0.001**
30	0.998	1.02	1.04	**1.01±0.002**
35	1.27	1.21	1.16	**1.18±0.005**
40	1.38	1.38	1.41	**1.39±0.002**
45	1.56	1.54	1.57	**1.55±0.003**

Table 2: Method of analysis in HPLC-Reverse phase

Parameters	Description
Instrument:	Thermo Fischer Scientific; RP-UHPLC
Instrument Model	Dionex Ultimate 3000
Detector:	UV Detector
Column	Thermo Scientific, ODS (250 mm x4.6 mm); Particle size 5µ
Flow rate	1.5 mL/min
Injection volume	10µL
Column temperature	30 ºC
Wavelength:	200 nm
Mobile phase	0.05 M Potassium dihydrogen orthophosphate (60): Acetonitrile (40)
Solvent mixture	Acetonitrile (50): Water (50)(%v/v)

$$\frac{Ua \times Sd \times p}{Sa \times Ud \times 100} \times 100$$

Where,
Sd = Standard dilution
Sa = Standard area
Ud =Sample dilution
Ua = Sample area
P = Potency of Standard Drug

Table 3: HPLC –RP analysis of blank

01	**BLANK**
Sample Name: BLANK	Injection Volume (µL): 10.0
Vial Number: RE2	Channel: UV_VIS_1
The sample for : Mr. Sankha Bhattacharya; RK university	Performed in: Supra drug and food testing research lab
Control Program: CYCLOPHOSPHAMIDE	Operator: Juhi Bhatt
Quantif. Method: CYCLOPHOSPHAMIDE	Instrument ID: U3000
Recording Time: 04/12/15 11:21:50	
Sequence: CYCLOPHOSPHAMIDE	

Table 4: Peak result for blank in HPLC

No.	Ret.Time (min)	Peak Name	Height (mAu)	Area (mAU*min)	Rel.Area (%)
1	0.97	n.a.	101.053	326.507	35.23
2	1.23	n.a.	33.291	600.357	64.77

Total:				926.864	100.00

Table 5: HPLC-RP analysis of standard drug of cyclophosphamide

02	**STANDARD**
Sample Name: STANDARD	Injection Volume (µL): 10.0
Vial Number: RE3	Channel: UV_VIS_1
The sample for: Mr. Sankha Bhattacharya; RK university	Performed in: Supra drug and food testing research lab
Control Program: CYCLOPHOSPHAMIDE	Operator: Juhi Bhatt
Quantif. Method: CYCLOPHOSPHAMIDE	Instrument ID: U3000
Recording Time: 04/12/15 11:28:22	
Sequence: CYCLOPHOSPHAMIDE	

Table 6: Peak result for standard

No.	**Ret.Time (min)**	**Peak Name**	**Height (mAu)**	**Area (mAU*min)**	**Rel.Area (%)**
1	2.12	n.a.	98.834	1285.156	100.00
Total:				1285.156	100.00

Table 7: IR interpretation of standard cyclophosphamide drug

Wave number	**Functional group**
3400cm^{-1}	N-H stretch(weak)
1100 cm^{-1}	N-H Stretch (medium)
1985 cm^{-1}	P=0 Stretch
1300 cm^{-1}	-CH_2-Cl
950 cm^{-1}	C-N
2370cm^{-1}	O=P-OH(singular –strong)

Table 8: IR interpretation of standard cyclophosphamide drug and physical mixture

Wave number	**Functional group**
1100 cm^{-1}	N-H Stretch (medium)
2370cm^{-1}	O=P-OH(singular –strong)
1300 cm^{-1}	-CH_2-Cl
3100 cm-1, 1790 cm^{-1}	Conjugated alkane
2750 cm^{-1}	OH-C=0
3690cm^{-1}	O-H(aliphatic)
1780 cm^{-1}	C=O(stretch)
1250 cm^{-1}	N-H (stretch)

Table 9: Solubility of cyclophosphamide in various solvent

Solvent	**Solubility**
Water	Soluble
Acetic acid	Rapidly soluble
Ethanol	Freely soluble

Acetic anhydride	Soluble
Diethyl ether	Soluble
Carbon tetrachloride	Soluble
Methanol (acetone free)	Rapidly soluble
Dichloromethane	Slightly insoluble (cloudy formation)
Glacial acetic acid	Slightly insoluble
Ethanol	Soluble

9. ACHOWLEDGEMENTS: The authors acknowledged Emcure Pharmaceuticals Ltd. Ahmedabad for gifting the cyclophosphamide drug, Supra drug and food testing and research laboratory-Himatnagar, for HPLC studies. B.Pharmacy college–Rampura & Ganpat University-Mehsana for providing facilities to carry out research work, MS University–metallurgy department for carrying out XRD and SEM analysis, and finally RK University to providing all necessary assistance.

10. CONFLICT OF INTEREST: Authors reports no conflict of interest.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

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RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

Formulation and Evaluation of Fast Disintegrating Metoprolol Succinate Sublingual Tablets

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ABSTRACT

In acute diseases, fast action of drug is required. Metoprolol succinate is used in hypertension and angina pectoris. Here we have prepared sublingual tablet of Metoprolol succinate using various superdisintegrants in concentration range of 1 to 10%. In vitro dissolution of formulation F7,8,25,26,27,28,31,32.35,36,37,38,39 and 40 showed more than 90 percent drug release within 3 minutes. Wetting time for all formulations was found to be 55 to 368 seconds. The water absorption ratios for all formulations were found to be 12.37 % to 97.06 %. The in vitro disintegration time for all formulation was found to be 10 second to 12 minute. Results shown that xanthan gum, cross povidone, colloidal silicon dioxide and alginic acid does not fulfill the requirement of disintegration time for sublingual tablet.

SUMMARY

This research work include formulation of fast disintegrating Metoprolol succinate sublingual tablet.

Keywords: Superdisintegrants, metoprolol succinate, sublingual, tablet, fast disintegrating

INTRODUCTION

The oral dosage form offers various advantages like ease of self administration, compactness and easy manufacturing. So it is considered as the most widely accepted and flexible route of drug administration. But common problem with oral dosage form is difficulty in swallowing for all age group. Especially

elderly, children, and patients who are mentally retarded, uncooperative or nauseated facing greater problem in swallowing oral dosage form *(1)*.

Fast disintegrating sublingual tablet is delivering the drug beneath the tongue and disintegrate rapidly within few minutes in presence of saliva. It offers various benefits like fast release of drug from the dosage form and bypasses the first pass hepatic metabolism of the Metoprolol succinate into the liver and it reaches the systemic circulation directly, so gives fast relief from the anginal pain and hypertension. Because bypassing of first pass hepatic metabolism, the bioavailability with drug is also increases. So, without need of swallowing, we can achieve fast release of drug. Also, the time required for onset of drug action for a conventional oral tablet is more which is generally not acceptable for acute disorders, but the sublingual drug delivery is most acceptable *(2)*.

Angina pectoris is acute disorder characterized by chest pain or discomfort due to coronary heart disease. In case of Hypertension (HTN), the blood pressure in the arteries is elevated *(3, 4)*. Metoprolol succinate is a cardio selective β1 adrenoreceptor blocker. It is mostly used in the treatment of acute disorders such as angina pectoris and also in chronic disease hypertension. It is a BCS (Biopharmaceutical Classification System) class-1 drug. It has high solubility and high permeability. Metoprolol succinate is freely soluble in water and methanol. The half life of Metoprolol succinate is approximately 3 to 4 hours. It undergoes extensive first pass hepatic metabolism resulting in 40% oral bioavailability. Hence the prepared sublingual tablet of Metoprolol succinate lead to enhance the bioavailability and avoidance of first pass hepatic metabolism *(5)*.

MATERIALS AND METHODS *(6-52)*:

Type here Metoprolol succinate (API), mannitol (Directly compressible material), microcrystalline cellulose (diluent and disintegrating agent), crosspovidone (super disintegrating agent), cross carmellose sodium (super disintegrating agent), xanthan gum (super disintegrating agent), chitosan (super disintegrating agent), colloidal silicon dioxide (super disintegrating agent), alginic acid (super disintegrating agent), agar (super disintegrating agent), guar gum (super disintegrating agent), karaya gum (super disintegrating agent), gellan gum (super disintegrating agent), polyvinyl-pyrrolidone K-30 (binder), sodium saccharine (sweetener) and talc (lubricant).

Formulation of Metoprolol Succinate Fast Disintegrating Sublingual Tablets:

Direct compression technique was used for formulation of sublingual tablets of Metoprolol succinate. All ingredients were first passed through 40# sieve. An accurate amount of drug and all excipients were homogenously blended using geometric dilution method. Here talc was added at last for lubrication and mix thoroughly. The tablets were prepared with the help of 7 mm flat punch by rotary tablet punching machine *(53, 54)*. (Table 1 to 5)

Pre-formulation study:

The drug-excipients compatibility study was performed by FT-IR spectroscopy *(53, 54)*.

Micromeritic properties of tablet powder mixture:

The flow properties were evaluated by various parameters like bulk density, tapped density, angle of repose, carr's index and hausner's ratio *(55, 56)*.

Evaluation of Tablets:

Appearance:

Tablets were evaluated for shape, colour, odor, taste etc *(55)*.

Thickness and Diameter:

The size of tablets was evaluated by Vernier calipers *(55)*.

Hardness:
The hardness was measured using Monsanto hardness tester. Three tablets were randomly selected from each formulation and the average hardness was noted *(54)*.

Friability:
This test was performed to determine the effects of friction and shock. Pre-weighed tablets was placed in the friabilator (roche friabilator) and rotated at 25 rpm (rotation per minute) for 4 minutes. The tablets were dedusted and reweighed, and the percentage friability was calculated using standard equation *(53, 54, 56, 58)*.

Weight variation:
Twenty tablets were selected randomly from each formulation, weighed individually and the average weight and % variation of weight was calculated *(53)*.

Wetting Time:
A piece of tissue paper was cut circularly (6.5 cm diameter) and placed on a petridish containing 6 ml of water at room temperature *(53, 54, 59)*. A tablet was placed on the surface of the tissue paper and the time required for the complete wetting of the tablet was noted.

Water Absorption Ratio:
A piece of tissue paper folded twice was kept in a petridish containing 6 ml of purified water. The tablet was placed on the tissue paper and allowed to wet completely. The wetted tablet was removed and reweighed *(56)*.
Water absorption ratio (R) is calculated using below equation;

$$R = 100 (W_a - W_b) / W_b$$

Where, W_b =Weight of tablet before absorption, W_a =Weight of tablet after absorption.

Disintegration time:
Official method as per USP:
In vitro disintegration time was determined using a modified disintegration method (n=5) by using disintegration tester at 37±0.5°C in distilled water. The tablet was carefully kept in a basket without covering plastic disks and 2 minutes is specified as the acceptable time limit for tablet disintegration *(54, 60)*.

.

RESULTS AND DISCUSSION

Type The use of superdisintegrant for the preparation of fast dissolving sublingual tablet is highly effective and easily available. Here various superdisintegrants were used for preparation of Metoprolol succinate tablet.

The pre-formulation study showed that there was no any interaction between Metoprolol succinate and excipients. Various properties of prepared tablet and preformulation parameters were evaluated. The results obtained are shown in a table. (Table 6 to 8)

Wetting time for all formulations was found to be 55 to 368 seconds. The water absorption ratios for all formulations were found to be 12.37 % to 97.06 %. The *in vitro* disintegration time for all formulation was found to be 10 second to 12 minute.

In vitro dissolution of formulation F7,8,25,26,27,28,31,32.35,36,37,38,39 and 40 showed more than 90 percent drug release within 3 minutes.

CONCLUSION

The fast disintegrating metoprolol sublingual tablets were prepared using various superdisintegrants. We found that cross carmellose sodium, agar, guar gum, karaya gum and gellan gum showed fast disintegration. So, these superdisintegrants can be successfully used for preparation of marketable formulation with good release rate.

TABLES

Table 1. Formulation of batch F1 to F8.

Ingredients	**Formulation Code (Quantity in mg per tablet)**							
	F1	**F2**	**F3**	**F4**	**F5**	**F6**	**F7**	**F8**
Metoprolol succinate	12.5	12.5	12.5	12.5	12.5	12.5	12.5	12.5
Mannitol	64	64	64	64	64	64	64	64
MCC	16	14	12	10	18	16	15	14
Xanthan gum	4	6	8	10	-	-	-	-
CCS	-	-	-	-	2	4	5	6
PVP K-30	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
Sodium saccharine	1	1	1	1	1	1	1	1
Talc	1	1	1	1	1	1	1	1
Final weight of tablet (mg)	100	100	100	100	100	100	100	100

Table 2. Formulation of batch F9 to F16 by Direct Compression Method

Ingredients	Formulation Code (Quantity in mg per tablet)							
	F9	**F10**	**F11**	**F12**	**F13**	**F14**	**F15**	**F16**
Metoprolol succinate	12.5	12.5	12.5	12.5	12.5	12.5	12.5	12.5
Mannitol	64	64	64	64	64	64	64	64
MCC	18	16	15	14	16	14	12	10
Chitosan	-	-	-	-	4	6	8	10
Cross povidone	2	4	5	6	-	-	-	-
PVP K-30	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
Sodium saccharine	1	1	1	1	1	1	1	1
Talc	1	1	1	1	1	1	1	1
Final weight of tablet (mg)	100	100	100	100	100	100	100	100

Table 3. Formulation of batch F17 to F24 by Direct Compression Method

Ingredients	Formulation code (Quantity in mg per tablet)							
	F17	**F18**	**F19**	**F20**	**F21**	**F22**	**F23**	**F24**
Metoprolol succinate	12.5	12.5	12.5	12.5	12.5	12.5	12.5	12.5
Mannitol	64	64	64	64	64	64	64	64
MCC	16	14	12	10	16	14	12	10
Colloiodal silicon dioxide	4	6	8	10	-	-	-	-
Alginic acid	-	-	-	-	4	6	8	10
PVP K-30	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
Sodium saccharine	1	1	1	1	1	1	1	1
Talc	1	1	1	1	1	1	1	1
Final weight of tablet (mg)	100	100	100	100	100	100	100	100

Table 4. Formulation of batch F25 to F32 by Direct Compression Method

Ingredients	Formulation code (Quantity in mg per tablet)							
	F25	**F26**	**F27**	**F28**	**F29**	**F30**	**F31**	**F32**
Metoprolol succinate	12.5	12.5	12.5	12.5	12.5	12.5	12.5	12.5
Mannitol	64	64	64	64	64	64	64	64

MCC	16	14	12	10	16	14	12	10
Agar	4	6	8	10	-	-	-	-
Guar gum	-	-	-	-	4	6	8	10
PVP K-30	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
Sodium saccharine	1	1	1	1	1	1	1	1
Talc	1	1	1	1	1	1	1	1
Final weight of tablet (mg)	100	100	100	100	100	100	100	100

Table 5. Formulation of batch F33 to F40 by Direct Compression Method

Ingredients	**Formulation code (Quantity in mg per tablet)**							
	F33	**F34**	**F35**	**F36**	**F37**	**F38**	**F39**	**F40**
Metoprolol succinate	12.5	12.5	12.5	12.5	12.5	12.5	12.5	12.5
Mannitol	64	64	64	64	64	64	64	64
MCC	16	14	12	10	16	14	12	10
Karaya gum	4	6	8	10	-	-	-	-
Gellan gum	-	-	-	-	4	6	8	10
PVP K-30	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
Sodium saccharine	1	1	1	1	1	1	1	1
Talc	1	1	1	1	1	1	1	1
Final weight of tablet (mg)	100	100	100	100	100	100	100	100

MCC - Microcrystalline cellulose, CCS - Cross carmellose sodium, PVP- Polyvinyl-pyrrolidone.

Table 6. Bulk density, tapped density, angle of repose, carr's compressibility index and hausner's Ratio

Sr. No.	Formulation code	Bulk density	Tapped density	Angle of repose	Carr's compressibility index	Hausner's ratio
1	F1	0.521	0.623	32.8	16.37	1.196
2	F2	0.524	0.626	29.5	16.29	1.195
3	F3	0.528	0.629	30.9	16.06	1.191
4	F4	0.529	0.632	28.7	16.30	1.195
5	F5	0.521	0.623	30.9	16.37	1.196
6	F6	0.523	0.62	30.4	15.64	1.185
7	F7	0.520	0.60	29.6	13.33	1.154
8	F8	0.521	0.62	29.7	15.97	1.190
9	F9	0.521	0.623	31.9	16.37	1.196
10	F10	0.523	0.62	30.3	15.65	1.184
11	F11	0.520	0.60	30.6	13.33	1.154
12	F12	0.521	0.62	29.1	15.97	1.190
13	F13	0.519	0.620	29.9	16.29	1.194
14	F14	0.513	0.619	31.3	17.12	1.207
15	F15	0.519	0.603	29.6	13.93	1.162
16	F16	0.521	0.62	30.1	15.97	1.190
17	F17	0.501	0.603	34.9	16.92	1.204
18	F18	0.513	0.589	35.3	12.90	1.148
19	F19	0.505	0.597	35.6	15.41	1.182
20	F20	0.507	0.595	34.1	14.79	1.174
21	F21	0.518	0.613	31.9	15.50	1.183
22	F22	0.522	0.619	30.3	15.67	1.186
23	F23	0.519	0.621	28.5	16.43	1.197
24	F24	0.515	0.617	26.9	16.53	1.198
25	F25	0.516	0.609	30.3	15.27	1.180
26	F26	0.520	0.614	30.2	15.31	1.181
27	F27	0.514	0.628	28.3	18.15	1.222
28	F28	0.516	0.620	28.9	16.77	1.202
29	F29	0.534	0.617	30.6	13.45	1.156
30	F30	0.530	0.624	30.8	15.06	1.177
31	F31	0.534	0.627	28.3	14.83	1.174
32	F32	0.529	0.624	28.9	15.22	1.180
33	F33	0.525	0.643	32.9	18.35	1.225
34	F34	0.523	0.625	31.5	16.32	1.195
35	F35	0.524	0.627	32.4	16.43	1.197
36	F36	0.531	0.629	29.1	15.58	1.185
37	F37	0.548	0.633	28.9	13.43	1.155
38	F38	0.542	0.639	30.4	15.18	1.179
39	F39	0.549	0.631	28.6	13.00	1.149
40	F40	0.535	0.647	26.9	17.31	1.209

Table 7. Hardness, thickness, diameter, percentage friability and weight variation of formulation F1 to F40

Formulation	Hardness (kg/cm^2)	Thickness (mm)	Diameter (mm)	% Friability	Average Weight (mg)

F1	3.4± 0.15	1.85±0.02	6.79±0.03	0.88±0.02	102.5±0.52
F2	3.3± 0.20	1.89±0.03	6.83±0.01	0.81±0.01	103.4±0.45
F3	3.7± 0.26	1.87±0.02	6.84±0.03	0.77±0.03	101.2±0.38
F4	3.2± 0.29	1.89±0.01	6.85±0.02	0.84±0.02	104.3±0.36
F5	3.6± 0.16	1.85±0.01	6.84±0.02	0.79±0.01	103.3±0.51
F6	3.7± 0.26	1.88±0.03	6.81±0.02	0.73±0.02	104.6±0.43
F7	3.5± 0.21	1.90±0.02	6.83±0.03	0.81±0.03	102.4±0.55
F8	3.6± 0.22	1.87±0.03	6.87±0.02	0.84±0.01	104.6±0.51
F9	3.5± 0.17	1.86±0.01	6.86±0.03	0.84±0.01	104.7±0.51
F10	3.8± 0.29	1.87±0.03	6.84±0.03	0.77±0.03	102.1±0.65
F11	3.6± 0.19	1.89±0.01	6.86±0.02	0.84±0.02	105.3±0.45
F12	3.7± 0.25	1.90±0.02	6.88±0.03	0.88 ± 0.02	104.5±0.58
F13	3.4 ± 0.18	1.84±0.01	6.89±0.03	0.85 ± 0.03	103.7±0.51
F14	3.3 ± 0.21	1.83±0.03	6.74±0.03	0.87 ± 0.04	101.1±0.28
F15	3.5 ± 0.11	1.85±0.01	6.86±0.02	0.81± 0.04	103.3±0.56
F16	3.6 ± 0.20	1.91±0.02	6.98±0.03	0.89 ± 0.03	102.5±0.36
F17	3.8± 0.27	1.89±0.03	6.89±0.02	0.86±0.02	103.6±0.54
F18	3.9± 0.32	1.76±0.03	6.74±0.04	0.79±0.03	102.2±0.63
F19	3.7± 0.21	1.87±0.02	6.76±0.02	0.82±0.02	103.3±0.45
F20	3.6± 0.24	1.93±0.02	6.78±0.03	0.87 ± 0.02	103.5±0.54
F21	3.5± 0.22	1.79±0.03	6.79±0.03	0.85±0.02	102.6±0.53
F22	3.7± 0.22	1.74±0.03	6.73±0.04	0.76±0.03	103.2±0.64
F23	3.8± 0.23	1.85±0.03	6.82±0.02	0.89±0.02	102.3±0.45
F24	3.5± 0.24	1.90±0.02	6.71±0.03	0.83 ± 0.02	101.5±0.51
F25	3.6± 0.23	1.84±0.03	6.84±0.02	0.84±0.04	102.6±0.51
F26	3.7± 0.32	1.78±0.03	6.71±0.04	0.89±0.03	103.2±0.63
F27	3.6± 0.21	1.85±0.02	6.74±0.02	0.88±0.02	102.3±0.51
F28	3.5± 0.24	1.92±0.04	6.79±0.03	0.91 ± 0.02	101.5±0.54
F29	3.3± 0.18	1.83±0.02	6.77±0.03	0.89±0.02	101.5±0.52
F30	3.4± 0.20	1.79±0.03	6.81±0.01	0.84±0.01	101.4±0.45
F31	3.5± 0.26	1.88±0.02	6.74±0.03	0.79±0.03	102.2±0.38
F32	3.4± 0.29	1.91±0.01	6.84±0.02	0.86±0.02	103.3±0.36
F33	3.6± 0.18	1.86±0.02	6.75±0.03	0.92±0.02	101.5±0.52
F34	3.4± 0.23	1.92±0.03	6.91±0.01	0.88±0.01	102.4±0.48
F35	3.3± 0.26	1.85±0.02	6.72±0.03	0.91±0.03	104.2±0.36
F36	3.6± 0.23	1.94±0.01	6.86±0.02	0.88±0.02	101.3±0.36
F37	3.5± 0.21	1.96±0.03	6.71±0.01	0.94±0.02	102.4±0.51
F38	3.6± 0.26	1.95±0.04	6.94±0.02	0.78±0.02	101.4±0.49
F39	3.4± 0.21	1.85±0.03	6.78±0.02	0.97±0.03	102.3±0.32
F40	3.3± 0.22	1.92±0.03	6.96±0.02	0.89±0.03	103.3±0.35

Table 8. Disintegration time, wetting time, water absorption ratio and drug content uniformity of formulation F1 to F

Formulation	Wetting time (sec)	Water absorption ratio	Disinteg-ration time	% Drug content	Cumulative % drug release in 3 min (%CCR)
F1	290 sec	63.76	> 10 min	99.28	20.3 ± 0.9
F2	270 sec	60.34	> 12 min	101.42	19.3 ± 0.7
F3	280 sec	72.41	> 10 min	102.85	20.1 ± 0.5
F4	260 sec	59.64	> 11 min	99.64	19.3 ± 0.4
F5	73 sec	69.78	57 sec	102.5	74.9 ± 0.9
F6	70 sec	70.87	36 sec	98.92	86.6 ± 0.9
F7	78 sec	76.37	24 sec	101.78	102.4 ± 1.3
F8	68 sec	78.76	33 sec	99.28	90.6 ± 0.8
F9	266 sec	37.13	> 8 min	101.45	21.3 ± 0.5
F10	260 sec	45.54	> 8 min	99.76	19.4 ± 0.7
F11	245 sec	38.98	> 8 min	100.65	22.5 ± 0.5
F12	250 sec	42.86	> 8 min	102.54	21.3 ± 0.4
F13	110 sec	44.74	7 min	101.65	24.3 ± 0.6
F14	100 sec	53.76	4 min	99.45	53.3 ± 0.8
F15	130 sec	64.95	120 sec	98.56	78.1 ± 0.5
F16	120 sec	58.74	3 min	101.34	59.3 ± 0.4
F17	260 sec	18.75	> 7 min	102.43	27.3 ± 0.4
F18	100 sec	24.04	> 7 min	99.65	29.4 ± 0.6
F19	96 sec	22.54	> 7 min	98.67	32.5 ± 0.3
F20	62 sec	18.51	> 7 min	101.76	31.3 ± 0.7
F21	368 sec	18.08	6 min	102.55	37.3 ± 0.4
F22	340 sec	16.83	6 min	101.44	38.9 ± 0.7
F23	240 sec	20.59	7 min	99.87	42.4 ± 0.5
F24	200 sec	12.37	8 min	100.87	41.3 ± 0.4
F25	120 sec	78.00	74 sec	101.54	97.3 ± 0.4
F26	100 sec	89.04	65 sec	100.56	98.9 ± 0.7
F27	78 sec	97.06	16 sec	99.87	102.4 ± 0.5
F28	56 sec	93.16	14 sec	100.87	101.3 ± 0.4
F29	145 sec	19.69	50 sec	99.76	85.7 ± 0.3
F30	150 sec	21.98	45 sec	101.67	88.9 ± 0.7
F31	130 sec	16.47	40 sec	99.76	92.4 ± 0.4
F32	134 sec	20.96	36 sec	100.54	101.3 ± 0.3
F33	101 sec	16.51	185 sec	101.43	83.5 ± 0.5
F34	200 sec	88.57	160 sec	99.73	88.9 ± 0.7
F35	160 sec	84.54	150 sec	101.55	91.2 ± 0.2
F36	180 sec	75.76	145 sec	99.98	97.1 ± 0.3
F37	70 sec	13.98	43 sec	100.76	93.8 ± 0.3
F38	65 sec	12.54	36 sec	98.87	94.9 ± 0.6
F39	55 sec	14.43	15 sec	101.23	97.5 ± 0.3
F40	60 sec	12.65	10 sec	100.56	99.1 ± 0.5

ACKNOWLEDGEMENT

The author thanks Alembic Pharma, Vadodara for providing gift samples of Metoprolol succinate to carry out this work. The author also thankful to management of School of Pharmacy, RK University, Rajkot for providing infrastructure and facilities for carried out this research work. Author also thanks GUJCOST, Gandhinagar for providing financial assistance for this work under minor research project grant.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

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Development And Validation Of A Stability-Indicating HPLC Method For The Determination Of Lurasidone Hydrochloride In Bulk And Degradation Kinetics Study In Basic Medium

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ABSTRACT

Method was RP HPLC using PDA detector at wavelength of 230 nm. Mobile phase was consist of Phosphate buffer (pH = 4.0) : Acetonitrile (45: 55), flow rate-1mL/min in Lichrospher ® 60 RP-HPLC, C8, (125mm x 4mm), 5µ column. Method has been validated as per ICH guidelines. Correlation coefficient for linearity - 0.9995, in the range of 100-400 µg/mL. LOD and LOQ for Lurasidone have been found to be 2.60 µg/mL and 7.88 µg/mL. Stress study was performed in acidic, basic, peroxide, photo and thermal conditions. It showed that degradation products didn't interfere in analysis. Hence method was specific and further investigated by performing degradation kinetics in presence of 1N NaOH at 50˚ C, 60˚ C, 70˚ C. Precision was performed and assessed by % RSD (was < 2%). Method could be used for routine QC testing and monitoring of stability of Lurasidone hydrochloride.

SUMMARY

Stability indicating RP-HPLC method and kinetic study in basic medium of LURASIDONE HCL was used for the determination of half-life and selection of new vehical for newer formulation.

Keywords: Lurasidone, Stability indicating method, validation, HPLC, Degradation kinetics

INTRODUCTION

Schizophrenia is a mental disorder characterised by abnormality in normal brain functioning etc. [1] Till date many antipsychotics were discovered and developed to treat schizophrenia. First generation antipsychotics, called as the typical antipsychotics which act by antagonizing the potent Dopaminergic D_2 receptors, effective in treating the positive symptoms but fail to control cognitive dysfunction. Second generation antipsychotics named as an atypical antipsychotic is effective in treating the positive as well as negative symptoms. Being its second generation antipsychotics they lower the risk of extrapyramidal symptoms [2].

Lurasidone ((3a*R*,4*S*,7*R*,7a*S*)-2-[((1*R*,2*R*)-2-{[4-(1,2-benzisothiazol-3-yl)-piperazin-1-yl]methyl}cyclohexyl)methyl]hexahydro-1*H*-4,7-methanisoindol-1,3-dione hydrochloride) is an antipsychotic drug approved by United States Food and Drug Administration (USFDA) for the treatment of schizophrenia on October 2010[3]. Lurasidone was developed by Dainippon Sumitomo Pharma Co. Ltd. in Japan. Lurasidone is a benzisothiazole derivative [4]. Like other antipsychotics Lurasidone acts by antagonizing dopamine D_2 receptors. It binds with serotonin receptors (5-HT_{2A}, 5-HT_7, and 5-HT_{1A}) and α_{2C} adrenergic receptors and improves cognitive functions through enhanced N-Methyl-D-aspartate (NMDA) mediated synaptic response [5, 6].

Stability indicating methods (SIM) are adopted for the analysis of stability samples. With the advent of International Conference on Harmonisation (ICH) necessities to establish this method has been clearly mandated. Stability indicating methods can give the detail on how the drug quality changes with the change in environmental factors like pH, humidity, temperature, light etc. SIM also helps a formulator to choose suitable vehicles to develop a new formulation [7]. According to literature survey there are several methods like HPLC[8-10] LC-MS[11,1] LC-MS-MS[12], are reported for determination of Lurasidone but there is no stability indicating method reported showing the degradation kinetics till date. Therefore we developed stability indicating HPLC method for determination of Lurasidone hydrochloride. The results are reported for determination of extent of influence of various stress conditions on the stability of the drug substance and a kinetics study of the degradation of Lurasidone in under basic conditions. Here in present study the developed method has been validated as per ICH guidelines[13].

MATERIALS AND METHODS

1. Materials and chemicals

Lurasidone was obtained from Torrent Pharmaceuticals, Ahmedabad as a gift sample. All chemicals and reagents like methanol, acetonitrile, potassium dihydrogen phosphate, sodium hydroxide, hydrochloric acid, hydrogen peroxide solutions were used of HPLC grade and given by Merck Chemicals, India.

2. HPLC instrumentation and chromatographic conditions

The method was developed using Agilent HPLC instrument equipped with Photo Diode Array Detector. Stationary phase Lichrospher ® 60 RP-select B, C8 (125mm x 4mm) x 5µm column (Merck). The mobile phase was consist of a mixture 45 volumes of Phosphate buffer (pH = 4.0) and 55 volumes of Acetonitrile, pumped at flow-rate of 1 mL per minute at ambient temperature. The accomplished detection wavelength was 230 nm.

3. Preparation of solutions

3.1. Standard stock solution of Lurasidone

Accurately weighed and transferred 100 mg of Lurasidone hydrochloride working standard in 100 mL volumetric flask (VF), pour 50 mL of methanol and sonicate to dissolve. Make up the volume up to mark.

3.2. Standard solution of Lurasidone

Transferred specified volume 5 mL from the standard stock solution to 20 mL volumetric flask. Diluted with mobile phase and mixed.

3.3. Acid induced degradation

Solution containing 1 mg of the drug in 1N HCl was prepared in methanol and kept overnight at 80˚ C. For HPLC analysis 5 mL aliquots were taken in to 20 mL VF and neutralised with 1N NaOH and diluted with methanol till mark.

3.4. Base induced degradation

Solution containing 1 mg of the drug in 1N NaOH was prepared in methanol and kept for 1 hour at 80˚ C. For HPLC analysis 5 mL aliquots were taken in to 20 mL VF and neutralised with 1N HCl and diluted with methanol till the mark.

3.5. Peroxide induced degradation

Solution containing 1 mg of the drug in 3% H_2O_2 was prepared in methanol and kept for 3 hr at room temperature. For HPLC analysis 5 mL aliquots were taken into 20 ml volumetric flask diluted with methanol up to the mark.

3.6. Thermal degradation

The Active Pharmaceutical ingredient (API) was kept at 80°C for 72 hours in hot air oven then made dilution in such way that the final concentration for Lurasidone hydrochloride 250 µg mL^{-1} achieved.

3.7. Photo degradation

The API was exposed to 200 watt h/m^2 for ultraviolet light in photo stability chamber for 24 hr then made dilution in such way that the final concentration for Lurasidone hydrochloride 250 µg mL^{-1} achieved.

4. Method Validation

4.1 Precision

Repeatability and intermediate precision studies were carried out by six replicate injections of standard preparation in different instrument, different instrument of same type and by different analyst. The result was recorded as % Relative Standard Deviation (% RSD).

4.2 Accuracy

Accuracy performed by recovery study at three levels at 80%, 100% and 120%. For each level three sets were prepared and % recovery was calculated.

4.3. Linearity

By plotting calibration curve of peak area v/s concentration, Linearity has been determined over 7 different concentrations. The linearity was found to be 100-400 µg mL^{-1}.

4.4 Limit of detection (LOD) and limit of quantitation (LOQ)

LOD and LOQ were found by the equation 1 as per ICH guidelines

$LOD = 3.3 \times \sigma / S$ and $LOD = 10 \times \sigma / S$ (1)

Where, σ was the S D of the response and S was mean of slope of the calibration curves.

4.5 Robustness

Robustness was performed by change in flow rate (± 0.1 mL/min), mobile phase composition (± 2%) and column oven temperature (± 5˚ C). Robustness was calculated in terms of % RSD.

4.6. Specificity

Specificity for the developed method was performed by recording blank chromatogram and compared with the standard. Further specificity was tested by performing forced degradation study including acid degradation, base degradation, peroxide degradation, photo degradation and thermal degradation. In all these tested conditions interference of degradation products with the main peak was determined.

4.7. System suitability

The System suitability parameters were evaluated by no. of theoretical plates (N) and tailing factor (T), Rt, Resolution.

5. Degradation kinetics under basic condition

The study was carried out at 50˚ C, 60˚ C and 70˚ C (experimental temperature) with the solution of 100 mg of drug with 1N NaOH. At pre-established time points the 5 mL aliquot has been transferred to 20 ml volumetric flask and neutralised with 1N HCl and diluted to the volume with methanol. The samples were analysed by HPLC with the optimized method.

RESULTS AND DISCUSSION

1. Method development and optimization

Primary objective of Stability indicating HPLC method development for determination of Lurasidone should be precise, accurate, robust and reproducible. All the degradation products should be well separated and the method should be very simple to be applied for routine analysis. During the method development several composition of mobile phases were tried and Optimised was a mixture 45 volumes of 13.6 gm of KH_2PO_4 dissolved in 1L of water and adjusted pH 4.0 with OPA and 55 volumes of ACN (1.0 mL/ min of flow rate). Lichrospher ® 60 RP-select B, C8 125mm x 4mm (Merck) column of 5 µm particles packing (Figure 2, 3). Detection was carried out at 230 nm using PDA detector.

2. Forced degradation

Forced degradation study data revealed that Lurasidone found to be highly unstable in basic and oxidative medium. It also gets degraded in presence of light. Basic condition led to 79.08% degradation (Figure 4). Oxidative stress led to 18.35% degradation (Figure5) and photolytic condition led to 34.54% degradation (Figure 6). While Lurasidone is stable in thermal condition as well as in acidic medium. (Table 1)

3. Method validation

Six replicate determinations were performed and % RSD for repeatability was found to be 0.11 and % RSD for intermediate precision was 0.18. The % RSD values were found < 2% that indicates the proposed method is precise (Table 2).

Accuracy was performed by standard addition method and results were recorded as % recovery in triplicates for each of the three levels (Table 3). The % recovery was found in the range of 99.97% - 101.89%.

Linearity shows the relationship between the responses against concentration of analyte. Linear correlation was found range of 100 µg mL^{-1} to 400 µg mL^{-1}. The equation for regression was

determined by plotting peak area versus concentration of Lurasidone in µg mL^{-1}(Table 4, Figure 7, 8).

LOD and **LOQ** were determined from the equations as per ICH guidelines. LOD and LOQ found for the proposed method were 2.60 µg mL^{-1} and 7.88 µg mL^{-1}.

Specificity ascertained by comparing the chromatogram of blank with the standard preparation. Interference of the degradation products with main peak was checked by Stress study. Drug substance was separated from the degradants impurities. Hence the developed method was proved to be specific.

System suitability was performed and found to be in acceptable range and system was suitable for intended analysis.

4. Degradation kinetics

Standard solution of Lurasidone was degraded at experimental temperature in 1N NaOH. Chromatograms were obtained found that the peak area of Lurasidone was reduced with time. The semi logarithmic plot of concentration of Lurasidone versus time in 1N NaOH (Figure 9) indicated apparent first order degradation behaviour. Using equation (2) where $[A_0]$ is the concentration of Lurasidone at time t=0 and $[A]_t$ is its concentration at time t, degradation rate constant (k) was determined. The slopes of the lines were found by linear regression analysis. Slopes were -4.8 x 10^{-3} h^{-1}, -5.2 x 10^{-3} h^{-1} and -7.1 x 10^{-3} h^{-1}
(r^2=0.9954) at 50˚ C, 60˚ C and 70˚ C respectively.

$$\text{Log } [A]_t = [\log A_0] - k\,t] / 2.303 \quad (2)$$

$$t_{1/2} = 0.693 / k \quad (3)$$

$$\text{Log } (k_1/k_2) = [E_a (T_2-T_1)] / 2.303\, RT_1T_2 \quad (4)$$

The half-life periods were calculated from the equation (3) and were found to be 55.58 min, 48.09 min and 40.44 min for 50˚ C, 60˚ C and 70˚ C respectively.

Equation (4) was derived from Arrhenius equation and applied for determination of Energy of Activation (E_a) for base mediated degradation kinetics. R is the gas constant (1.987 cal mol^{-1}). Derived Energy of Activation (E_a) was 14.33 KJ mol^{-1}

CONCLUSION

As per ICH guideline developed method was validated. The results of forced degradation study revealed that Lurasidone is susceptible to degradation in alkaline, light, and oxidative stress. Obtained degradation of products are well separated from the peak of Lurasidone and peak purity index were found to be more than 0.9999.

The kinetics was performed in base catalysed degradation of Lurasidone at 50˚ C, 60˚ C and 70˚ C. The reaction found to be of first order reaction with half-life periods 55.58 min, 48.09 min and 40.44 min for 50˚ C, 60˚ C and 70˚ C respectively. And the Energy of Activation (E_a) was 14.33 KJ mol^{-1}.

Validation of the method shows that proposed method is suitable for intended use. The present method found as linear in the range of 100 µg ml^{-1}- 400 µg ml^{-1}as well as accurate and precise with respect to repeatability and intermediate precision. Lurasidone HCL was separated from the degradation products after force degradations study. The results revealed the method is robust with deliberate changes in optimised HPLC conditions. From the above results it was found that this method can be applied for QC analysis of active pharmaceutical ingredient in bulk substance.

FIGURES

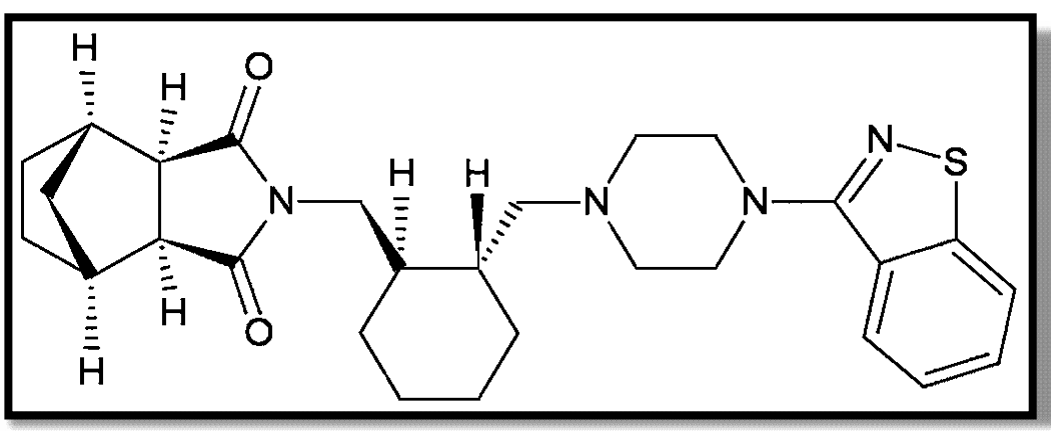


Fig. 1: Structure of Lurasidone

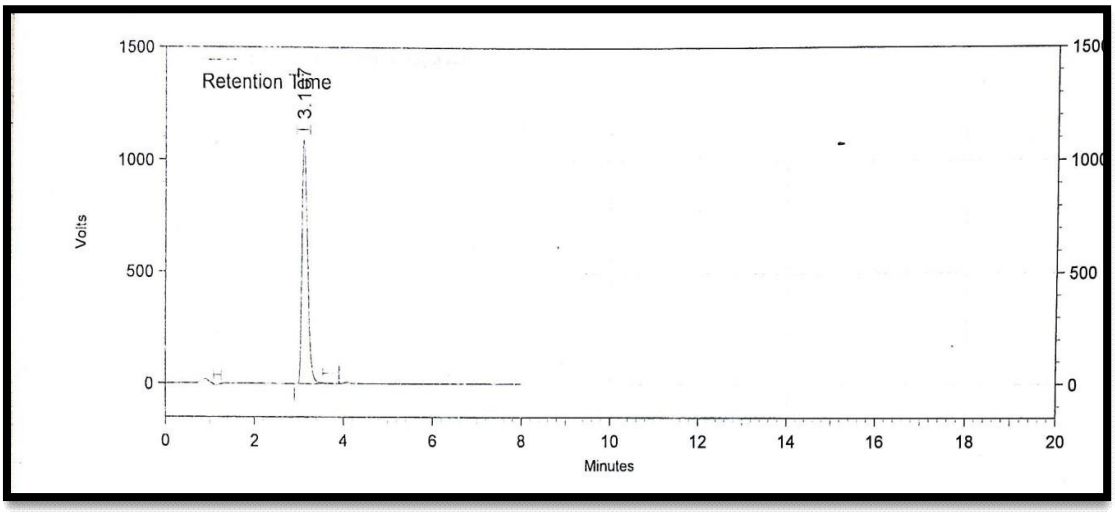


Fig. 2: HPLC Chromatogram of Lurasidone standard preparation (250 µg mL^{-1})

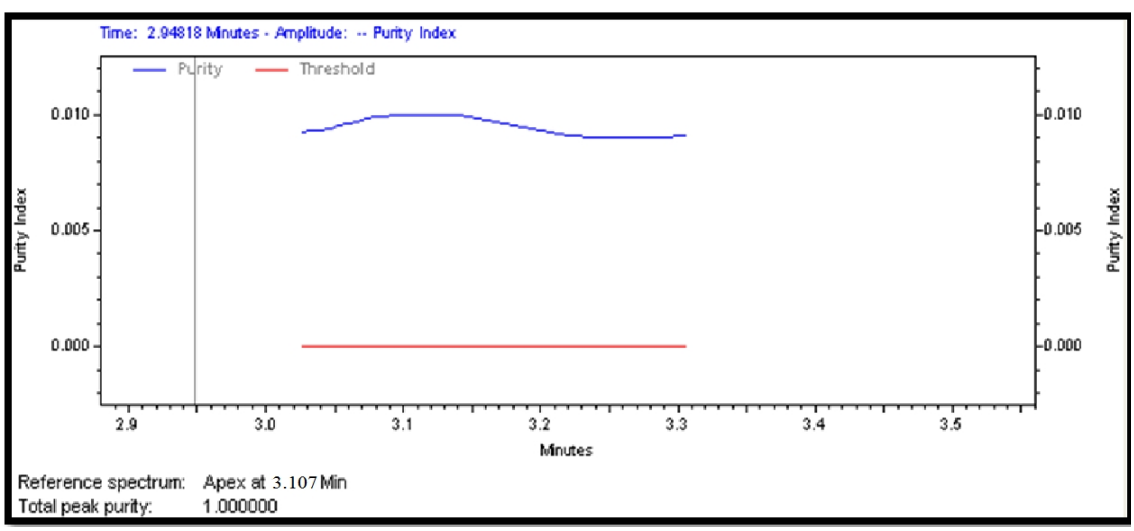


Fig. 3: Peak purity spectra for Lurasidone standard preparation (250 µg mL^{-1})

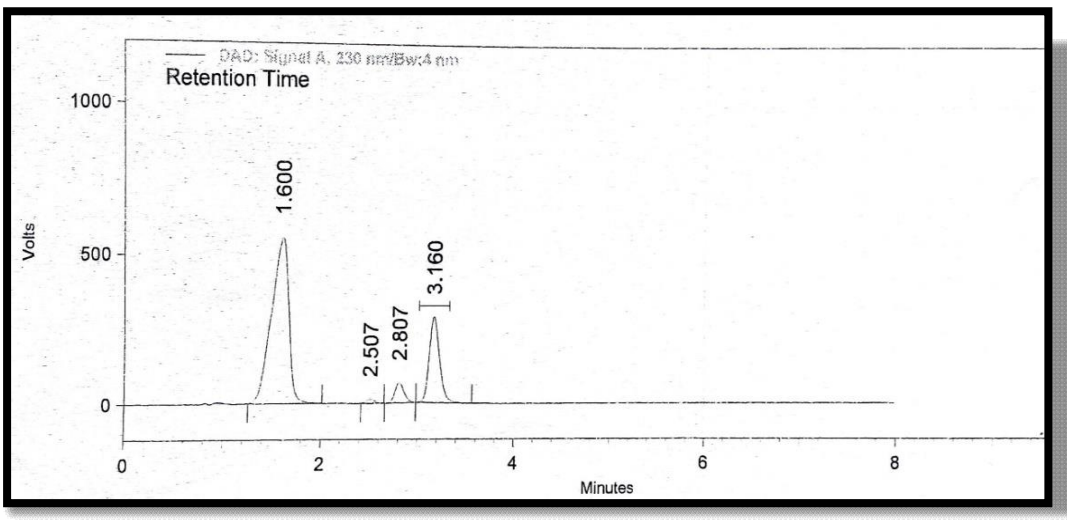


Fig. 4: Chromatogram of base degraded Lurasidone

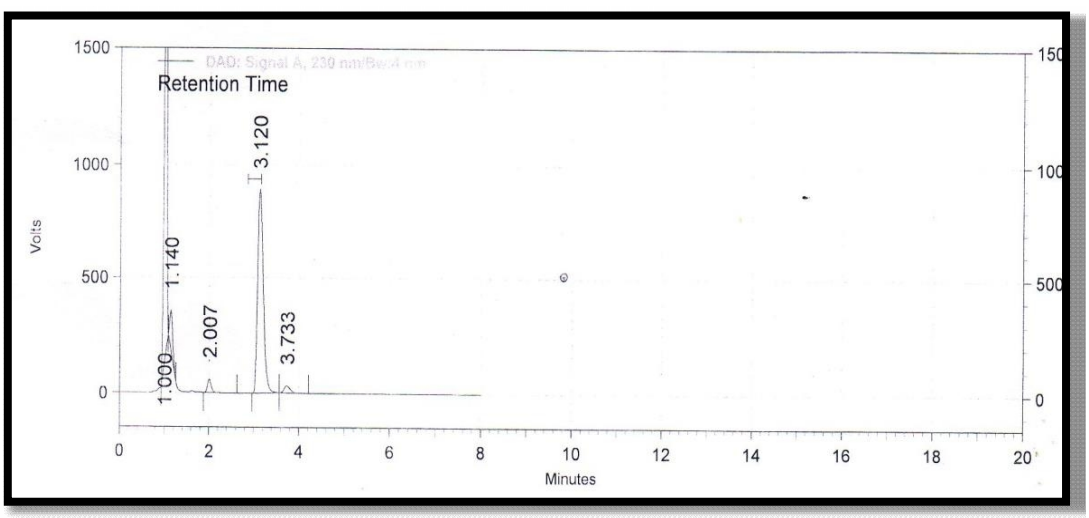


Fig. 5: Chromatogram of peroxide degraded Lurasidone

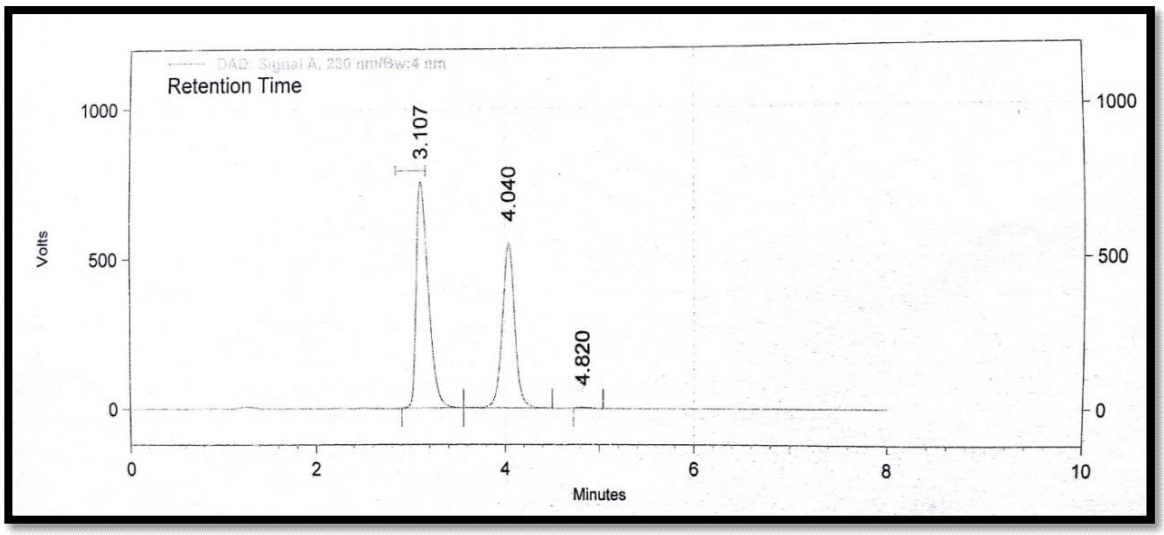


Fig. 6: Chromatogram of photo degraded Lurasidone

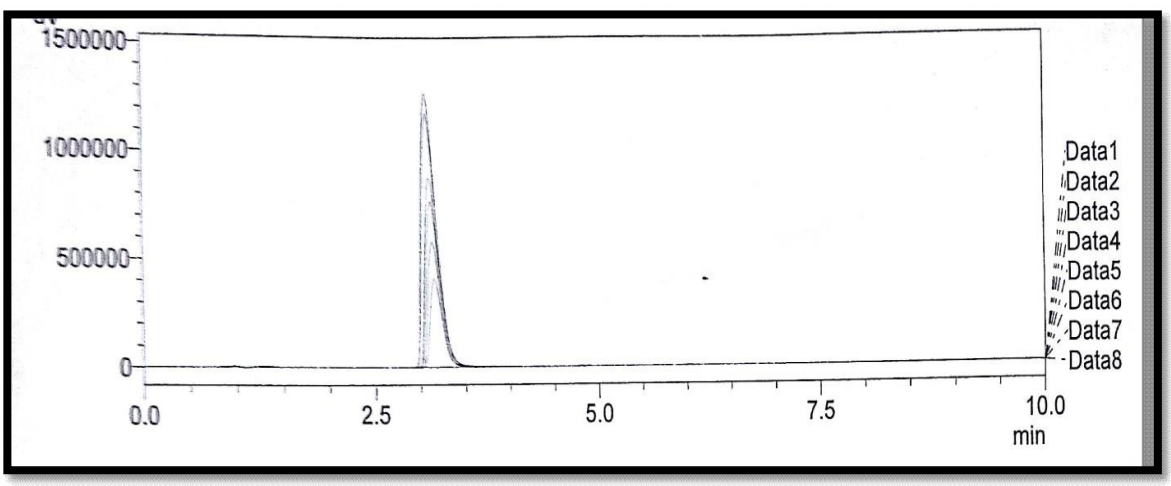


Fig. 7: Linearity overlain spectra of Lurasidone

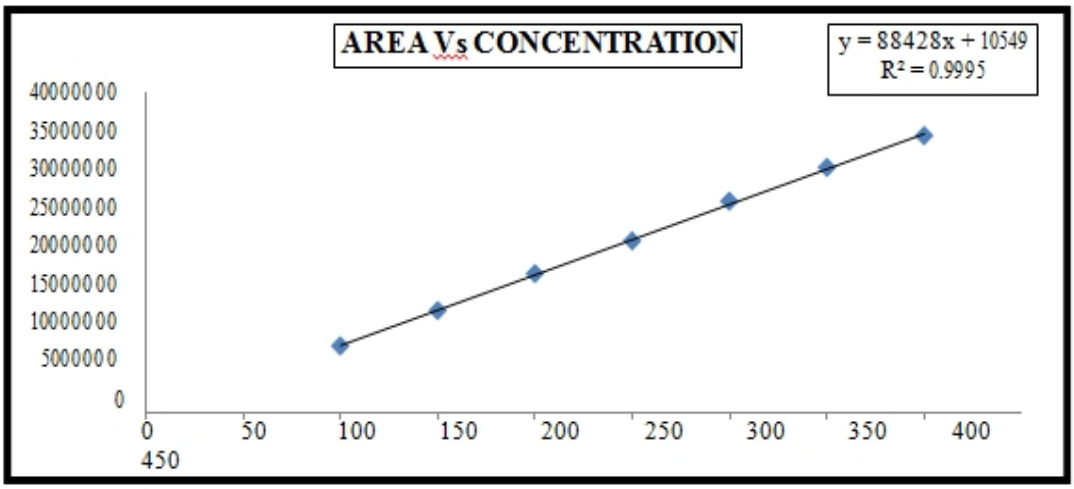


Fig. 8: Linearity plot of Lurasidone

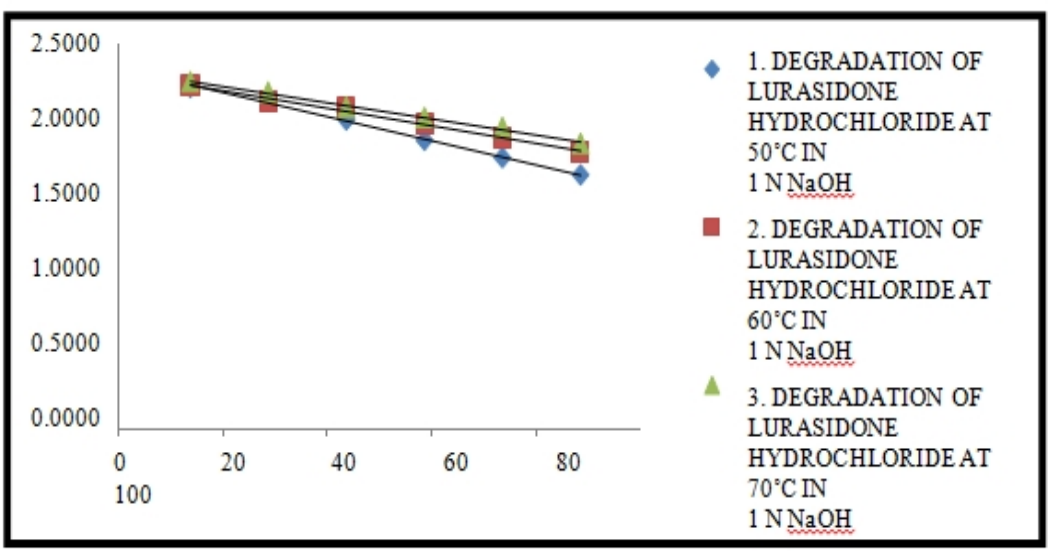


Fig. 9: Semi logarithmic plot of concentration of Lurasidone versus time in 1N NaOH

TABLES

Table 1: Forced degradation study of Lurasidone at different stress condition

Degradation condition	% Degradation	Resolution	Peak purity index
Acid/1 N HCl/80˚C/ 24 hr	**ND**	-	1.0000
Base/ 1 N NaOH/80˚C/ 1 hr	79.08	2.04	1.0000
Peroxide/ 3% H_2O_2/ RT/ 3 hr	18.35	5.93	1.0000
Photo/200 watt h/m^2/UV/ 24 hr	34.54	3.99	1.0000
Thermal/ 80˚C/72 hr	**ND**	-	1.0000

Table 2: Data indicating repeatability of Lurasidone

Concentration ($\mu g\ mL^{-1}$)	Peak area SD		% RSD	
250	Repeatability	Intermediate precision	Repeatability	Intermediate precision
	24989	39807	0.11	0

Table 3: Data indicating Accuracy

Level	Total concentration	Expected amount	Recovered amount	% Recovery	% RSD
80%	180.33	80	80.33	100.42	0.27
	180.35	80	80.35	100.44	
	179.97	80	79.97	99.97	
100%	201.89	100	101.89	101.89	0.14
	201.83	100	101.83	101.83	
	201.63	100	101.63	101.63	
120%	221.07	120	121.07	100.89	0.50
	220.03	120	120.03	100.03	
	221.10	120	121.10	100.91	

Table 4: Data indicating Linearity of Lurasidone

Linearity solution	Concentration (µg/mL)	Area
Level-1	100	8782962
Level-2	150	13209876
Level-3	200	17803079
Level-4	250	21941596
Level-5	300	26892187
Level-6	350	31099140
Level-7	400	35093295
Correlation Coefficient (r^2)	0.9995	
Slope of regression line	88428	
Y-intercept	10549	

ACKNOWLEDGEMENT

We are thankful to Torrent Pharmaceuticals for providing with Lurasidone API. We thank Oasis Test House for their kind help and support for providing with the needed instruments.

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ANATOMY, FLUORESCENCE ANALYSIS AND PHYTOCHEMICAL SCREENING OF *Stachytarpheta indica*

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ABSTRACT

Stachytarpheta indica is a small perennial erect shrub. The whole plant or its parts are used medicinally. It has not been studied for its anatomy and pharmacognosy. In this work anatomy of various parts, fluorescence analysis and phytochemical screening of plant extracts has been investigated. The investigated parameters are helpful in taxonomy and pharmacognosy.

SUMMARY

The investigated parameters i.e. fluorescence analysis and phytochemical screening of *Stachytarpheta indica* are helpful in taxonomy and pharmacognosy.

Keywords: *Stachytarpheta indica*, Anatomy, Pharmacognosy, Phytochemicals

INTRODUCTION

Stachytarpheta indica belonging to Verbenaceae family and is a small perennial erect shrub reaching up to 90 - 180 cm height. It is one of the important medicinal plants commonly found in South Gujarat region of Western India. The whole plant is useful in the treatment of diabetes (Adjanahoun*et al*., 1991). Plant is also useful in veneral diseases, erysipeals, dropsy and stomach ailments; juice employed to remove cataract and decoction as an abortifacient (Ambasta, 1986).Infusion of stem bark is used in diarrhea and dysentery (Ambasta, 1986).In Brazil, the triturated fresh leaf is applied to ulcers (Watt and Breyer - Brandwijk, 1962). Leaf paste is applied for skin troubles. Decoction of leaves is used for ulceration of nose (Shamnugam *et al.*, 2009). It is revealed from literature survey that the pharmacognostic studies on this species is lacking. Hence, the present study carried out to evaluate various pharmacognostic standards.

MATERIALS AND METHODS

Fresh plant materials were collected from the campus of VNSG University, Surat and were sun dried over a period of 48h. The fluorescent and phytochemical analyses were performed by using standard methods (Trease and Evans, 1986; Harborne, 1973). For the anatomical studies, 8 to 10 µm thin sections were taken and stained with safranin O and FCF fast green following Berlyn and Miksche (1976).

RESULTS AND DISCUSSION

Results

Anatomical Studies

Stem

It has uniseriate epidermis, hypodermis of collenchyma and parenchymatous cortex. The pith is parenchymatous and encircled by four collateral vascular bundles (Fig. 1A).

In old stem secondary xylem has roundish, solitary vessel elements, uniseriate xylem rays and polygonal xylem fibers with thick lignified cell wall. Secondary phloem is made up of sieve tube elements, companion cells and many phloem parenchyma cells. Small groups of fibers are observed external to secondary phloem (Fig. 1B). Node is unilacunar single trace. Leaf trace is arc shaped (Fig. 1C).

Root

Young root has narrow cortex of parenchyma and tetrarch xylem. Pith is small, made up of parenchyma cells. Secondary xylem has solitary vessel elements, uniseriate xylem rays and fibers. Patches of fibers are observed embedded in secondary phloem (Fig. 1D, E).

Root periderm has phellum of rectangular cells with suberized wall and phelloderm of tangentially enlarged parenchymatous cells. Certain phellum cells have tannin (Fig. 1D, E).

Leaf

Lamina has short palisade cells and 6 - 8 layers of spongy cells. Adaxial epidermal cells are larger than abaxial ones. Their cell wall is highly sinous. Anomocytic stomata are present on both surfaces (Fig. 1F, 2A).

Midrib and petiole have hypodermis of collenchyma and large parenchymatous cortex. Midrib has a main central, crescentic collateral vascular bundle and 2 - 3 small collateral strands on its adaxial face. The vasculature of petiole has a large incurved collateral strand and two adaxial bundles, each in one corner (Figs. 2B, C).Eglandular filiform trichomes are present on lamina, midrib, petiole and stem. Certain trichomes have warted cell wall and bulbous lower cells (Fig. 1A).

Fluorescence analysis and phytochemical screening

Stachytarpheta indica yields course powder and color is yellowish brown. The fluorescence analysis is presented in (Table 1). Qualitative phytochemical analysis of the different extract showed the presence of Alkaloid, Tannin, Flavonoid, Proteins, Steroids, Carbohydrates, Fats and Fixed oils (Table 2).

Discussion

Anatomical investigations showed that the Stachytarpheta indica exhibits distinguishing characters Viz.; eglandular trichomes, four collateral bundles in stem, anomocytic stomata, unilacunar node, crescent shape vascular strand in midrib and presence of adaxial bundles in midrib and petiole. These micro-characters have pharmacognostic value.

The fluorescent analysis data may help to identify authentic drug. The phytochemical screening confirmed the presence of active phyto constituents like Alkaloid, Tannins, Flavonoid, Steroids, Carbohydrates, Fats and Fixed oil. These parameters are help to identify the correct species of the plant as such analysis has been not carried out earlier.

CONCLUSION

From the present study it was revealed that several microscopic features, fluorescence analysis and phytochemical screening of *Stachytarpheta indica* are helpful in taxonomy and pharmacognosy.

FIGURES

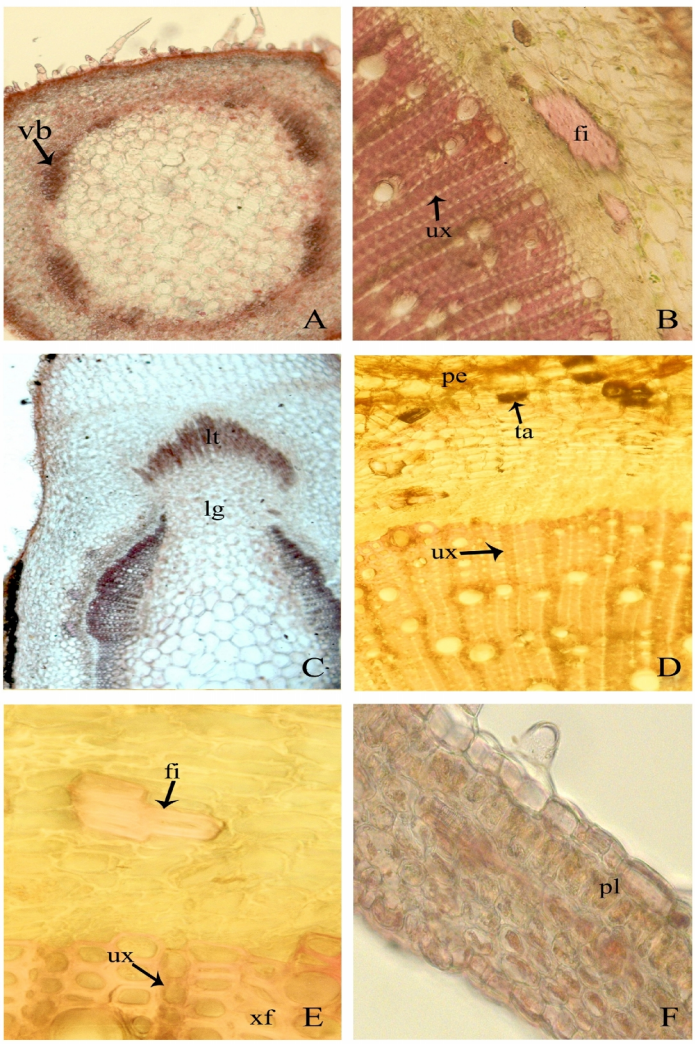


Fig. 1 A - F, transection. A, B, stem; C, node; D, E, root; F, lamina. A, B, C, D, F, X480; E, X800. (fi - fibers; lg - leaf gap; lt - leaf trace; pe - phellum; pl - palisade cells; ta - tannin; ux - uniseriate xylem rays; vb - vascular bundle).

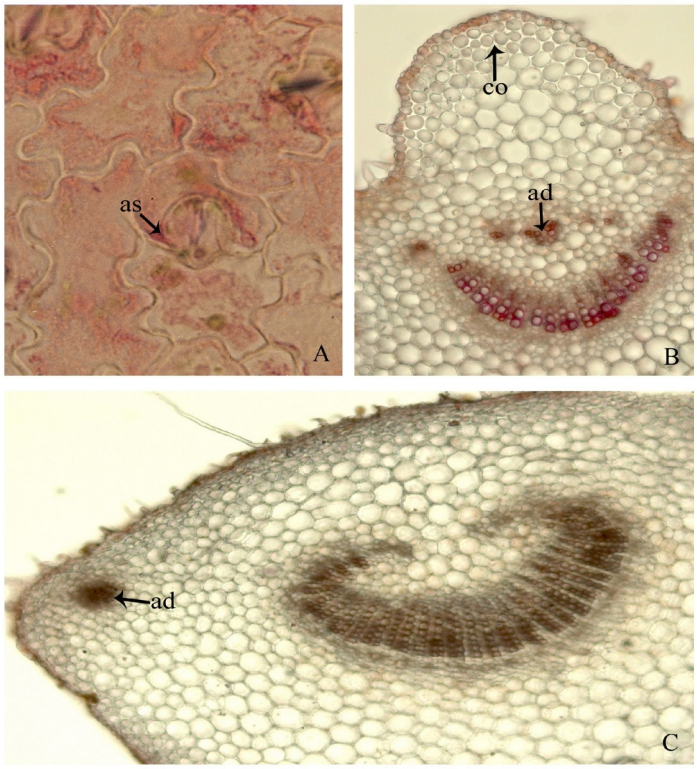


Fig. 2 A - C. A, surface view of adaxial epidermis of lamina. B, C, transection. B, midrib ; C, petiole. A, X800 ; B, C, X480. (ad - adaxial bundle ; as - anomocytic stomata ; co - collenchyma).

TABLES

Table 1: Fluorescence analysis of *Stachytarpheta indica*

Treatment	Ordinary light	Ultraviolet light	
		254 nm	366 nm
Powder as such	Yellowish Brown	Brown	Blackish Brown
Powder + Distill water	Orange	Brown	Yellow
Powder + HCL	Yellow	Creamy White	Yellowish Orange
Powder + H_2SO_4	Creamy White	Orange	Yellow
Powder + Iodine Solution	Brick Red	Black	Black
Powder + Acetic acid	Orange	Brown	Yellowish Orange

Table 2: Preliminary phytochemical screening of the plant extract

Phytochemicals	Test	Aqueous extract	Petroleum ether extract	Alcohol extract
Alkaloid	Dragendroff's test	+	-	-
Tannins	Ferric chloride test	+	-	-
Flavonoid	Alkaline reagent test	+	+	-
Steroids	Salkowski test	-	+	+
Carbohydrates	Molish's test	+	+	+
Fats and Fixed oil	Copper sulphate test	-	+	+

+ indicate present; - indicate absent.

ACKNOWLEDGEMENT

We are thankful to Dr. Y. J. Thanki for giving us plant authentication based on flora of India.

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Proceedings of RK University's First International Conference on Research & Entrepreneurship (Jan. 5th & Jan. 6th, 2016)

(Proceedings available for download at rku.ac.in/icre)

RK University's First International Conference on Research & Entrepreneurship (ICRE 2016)

Isolation And Identification Of B-Sitosterol From The Stem Bark Of Three Indigenous *Cordia* Species, Using The Preparative TLC Method

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ABSTRACT

The present study aimed to isolate and identification of β-sitosterol from the stem barks of three *cordia species, C. macleodii, C. dichotoma and C. rothi*. For isolation of β–sitosterol, Unsaponifiable fractions of all three species were introduced to preparative TLC (20cm*20cm) using Hexane: Diethyl Ether: Glacial Acetic Acid (6:4:0.2) as mobile phase. The detection of spot of β–sitosterol was accomplished under UV light (366 nm) and corresponding spot was scrapped out at 0.35 Rf and collected separately along with the silica gel. Scrapped compounds were purified by chloroform and subjected to HPTLC study for identification with reference standards. Both, standard and sample are separated on same Rf 0.35. Similarities of this Rf value suggest that all three species contained β–sitosterol and further it is confirmed by comparison through super imposable UV spectra. So the compound was concluded as β-sitosterol.

SUMMARY

Isolation and Identification of β sitosterol from three medicinal *cordia* species.

Keywords: C. macleodii, C. dichotoma, C. rothi, HPTLC, β Sitosterol, Phytosterols, Preparative TLC

INTRODUCTION

Cordia species found as shrub and trees in the family Boraginaceae. About 300 species have been identified worldwide, mostly in warmer regions**.** *Cordia species* from boraginaceous family is one of the rich sources to get therapeutic important compound. *Cordia species*are widely distributed in the moist & dry deciduous forest up to 1200 m in peninsular India, mostly from Chota Nagpur to Tamilnadu and Gujarat.*(1)* The genus *Cordia* (Boraginaceous), a known source of benzoquinones, naphthoquinones, *(1)* hydroquinone, triterpenes, *(2)* polyphenols and flavonoid comprises about 250 species distributed throughout the New World. Many compounds originally isolated from *Cordia* species have been reported as presenting several biological activities such as antifungal, larvicidal, anti–inflammatory and anti–androgenic.*(2,3)*

Cordia macleodii Hook. (Boraginaceae), native to India, has been traditionally reported to be used as aphrodisiac, for mouth sores and especially for jaundice.*(4)* Though the stem bark of this plant has been highlighted for different enthnopharmacological properties*(5)* research study report on phytochemical studies is lacking.*(6)* Sometimes it is observed that people use the taxonomically different species for the same use, just because of morphological similarities or a common vernacular names e.g. *Calotropis sp., Sida sp., Ocimum sp.* etc. It is the duty of a scientist to work out the scientific rationales for such kind of use. Therefore, in the present study an attempt has been made to evaluate the phytochemicals of stem bark of *Corida macleodii* along with other allied species *Cordia dichotoma* (Forest F.) and *Cordia rothi* (Roem & Schulte).

In this research work, a simple and accurate preparative TLC and HPTLC methods has been established for isolation and identification β–sitosterol in the dried stem bark powder of *C. macleodii*, *C.dichotoma* and *C.rothi*.

MATERIALS AND METHODS

Plant Material

Fresh stem bark of *C. macleodii* was collected from Balakgir district of Orissa, India in the month of April–May 2009 and *Cordia rothi* & *Cordia dichotoma* barks were collected from their natural habitat, Jamnagar (Gujarat), India in month of April–May 2009.The collected samples were identified, authentified by using various floras and texts and also matched with Pharmacognosy departmental herbarium of Gujarat Ayurved University, Jamnagar, Gujarat.

Chemicals and Reagent

Standard compound β–sitosterol purchased from Sigma-Aldrich. Other chemicals like Petrolium ether, Chloroform, Methanol, Hexane, di ethyl ether (Loba-India), used in this work were procured from Merck india LTD. Analytical TLC plates used in the studies were: Silica gel 60 F254 TLC plate (Merck, Darmstadt, Germany)

Standard stock solutions

Accurately weighed 10 mg β–sitosterol was dissolved in 10 ml Chloroform. This stock solution was used for further study.

Unsaponification of Sample *(7)*

A correctly weighed petroleum ether extract (1.01) was taken in volumetric flask and add 40 ml of 20% alcoholic KOH and kept overnight. On next day, the mixture was refluxed for 6hrs and then cooled at room temperature. Two times volume of distilled water was added and extracted with ether. Combined ethereal extract was washed with distilled water till neutral with litmus paper and dried over anhydrous sodium sulphate. After this, ether was evaporated to obtain un-saponifiable portion.

Solvent System Designing*(8, 9)*
Multi component solvent system was designing using triangle series founded by Snyder. Here, TLC was used for the designing of multi-component solvent system. Finally from different solvent, multi component solvent system, Hexane: Di ethyl ether: Glacial Acetic Acid in the proportion of 6:4:0.2 designing for the ideal and maximum separation for unsaponifiable fractions.

Isolation and purification of compounds by Preparative TLC
The unsaponifiable fractions of all three plants were dissolved in the chloroform of required solubility and this solution was spotted on TLC plates with reference standard. Then the TLC plates were run by specific solvent system, Hexane: Di ethyl ether: Glacial Acetic Acid in the proportion of 6:4:0.2. and were view individually in UV chamber on 366 nm and with the Liberman buchard spraying reagent. (Grayish violet) After identification of interest compound spot it was subjected to preparative TLC.

In preparative TLC (20*20 cm) method, Concentrate sample was loaded. Band spotting was done so that highest amount of sample can be loaded on the plates which facilitate the maximum separation of the material of interest. The plate was allowed to run in the mobile phase. After complete development the plate was dried out. The finding of spot of β–sitosterol was accomplished under UV light. (366 nm) Each of the fluorescent spots, confirm with those of standard reference compound noted down. Corresponding spot was separated out at 0.35 Rf (grayish violet). The band that shows the 0.35 R_F was marked and the interested compound was scrapped out and collected separately along with the silica gel along.

The separated compound was redissolved in chloroform. Afterward the solution was filtered and the solvent was evaporated and purified with chloroform. Then it was subjected to HPTLC for the identification with reference standard.

Identification of compound by HPTLC

Instrumental conditions:
HPTLC was performed on 5 cm × 5 cm aluminum backed plates coated with 0.2 mm layers of silica gel 60 F254 (Merck, Germany). Samples were applied in band with a Linomat V applicator CAMAG (Switzerland), equipped with a 100- µ L syringe, band length 8 mm to 10 mm, track space 8 to 14 mm (as required). Plates were developed vertically in a CAMAG twin trough chamber previously saturated with mobile phase vapor for 15 to 20 min at room temperature. After development, the plates were dried at room temperature and observed in a CAMAG UV cabinet. Densitometry scanning of the plates was performed in densitometer scanner 4 with winCATS software (CAMAG, Switzerland) in reflectance-absorbance mode; scan speed 10 mm/s.

RESULTS AND DISCUSSION

The present study was undertaken to develop a rapid isolation and identification method of β-sitosterol as single and in all three barks samples.

Optimization of the Chromatography

Various mobile phases, single solvents, as well as various blends of solvent mixtures in varying proportions, were tried to achieve optimum resolution. The mobile phase Hexane: Di ethyl ether: Glacial Acetic Acid in the proportion of 6:4:0.2 showed better resolution of both β-sitosterols (0.35 RF) (Figure 1A, 1B.1C). The selection of wavelength was based on maximum absorbance for optimum sensitivity.

CONCLUSION

By using preparative TLC method, β-sitosterol was isolated from petroleum ether extract of the stem barks of all three plants and identified by HPTLC with standard compound. It is first time reported in stem barks of all these plants. Preparative TLC is the method for separation of the active principles on the basis of their R_f values. By this method the purity can obtained up to the mark. It is also a technique to separate mg/gm level of the sample. Therefore, the proposed TLC method being simple, fast, and cost effective It will be very useful as routine quality control method for detection of β–sitosterol as such and in various medicinal plants.

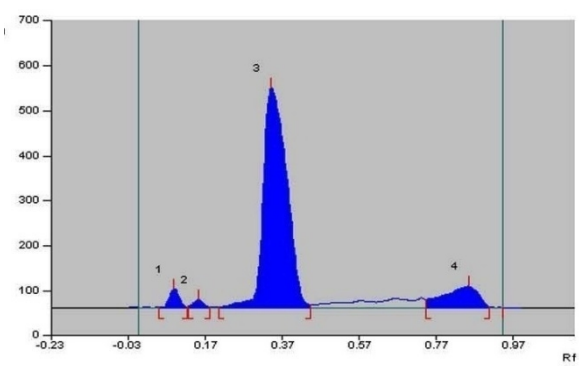
FIGURES



1A

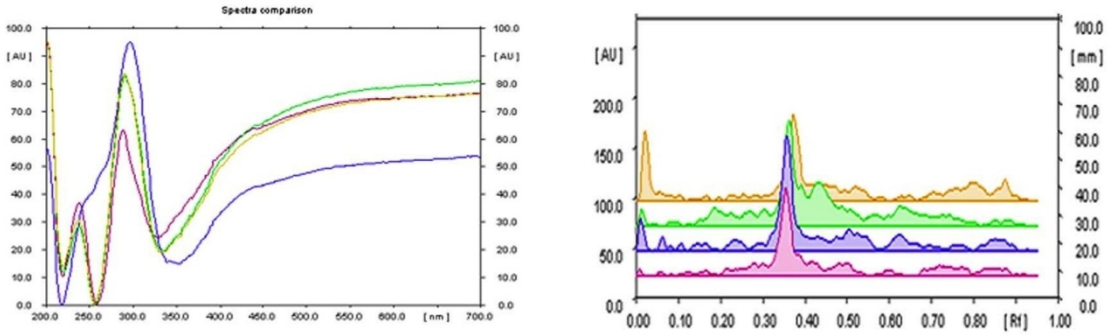


1B



1C

Figure: 1 Identification of β–sitosterol, **A (366 nm):** T1-Standard compound, T2-*C.macleodii* unsap, *C.dichotoma* unsap, *C. rothi* unsap, **B:** After spray with L.B. reagent, **C:** Densitogram of β–sitosterol



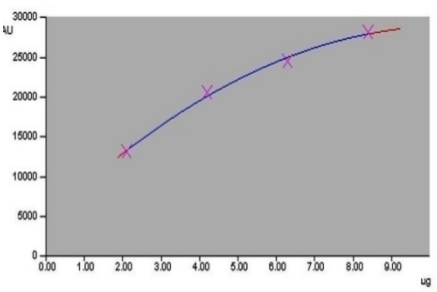
UV comparison of all track

2A

3D graph of all track

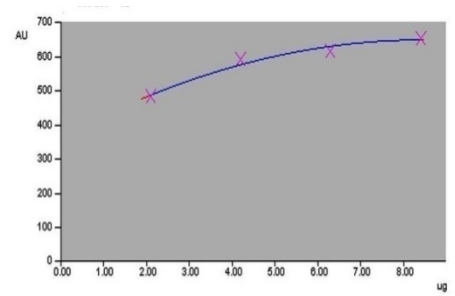
2B

Figure: 2 A: comparison of super imposable UV spectra, **B:** 3D graph of all tracks with standard



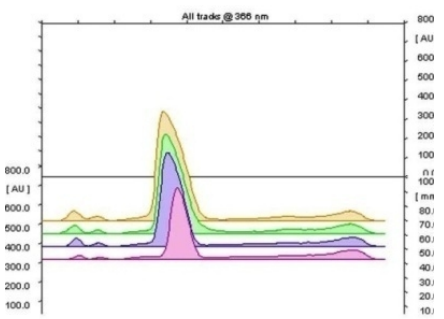
Liniarity via Area

3A



Liniarity via heaight

3B



3D Graph of β-Sitosterol

3C

Figure: 3 A: Linearity via area, **B:** Linearity via height**, C:** 3D graph of linearity

TABLES

Table 1: Identification of β–sitosterol with isolated compound of all three stem barks.

Sample	366 nm (long UV)		After spray with L.B. reagent	
	No. of Spot	R_f Value	No. of Spot	R_f Value
Track–1	1	0.35	1	0.35
Track–2	3	0.23,0.36,0.50	2	0.36,0.50
Track–3	2	0.36,0.43	1	0.36
Track–4	3	0.37,0.52,0.88	1	0.37

Table no. 1 showed the identification of β–sitosterol in all barks. Standard compound separate on 0.35 R_f value at 366 nm and derivetization with L.B. reagent. Isolated compounds of barks were also separated at 0.35 R_F value which suggested that β–sitosterol present in all three barks. It is also confirmed by comparison through super imposable UV spectra. (Fig.2A). 3D graph of all track with standard have been shown in figure no 2B.

Table 2. Linearity of β–sitosterol

Sample	254 nm (Short UV)		366 nm (long UV)		After spray with L.B. reagent	
	No. of Spot	R_f Value	No. of Spot	R_f Value	No. of Spot	R_f Value
Track–1	1	0.35	1	0.35	1	0.35
Track–2	1	0.35	1	0.35	1	0.35
Track–3	1	0.35	1	0.35	1	0.35
Track–4	1	0.35	1	0.35	1	0.35

The calibration curves of β–sitosterol was prepared by plotting peak areas verses different concentration in the range 2.5–10 µg found to be linear as shown in table 2. Linearity have been shown in figure no 3A, 3B, 3C. Calibration parameters (**Regression mode-Polynomial**) for β–sitosterol via area, Y=4479.958+4636.991*X, R^2=0.998, sdv–3.20% and via height, Y=362.722+67.382*X, R^2=0.982, sdv–4.10%.

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