



**CENTRE OF
EXCELLENCE
IN PROCESS
INTENSIFICATION**

ABOUT THE DEPARTMENT



PROFESSOR SUNIL S. BHAGWAT

B. Chem. Engg (Mumbai, 1984), M. Chem. Eng. (Mumbai, 1986), Ph.D. (Tech)(Mumbai, 1989)

Professor in Department of Chemical Engineering

Coordinator, Centre of Excellence in Process Intensification (COE-PI) (TEQIP Phase-II)

ABOUT ICT

The Institute of Chemical Technology (ICT), a Deemed University under Section 3 of UGC Act of 1956, was a Lead Institution under the Technical Education Quality Improvement Program (TEQIP) Phase –I of the Government of India. The purpose of TEQIP is to enhance capacities of institutions to become dynamic, quality conscious, efficient and responsive to rapid economic and technological developments occurring at both, and the national and international levels. ICT made impressive progress under Phase –I. In the second phase of TEQIP-II, ICT has been selected for funding of Centre of Excellence in Process Intensification by

MHRD, GoI, for the period September, 2013 to March, 2017 with total grant of Rs. 5.0 Cr.

ABOUT CENTRE OF EXCELLENCE IN PROCESS INTENSIFICATION (COE-PI):

The Centre of Excellence in Process Intensification has been established under TEQIP-II, sponsored by MHRD, GoI. The Centre has taken up research activities to help industries to modify their processes with an objective of achieving reduction in the energy consumption and environmental impact. The centre works on real life problems, train research students as well as improve the undergraduate education

by incorporating principles of process intensification. In addition, the Centre has taken up few projects related to social relevance related to water purification, indigenous generation of electricity in rural area, improvement of energy efficiency in cooperative dairy industry.

The centre is carrying out interdisciplinary research which includes Chemical Engineering, Polymer Science and Engineering, Textiles, Food Engineering and Technology, Basic chemistry, Oils and Oleo chemicals, Dye-stuff Technology branches. The centre will thrive to improve the existing chemical processes in terms of reduction in number of steps, equipment, time, and space and energy.

Head & Nodal officer		Name	Email ID
Head of the Institute		Prof. G. D. Yadav, Vice-Chancellor	gd.yadav@ictmumbai.edu.in
CoE-PI Coordinator		Prof. S.S. Bhagwat	ss.bhagwat@ictmumbai.edu.in
Nodal Officer		Prof. V. K. Rathod	vk.rathod@ictmumbai.edu.in

STEERING COMMITTEE

Sr. No.	Name	Designation	Email
1	Professor S. S. Bhagwat	Coordinator	ss.bhagwat@ictmumbai.edu.in
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4	Professor V. G. Gaikar	Member	vg.gaikar@ictmumbai.edu.in
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6	Dr. Shirish Karve	Member (Industry)	shirish.karve@gmail.com
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8	Dr. Ravi Mariwala	Member (Industry)	ravi.mariwala@gmail.com

COE-PI OFFICE STAFF:

Miss. Supriya Gavali (from March, 2014 to till today)

PUBLICATIONS:

Sr. No.	Journal Name	Title of the Paper	Authors Name
1	Indian Journal of Scientific Research (IJSR)	Dyeing of Nylon using nano disperse dyes	Dr. R. D. Kale
2	Chemical Engineering Science	On nature of mass transfer near liquid-liquid interface in the presence of Marangoni instabilities (In Press)	Dr. C. S. Mathpati
3	ICMST -2016 Conference	Microalgae as Sustainable Energy and Its Cultivation (ICMST-2016, will be published in Advanced Science Letters of American Scientist Publisher)	Dr. C. S. Mathpati

4	International Journal: Ultrasonics Sonochemistry	Intensified Oxalic Acid crystallization using ultrasonic reactors: Understanding effect of operating parameters and type of ultrasonic device.	Dr. P. R. Gogate
5	International Journal: Ultrasonics Sonochemistry	Improved crystallization of ammonium sulphate using ultrasound assisted approach with comparison with the conventional approach	Dr. P. R. Gogate
6	Current catalysis	Microwave assisted Biginelli reaction using MoO ₃ -SiO ₂ as bi-functional catalysts (Communicated)	Prof. R. V. Jayaram
7	Catalysis Communications	Microwave assisted synthesis of 4(3H)-Quinazolines using MoO ₃ -SiO ₂ as bi-functional catalysts (Communicated)	Prof. R. V. Jayaram
8	ChemCatChem	Sorption-Enhanced Steam Reforming of Glycerol over Ni-hydrotalcite: Effect of Promotion with Pt	Dr. P. D. Vaidya
9	International Journal: Hydrogen Energy	Tailored Ce- and Zr-doped Ni/hydrotalcite materials for superior sorption-enhanced steam methane reforming	Dr. P. D. Vaidya
10	Energy Technol	New hybrid materials for improved hydrogen production via sorption-enhanced steam reforming of butanol	Dr. P. D. Vaidya
11	Under Preparation	Three phase partitioning as a novel approach for the rapid extraction of Marmelosin (Imperatorin) from Aegle Marmelos fruit and determination of antioxidant activity	Prof. V. K. Rathod
12	Under Preparation	Simultaneous determination of Corosolic acid from Lagerstroemia speciosa by using three phase partitioning	Prof. V. K. Rathod
13	Under Preparation	Rapid determination of Corosolic acid from lagerstroemia speciosa by using microwave assisted extraction	Prof. V. K. Rathod
14	Under Preparation	Ultrasound assisted extraction of Corosolic acid from lagerstroemia speciosa	Prof. V. K. Rathod
15	Under Preparation	Microwave assisted extraction of Marmelosin from Aegle Marmelos	Prof. V. K. Rathod
16	In Preparation	Novel metallopeptide hybrid complex nano-materials-enantioselective Self assembly	Prof. B. M. Bhanage

17	In Process	Ascertain the feasibility of using the diesel engine variant of the process in a rural area to meet the electricity and water demands on the area	Dr. V. H. Dalvi
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PATENTS:

Sr. No.	Title	Inventor
1	Zinc Oxide Peptide Nanomaterials and Method Of Preparation Thereof.	Prof. B. M. Bhanage
2	An efficient synthetic methodology to synthesize 2-chloro alkyl ethanoate compounds catalyzed by Li.	Prof. B. M. Bhanage
3	A robust Bio-catalytic methodology to synthesize (2E)-bot-enoate compounds using lipase and Super	Prof. B. M. Bhanage
4	Eco-Friendly Methodology for synthesis of 2, 2-Dimethyl propanoate compounds catalyzed by lipase.	Prof. B. M. Bhanage
5	An improved process for nucleophilic aromatic fluorination	Prof. M. S. Degani
6	Integrating enhanced power generation with low to medium thermal treatment of an aqueous stream.	Dr. V. H. Dalvi
7	Rotating disc impinging jet contactor (In Process)	Dr. C. S. Mathapati

THE FOLLOWING PROJECTS ARE CURRENTLY GOING ON UNDER THE CENTRE:

- | | | |
|--|---|--|
| 1. Dyeing of Polyester & it's blend using Nano-emulsions | and Computational Fluid Dynamics | Synthesis of Bioactive colorants lutein/lycopene/indigoid/azulenes |
| 2. Sorption-Enhanced Reforming process for Hydrogen production | 7. Process development of Nanostructure Metal oxides by sonochemical techniques | 12. Enzymatic Process Intensification for the manufacture of structure lipids to enhance the yield |
| 3. Process intensification through catalytic process- Microwave Assisted Bifunctional Catalysis for Tandem Reactions | 8. Extraction of Natural Ingredients Using Novel Extraction Techniques | 13. Validation of new hand-pump design for water disinfection: Field Trials** |
| 4. Process Intensification of Crystallization Using Sonochemical Reactors | 9. Development of eco-friendly and cost effective extraction technologies using supercritical carbon dioxide" | 14. Micro Hydro Electricity Production: Electricity Generation for Lighting and Irrigation using natural flow of irrigation Canal and its Performance Evaluation** |
| 5. Microwave assisted halogenations reactions using flow reactor | 10. Microwave,Ultrasound, Solar energy assisted preparation of Metal Oxide Nanomaterials | 15. Open Source Process Simulator for Process Intensification** |
| 6. Design aspects of two-opposed-jet micro-extractor: Experimental | 11. Microwave Assisted/enzyme mediate extraction/ | |

16. Heat Based refrigeration technology implemented in Dairy for milk chilling**

(** A project related to technology transfer to rural India has been added as requested by NPIU)

COE-PI PROJECT REPORT:

Project Title: **Dyeing of**

Polyester and its blend using Nanoemulsions

Project investigator: **Dr**

Ravindra D Kale

Coinvestigator: **Dr. Amit P.**

Pratap

1. Objectives:

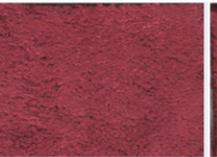
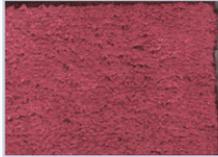
- Explore the possibility of dyeing Nylon with these nanoemulsions
- Dyeing of Polyester by

thermosol method using nanoemulsion

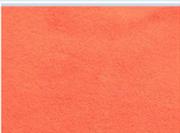
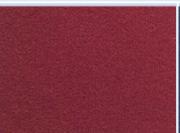
- Dyeing of polyester/Lycra blend with Crude disperse dyes using nano emulsion
- Dyeing of Aramide blend with Crude disperse dyes using nano emulsion
- Study the stability of nanoemulsion

2. Outcome till date:

3.1 Polyester and lycra blend :

Temp(°C)/Dyeing Methods	Control Dyed Samples (CDS)	Nanoemulsion Methods		
		Phase Inversion Composition	Ultrasonication	Spontaneous
100				
110				
120				
130				

3.2 Nylon

Substrate: Nylon		Shade: 3%	
Machine: HTHP		Dyeing Time: 60min	
Dyeing Temp: 100°C			
	Nanodispersed Dye		
	Orange RL 200%	Rubine GFL 200%	Navy Blue 2GD H/C
Dyeing with commercial dye			
Nanoemulsion Dyeing Method			

3.3 Thermosol dyeing of polyester

Substrate: Polyester		Shade: 3%	
Machine: Thermosol Tech.		Dyeing Time: 2min	
Curing temperature: 200°C			
	Nanodispersed Dye		
	Orange RL 200%	Rubine GFL 200%	Navy Blue 2GD H/C
Dyeing with commercial dye			
Nanoemulsion Dyeing Method			

3. ACHIEVEMENTS TILL DATE (DELIVERABLE: PUBLICATION, CONFERENCE, PATENTS):

Publication

Sr. No.	Title And Authors	Journal/Conference	Vol. No.	Pages	Year
a)	Dyeing of Nylon using nano disperse dyes R.D. Kale, Amit Pratap, Prerana Kane, Vikrant Gorade	Indian Journal of Scientific Research (IJSR) (ISSN : 0976-2876 (Print) ISSN: 2250-0138 (Online))	Accepted Manuscript		

Conference:

b)	Dyeing of Polyester and its blend using nanoemulsions. R.D. Kale, Amitb P. Pratap, Vikrant Gorade, Prerana Kane	<i>Won best presentation award at "CoE, TEQIP-II - Conclave for Research Excellence through Collaboration" organized by The Centre of Excellence (CoE), College of Technology, GB Pant University of Agriculture & Technology, Uttarakhand.</i>	-	-	06 th - 08 th October 2016
c)	Dyeing of Nylon using nano disperse dyes R.D. Kale, Amit Pratap, Prerana Kane, Vikrant Gorade	Presented a paper in International Conference on Contemporary Issues in Science, Engineering and Management, Gandhi Institute For Technology (GIFT) at Gramadiha, Bhubaneswar, Orissa.	-	-	18 th & 19 th February 2017

MOU FROM INDUSTRIAL PARTNER INTERESTED IN ABSORBING THE DEVELOPED TECHNOLOGY

- We have been carrying out bulk trials in **Reliance Industry, Ahmadabad** and who want to adopt this technology resulting in **consultancy of Rs. 6 Lakh**

Project Title:

Microwave, Ultrasound, Solar Energy assisted preparation of metal oxide nanomaterials

Project investigator: Prof. B. M. Bhanage

Objective: Synthesis of metal and metal oxide nanoflowers using alternative form of energy, such as solar energy, ultrasound and microwave.

Outcome till Date:

1. Synthesis of Zinc oxide Nanoflowers

Synthesis of zinc oxide nanoflowers by using microwave energy, sonication energy and by heat is well known idea. Research of synthesis of nanomaterials is shifting to synthesis using greener and Cost effective way. Flower shaped nanoparticles has lot of potential in immediate future application. Flower-shaped nanoparticles of zinc oxide could trigger the growth of new blood vessels reported in Nature India.

Such nanoparticles will be useful in repairing damaged blood vessels and restoring blood flow to damaged tissues. It will be useful for treatment of cardiovascular diseases, ischemic heart, limb diseases and wound healing. It also

has immediate application in catalysis, advance materials, drug delivery, biosensors and etc.

We have achieved major breakthrough in this area by using solar energy. we prepare zinc oxide nanoflowers using solar energy, benign chemicals and without any lens incorporated instrument. It takes 90-100 minutes (Depend on weather) under direct solar light.

The synthesized zinc oxide nanoflowers were confirmed using XRD and UV-Vis spectrometer

and also characterized with help of FE- SEM, DSC-TGA techniques. This innovation is applied for Indian

2. Synthesis of Novel Inorganic-peptide chiral nanomaterial.

Alkaline hydrothermal systems known as candidates for the origin and evolution of life on the primitive Earth. In this project we discovered synthesis of amide linkage in water alkaline hydrothermal condition.

The one pot formation of peptide from single or multiple amino acids with zinc in water through Enantioselective discrimination, showing highly complex hybrid self-assemble nanomaterials in

Uniform size and shape. This protocol facilitates formation of needle type nanoflowers, nanotube, plate type nanoflowers.

Chirality is one of the most fascinating occurrences in the natural world. A chiral molecule is one that has two mirror-image forms which are non-superimposable in three dimensions. The mirror-image forms of the chiral molecule are classified as enantiomers. Chirality plays an important role in the fields of chemistry, pharmacology, biology and medicine. These self-assemble zinc-peptide nanomaterials also exhibit chiroselective geometrical property. Currently we are carrying out characterization using FEG-SEM.

Foldamers:

Foldamer refers to a molecule those folds into a structurally stable state in solution. The design of foldamer design promises new routes to important compounds for use in sensors, smart materials,

and catalysts. Proteins and peptides are an important class of natural foldamers that carry out a host of essential functions in biology, including molecular recognition, information storage, catalysis, and controlled crystallization of inorganic materials.

Heterochiral peptides are one such class of biomimetics with a potential for greater structural diversity than peptides consisting solely of L-amino acids.

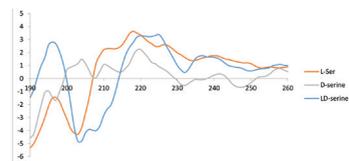
In this work, we discover novel approach for designing heterochiral peptides that can used as foldamers. Incorporating D-amino acids into peptides also reduces their susceptibility to proteolysis, making them useful for biomedical applications. Periodic heterochiral sequences yield interesting topologies, generally inaccessible to ribosomally encoded proteins. However, novel conformations can be attained by peptides by combinations of L- and D-amino acids.

Circular dichroism spectroscopic measurements were performed to determine the secondary structure of metallopeptide in 0.1% acetic acid solution. L-serine and LD-serine (racemic mixture of serine) shows characteristic helix state as compared to literature reported and CD spectra have been shown in Fig.

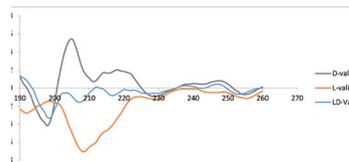
Due to lack of reported evidence, the unambiguous assignment of a CD spectrum to a specific

helical secondary structure in this case has not been possible to date. However there is

agreement from theoretical analyses which have suggested an 8-helix for this Circular dichroism spectra.



L-serine and LD-Serine show negative maxima at 203nm and intense and broad positive maxima spreading between 210nm to 250 nm. This indicates that helical orientation of Zn-(L-serine peptide) and Zn-(LD-Serine peptide) is same. D-serine shows positive peak at 203nm and negative spreading between 230nm to 250nm. All three curves shows minima at 232nm and 248nm.



L-valine and LD-Valine show negative maxima at 198nm and intense and broad positive maxima spreading between 210nm to 250 nm. This indicates that helical orientation of Zn-(L-Valine peptide) and Zn-(LD-Valine peptide) is same. D-Valine shows positive peak at 198nm and negative spreading between 230nm to 250nm. All three curves shows minima at 230nm and 248nm.

Achievements: Patents

1. Zinc Oxide Peptide Nanomaterials and Method of Preparation Thereof.

- PCT Application: (PCT/IB16/56356-01), Indian Patent application no. 201621036137
- An efficient synthetic methodology to synthesize 2-chloro alkyl ethanoate compounds catalyzed by Li.
- A robust Bio-catalytic methodology to synthesize (2E)-butenoate compounds using lipase and Super. Indian Patent Application: 201621043666
- Eco-Friendly Methodology for synthesis of 2,2-Dimethyl propanoate compounds catalyzed by lipase. Indian Patent Application: 201621043667

Publications :

- Novel metallopeptide hybrid complex nanomaterials-enantioselective Self-assembly. (In preparation).

Project Title: Micro Hydro Electricity Production: Electricity Generation for Lighting and Irrigation using the natural flow of irrigation Canal and its Performance Evaluation.

Project investigator: Dr. D. V. Pinjari

Objectives:

Social

- Distributed scale electricity generation for solving intermittent electricity supply problem in rural parts of India.
- It is one of the renewable and nonpolluting source of energy which can be harnessed for generating electricity at the location.

- It is beneficial for the sustainable development of rural parts of India.

Technological

- Pilot scale electricity generation using kinetic energy of flow (rivers, canals etc.)
- Based on pilot plant performance, detailed model will be developed for overall estimation of power production.
- Studying various parameters affecting electricity generation, this includes design of the blades, mass flow rate of water in the river/canal.

Outcome till date:

- Hydropower is a method of generating electricity that uses moving water (kinetic energy) to produce electricity. Small-scale hydropower has been used as a common way of generating electricity in isolated regions since end of 19th century. Small-scale hydropower systems can be installed in small rivers, streams or in the existing water supply networks, such as irrigation canal, drinking water or wastewater networks. In contrast with large-scale hydropower systems, small-scale hydropower can be installed with little or negligible environmental impact on wildlife or ecosystems, mainly because the majority of small hydropower plants are run-of-river schemes

or implemented in existing water infrastructure. Due to its versatility, low investment costs, and as a renewable energy source, small-scale hydropower is a promising option for producing sustainable, inexpensive energy in rural or developing areas.

- Micro hydro power systems are able to generate electricity by using the movement of water from small streams to rotate a wheel or turbine in order to spin a shaft. The shaft's motion is used to power an alternator or generator that converts the rotational energy to electricity. The major advantage of hydroelectricity is elimination of the cost of fuel. Hydroelectric plants are immune to increases in the cost of fossil fuels such as oil, natural gas or coal, and do not require fuel to be imported. Hydroelectric plants tend to have longer lives than fuel-fired generation, with some plants now in service having been built 50 to 100 years ago. Operating labour cost is usually low since plants are automated and have few personnel on site during normal operation.
- For the experimentation of the micro hydro project site was selected such that where water velocity was about 1.8 m/sec and volumetric flow about 30,000 Liter/sec. Both sides of channel is concreted so we get a maximum water flow also there is hard rock at the site so required civil work

was less. The selected was sited under Nira Right Bank Cannel (Water Resource Department GoM). To perform any activity on the site, we got prior permission from Chief Secretary Water Resource Department also from Executive Engineering Nira Right Bank Cannel Pune. According to the water flow pattern, we designed and developed blades (called hydro mill) and successfully installed on the site. Rotation of blades due to the flow of water is measured. Practically, we got 14 RPM whereas theoretically it was 15. To generate electricity the alternator requires 1500 RPM, whereas we got 14, so, it is necessary to couple the gearbox with increasing RPM. We designed the gearbox having ratio 1:100 and having sufficient torque run 10KVA alternator. Right now water is available in cannel but, water level is not enough to rotate the blades. (Total depth is 3m, now water is at 2 m level) we have to wait for days to perform experiments.

- Planing to participate and present paper at “3rd Conference on 'Operation & Maintenance and Renovation, Modernizat'ion, Uprating and Life Extension of Hydro Power Plants' - 22-23 August 2017 at New Delhi.”

Project Title: Microwave assisted Halogenation reactions using flow reactor

Project investigator: Prof. M. S. Degani

1. Objectives

Development of nucleophilic aromatic fluorination process for 4-chloronitrobenzene and 2,4-dinitro chlorobenzene using combination of modern technologies namely microwave heating and continuous flow reactor in order to improve energy efficiency, yield, reaction time, aspects of green chemistry.

2. Outcomes

- Reactions were standardized with 4-nitro chlorobenzene and 2,4-dinitro chlorobenzene as substrates.
- Precipitated KF was found to be the optimum fluorinating reagent compared to normal KF and freeze dried KF. 3 equivalents of KF were found to be optimum.
- DMSO was found to be the optimum solvent for the reaction in conventional heating and microwave assisted reactions.
- Reaction required 24h in conventional heating or 1h of intermittent heating with microwave. With flow reactor, due to difficulties in handling solids inside flow reactor these parameters did not work.
- Methanol was explored as solvent with flow reactor. However though the starting material was consumed completely the reaction led to formation methoxy

derivative instead of fluorinated derivative.

- TBAF as fluorinating agent with acetonitrile as solvent in flow reactor led to successful conversion to expected fluorinated product 2, 4 dinitro fluorobenzene.

Other outcomes

The continuous flow reactor set up has been utilized for optimization of various other reactions like synthesis of intermediate of bupropion and in-situ preparation of dimethyl dioxirane for oxidation of organic compounds.

The infrared spectrophotometer has been utilized by number of students for characterization of molecules. Several publications have resulted using this.

1. Lele, A. C.; Raju, A.; Khambete, M. P.; Ray, M. K.; Rajan, M. G. R.; Arkile, M. A.; Jadhav, N. J.; Sarkar, D.; Degani, M. S. Design and Synthesis of a Focused Library of Diamino Triazines as Potential Mycobacterium Tuberculosis DHFR Inhibitors. ACS Med. Chem. Lett. 2015, 6 (11), 1140–1144. DOI: 10.1021/acsmchemlett.5b00367
2. Lele, A. C.; Khambete, M. P.; Raju, A.; Ray, M.; Rajan, M. G. R.; Degani, M. S. Design and Synthesis of Novel Mycobacterium Tuberculosis DHFR Inhibitors. Int. J. Pharma Sci. Res. 2016, 7 (6), 2352–2356.
3. Shelke, R. U., Degani, M. S., Raju, A., Ray, M. K. and Rajan,

M. G. R. (2016), Fragment Discovery for the Design of Nitrogen Heterocycles as Mycobacterium tuberculosis Dihydrofolate Reductase Inhibitors. Arch. Pharm., 349: 602–613. doi: 10.1002/ardp.201600066

3. Publication/ Patents/ Conference related to Project

- A provisional patent has been filed for “An improved process for nucleophilic aromatic fluorination” with number TEMP/E-1/29091/2016-MUM.
- Attended conference Flow Chemistry India 2017 organized by SELECTBIO India on January 18-19, 2017 at Ramada Powai Hotel & Convention Centre in Mumbai under the auspices of the Flow Chemistry Society.

Project Title: Sorption-Enhanced Hydrogen Production

Principal Investigator: Dr. P. D. Vaidya

Objectives:

1. Study of Sorption-enhanced steam reforming (SESR) of model compounds such as methane, ethanol, glycerol and butanol.
2. Study of carbonation and calcinations reactions with cyclic SER and breakthrough curves.

Outcomes till date:

- Several different hybrid materials based on calcium and hydrotalcites were synthesized and employed

for Sorption enhanced reforming studies.

- Sorption-enhanced steam reforming of methane, ethanol, glycerol and butanol was extensively studied using the (catalyst + sorbent) combinations and hybrid materials.
- The influence of reaction variables such as temperature, S/C ratios and sorbent mass fraction on hydrogen production was studied.
- The synthesized hybrid materials were successful in reporting over 90 mol % of H₂ at much lower temperatures (<773 K) and atmospheric pressures.
- The materials remained stable over several cycles of carbonation and regeneration which justifies its large scale applicability.

Facility created under CoE-PI

Air compressor and analytical weighing balance have been procured.

4. Publication/Patents/ Conference related to Project:

International Publications:

1. Dewoolkar K. D. and Vaidya P. D., ChemCatChem, 2016, 8, 3499-3509.
2. Dewoolkar K. D. and Vaidya P. D., Energy Technol. 2017, DOI: 10.1002/ente.201600645
3. Dewoolkar K. D. and Vaidya P. D., Int. J. Hydrogen Energy, 2017, DOI: 10.1016/j.ijhydene.2017.06.235.

Project title: Process Intensification for Extraction of Turmeric and Pepper Oleoresin by Enzyme-Assisted Supercritical Carbon Dioxide

Project investigator: Prof. R. S. Singhal

Objectives:

1. To study the effect of enzyme pretreatment on the extraction time required for Supercritical fluid extraction (SCFE) and to optimize the process for extraction of curcuminoids from Curcuma longa for maximum recovery of product.
2. To study the effect of enzyme pretreatment on the extraction time required for SCFE and to optimize the process for extraction of pepper oleoresin from Piper nigrum for maximum recovery of product.

Outcome till date:

Extensive literature survey was done to understand the basics, working and applications of supercritical fluid extraction technique (SCFE).

Part I

1. Theoretical modeling of extraction parameters was done to predict the possible pressure and temperature combination at which maximum extraction of curcuminoids from rhizomes of Curcuma longa would be obtained. Hildebrandt solubility parameter predicted the pressure of 400 bar and temperature of 40°C.

- Optimization of supercritical fluid extraction conditions like extraction pressure, temperature, time, quality and quantity of co-solvent for maximum extraction of turmeric oleoresin from rhizomes of *Curcuma longa* was done. A pressure of 350 bar at 65°C for 150 minutes using 30% ethanol as a co-solvent gave maximum extraction of oleoresin and curcuminoids with 56.7% and 13.9% relative extraction efficiency compared to Soxhlet extraction with acetone.
- Selection of Stargen® as an enzyme for pretreatment was done and enzymatic activities of Amylase and Glucoamylase were determined using DNSA and GOD-POD assays.
- Enzyme pretreatment studies were done with respect to enzyme load, pH, temperature and time followed by SCFE for enhanced yield of turmeric oleoresin. Using 2% (w/w) Stargen® at pH 3.5 and 30°C for 10 h, gave maximum extraction of oleoresin and curcuminoids with 70.2% and 168.6% relative extraction efficiency.
- Development of HPLC technique for analysis of curcuminoids. The optimized extracts viz. Soxhlet extracted oleoresin, SCF extracted oleoresin and enzyme assisted SCF extracted oleoresin were analyzed using HPLC

for detection of three curcuminoids.

Part 2

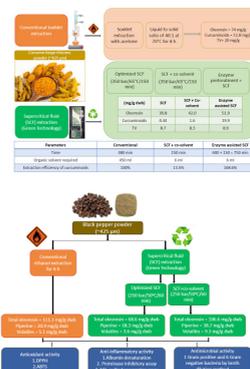
- Theoretical modeling of extraction parameters was done to predict the possible pressure and temperature combination at which maximum extraction of piperine from *Piper nigrum* berries would be obtained. Hildebrandt solubility parameter predicted the pressure of 300 bar and temperature of 40°C.
- Development of HPLC technique for detection of piperine.
- Optimization of extraction pressure, temperature, time and co-solvent for maximum extraction of piperine from *Piper nigrum* berries was done. A pressure of 250 bar at 50°C for 60 minutes using 30% ethanol as a co-solvent gave maximum extraction of oleoresin and piperine with 95.9% and 184.7% relative extraction efficiency compared to solvent extraction with ethanol.
- The piperine rich pepper oleoresin showed significant and comparable antioxidant, anti-inflammatory and anti-microbial activities against the respective standards.

Facility created under CoE-PI: Dionex HPLC Ultima 3000 with photo diode array (PDA), fluorescence and RI detector.

Publication/ Patents/ Conference related to Project: Publication(s) in process.

Graphical abstract:

Part 1



Project Title: Cold Storage Facility for Post-Harvest Preservation of Fruits and Vegetables Using Solar & Biomethane Heat Based Refrigeration.

Project investigator: Prof. S. S. Bhagwat

Objectives :

- To develop a cost effective heat based refrigeration technology for milk chilling.
- To setup an industrial scale demo unit of heat based cold storage facility for post-harvest preservation of fruits and vegetables in rural area.
- Study thermodynamic properties of novel working fluid for Heat based refrigeration system.

Outcome till date :

- The industrial heat based refrigeration technology is successfully implemented at Gokul Dairy, Kolhapur (Borwade Milk Chilling Centre). The technology is combination of Vapour Absorption refrigeration (VAR) system and Vapour

Compression System (VCR) system (Combo VAR-VCR technology). This process operates partially on heat available from briquette fired boiler for milk chilling. It consumes very less electricity in comparison with VCR system. Gokul Dairy has achieved saving of Rs. 7,000/day upto April-2016 which has increased upto Rs. 9,000/day after full implementation of Combo VAR-VCR technology.

- The vapour pressure measurements and other physiochemical properties were determined of novel salt mixture which is non-corrosive and cost effective which can replace commercially available LiBr-Water working fluid pair.

Facility created under CoE-PI

- High Pressure Stirring Autoclave to study thermodynamic properties of novel working fluids for absorption refrigeration system.
- Energy Efficient Air-Cooled Electric Chillers has been purchased and installed in ICT, Oils Building in Computer Laboratory.

Project Title: Open Source Process Simulators for Process Intensification

Project investigator: Dr. V. H. Dalvi

Objectives

- To develop a process which improves the performance of gas power cycles and also

integrates low to medium thermal treatment of an aqueous stream.

- To use a low grade source of energy such as solar energy, biomass or geothermal energy for the thermal treatment of the aqueous stream.
- To use any aqueous stream instead of a highly purified demineralised stream as a source of steam which augments the power output of the gas power cycle thus saving purification cost.
- To compare the performance of the above process with the standard gas power cycles such as the Brayton Cycle and the Cheng's Cycle
- To check the feasibility of using a diesel engine in place of a gas turbine in the above process.
- To develop a novel process simulator by interfacing Python and Microsoft Excel to simulate the above process.

Outcome till Date

- We have created a novel process simulator using both Python and Microsoft Excel to simulate our process.
- We have successfully developed a process which improves the performance of a gas power cycle and integrates low to medium thermal treatment for an aqueous stream for sterilization which can then be sent for additional treatment.

- We have demonstrated a greater performance of the above cycle compared to the Brayton Cycle and Cheng Cycle using seawater as the aqueous stream.
- Achieved improved performance while using a diesel engine in place of the gas turbines in the cycle.
- Reduced the exhaust gas temperature from 700 oC (Brayton Cycle) to 150 oC in the novel process by effectively recovering the waste heat from the exhaust combustion gases.

Publication/ Patents/ Conference related to Project

- A patent has been filed for the above invention of integrating enhanced power generation with low to medium thermal treatment of an aqueous stream.
- We are currently working on a paper to ascertain the feasibility of using the diesel engine variant of the process in a rural area to meet the electricity and water demands on the area.

Project title:

Process intensification in extraction of natural ingredients using novel extraction techniques.

Project investigator: Prof. V.K. Rathod

Introduction:

There is substantial increase in the field of extraction of phytochemicals from various natural sources. Plants are storehouse of secondary

metabolite responsible for various pharmacological activities. Phytochemicals are mostly present in trace amount and are present inside the cell wall of plants. Therefore there is a need to develop and design an experimental method for extraction of biomolecules from various natural sources. In this present context two natural sources are screened for potent bioactive.

1. Fruits of *Aegle marmelos* have numerous uses in Ayurvedic medicine. The unripe fruit is used as an astringent, digestive and stomachic. The unripe fruit has been reported to show antiviral activity and proved to be potent hypoglycaemic agent. It has also been reported with antifungal and anthelmintic activity. Marmelosin also called as imperatorin is a major chemical constituent of the fruit and has been proved to have an anticancer, antibacterial and anti-inflammatory activity.
2. *Lagerstroemia speciosa*, which is also known as the banaba plant has a long history of medical applications including blood pressure support and diabetes and kidney-related diseases. It is also called as pride of India and is native to Southern Asia. The major active component in banaba is corosolic acid and it is responsible to show anti-diabetic, anti-inflammatory, antiobesity activity.

3. *Polyalthia longifolia* commonly called as false ashoka tree is an evergreen tall, handsome plant used as ornamental street tree due to its effectiveness in combating noise pollution. Its leaves are aromatic and mostly used in traditional medicine. Leaves are store house of flavonoids such as quercetin and rutin. Quercetin and rutin is widely known for its immense pharmacological activities such as inflammation, cancer and arthritis.

The traditional solvent extraction technique for extraction suffers from various disadvantages such as large solvent requirement, higher extraction time, lower yield and higher extraction temperature. Hence, there is a need to develop an efficient, robust and economical extraction method. The main advantages of novel techniques include reduced extraction time and reduced solvent consumption with enhancement in yield. Applications of various novel techniques such as microwave assisted extraction, microwave assisted micellar extraction, ultrasound assisted extraction and supercritical CO₂ extraction for the extraction of Marmelosin from *Aegle marmelos*, Corosolic acid from *Lagerstroemia speciosa* and flavonoids from *Polyalthia longifolia* have not been reported in the literature.

Objectives:

The overall objective of the study is as follows:

- To carry out screening of

phytochemicals from various natural sources.

- To develop analysis method for quantification of marker compounds.
- Process intensification using microwave assisted extraction and ultrasound assisted extraction.
- To carry out separation and purification of biomolecules by using various techniques.

Outcome till date:

- Extraction of Corosolic acid from *Lagerstroemia speciosa* by using ultrasound assisted extraction
- Three phase partitioning of Corosolic acid from *Lagerstroemia speciosa*.
- Microwave assisted extraction of Corosolic acid from *Lagerstroemia speciosa*.
- Extraction of Marmelosin from *Aegle marmelos* by using microwave assisted extraction
- Three phase partitioning of Marmelosin from *Aegle marmelos*.
- Extraction of rutin and quercetin from *Polyalthia longifolia* and process optimization by using different extraction technique (In progress).

Result:

Based on the experiment performed and optimization of various parameters following result are obtained as reported in the table.

Corosolic acid from Lagerstroemia speciosa :

Method	Time (min.)	Temperature (°C)	Solvent to solute ratio (ml/g)	Yield (mg/g)
Soxhlet Extraction	300	50	30:1	14.0
TPP	120	30	40:1	9.34
UAE	15	50	40:1	8.93
MAE	1.0	50	20:1	9.68

Marmelosin from Aegle marmelos :

Method	Time (min.)	Temperature (°C)	Solvent to solute ratio (ml/g)	Yield (mg/g)
Soxhlet Extraction	300	50	30:1	0.258
TPP	180	30	50:1	0.343
MAE	01	50	20:1	0.548

- The result indicates that microwave assisted extraction helps to improve yield at lower time, less solvent and energy consumption.

Facility created under COE-PI:

- Laminar Air Flow
- HPLC & Chiral Column

Publication related to project under preparation

- Three phase partitioning as a novel approach for the rapid extraction of Marmelosin (Imperatorin) from Aegle Marmelos fruit and determination of antioxidant activity
- Simultaneous determination of Corosolic acid from Lagerstroemia speciosa by using three phase partitioning
- Rapid determination of Corosolic acid from

lagerstroemia speciosa by using microwave assisted extraction

- Ultrasound assisted extraction of Corosolic acid from lagerstroemia speciosa
- Microwave assisted extraction of Marmelosin from Aegle Marmelos.

Future work plan

- Extraction of Marmelosin from Aegle marmelos by using ultrasound assisted extraction
- Extraction of Corosolic acid from Lagerstroemia speciosa by using three phase partitioning coupled with ultrasound.
- Antidiabetic activity of Marmelosin and Corosolic acid from Aegle marmelos and Lagerstroemia speciosa.
- Extraction of rutin and quercetin from Polyalthia longifolia leaves by using

novel extraction technique.

Project Title:

Design aspects of two-opposed-jet micro-extractor: Experimental and \ Computational Fluid Dynamics

Project investigator: Dr. C. S. Mathpati

Objectives:

- Study of liquid-liquid extraction using impinging jet contactor and comparison.
- To study nature of mass transfer near liquid-liquid interface.

Outcome:

- Liquid-liquid extraction experiments were done in small setup: phenylacetic acid, water and heavy normal paraffin
- Data collected for ANN study from literature to develop empirical correlation for

mass transfer coefficient.

- The nature of mass transfer statistics along with influencing parameters of flow around a rising drop, in the presence of Marangoni instabilities has been studied using simultaneous PIV-PLIF technique. The vorticity field in presence of Marangoni instabilities shows marked differences compared to the case where mass transfer is absent, and shows properties similar to high Reynolds number turbulence.

Deliverables achieved

- The designed pilot plant can be used successfully for liquid-liquid extraction.
- CFD study for rotating disc with impinging jet extractor done in OpenFOAM.
- Energy transfer rates and scalar transfer rate are intimately related to each other, through which we can infer that the mass transfer process near the drop interface is dominated by small scale flow structures. Based on the evaluated energy transfer rates mass transfer coefficient in time has been calculated.

Facility created under CoE-PI

Design Expert software from Statease was purchased for design of experiments. Altair's Hypermesh software was purchased from Design Tech Systems is useful for proper structured meshing.

Publication/Patents/

Conference related to Project:

In Research Journal:

1. Khadamkar H. P., Khanwale M. A., Sawant S. S., Mathpati C. S. (2017). On nature of mass transfer near liquid-liquid interface in the presence of Marangoni instabilities. Chemical Engineering Science, in press.

In Conference:

S. S. Sawant, B. D. Gajbhiye, C. S. Mathpati, Reena Pandit, A. M. Lali, "Microalgae as Sustainable Energy and Its Cultivation", ICMST-2016, will be published in Advanced Science Letters of American Scientist Publisher

Patents: (1) Rotating disc impinging jet contactor (In

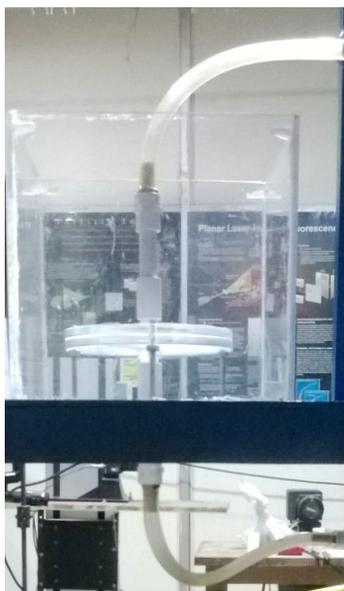


Figure 1. Two opposed jet PIV set-up

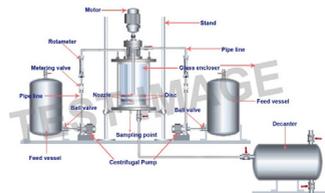


Figure 2: Schematic of two opposed jet micro extractor set-up

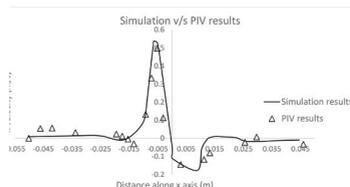


Figure 3. Comparison of simulation and experimental (PIV) results.

Project Title:

Process intensification of crystallization using sonochemical reactors.

Project investigator: Dr. P. R. Gogate

Objectives:

1. Understanding the effect of ultrasound on crystallization process of different commercially important products
2. Optimization of operating parameters
3. Study of antisolvent crystallization and cooling crystallization
4. Evolution of sound scale up strategies for possible industrial exploitation of the sonocrystallization

Highlights of the Important Design related information:

- Use of ultrasound helps in reduction in the average size of crystals as compared to the conventional stirring based approach
- Use of ultrasound intensifies the process by reducing the induction time and also changing the metastable zone width
- Ultrasound also helps in obtaining the correct polymorph identified based on crystal shape
- Crystal size and morphology can be controlled with the application of ultrasound
- Design of pilot scale configuration has been successful and applied for experiments

Research Highlights:

The work compared the efficacy of ultrasonic bath and horn for intensifying the cooling crystallization of oxalic acid for the first time. The effect of various parameters such as temperature, crystallization time, irradiation time, ultrasonic frequency and ultrasonic power on the crystal characteristics has been investigated. The average particle size of oxalic acid crystals reduced with an increase in irradiation time and ultrasonic power dissipation. Comparison of crystal characteristics obtained in ultrasonic bath with the ultrasonic horn under optimized parameters revealed that the average mean size in the case of ultrasonic horn

and bath were only marginally different. The studies related to the effect of different cooling modes using ultrasonic horn revealed that a narrow size distribution and lower average size of 357.2 μm was obtained for the cooling using ice cold water as compared to air cooling where observed mean size was 439.7 μm . A narrow particle size distribution was also obtained in the scale-up studies using ultrasonic horn with mean size of 96.70 μm , which was significantly lower than 161.1 μm obtained under conventional approach. Overall significant process intensification benefits have been established for the ultrasound assisted approach for cooling crystallization of oxalic acid.

The application of low intensity ultrasonic irradiation for improving the cooling crystallization of Mefenamic Acid was also investigated for the first time. The crystal shape and size has been analyzed with the help of optical microscope and image analysis software. The effect of ultrasonic irradiation on crystal size, particle size distribution (PSD) and yield has been investigated, also establishing the comparison with conventional approach. It has been observed that application of ultrasound not only enhances the yield but also reduces the induction time for crystallization as compared to conventional cooling crystallization technique. In the presence of ultrasound, the maximum yield was obtained at optimum conditions of

power dissipation of 30 W and ultrasonic irradiation time of 10 min. The yield was further improved by application of ultrasound in cycles where the formed crystals are allowed to grow in the absence of ultrasonic irradiation. It was also observed that the desired crystal morphology was obtained for the ultrasound assisted crystallization. The conventionally obtained needle shaped crystals transformed into plate shaped crystals for the ultrasound assisted crystallization. The particle size distribution was analyzed using statistical means on the basis of skewness and kurtosis values. It was observed that the skewness and excess kurtosis value for ultrasound assisted crystallization was significantly lower as compared to the conventional approach. The overall process intensification benefits of mefenamic acid crystallization using the ultrasound assisted approach were reduced particle size, increase in the yield and uniform PSD coupled with desired morphology.

Publications/presentations related to the project:

- P. N. Patil, P.R. Gogate, L. Csoka, A. Dregelyi-Kiss, M. Horvath, Intensification of Biogas production using pretreatment based on hydrodynamic cavitation, *Ultrasonics Sonochemistry*, 30, 79-86, 2016
- S. R. Iyer, P. R. Gogate, *Ultrasound Assisted Crystallization of Mefenamic*

Acid: Effect of operating parameters and comparison with conventional approach, *Ultrasonics Sonochemistry*, 34, 896-903, 2017

- R.S. Vishwakarma, P.R. Gogate, Intensified oxalic acid crystallization using ultrasonic reactors: Understanding effect of operating parameters and type of ultrasonic device, *Ultrasonics Sonochemistry*, 39, 111-119, 2017
- A.V. Mohod, P.R. Gogate, Improved crystallization of ammonium sulphate using ultrasound assisted approach with comparison with the conventional approach, *Ultrasonics Sonochemistry*, Forwarded for Publication, 2017

Industry Interactions:

- One Consultancy Project with SRF on exploring application of cavitation for crystallization and waste water treatment
- Couple of projects with Kopran research laboratories and Sigachi industries in the area of application of cavitational reactors for wastewater treatment
- One sponsored project with Unilever on treatment of grey water using cavitation and ozone
- Interactions with invited lectures at Mylan Laboratories in the area of Crystallization
- Interaction with Atul in the area of wastewater treatment

for exploring the application of cavitational reactors

Project Title: Bifunctional catalysts - microwave assisted tandem reactions

Project investigator: Prof. R. V. Jayaram

Objectives:

- To explore the catalytic efficiency of bi-functional catalysts with acidic/ basic and oxidative active sites.
- To improve the activity/selectivity of the catalysts for the reactions chosen by MW irradiation
- To explore a new methodology for industrially relevant products
- To check the reusability of the catalysts

Outcome till date:

A. $\text{MoO}_3\text{-SiO}_2$ bi-functional catalyst:

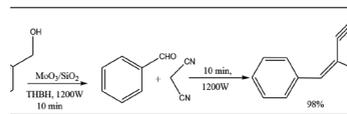
The following catalysts were prepared with various composition of MoO_3

Sr. No.	Mole % of Mo loading on SiO_2	Formula
1	5	$5\% \text{MoO}_3\text{-SiO}_2$
2	10	$10\% \text{MoO}_3\text{-SiO}_2$
3	15	$15\% \text{MoO}_3\text{-SiO}_2$
4	20	$20\% \text{MoO}_3\text{-SiO}_2$

Prepared catalysts were characterized by XRD, EDX, IR,

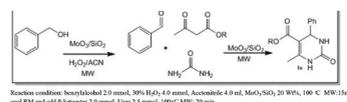
BET, TPD and DSC-TGA

a. Synthesis of α,β -unsaturated amides using $\text{MoO}_3\text{-SiO}_2$ bi-functional catalysts.



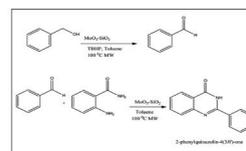
Reaction Condition : Benzyl alcohol 1.00 mmol, TBHP 2.5 mmol, Catalyst 20 Wt%, 100°C after completion of reaction add malanonitrile 1.1 mmol

b. Synthesis of dihydropyrimidinones by using $\text{MoO}_3\text{-SiO}_2$ bifunctional catalysts.



Reaction condition: benzylalcohol 2.0 mmol, 30% H_2O_2 4.0 mmol, Acetonitrile 4.0 ml, $\text{MoO}_3/\text{SiO}_2$ 20 Wt%, 100 °C MW:15min and RNH and add phenylurea 2.0 mmol, Urea 2.5 mmol, 100°C MW: 20 min.

c. Synthesis of 2-phenylquinazolin-4(3H)-one and its derivatives using $\text{MoO}_3\text{-SiO}_2$ catalysts.



Reaction condition: - Benzyl Alcohol 1.0 mmol, TBHP 3.0 mmol, Catalyst 30 mg, Temp: 100 °C, 455 watt after completion of reaction added o-aminobenzamide 1.0 mmol

d. Industrial Application - Preparation of 2-aryl-5,6-dihydropyrimidinone (herbicide) using $\text{MoO}_3\text{-SiO}_2$ bi-functional catalyst

B. Mixed metal oxides as reusable catalysts:

Sr. No.	Formula	% (Wt/Wt) of GdMoO_4
1	$5\% \text{Gd-MoO}_4/\text{ZnO}$	(5%)

2	10%Gd-MoO ₄ /ZnO	(10%)
3	15%Gd-MoO ₄ /ZnO	(15%)
4	20%Gd-MoO ₄ /ZnO	(20%)
5	25%Gd-MoO ₄ /ZnO	(25%)

Characterization of GdMoO₄/ZnO catalysts

a. Synthesis of 2-phenylquinazolin-4(3H)-one and its derivatives using GdMoO₄/ZnO catalysts.

Publication/ Patents/

Conference related to Project :

1. Microwave Assisted Biginelli Reaction using MoO₃-SiO₂ as Bi-Functional Catalysts. R. V. Jayaram*, S. V. Katkar and Priya Singh *Journal: Current catalysis (Communicated)*
2. Microwave assisted synthesis of 4(3H)-Quinazolines using MoO₃-SiO₂ as bi-functional catalysts. R. V. Jayaram*, S. V. Katkar, D. V. Hase and A. Mathakar *Journal: Catalysis Communications (Communicated)*

Project Title:

Enzymatic process Intensification for the manufacturing of structured lipids To enhance the yield

Project investigator: Dr. J. T. Waghmare

INTRODUCTION:

High levels of fats in the diet are associated with many health related risks such as CVD and certain cancers. With growing awareness and widely publicized reports on the detrimental effects of high fatty foods; people have become conscious about what they eat. Unanimously, health communities acknowledged the taxing effects of high fat diet and have set a maximum level of fat consumption. Since more than 30% of total calories in diet come from fat, fat substitutes are becoming new trend in the market which provide lower calories without compromising on taste.

Structured lipids (SL) are defined as triacylglycerols (TAG) restructured or modified to change the fatty acid composition and/or their positional distribution in glycerol molecules by chemical or enzymatic processes. SL may provide the most effective means of delivering desired fatty acids for nutritive or therapeutic purposes. SL can also be synthesized to improve or change the physical and/or chemical characteristics of TAG such as melting point, solid fat contents, iodine, and saponification number.

Structured lipids are constituents of functional foods and are also known as nutraceuticals. An early definition of structured lipids was “novel triglyceride mixtures produced by hydrolysis and interesterification, by traditional chemical methods or by genetic or environmental modification of oil-producing cells”. A later

definition proposed by Casimir Akoh was “triacylglycerols containing mixtures of short-chain or medium chain, or both, and long chain fatty acids, preferably in the same glycerol molecule in order to exhibit their maximum potency”. They are fatty acid based substances having fat like properties but follow different metabolic pathway unlike conventional triglycerides. There are many other terms that have a similar meaning to SL, including structured triacylglycerols/triglycerides, which are also often used. Other terms such as tailor made fats, inter-esterified fats, modified fats, restructured fats, novel triacylglycerols, etc have also appeared in the scientific literature. The terms overlap each other and the range they cover is often unclear or inconsistent from case to case.

SYNTHESIS (REACTIONS & MECHANISM) & PRODUCTION:

The terms used in this regard are as follows:

- **ACIDOLYSIS:** A reaction between an ester, such as a triacylglycerol, and a fatty acid in the presence of an acidic or enzymatic catalyst. The reaction results in an exchange of acyl groups.
- **ALCOHOLYSIS:** A reaction between an ester, such as a triacylglycerol and an alcohol, such as glycerol or methanol, in the presence of catalysts. The reaction results in an exchange of the alcohol moiety.

- **ESTER - ESTER EXCHANGE:** A reaction between an ester, such as triacylglycerol, and another ester, such as another triacylglycerol or an ethyl ester, in the presence of an alkaline catalyst or enzymatic catalyst. The reaction results in an exchange of acyl groups between two esters.
- **ESTERIFICATION:** A reaction between an acid and an alcohol, or between compounds containing carbonyl and hydroxyl groups.
- **INTERESTERIFICATION:** A general term for the reactions between an ester and a fatty acid, an alcohol,

or another ester. It is called as alkaline catalyzed randomization of oils and fats.

- **TRANSESTERIFICATION:** A term often used for ester-ester exchange.

There are two ways to synthesize Structured Lipids:

1. Chemical Interesterification
2. Enzymatic Interesterification

ENZYMATIC VS. CHEMICAL INTERESTERIFICATION

Beside the use of pure chemicals, microbial lipase enzymes are applied as biochemical catalysts in the enzymatic interesterification. In contrast

to the chemical process, the enzymatic process is more selective, as the lipase enzymes may act very selectively in space, i.e they attack only well-defined ester bonds. Enzymatic interesterification is preferred when a well-defined triglyceride composition is required. While on one hand chemical interesterification is a faster route, enzymatic interesterification is a much slower process and is more sensitive to impurities and reaction conditions. Chemical interesterification produces randomly structured lipids and enzymatic interesterification produces specific, region-selective structured lipids.

Chemical interesterification	Enzymatic interesterification
Low processing cost (batch reactor)	High processing cost (continuous plug flow reactor, lipase)
High processing lost (oil saponification)	Minimum processing loss
Low oxidative stability (tocopherol loss)	No change in oxidative stability
High levels of reaction by products (MAG,DAG, glycerol)	Low levels of reaction by products
Flavour reversion problem	No flavor reversion
Highly reproducible and easily controlled	More complex operation and control

CHEMICAL INTERESTERIFICATION:

- a) Ester-Ester interchange can be achieved without catalyst at high temperatures (>200).
- b) In the presence of an acid, alkali or metal, however, the reaction may take place under much milder conditions (20-100 0 C).
- c) The most effective catalysts are the alkali (m) ethylates and sodium/potassium

alloys.

Chemical interesterification is a two stage process. The metal catalyst normally acts as a “pre-catalyst”; the real catalyst being formed in the initial reaction stage. The redistribution of fatty acids can be directed or left to operate at random. In the second case, equilibrium, corresponding to the law of probability, is reached: the fatty acids are redistributed non-selectively

throughout the different esters. Interesterification can also be directed to a certain extent, e.g. by segregation of the newly formed, high melting esters from the reaction mixtures through controlled crystallization. Random Interesterification is commonly used whereas directed Interesterification applies to special cases only.

ENZYMATIC INTERESTERIFICATION:

Enzymatic Interesterification (EIE) is the catalytic reaction that occurs when an enzyme is introduced into oil and rearranges the fatty acids on the glycerol backbone of a triglyceride. Triglycerides are either liquid or solid at room temperature. The rearrangement of the fatty acids that occurs with enzymatic interesterification provides structure and functionality to triglycerides at room temperature. This process adjusts the melting properties, increasing functionality and plasticity in food production applications. One of five different ways of altering melting property profiles, enzymatic interesterification is unlike the more widely used partial hydrogenation method in that it produces no trans fatty acids and lowers saturated fat content. The reaction between the oils and the enzyme causes the fatty acids of the triglycerides to exchange positions to the -1 and -3 glycerides, thereby altering the chemical composition and physical properties of the fat. Using a 1, 3-specific enzyme, only the fatty acids in the 1,3 positions are shifted around, and the 2-position is untouched. Unsaturated fatty acids in the 2-position are more often found naturally in plant lipids or vegetable oil sources. Allowing the rearrangement of the saturated fatty acids to occur in the 1-position and 3-position increases the amount of bioavailable unsaturated fatty acids.

LIPASES

Lipases, or TAG hydrolases are heterogeneous class of enzymes, which may be obtained from microbial, animal or plant sources. Lipases are known for their physiological function in the digestion and absorption of lipids. These properties are now used in various biotechnological applications. Unlike chemical synthesis, enzymatic catalysis does not fully require fully refined and anhydrous oils and the environmental impact of the enzymatic process is less severe. Chemical catalysis often requires additional purification steps and higher temperatures, which consumes more energy. Depending on the reaction system, lipases are able to catalyze not only hydrolysis, but also synthesis reactions such as esterifications, acidolysis or alcoholysis. However, the greatest advantage of lipases resides in their chemo-, region-, and also stereoselectivity, which makes them attractive for use in academia and industry.

The lipase enzymes used today in enzymatic interesterification can be classified into three groups.

- NON - SPECIFIC LIPASES:** These catalyze the decomposition of the glycerides into fatty acids and glycerol with the formation of mono- and diglycerides as a by-product. In the absence of water, the reaction becomes a random interesterification.
- 1, 3 SPECIFIC LIPASES:** These activate the liberation

of fatty acids in the 1st and the 3rd position, and replace them by other fatty acids. Examples are *Aspergillus Niger*, *Mucor Javanicus* and a certain members of *Rhizopus* family.

- FATTY ACID SPECIFIC LIPASES:** They have a preference for particular fatty acid. For example, *Geotrichum candidum* is specific for the hydrolysis of glycerides containing some particular fatty acids (i.e. long chain fatty acids with a double cis bond at 9th position).
- 1, 3 Specific Lipases are generally applied. The reaction in a 1,3 specific enzymatic interesterification can be represented as follows:

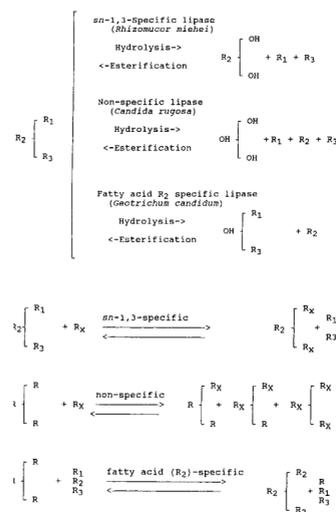


Figure 15 Specificity of triacylglycerol lipases in interesterification and transesterification
1., R₁, R₂, R₃, fatty acid/acyl moieties.

Figure 1 specificity of triacylglycerol lipases in interesterification and transesterification: R₁,R₂,R₃, fatty acids/acyl moieties

As lipases act in aqueous media, the hydrolysis reaction of the triglyceride substrates-which are insoluble in water-occurs at the lipid/water interface. Most lipases are stable in organic media and they can also catalyze acyl transfer reactions. Active sites of lipases contain three amino acid residues, namely histidine, serine and aspartic acid. The catalytic triad is present in all lipases except that aspartic acid can sometimes be replaced by glutamic acid as in the case of lipase from *Geotrichum candidum*. As lipases operate at oil/water interface, they have to first modify their structure for the substrate to reach the active site. 3-D structure of various lipases revealed the existence of a flap (lid) covering the active site. The phenomenon of interfacial activation corresponds to the opening of this flap when the lipase binds to the interface. This change of structure provides free access to the catalytic triad that was previously buried within the 3-D structure of the enzyme.

SELECTIVITY:

Selectivity is the one of the most important properties of the lipases. It can be defined as comparative differences in rates of certain reactions. Lipases may exhibit one or several types of selectivity corresponding to a preferential action from a kinetic point of view. Substrate selectivity is due to the chemical nature of the substrate with some lipases preferring to hydrolyze a certain type of acylglycerol. For example, TAGs are most preferred substrate for pancreatic lipases, which

hardly attack the MAGs. Stereoselectivity of a lipase is its ability to preferentially hydrolyze either the sn-1 or sn-3 position of TAGs.

FACTORS AFFECTING LIPASE ACTIVITY:

1) pH and TEMPERATURE:

While lipases of fungal origin usually have an optimum pH in the neutral or slightly acidic range, lipases produced by bacteria exhibit maximum activity with a neutral or slightly alkaline pH. Lipases usually operate in mild conditions of temperature and pressure. Their optimum temperature is often between 30-50°C with microbial lipases being more thermally stable than plant or animal lipases. However, some enzymes are active at extreme temperature, e.g, the lipase from *Antrodia cinnamomea* shows maximum activity at 45°C and it remains active upto 80°C. Also immobilized enzymes from *Candida Antarctica B* are highly stable and can be used for months under process conditions at temperature of 60-70°C.

2) THERMODYNAMIC WATER ACTIVITY:

The use of lipases as biocatalysts for organic synthesis requires the presence of a certain amount of water in order to maintain the lipase in an active 3-D structure resulting from an interfacial activation mechanism. Indeed, a

minimum hydration of the enzyme is essential to make the active site assessable to the substrate and thus ensure its lipolytic activity. In order to catalyze the acyl transfer reactions, it is therefore necessary to work in a limited aqueous phase so as to ensure active structure while limiting the reverse hydrolysis reaction. The equilibrium shift can be achieved by eliminating one of the reaction products through evaporation, crystallization, specific solvent extraction, adsorption or distillation. Thus aqueous microenvironment is an important control parameter in enzymatic synthesis and is governed by the thermodynamic water activity (a_w). By definition it is the ratio of the vapor pressure of water in gas phase over the liquid phase. Thus, this parameter reflects the amount of unbound or free water available as substrate. Water activity determines the performance of synthesis, namely its yield, selectivity and stability. By studying water activity, one can define the optimum conditions of hydration necessary for maximum enzyme activity in acyl transfer catalysis while limiting competitive hydrolysis. Sorption isotherms of the enzyme, representing its water content plotted against water activity at constant temperature can be studied to determine the moisture

content corresponding to optimum aw. For most lipases, water activity is generally between 0.25 and 0.45, which corresponds to moisture contents between 0.5% and 1%.

3) IMMOBILIZATION:

Enzyme immobilization is the process of attaching enzymes to appropriate support materials by inclusion, adsorption, or covalent bonding. Immobilization process aims at stabilizing lipases and at reducing their cost per use in order to make enzymatic process industrially viable. Enzymatic lipases which are immobilized can be easily separated from reaction mixtures via simple physical processes like filtration.

4) EFFECT OF SOLVENT:

Factors needed to be taken into account when choosing a proper solvent for a given enzymatic reaction:

- Solvent density and Viscosity.
- Surface tension.
- Toxicity.
- Flammability.
- Cost.

The Polarity of solvent affects the lipase-catalyzed reactions.

Organic solvents such as n-hexane play key roles, including and increase in the solubility of non-polar substrates and the shifting of the reaction towards ester synthesis rather than hydrolysis. Lipases tend to be more active in n-hexane

than in other solvents such as iso-octane, acetone, petroleum ether, toluene and ethyl acetate.

In solvent free systems, suitable for food products, the absence of solvent facilitates downstream processing as the elimination of solvent offers significant costs saving and high product purity. Solvents however are more useful in high viscous substrates where poor mixing may result in slower reactions. Here solvents act to promote mixing by reducing the viscosity of reaction mixture.

Taking into consideration the toxicity and storage & handling problems of solvents, Ionic Liquids are the new trend in this aspect. They can be made as per required polarity and perform similar functions as organic solvents with comparable efficacy. An added advantage of Ionic liquids is that they are eco-friendly and less toxic than organic solvents.

PRODUCTION & PROCESSING

• ONE-STEP PROCESS:

Interesterification carried out of acidolysis of TAG with fatty acid or by transesterification between two TAGs using 1, 3-regiospecific lipases. Theoretically 66.66% conversion can be achieved but usually <40% incorporation can be achieved in single step process. To improve one-step enzymatic process operational stability of enzymes is improved by immobilization.

• MULTI-STEP PROCESS:

Although structured lipids can be synthesized in a single step process using 1, 3 specific lipases, it is extremely difficult to convert all formed intermediate DAG into desired TAGs. Furthermore, many purification steps required for removing by-products make its industrial application less attractive. The best known two-step approach is as follows:

- (1) Alcoholysis of neutral oil/ fat using specific 1,3 lipases, yielding 2-MAG.
- (2) Purified 2-MAG esterified with two equivalents of FA into desired TAG of high yield and purity. The use of alcoholysis instead of acidolysis allows avoiding acyl migration.

LITERATURE SURVEY:

Mounika.C and Yella.R prepared palm olein enriched with medium chain fatty acids by lipase acidolysis. Objective of his work was to incorporate medium chain fatty acids, caprylic and capric acids to palm olein by 1,3-specific lipase acidolysis and thus improve its nutritional quality and widen its food and other applications. Enzyme used by him was Lipozyme RMIM and Pancreatic lipase. Fatty acid used were Caproic acid (C6:0), caprylic acid (C8:0) and capric acid (C10:0)

Conclusion:

Nutritional quality of palm olein was improved by reducing palmitic acid and enriching with medium chain fatty acids and also widened its applications in

foods pharmaceuticals.

P.A. Nunes et al Produced olive oil enriched with medium chain fatty acids catalysed by commercial immobilised lipases.

Objective: is the selection of the best system type and biocatalyst for the production of MLM, by acidolysis of virgin olive oil with caprylic or capric acids.

Enzyme: *T. lanuginosa* (Lipozyme TL IM), *R. miehei* (Lipozyme RM IM) and *C. antarctica* (Novozym 435) and Porcine pancreatic lipase

Fatty acid: Caprylic acid (C8:0), Capric acid (C10:0)

Conclusion:

- Lipozyme RI IM, and Novozym 435, were the biocatalysts that presented the highest operational stability, together with high Incorporation levels and low acyl migration for the production of structured lipids by acidolysis of caprylic or capric acid with virgin olive oil, in batch mode.
- Therefore, these biocatalysts seem to be the most adequate for process implementation aimed at the production of MLM rich in caprylic and capric acids.

E. Hita and A. Robles Produced structured triacylglycerols (STAG) rich in docosahexaenoic acid (DHA) in position 2 by acidolysis of tuna oil catalyzed by lipases

Enzyme: *Rhizopus oryzae* (Lipase D) and *Rhizopus delemar* (Lipase Rd),

Fatty acid: caprylic acid (CA) and docosahexaenoic acid (DHA)

MacKenzie AD, Stevenson DE Produced high-oleic acid tallow fractions using lipase-catalyzed directed interesterification, using both batch and continuous processing.

- Immobilized lipases were used to catalyze batch-directed interesterification of tallow, resulting in oleins containing significantly higher levels of unsaturated fatty acids than obtained by fractionation without lipase.
- This method may form the basis for a process to produce highly mono-unsaturated tallow fractions for use in food applications
- Enzyme : Novozym 435 and Lipolaset
- Fatty acid : lightly fractionated tallow product.
- Conclusion
- Lipase-catalyzed directed batch interesterification of tallow was an effective means to increase the separation between saturated and unsaturated fatty acids compared with simple fractionation.
- The presence of immobilized lipase consistently produced more saturated stearins and less saturated oleins than no-enzyme controls under equivalent conditions.

- The process was affected detrimentally by any addition of water to the reaction medium; FFA levels increased considerably, but there was little or no benefit from increased enzyme activity.

- The main drawback with the batch approach is that crystallization and interesterification occur in the same vessel.

- The continuous reactor used in this study had considerable advantages when used in directed interesterification of tallow, compared with a batch process.

A. Robles et al Synthesized of structured lipids by two enzymatic steps: Ethanolysis of fish oils and esterification of 2-monoacylglycerols

Enzyme: Novozym 435 (N-435) from *C. antarctica*, Lipozyme RM,IM from *Mucor miehei*, lipases D and DF from *R. oryzae*

Fatty acid: Cod liver oil and tuna oil

Gabriella H. et al Integrated enzymatic production of specific structured lipid and phytosterol ester compositions

Enzyme: *Rhizopus oryzae* (Lipase D) and *Rhizopus delemar* (Lipase Rd), *Rhizomucor miehei*

Fatty acid: Sunflower oil and caprylic acid (CA)

EXPERIMENTAL WORK:

CHEMICAL ESTERIFICATION

Batch no.	Fatty acid	Alcohol	Catalyst	Solvent	Stirring rate (rpm)	Pressure	Temperature (°C)	Batch size	Acid value
1	oleic	Glycerol	Nil	Nil	380	Atm. pressure	120	300gm	74.2175
2	Oleic	Glycerol	Nil	Nil	380	Atm. pressure	140	300gm	70.8569
3	Oleic	Glycerol	Nil	Nil	380	Atm. pressure	160	300gm	71.987
4	Oleic	Glycerol	Sulphuric acid	Nil	380	Under vacuum	90	300gm	65.287
5	Oleic	Glycerol	Sulphuric acid	Nil	380	Under vacuum	110	300gm	75.847
6	Oleic	Glycerol	Sulphuric acid	Nil	380	Under vacuum	125	300gm	72.587
7	Oleic	Glycerol	Sulphuric acid	Toluene	380	Atm. pressure (nitrogen applied)	120	300gm	23.060
8	Oleic	Glycerol	Sulphuric acid	Toluene	380	Atm. pressure (nitrogen applied)	140	300gm	22.568
9	Oleic	Glycerol	Sulphuric acid	Toluene	380	Atm. pressure (nitrogen applied)	160	300gm	22.280
10	Capric	Glycerol	Sulphuric acid	Toluene	380	Atm. pressure (nitrogen applied)	120	300gm	15.634
11	Capric	Glycerol	Sulphuric acid	Toluene	380	Atm. pressure (nitrogen applied)	140	300gm	17.968
12	Capric	Glycerol	Sulphuric acid	Toluene	380	Atm. pressure (nitrogen applied)	160	300 gm	17.562

ENZYMATIC ESTERIFICATION

1. ENZYME- LIPOZYME RM (RHIZOMUCOR MIEHEI)

A. Effect of various solvents on enzymatic glycerolysis of capric acid and caprylic acid

Batch no.	Fatty acid	Alcohol	Enzyme	Solvent	Stirring rate (rpm)	Temperature (°C)	Acid value	Peroxide value
01	Caprylic	Glycerol	Lipozyme RM	Nil	150	50	262.1	3.92
02	Caprylic	Glycerol	Lipozyme RM	Nil	150	60	249.3	3.12
03	Capric	Glycerol	Lipozyme RM	Hexane	150	37	101.8	1.98
04	Capric	Glycerol	Lipozyme RM	Hexane	150	45	100.9	1.67
05	Capric	Glycerol	Lipozyme RM	Isooctane	150	37	117	2.12
06	Capric	Glycerol	Lipozyme RM	Isooctane	150	45	112	2.01
07	Capric	Glycerol	Lipozyme RM	Isooctane	150	50	186.4	1.24
08	Capric	Glycerol	Lipozyme RM	Isooctane	150	60	167.74	0.849
09	Capric + Caprylic	Glycerol	Lipozyme RM	Isooctane	150	85	101.56	2.31
10	Capric + Caprylic	Glycerol	Lipozyme RM	Isooctane	150	100	95.67	1.71
11	Caprylic	Glycerol	Lipozyme RM	Hexane	150	37	126.32	2.10
12	Caprylic	Glycerol	Lipozyme RM	hexane	150	45	134.87	2.80

B. Effect of various molar ratios of capric acid and glycerol on enzymatic glycerolysis of capric acid

Sr. no	Molar ratio (capric acid: glycerol)	Enzyme	Temperature (°C)	Acid value
01	1:1	Lipozyme RM	60-70	30.63
02	2:1	Lipozyme RM	60-70	36.82
03	3:1	Lipozyme RM	60-70	58.26
04	4:1	Lipozyme RM	60-70	75.18
05	3:1	Lipozyme RM	60-70	20.26
06	4:1	Lipozyme RM	60-70	32.80

C. Effect of temperature on enzymatic glycerolysis of capric acid

Sr. no	Enzyme	Temperature (°C)	Acid value
01	Lipozyme RM	50-54	68.46
02	Lipozyme RM	55-59	62.86
03	Lipozyme RM	60-64	58.35
04	Lipozyme RM	65-70	55.23

D. Effect of enzyme loading on enzymatic glycerolysis of capric acid

Sr. no	Enzyme loading (%)	Temperature (°C)	Acid value
01	4	65-70	55.67
02	6	65-70	57.39
03	8	65-70	58.93

2. ENZYME- LIPASE PS (PSEUDOMONAS AERUGINOSA)

A. Effect of various solvents on enzymatic glycerolysis of capric acid and caprylic acid

Batch no.	Fatty acid	Alcohol	Enzyme	Solvent	Stirring rate (rpm)	Temperature (°C)	Acid value	Peroxide value
01	Caprylic	Glycerol	Lipase PS	Nil	150	70	262.1	5.12
02	Caprylic	Glycerol	Lipase PS	Hexane	150	70	249.3	4.97
03	Caprylic	Glycerol	Lipase PS	Isooctane	150	70	101.8	5.58
04	Capric	Glycerol	Lipase PS	Nil	150	70	100.9	3.86
05	Capric	Glycerol	Lipase PS	Hexane	150	70	117	3.42
06	Capric	Glycerol	Lipase PS	Isooctane	150	70	134.87	4.67

B. Effect of various molar ratios of capric acid and glycerol on enzymatic glycerolysis of capric acid

Sr. no	Molar ratio (capric acid: glycerol)	Enzyme	Temperature (°C)	Acid value
01	1:1	Lipase PS	60-70	120.39
02	2:1	Lipase PS	60-70	135.82
03	3:1	Lipase PS	60-70	142.21
04	4:1	Lipase PS	60-70	168.95
05	3:1	Lipase PS	60-70	102.85
06	4:1	Lipase PS	60-70	124.1

C. Effect of temperature on enzymatic glycerolysis of capric acid

Sr. no	Enzyme	Temperature (°C)	Acid value
01	Lipase PS	50-54	179.6
02	Lipase PS	55-59	175.9
03	Lipase PS	60-64	163.52
04	Lipase PS	65-70	161.29

D. Effect of enzyme loading on enzymatic glycerolysis of capric acid

Sr. no	Enzyme loading (%)	Temperature (°C)	Acid value
01	4	65-70	165.27
02	6	65-70	173.81
03	8	65-70	170.73

DISCUSSION

Chemical esterification

We performed 12 batches of chemical esterification. Out of 12 batches in first 9 batches Oleic acid was used as a fatty acid and in remaining 3 batches capric acid used. The aim of the using Oleic acid first was to optimize the reaction parameters. The batches conducted at atmospheric pressure as well as under vacuum with varying temperature gave product of high acid value.

Enzymatic esterification

Two enzyme were used namely Lipozyme RM and Lipase PS. Different reaction parameters were optimized in order to get the maximum yield of MCT. The different reaction parameters optimized were the various solvents, molar ratio of capric acid and glycerol, temperature and percentage of enzyme loading.

1. ENZYME: LIPOZYME RM

A. Effect of various solvents on enzymatic glycerolysis of capric acid and caprylic acid

It is seen that a good yield of triglyceride synthesis can be achieved with immobilized lipase with organic solvents. The products were analysed by GC-MS. When the sample was analyzed different peaks were obtained for different components like unreacted

glycerol, capric /caprylic acid, monoglycerides, diglycerides and triglycerides.

B. Effect of various molar ratios of capric acid and glycerol on enzymatic glycerolysis of capric acid

While studying the effect molar ratio of capric acid: glycerol the moles of capric acid changed from 1 to 4 moles keeping the amount of glycerol constant. As the molar ratio of capric acid: glycerol increased from 1:3 to 1:4, the proportion of TG was also increased, but the amount of un-reacted acid was also increased. Therefore the molar ratio of 1:3 was fixed for investigation of the effects of the other parameters on MCT synthesis.

As the molar ratio was increased from 1:1 to 1:4, the fatty acids were left out due to the variation in the proportions of the fatty acid used in the esterification reaction. This indicates that there is no economic advantage in using high substrate molar ratios.

The effect of increased molar content of glycerol on the yield of MCT keeping the moles of Capric acid was also studied. But since the yield of MCT was very low in this case, these results were not considered for MCT yield.

C. Effect of temperature on

enzymatic glycerolysis of capric acid

As the temperature was increased from 50°C to 70°C the yield of MCT also increased. hence for further studies 65-70°C temperature was fixed at which the maximum yield of MCT.

D. Effect of enzyme loading on enzymatic glycerolysis of capric acid

This study was performed by varying enzyme load from 4% to 8%. At 6% enzyme loading highest yield of MCT was obtained. Hence, in order to minimize the overall cost of the process an enzyme load of 6% was sufficient.

2. ENZYME: LIPASE PS

A. Effect of various solvents on enzymatic glycerolysis of capric acid and caprylic acid

Product obtained by Glycerolysis of capric and caprylic acid using enzyme lipase PS had higher acid value as compared to those obtained by enzyme lipozyme RM. Moreover yield of the product was almost similar to that of enzyme lipozyme RM.

B. Effect of various molar ratios of capric acid and glycerol on enzymatic glycerolysis of capric acid

By varying moles of capric acid and keeping glycerol constant, acid value of final product was increased. As moles of

capric acid changed from 1 to 4 moles keeping the amount of glycerol constant the acid value increased from 120.39 to 142.21. As the molar ratio of capric acid: glycerol increased from 1:3 to 1:4, the proportion of TG was also increased, but the amount of unreacted acid was also increased.

As the molar ratio was increased from 1:1 to 1:4, the fatty acids were left out due to the variation in the proportions of the fatty acid used in the esterification reaction. This indicates that there is no economic advantage in using high substrate molar ratios.

The effect of increased molar content of glycerol on the yield of MCT keeping the moles of Capric acid was also studied. But since the yield of MCT was very low in this case, these results were not considered for MCT yield.

C. Effect of temperature on enzymatic glycerolysis of capric acid

No significant effect of temperature was seen on glycerolysis of capric acid.

D. Effect of enzyme loading on enzymatic glycerolysis of capric acid

This study was performed by varying enzyme load from 4% to 8%. At 8% enzyme loading highest yield of MCT was obtained. Hence, in order to minimize the overall cost of the process an enzyme load of 8% was sufficient.

CONCLUSION:

Compared to enzymatic process chemical esterification is efficient process in terms of acid value

and yield of final product. But when it comes to disadvantage chemical esterification leads to a extremely dark coloured product at higher cost. It is not energy intensive process.

Enzymatic esterification is energy intensive process as it requires less energy but acid value of final product is extremely high as compared to chemical product.

Two different enzymes were used and both of them gave product with totally higher acid value than those obtained by chemical method.

FUTURE WORK:

- Same batches will be carried out by using Sonication technique and results will be observed.
- Same batches will be carried out by replacing the enzyme lipozyme RM with Candida antarctica Novozyme 435.
- Studying the impact of enzyme mixture on product formation.
- Enzymatic Interesterification of MCT oil.
- Applications of MCT oil.

Project Title:

PROCESS DEVELOPMENT OF NANO STRUCTURE METAL OXIDES BY SONOCHEMICAL METHOD

Project investigator: Dr. S. T. Mhaske

Synthesis of nanostructured Titanium Dioxide and effect of reaction parameters.

Titanium dioxide (TiO_2) is an inorganic metal oxide which is widely used in the Surface Coating and the Polymer industry as a pigment and a filler.

It is also noteworthy used as an additive in sunscreen and food coloring. TiO_2 mainly exists in two crystalline polymorphs viz.; Anatase and Rutile. In the anatase form, particularly, TiO_2 is a photocatalyst under ultraviolet (UV) light, generating radical species that cause degradation of surface which, it is in contact. If it is used as a pigment or filler in exterior application, the materials can be degraded, which ultimately result in a marked reduction in the material's properties. An effort was made to synthesize nanostructured TiO_2 via sol-gel technique to obtain a 100% rutile polymorph of nanostructured TiO_2 . The sol-gel synthesis technique was suitably modified by incorporating ultrasound to study the effect of cavitation on the phase transformation, particle size, crystallinity and morphological (scanning electron microscopy) properties of the obtained nano- TiO_2 .

Synthesis of nanostructured Zirconium Dioxide and effect of reaction parameters.

Abstract:

Zirconia materials owing to their properties are of widespread application. Pure zirconia in equilibrium state exists in three polymorphic form: monoclinic below $\sim 1170^\circ\text{C}$, tetragonal in the temperature range $\sim 1170\text{-}2370^\circ\text{C}$ and cubic above $\sim 2370^\circ\text{C}$. The properties of zirconia forms of higher symmetry are very often preferable to monoclinic one. Yttrium oxide is usually used as the stabilization component of higher symmetry zirconia.

The increasing interest in the nanotechnology of zirconia materials is noteworthy.

Conclusions:

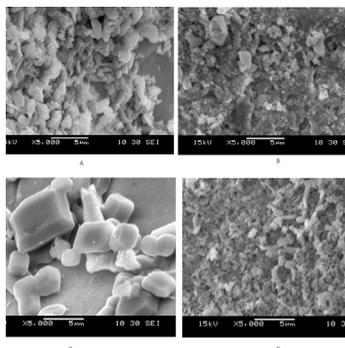
1. Zirconium dioxide was synthesized by both the conventional and the ultrasound assisted precipitation synthesis.
2. Various parameters of the synthesis were studied and an optimum parameter window with respect to the synthesis of Tetragonal ZrO_2 was established.
3. From the results received, the optimum parameter values were:
 - a. Calcination Temperature of $500^\circ C$
 - b. Ultrasonic amplitude of 40% corresponding to power input of 27.11 W

In-situ synthesis of CeO_2/ZnO composite nanoparticles and its application in degradation of Rhodamine B using sonocatalytic & photocatalytic method

Abstract:

Individual CeO_2 , ZnO and bimetal oxide core shell CeO_2/ZnO nanoparticles were synthesized without any stabilizers using in-situ precipitation technique. The

structure, morphology and particle size of the synthesized nanoparticles were analyzed by using X-ray powder diffraction, scanning electron microscopy and Transmission electron microscopy. Results reveal that the phase structures of CeO_2/ZnO are composed by cubic (fluorite) phase of CeO_2 and hexagonal (wurtzite) phase of ZnO . Structure characterization of core shell particles by Transmission electron microscopy indicates that the ZnO shell is around 5 nm in thickness and CeO_2 core is 9 nm in diameter. The effectiveness of the synthesized CeO_2/ZnO used as catalysts for the photocatalytic well as sonocatalytic degradation of Rhodamine B dye has also been investigated. The results showed that Photocatalysis was more efficient than sonocatalysis in degradation of Rhodamine B.



SEM images of NUS and US synthesized specimens. A: NUS 600 at 5000X, B: NUS 700 at 5000X, C: US 500 at 5000X, D: US 700 at 5000X.

Project Name: MICROWAVE ASSISTED / ENZYMES MEDIATED EXTRACTION/ SYNTHESIS OF BIOACTIVE COLORANTS INDIGOID / AZULENE / C-C BOND FORMATION.

Project investigator: Prof. N. Sekar

Research Objective:

Process intensification and scaling up the reaction yields of Diketo-pyrrolo-pyrrole (DPP) and indigo/ Microwave Extraction of essential oil from bottle brush leaves and black cardamom

Outcome till date

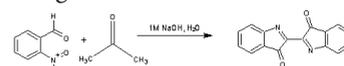
Synthesis Part

Research Objective:

Process intensification and scaling up the reaction yields of Diketo-pyrrolo-pyrrole (DPP) and indigo

Detailed Progress report:

Reaction No: 1- Synthesis of Indigo



Chemical Table:

Sr No.	Chemical/Reagent	Mol wt. g/mol	mMoles	Wt/ml	
1)	2-nitrobenzaldehyde	151.12	3.3mmole	0.5	
2)	NaOH	40		2.5ml	1M
3)	Acetone	58.08		5ml	
4)	Dist. Water	18		5ml	

Ref: doi: 10.1021/ed084p1004; J. Chem. Educ., 2007, 84 (6), p 1004

Procedure:

- One equivalent of o-nitrobenzaldehyde was dissolved in 5ml of acetone and 5ml of water.
- Slowly dropwise 2.5 ml of 1M NaOH was added with stirring
- Within few seconds of NaOH addition blue coloration indicating the formation of indigo was observed
- The addition was continued till the precipitate formation seized.
- The precipitate was neutralized with dil HCl acid and washed several times with water to remove base; filtered on suction pump.
- The product was dried in oven for 3 hrs at 110 oc and was weighted.

Observation:

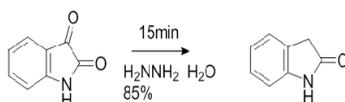
Weight of the product =0.090mg

Theoretical yield = 0.249g

Therefore % yield = $[(0.09/0.249) \times 100]$
=36%

Results and Conclusion:

- The required dye was synthesized as per published literature and the yield was too in the expected practical yield reported in literature.
- melting point of the compd being very high >300 oc hence the product needs to be further characterized luike UV in DMF (lit value=410 nm for indigo in DMF solvent)

Reaction No: 2- Synthesis of indoline -2, 3-one**Procedure:**

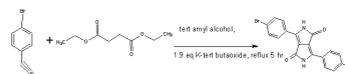
- The isatin was dissolved in

98% of hydrazine solution and stirred at room temperature for 30minutes. Poured in cold water to give precipitate.

- The reaction was worked up and extracted in ethyl acetate.
- The compound was recrystallised from ethyl acetate.

Observation:

The TLC of the recrystallised compound was done which showed a single spot

Reaction No: 3- Synthesis of DPP**Chemical Table:**

Sr No.	Chemical/Reagent	Mol wt. g/mol	mMoles	Wt/ml	Eq. wt.
1)	4-Bromobenzonitrile	182.02	3.7	0.6	1
2)	Diethyl succinate	174.99 d=0.805g/cc			1.4
3)	Pota- tert-butaoxide				1.9
4)	2-methyl 2-butanol			2ml	

Procedure:

- Under the positive pressure of nitrogen 4-bromo benzonitrile, and potassium tert butaoxide was taken in a two neck round bottom flask fitted with reflux condenser.
- At 20-25 oc Diethyl succinate was added using dropping funnel in portions.
- The reaction was allowed to come to room temperature and heated to 100 oc for 2hrs, further was refluxed at 120 oc
- After the reaction is completed it is allowed to cool down and monitored by TLC (ethyl acetate + hexane12%)
- The reaction was monitored by TLC every hr to check the product formation.
- Acid work up was done and the product was portioned between MDC (Methylen dichloride) and water dried over sodium sulfate

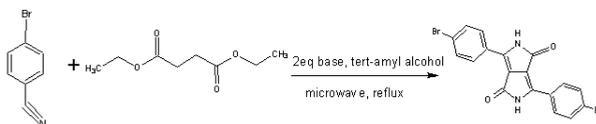
Observation:

TLC No.	Reaction time (Hrs)	Colour of reaction	Temperature oc	spots on TLC
1	2	reddish	100	Fluorescent spot in reaction mix along with Sm
2	4	reddish	120	Fluorescent spot in reaction mix along with Sm
3	5	Red-brown	120	Fluorescent spot in reaction mix along with Sm

(SM-starting Material)

Results and Conclusion:

1. A fluorescent product was obtained at the end of the reaction, but no further purification or characterization was done.

Reaction No: 4- Microwave Assisted synthesis of DPP using organic bases**Chemical Table:**

Sr No.	Chemical/Reagent	Mol wt. g/mol	mMoles	Wt/ml	Eq. wt.
1)	4-Bromobenzonitrile	182.02	1.09	0.200 mg	1
2)	Diethyl succinate	174.99	1.526	0.254 ml	1.4
3)	Piperidine	85.5	2.18	0.215 ml	2
4)	2-methyl 2-butanol	88.15	-	5 ml	-

Procedure

1. 4-bromo benzonitrile, diethyl succinate, piperidine are added and mixed well
2. The reaction mixture is kept in the microwave for 30 seconds at 160 watt power, temp was set at 90oc while the stages were set at 1
3. After the reaction is completed it is allowed to cool down and monitored by TLC (ethyl acetate + hexane12%)

Observation:

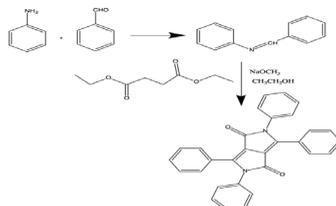
Reaction no	Base	Reaction time	Colour of reaction	MW Energy (watts)	spots on TLC
1	piperidine	30sec	colourless	160	No spot apart from SM
2	piperidine	60sec	colourless	160	No spot apart from SM
3	piperidine	300sec	colourless	240	No spot apart from SM

(SM-starting Material)

Results and Conclusion:

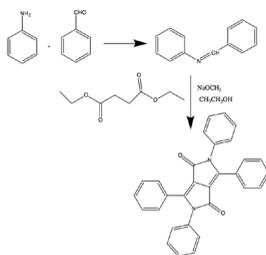
2. No color change is observed in the reaction
3. No spot in the reaction mixture was obtained other than those for reactants on TLC in UV light
4. Hence the reaction needs to be done at still high energy (>240watts) and for longer time(>5mins)
5. Similar reaction needs to be performed using stronger bases like diisopropylamine under similar conditions

Reaction No: 5- Microwave Assisted synthesis of N-phenyl substituted DPP



Work Up of reaction: Filtration, Drying and purification of Schiff Base was done and dark yellow liquid was synthesized.

Reaction No 6:- Microwave Assisted synthesis of N-phenyl substituted DPP



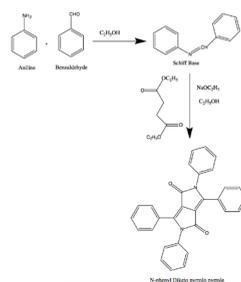
Chemical Table:

Sr No.	Chemical/ Reagent	Mol wt. g/mol	mMoles	Wt/ml	Eq. wt.
1)	Aniline	93.13	21.48	2 ml	1
2)	Benzaldehyde	106.121	21.48	2.2 ml	1
3)	Schiff Base				
4)	Diethyl succinate	174.99			
5)	Sodium methoxide	54.02			
6)	Ethanol	46.06	-	10 ml	-

Procedure:

1. Aniline and Benzaldehyde is mixed in methanol and stirred for few minutes
2. After the reaction is completed it is monitored by TLC (ethyl acetate + hexane25%)
3. Starting material is observed on the TLC
4. Reaction Mixture is kept under reflux for proper mixing and formation of Schiff Base

Reaction No 7: - Microwave Assisted synthesis of N-phenyl substituted DPP



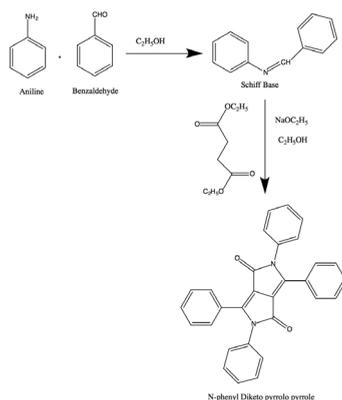
Chemical Table:

Sr. No.	Chemical/Reagent	Mol wt. g/mol	mMoles	Wt/ml	Eq. wt.
1)	Schiff Base	181.2	0.021	3.8 gm	2.2
2)	Diethyl succinate	174.99	0,009	1.12 ml	1
3)	Sodium eethoxide	54.02	0.063	1.5 gm	3
4)	Ethanol	46.06	-	40ml (25ml+5ml+10ml)	-

Procedure:

1. Set up of the reaction is done as per the discussed work plan
2. Sodium is added to the round bottom in small pieces in present of ethanol solution to form sodium ethoxide in the reaction mixture which will in turn act as the required strong base
3. Diethyl succinate is added to the reaction to the mixture under inert conditions
4. White colour semi solid material formation is observed after sometime
5. Dropwise addition of Schiff base dissolved in excess of ethanol is done and the mixture is stirred properly for an hour
6. The reaction is cooled down and is kept for reflux overnight

Reaction No 8: Microwave Assisted synthesis of N-phenyl substituted DPP



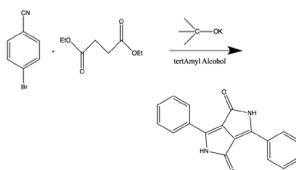
Chemical Table:

Sr No.	Chemical/Reagent	Mol wt. g/mol	mMoles	Wt/ml	Eq. wt.
1)	Schiff Base	181.2	0.021	3.8 gm	2.2
2)	Diethyl succinate	174.99	0,009	1.12 ml	1
3)	Sodium eethoxide	54.02	0.063	1.5 gm	3
4)	Ethanol	46.06	-	40ml (25ml+ 5ml+10ml)	-

Result:

TLC of the resultant mixture showed a drag of the final product with reactants already present in it and hence no adequate result was obtained from this route.

Reaction No 9: - Microwave Assisted synthesis of DPP using Potassium tert Buxoide as base



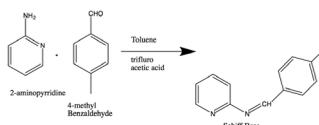
Chemical Table:

1)	Bromobenzonitrile	182.02	2.75	0.500 gm	1
2)	Diethyl succinate	174.99	0.92	0.153 ml	0.33
3)	Potassium tertButoxide	112.21	3.27	0.367 gm	1.19
4)	Tert Amyl alcohol	88.15	-	100 ml	-

Procedure:

1. Potassium tertbutoxide and bromobenzonitrile was mixed and added in a round bottom flask with excess of tertamyl alcohol in inert conditions
2. The mixture was heated for 5 mins in the microwave at 240 watt and temperature was set to 80
3. Dropwise addition of diethyl succinate was done with temperature set to be 100 and kept for 5 minutes in microwave
4. TLC was checked for the resultant mixture and no spots of DPP (resultant mixture) were observed on TLC paper

Reaction No 10: - Preparation of Schiff Base(intermediate) for preparation of N-phenyl DPP



Chemical Table:

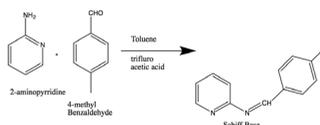
Sr No.	Chemical/Reagent	Mol wt. g/mol	mMoles	Wt/ml	Density g/m3	Eq. wt.
1)	4-methylbenzaldehyde	120.15	10	1.17 ml	1.019	1

2)	2-aminopyridine	94.11	11	1.03 gm	-	1.1
3)	TriFluoro Acetic Acid	114.02	0.5	2 ml	1.49	0.005
4)	Toluene	92.14	-	25ml	0.867	-

Procedure:

- As shown in the table 1.03gm of 2-amino pyridine and 1.17ml 4-methyl benzaldehyde are mixed in presence of toluene as solvent and kept for reflux for 2 hours
- Trifluoro acetic acid is added in the reaction and reaction is kept for 12 hours

Reaction No11: - Preparation of Schiff Base(intermediate) for preparation of N-phenyl DPP



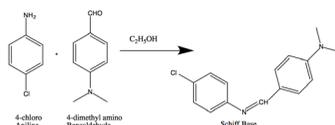
Chemical Table:

Sr No.	Chemical/Reagent	Mol wt.g/mol	mMoles	Wt/ml	Density g/m ³	Eq. wt.
1)	4-methylbenzaldehyde	120.15	10	1.17 ml	1.019	1
2)	2-aminopyridine	94.11	11	1.03 gm	-	1.1
3)	TriFluoro Acetic Acid	114.02	0.5	2 ml	1.49	0.005
4)	Toluene	92.14	-	25ml	0.867	-

Result:

- Reaction Mixture was allowed to cool down for a while
- TLC was conducted which gave no good results as the product still had starting material present in it and the result was not satisfactory

Reaction No12: Synthesis of Schiff base for preparation of N-phenyl DPP



Chemical Table:

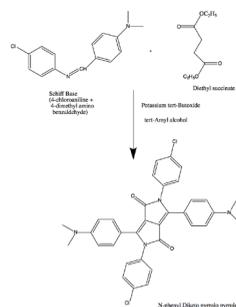
Sr No.	Chemical/Reagent	Mol wt. g/mol	mMoles	Wt/ml	Eq. wt.
1)	4-chloroaniline	127.57	7.84	1 gm	1
2)	4-dimethyl amino Benzaldehyde	149.19	7.84	1.17 gm	1
3)	Ethanol	46.06	-	10 ml	-

Procedure:

- 4-chloroaniline and 4-dimethyl amino benzaldehyde was mixed in 10 ml of ethanol and kept under reflux for 1.5 hours and the solid yellow product was seen forming after sometime

- The reaction was allowed to cool down and the yellow solid (Schiff base) is filtered out
- Schiff base is collected separately and stored.

Reaction No 13: Synthesis of N-phenyl DPP through Schiff base



Chemical Table:

Sr No.	Chemical/Reagent	Mol wt.g/mol	mMoles	Wt/ml	Eq. wt.
1)	Schiff Base	258.5	2.32	0.6 gm	2.1
2)	Diethyl succinate	174.99	1.104	0.184ml	1
3)	Potassium tert-butoxide	112.21	2.32	0.260gm	2.1
4)	Tert-Amyl alcohol	88.15	-	10ml	-

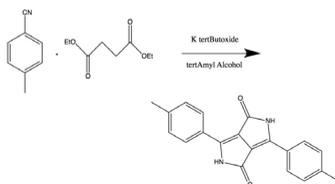
Procedure:

- Diethyl succinate was added along with potassium tert-butoxide with 7 ml of tert amyl alcohol
- Schiff base prepared from the previous reaction was added to the reaction mixture after dissolving it in the remaining 3 ml of the solvent.
- The reaction was heated upto 100 degree Celsius.
- After 1 hour TLC was checked followed by stirring for 4 hours
- TLC was again checked and the observations were noted

Result and Observation:

- When the first TLC was spotted a slight light green fluorescence was seen in the product
- When the reaction was continued to reflux for 4 hours the TCL showed no traits of fluorescence in the product and contained starting material spots in it
- After keeping the reaction overnight when third TLC was taken it 40%ethyl acetate to hexane it corresponded with starting material spots which meant the starting material wasn't consumed and the reaction wasn't going forward
- Another TLC was checked in 10% MeOH in CHCl₃ as DPP are generally insoluble in many of the solvents but that too didn't show any satisfactory results with product corresponding with Schiff base

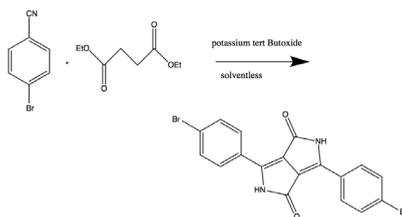
Reaction No 14: Synthesis of DPP through 4 methyl benzonitrile



Chemical Table:

Sr No.	Chemical/Reagent	Mol wt. g/mol	mMoles	Wt/ml	Eq. wt.
1)	4-methyl benzonitrile	117.148	41.87	5.00 ml	1
2)	Diethyl succinate	174.99	58.62	9.72ml	1.4
3)	Potassium tertButoxide	112.21	79.553	8.92 gm	1.9
4)	Tert Amyl alcohol	88.15	-	20 ml	-

Reaction No 15 : Solvent-less synthesis of DPP



Chemical Table:

Sr No.	Chemical/Reagent	Mol wt. g/mol	mMoles	Wt/ml	Eq. wt.
1)	Bromobenzonitrile	182.02	2.75	1 gm	1
2)	Diethyl succinate	174.99	0.92	0.3 ml	0.33
3)	Potassium tertButoxide	112.21	3.27	0.735 gm	1.19

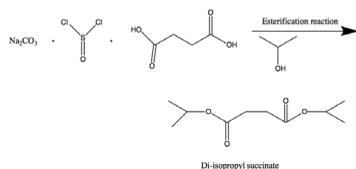
Procedure:

- Potassium tert butoxide and bromo benzonitrile was mixed at 20 c for one hour in inert conditions
- Dropwise addition of diethyl succinate is carried out through a syringe in a span of half n hour
- The mixture was allowed to rise to 100 c and temperature was maintained between 105-120 c for 3 hours
- The mixture was cooled to 65 c and acetic acid was added to neutralize the reaction and kept for 1 hour reflux
- Later the precipitate was washed off through ethanol and water.

Observation:

- Red colour was seen forming during the reaction but no black residue particles were noticed in the reaction mixture.
- TLC didn't show any satisfactory results

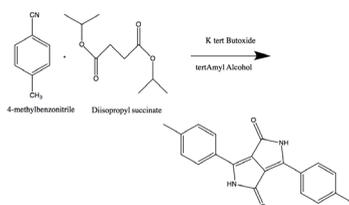
Reaction No 16: Synthesis of Di-isopropyl succinate



Chemical Table:

Sr. No.	Chemical/Reagent	Mol wt. g/mol	mMoles	Wt/ml	Eq. wt.
1)	Sodium carbonate	105.98	84.68	8.975 ml	2
2)	Thionyl chloride	118.97	84.68	6.15 ml	2
3)	Succinic Acid	118.09	42.34	5 gm	1
4)	Iso propyl alcohol	60.1	84.68	50 ml	2

Reaction No 17: Synthesis of DPP from succinic ester condensation



Chemical Table:

Sr No.	Chemical/Reagent	Mol wt. g/mol	mMoles	Wt/ml	Density g/cm ³	Eq. wt.
1)	4-methyl benzonitrile	117.148	8.53	1.02 ml	0.985	1
2)	Diisopropyl succinate	202.25	2.815	0.6 ml	0.99	1.4
3)	Potassium tertButoxide	112.21	10.236	1.15 gm	-	1.9
4)	Tert Amyl alcohol	88.15	-	20 ml	-	-

Procedure:

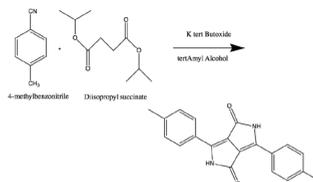
- 1) 4-methyl benzonitrile is added to the mixture of potassium tertutoxide and 10 ml of tertAmyl alcohol (2-methyl 2-butanol) at 20-25 c temperature.
- 2) Dropwise addition of diisopropyl succinate in tert Amyl alcohol was done over a period of 1.5 hours and mixture was kept at 100 c for 3 hours
- 3) The reaction mixture was dried by separating out the solvents (isopropyl alcohol and tertAmyl alcohol)
- 4) Resultant dried paste was mixed with 20 ml of methanol and 0.3 ml of acetic acid and allowed to reflux.

Observation:

- 1) No Red color was seen in the mixture after two hours of reflux which shouldn't be the case (according to the paper referred) in the formation of DPP

Note: the diisopropyl succinate used in this reaction is prepared in the previous experiment and not the one available in the market is used.

Reaction No 18: Synthesis of DPP via Condensation method



Chemical Table:

Sr No.	Chemical/Reagent	Mol wt. g/mol	mMoles	Wt/ml	Density g/cm ³	Eq. wt.
1)	4-methyl benzonitrile	117.148	8.53	1.02 ml	0.985	1
2)	Diisopropyl succinate	202.25	2.815	0.6 ml	0.99	1.4
3)	Potassium tertButoxide	112.21	10.236	1.15 gm	-	1.9
4)	Tert Amyl alcohol	88.15	-	20 ml	-	-

Procedure:

- 5) 4-methyl benzonitrile is added to the mixture of potassium terttutoxide and 15 ml of tertAmyl alcohol (2-methyl 2-butanol) at 20-25 c temperature.
- 6) Dropwise addition of diisopropyl succinate in tert Amyl alcohol was done over a period of 1.5 hours and mixture was kept at 100 c for 3 hours
- 7) The reaction mixture was dried by separating out the solvents (isopropyl alcohol and tertAmyl alcohol)
- 8) Resultant dried paste was mixed with 20 ml of methanol and 1 ml of acetic acid and allowed to reflux for 2 hours

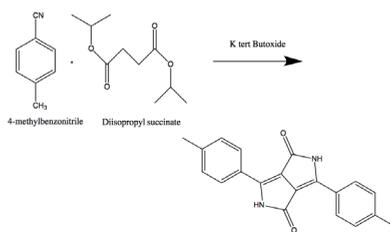
Observation:

- 1) Red colour was obtained after the reaction and further work up was done
- 2) After addition of water no precipitates were observed at all
- 3) The TLC also didn't show any satisfactory results

Note: The diisopropyl succinate used in this reaction is the procured one from the market. Due to its lack of availability we were bound to synthesize it and use it as per the previous reaction.

Reaction No 19: Solvent-less synthesis of DPP

Aim: To synthesize DPP without using tertAmyl alcohol as it is difficult to remove it in inert conditions



Chemical Table:

Sr No.	Chemical/Reagent	Mol wt. g/mol	mMoles	Wt/ml	Density g/cm ³	Eq. wt.
1)	4-methyl benzonitrile	117.148	17.07	2.04 ml	0.985	1
2)	Diisopropyl succinate	202.25	5.63	1.15 ml	0.99	0.33
3)	Potassium tertButoxide	112.21	20.484	2.3 gm	-	1.2

Procedure:

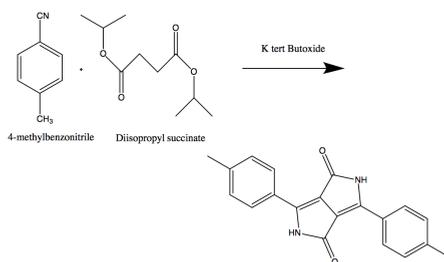
- 1) Potassium tert Butoxide is taken in a two neck RB and 4-methyl benzonitrile is added to it at 15-20 c temperature.
- 2) Dropwise addition of diisopropyl succinate is done in 1 hour
- 3) The reaction mixture is allowed to heat at 120c temperature for 3 hours so that the material melts and a paste is formed that mixes properly with each other.
- 4) The mixture is allowed to cool at 65c temperature and the work up is done
- 5) 15 ml of methanol and 1 ml of acetic acid is added to the reaction mixture and refluxed for 2 hours
- 6) The mixture is then washed with water and if precipitates exist they are mixed into methanol to remove the impurities and filtered further.
- 7) The resultant mixture is dried overnight and collected

Observation and Result:

- 1) The mixture was dissolved in water and it was observed that it is insoluble in it
- 2) After drying the mixture was collected separately and it shows brilliant red colour
- 3) The resultant powder weighed around 0.235 gms with a yield of 12%
- 4) A sample of the product was given for NMR and results are awaited for confirmation of the product.

Reaction No 20: Solvent-less synthesis of DPP

Aim: After getting success in the last scheme, a bigger batch of solvent less synthesis of DPP was put up (5 gms)



Chemical Table:

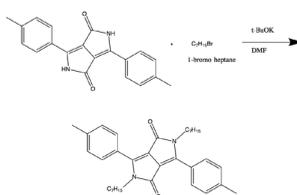
Sr No.	Chemical/Reagent	Mol wt. g/mol	mMoles	Wt/ml	Density g/cm ³	Eq. wt.
1)	4-methyl benzonitrile	117.148	42.65	5.1 ml	0.985	1
2)	Diisopropyl succinate	202.25	14.075	2.875 ml	0.99	0.33
3)	Potassium tertButoxide	112.21	51.18	5.75 gm	-	1.2

Procedure: Similar as Reaction 19.

Result: Pending

Reaction No 21: N-alkylation of DPP

Aim: To make the insoluble DPP moiety soluble by addition of -R group (N-alkylation) for fluorescence to enhance



Chemical Table:

Sr No.	Chemical/Reagent	Mol wt.g/mol	mMoles	Wt/ml	Density g/cm ³	Eq. wt.
1)	4-methyl phenyl DPP	316	2.215	0.7 gm		1
2)	1-Bromo heptane	179.10	7.310	1.15ml +1 ml	1.14	3.3
3)	Potassium tertButoxide	112.21	9.03	1.013 ml + 1gm	-	4.077
4)	DMF	73.09		25ml + 10ml		

Procedure:

- 8) 4-methyl phenyl DPP is added with DMF (15ml) and potassium tert Butoxide in a round bottom flask in presence of nitrogen at room temperature.
- 9) The resulting mixture was stirred for 30 mins and then 1-bromoheptane was added slowly in a span of half n hour.
- 10) After stirring for additional 1 hour at room temperature the mixture was heated to 60c and stirred overnight.
- 11) TLC of the mixture was seen which still showed the remains of DPP in it and hence additional of 1 ml of 1-bromoheptane and 1 gm of potassium tert-Butoxide was added to the same quantity of DPP moiety and stirred at 95c temperature overnight.
- 12) The resulting mixture was cooled and work up was done

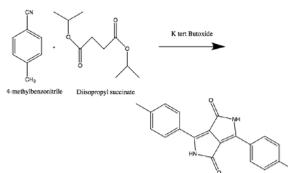
Observation and Results:

- 1) The mixture was poured into cold water and stirred for few minutes for it to precipitate out the product
- 2) The product was then filtered and dried for 5-6 hours

- TLC was checked which showed 2 spots along with the product and some unreacted DPP
- Column chromatography was done for more precise conclusion

Reaction No 22: Solvent-less synthesis of DPP

Aim: To synthesize more than 1 gm of DPP for further modifications



Chemical Table:

Sr No.	Chemical/Reagent	Mol wt. g/mol	mMoles	Wt/ml	Density g/cm ³	Eq. wt.
1)	4-methyl benzonitrile	117.148	85.3	10.2 ml	0.985	1
2)	Diisopropyl succinate	202.25	28.15	5.75 ml	0.99	0.33
3)	Potassium tertButoxide	112.21	102.36	11.5 gm	-	1.2

Procedure:

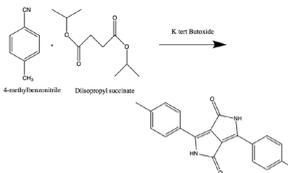
Similar as Reaction 19.

Observation and Result:

- As per the earlier procedure the reaction mixture failed to give red color paste and after completion of the reaction showed white unreacted contains of potassium tert-Butoxide in it
- TLC didn't give any desired result
- REACTION FAILED: Due to the usage of old potassium tert-Butoxide

Reaction No 23: Solvent-less synthesis of DPP

Aim: To produce 1 gm of DPP for further chemistry on it



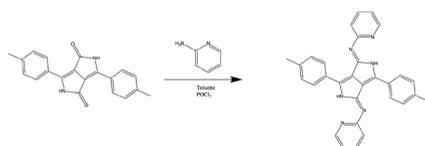
Chemical Table:

Sr No.	Chemical/Reagent	Mol wt. g/mol	mMoles	Wt/ml	Density g/cm ³	Eq. wt.
1)	4-methyl benzonitrile	117.148	42.65	5.1 ml	0.985	1
2)	Diisopropyl succinate	202.25	14.075	2.875 ml	0.99	0.33
3)	Potassium tertButoxide	112.21	51.18	5.75 gm	-	1.2

Procedure: Similar as Reaction 19.

Result:

- Brilliant red color DPP was obtained after filtration and drying of the product overnight
- The DPP formed had yield of 8.3% (1.116 gms)



Chemical Table:

Sr No.	Chemical/Reagent	Mol wt. g/mol	mMoles	Wt/ml	Eq. wt.
1)	DPP	316	1	0.316 gm	1
2)	2-amino pyridine	94.11	2.5	0.235 gm	2.5
3)	POCl ₃	153.33	8	0.75 ml	8
4)	Toluene		-	15 ml	-

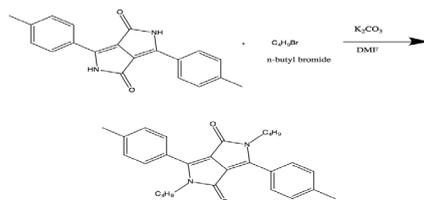
Procedure:

- 1) DPP and 2-amino pyridine was mixed at room temperature in absolute toluene and stirred for few mins
- 2) POCl₃ was added drop-wise over a period of 20 mins and refluxed in inert atmosphere
- 3) The reaction was monitored by TLC and later on the reaction was kept for reflux overnight

Observation and Result:

- 1) 2 fluorescent spots were observed in 50% ethyl acetate-hexane apart from multiple spots
- 2) The reaction mixture hasn't changed the color and shows traits of unreacted DPP in it
- 3) Column Chromatography is yet to be done for final results

Reaction No 25: N-alkylation of DPP



Chemical Table:

Sr No.	Chemical/Reagent	Mol wt. g/mol	mMoles	Wt/ml	density	Eq. wt.
1)	DPP	316	0.633	0.200 gm	-	1
2)	n-butyl bromide	137.01	2.532	0.272 ml	1.276	4
3)	K ₂ CO ₃	138.205	2.532	0.35 gm	2.43	4
4)	DMF		-	7 ml		-

Procedure:

- 1) DPP and dried K₂CO₃ was added and stirred for 15-20 mins at room temperature under nitrogen
- 2) N-butyl bromide was added drop-wise and reaction mixture was stirred for 35-40 mins at room temperature
- 3) The mixture was then heated at 85-90 c temperature for 3 hours til the mixture turns orange.

Observation and result:

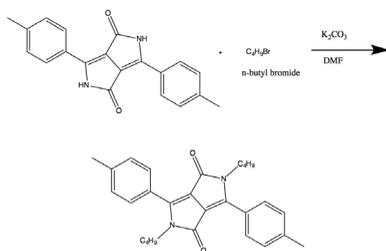
- 1) TLC was checked which showed bright yellow spot along with a light orange spot and DPP was

totally consumed

- 2) After cooling, the work up of the mixture was done, filtered and dried overnight
- 3) Orange powder is obtained weighing 0.150 gms with a yield of 63%

Reaction No 26: N-alkylation of DPP

Aim: After the success in previous reaction, aim is to put up a slightly bigger batch of DPP in order to get as much as N-alkylated DPP formed for further schemes



Chemical Table:

Sr No.	Chemical/Reagent	Mol wt. g/mol	mMoles	Wt/ml	density	Eq. wt.
1)	DPP	316	1.899	0.600 gm	-	1
2)	n-butyl bromide	137.01	7.596	0.816 ml	1.276	4
3)	K_2CO_3	138.205	11.215	1.55 gm	2.43	5.9
4)	DMF		-	21 ml		-

Procedure:

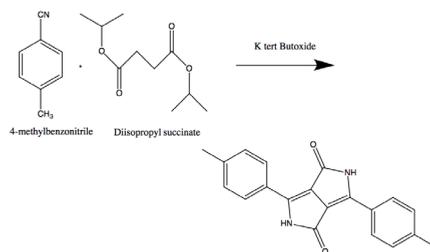
- 4) DPP and dried K_2CO_3 was added and stirred for 15-20 mins at room temperature under nitrogen
- 5) N-butyl bromide was added drop-wise and reaction mixture was stirred for 35-40 mins at room temperature
- 6) The mixture was then heated at 60 c temperature for 3 hours and 0.5 gms of more base was added for the reaction mixture to turn orange and consume all the DPP
- 7) The reaction mixture was then heated at 90c temperature for 2 hours

Observation and result:

- 4) TLC was checked which showed bright yellow spot along with a light orange spot and DPP was totally consumed
- 5) After cooling, the work up of the mixture was done, filtered and dried overnight and collected
- 6) Total quantity of 0.56 gms was produced
- 7) Both the dried product as well as the previous reaction product were mixed and recrystallized with DCM and n-Hexane couple of times to discard the impurities and get the pure product
- 8) Finally, Column chromatography was done and the orange and yellow spots were separated with few impurities still present in the product
- 9) The product sample is given for NMR and results are awaited.

Reaction No 27: Solvent-less synthesis of DPP

Aim: To synthesize DPP for further modifications



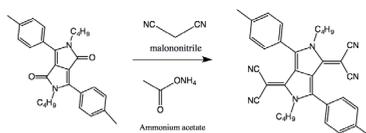
Chemical Table:

Sr No.	Chemical/Reagent	Mol wt. g/mol	mMoles	Wt/ml	Density g/cm ³	Eq. wt.
1)	4 - methyl benzonitrile	117.148	21.3375	2.55 ml	0.985	1
2)	Diisopropyl succinate	202.25	7.0375	1.4375 ml	0.99	0.33
3)	Potassium tertButoxide	112.21	25.59	2.875 gm	-	1.2

Observation and Result:

- 1) The reaction was carried out in the similar manner as discussed in earlier schemes with the same parameters.
- 2) Mixing of potassium tert butoxide and 4-methyl benzonitrile didn't give red color unlike previous methods
- 3) As the reaction was carried out further, there was no color change observed after 4 hours and when a sample was tested it showed solubility in water
- 4) It was concluded that due to poor grade of potassium tert butoxide this reaction failed to give desired results.

Reaction No 28: Condensation of N-alkylated DPP with malononitrile

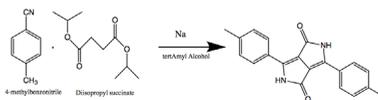


Procedure:

- 5) N-alkylated DPP purified from the previous reaction was mixed with malononitrile and 1 ml of ammonium acetate in a Dean Stark apparatus
- 6) Toluene is added as the solvent and the azeotropic mixture of toluene/water is set up and heated for 36 hours.
- 7) The reaction is monitored by TLC

Observation and Result:

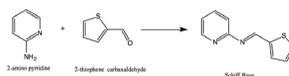
- 1) When the reaction is stopped one solid state compound is present with a liquid compound
- 2) Work up is done with ethyl acetate and water and TLC showed impurities
- 3) When filtered the solid is obtained in a form of gray colored cluster which didn't run on TLC not showing any satisfactory results

Reaction No 29: Synthesis of DPP via sodium tert Amylate solution**Aim:** To synthesize DPP by using sodium as a base to increase its yield**Chemical Table:**

Sr No.	Chemical/Reagent	Mol wt. g/mol	mMoles	Wt/ml	Density g/cm ³	Eq. wt.
1)	4-methyl benzonitrile	117.148	17.07	2.04 ml	0.985	1
2)	Diisopropyl succinate	202.25	8.535	1.75 ml	0.99	0.5
3)	Sodium	23	25.605	0.6 gm	-	1.2
4)	Tert amyl alcohol	88.15	-	25 ml	-	-

Procedure:

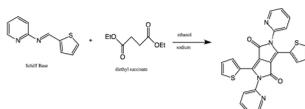
- 1) The apparatus was set up same as the previous reactions for synthesis of DPP
- 2) 20 ml of tert amyl alcohol was taken in a flask and sodium was added to it to form a sodium tert amylate solution
- 3) After 2 hours with all possible parameters taken into consideration sodium was not able to dissolve in the solvent due to impure sodium metal

Reaction No 30:- Preparation of Schiff Base

Sr No.	Chemical/Reagent	Mol wt. g/mol	mMoles	Wt/ml	Eq. wt.
1)	2-amino pyridine	94.11	10.62	1 gm	1
2)	2-thiophene carboxaldehyde	112.15	10.62	0.99 ml	1
3)	Ethanol	46.06	-	10 ml	-

Procedure:

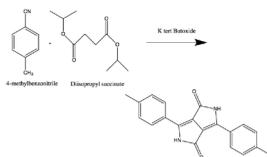
- 1) 2-amino pyridine and 2-thiophene carboxaldehyde is mixed in ethanol and stirred for 5 hours at RT
- 2) The reaction is monitored by TLC after every 30 mins
- 3) Color change in the reaction is observed once the reactants are used up
- 4) Excess of ethanol is distilled off through rotatory flask and the wet paste is filtered with washing with ethanol
- 5) The product is dried and collected

Reaction No 31: - Preparation of Schiff Base

Sr. No.	Chemical/Reagent	Mol wt. g/mol	mMoles	Wt/ml	Eq. wt.
1)	Schiff Base	188.25	10.6	2 gm	1

2)	Diethyl succinate	174.99	5.31	0.99 ml	0.5
3)	Sodium	23	23.32	1 gm	2.2
4)	Ethanol	46.06	-	25 ml	

Reaction No 32: Solvent-less synthesis of DPP



Chemical Table:

Sr No.	Chemical/Reagent	Mol wt. g/mol	mMoles	Wt/ml	Density g/cm ³	Eq. wt.
1)	4-methyl benzonitrile	117.148	42.65	5.1 ml	0.985	1
2)	Diisopropyl succinate	202.25	14.075	2.875 ml	0.99	0.33
3)	Potassium tertButoxide	112.21	51.18	6 gm	-	1.2

Procedure:

- 1) As per the previous success obtained in the solventless synthesis of DPP, this reaction was set up accordingly to get the maximum yield possible
- 2) After mixing of the reactants the mixture was heated at 120c temperature for 3 hours
- 3) Due to improper mixing of the reactants the potassium tert butoxide didn't melt properly to form a thick red paste as expected
- 4) Even after adding some solvent (tert amyl alcohol) in very less quantity, the mixing didn't happen and the reaction failed.

Reaction No 33: Synthesis of DPP via sodium tert Amylate solution

Aim: To synthesize DPP by using sodium as a base to increase its yield.



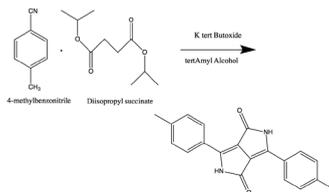
Chemical Table:

Sr No.	Chemical/Reagent	Mol wt. g/mol	mMoles	Wt/ml	Density g/cm ³	Eq. wt.
1)	4-methyl benzonitrile	117.148	17.07	2.04 ml	0.985	1
2)	Diisopropyl succinate	202.25	8.535	1.75 ml	0.99	0.5
3)	Sodium	23	25.605	0.6 gm	-	1.2
4)	Tert amyl alcohol	88.15	-	25 ml	-	-

Procedure:

- 1) The apparatus was set up same as the previous reactions for synthesis of DPP
- 2) Unlike last time, pure grade of sodium was mixed in tert amyl alcohol solvent to form sodium tert Amylate solution
- 3) Even after constant stirring for 2 hours at 70c temperature, sodium doesn't solubilizes in tert amyl alcohol
- 4) Hence, the reaction fails

Reaction No 34: Synthesis of DPP from succinic ester condensation



Chemical Table:

Sr No.	Chemical/Reagent	Mol wt. g/mol	mMoles	Wt/ml	Density g/cm ³	Eq. wt.
1)	4-methyl benzonitrile	117.148	8.53	1.02 ml	0.985	1
2)	Diisopropyl succinate	202.25	2.815	0.6 ml	0.99	0.33
3)	Potassium tertButoxide	112.21	10.236	1.15 gm	-	1.2
4)	Tert Amyl alcohol	88.15	-	10 ml		-

Procedure:

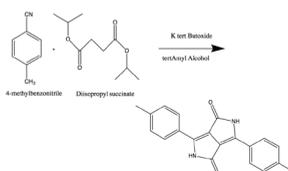
- 1) 4-methyl benzonitrile is added to the mixture of potassium terttutoxide and 10 ml of tertAmyl alcohol (2-methyl 2-butanol) at 20-25 c temperature.
- 2) Dropwise addition of diisopropyl succinate in tert Amyl alcohol was done over a period of 1.5 hours and mixture was kept at 100 c for 3 hours but no colour change was observed
- 3) The reaction was heated for 12 hours at 110c temperature and the color change was noted

Observation: No Red color was seen in the mixture after 16 hours of the reaction

Reaction No 35: Microwave Assisted Synthesis of DPP

Chemical Table:

Sr No.	Chemical/Reagent	Mol wt. g/mol	mMoles	Wt/ml	Density g/cm ³	Eq. wt.
1)	4-methyl benzonitrile	117.148	1.37	0.25 ml	0.985	1
2)	Diisopropyl succinate	202.25	0.4521	0.087 ml	0.99	0.33
3)	Potassium tertButoxide	112.21	1.644	0.1835 gm	-	1.2
4)	Tert Amyl alcohol	88.15	-	2.5 ml		-



Procedure:

- 1) Add 4 methyl benzonitrile and potassium tert butoxide to the G10/G30 vial and close it immediately so it is not in contact with air
- 2) Add dissolved di-isopropyl succinate in tert Amyl alcohol to the vial
- 3) Set the required data in the microwave (1). Name of the reaction (2) Standard route (3) Heat as fast as possible (4) Hold time (here 10 mins) (5) Cooling time (here 5 mins) and press Start

- Once the reaction is completed a TLC is taken to check whether the starting material is consumed or not
- Once confirmed, methanol is added to the reaction mixture and kept it on reflux for 2 hours
- It is necessary to neutralize the reaction mixture by acetic acid before refluxing
- Water is added at the end of reaction and filtered

Observation:

- Once the reaction mixture was dried not much precipitate of the compound were found
- The mixture shows no traces of DPP

Reaction No 37: Microwave Assisted Synthesis of DPP

Chemical Table:

Sr No.	Chemical/Reagent	Mol wt. g/mol	mMoles	Wt/ml	Density/cm ³	Eq. wt.
1)	4-bromol benzonitrile	182.02	1.49	0.250 gm	-	1
2)	Diisopropyl succinate	202.25	0.4521	0.087 ml	0.99	0.33
3)	Potassium tertButoxide	112.21	1.644	0.1835 gm	-	1.2
4)	Tert Amyl alcohol	88.15	-	2.5 ml		-

Procedure:

- Add 4 methyl benzonitrile and potassium tert butoxide to the G10/G30 vial and close it immediately so it is not in contact with air
- Add dissolved di-isopropyl succinate in tert Amyl alcohol to the vial
- Set the required data in the microwave (1) Name of the reaction (2) Standard route (3) Heat as fast as possible (4) Hold time (here 10 mins) (5) Cooling time (here 5 mins) and press Start
- Once the reaction is completed a TLC is taken to check whether the starting material is consumed or not

Observation:

- The reaction showed a lump of solid present in the vial
- Tlc also confirmed that DPP wasn't fully formed showing traces of starting material
- Methanol and a drop of acetic acid is added to the reaction mixture and kept on stirring overnight to see if precipitates are formed instead of the emulsion seen at present
- After 48 hours of stirring the mixture, no precipitates were found.

Extraction Part

Scheme No:1

Extraction of essential oil from bottle brush leaves by

In this I have extracted the essential oil from leaves of bottle brush solvent free

I have done the optimization of the yield of extracted oil by considering the three major factor affecting yield using Design of experiments

Also compare of yield with convention hydro distillation is done

Also the SEM of the extracted leaves are done and comparison also done

GC of sample is done

Factor studied for DOE

Sr. No.	Factor to be studied	Higher Level	Lower level
1	Time	60	45
2	Power of microwave	8	6
3	Temperature	105	100

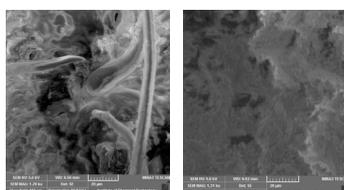
Result table

RUN	Exp no.	A	B	C	AB	AC	BC	ABC	Yeild
1	2	-0.32	-0.32	-0.32	0.32	0.32	0.32	-0.32	0.32
2	4	0.27	-0.27	-0.27	-0.27	-0.27	0.27	0.27	0.27
3	3	-0.42	0.42	-0.42	-0.42	0.42	-0.42	0.42	0.42
4	6	0.38	0.38	-0.38	0.38	-0.38	-0.38	-0.38	0.38
5	7	-0.29	-0.29	0.29	0.29	-0.29	-0.29	0.29	0.29
6	8	0.25	-0.25	0.25	-0.25	0.25	-0.25	-0.25	0.25
7	1	-0.35	0.35	0.35	-0.35	-0.35	0.35	-0.35	0.35
8	5	0.35	0.35	0.35	0.35	0.35	0.35	0.35	0.35
	Σ	-0.13	0.37	-0.15	0.05	0.05	-0.05	0.03	
	(Σ)^2/8	-0.01625	0.04625	-0.01875	0.00625	0.00625	-0.00625	0.00375	

Anova analysis

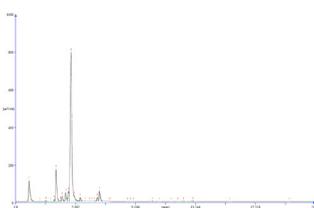
Source	SS	[SS]	D. F.	M. S. S.	F calculation
A	-0.01625	0.01625	1	0.01625	2.88888889
B	0.04625	0.04625	1	0.04625	8.22222222
C	-0.01875	0.01875	1	0.01875	3.33333333
AB	0.00625	0.00625	1	0.00625	
AC	0.00625	0.00625	1	0.00625	
BC	-0.00625	0.00625	1	0.00625	
ABC	0.00375	0.00375	1	0.00375	
TOTAL		0.10375	7		

- From anova table F value for A, B, C factors we get
- From table of F critical we get is 7.708
- So only B (Microwave Power) is having more value i. e. 8.22 so factor B impact on yield
- sum of square (s.s.) of the factor B is +0.04 so +ve value of factor B (Microwave Power) increases the yield of extraction of bottle brush leaves
- SEM Images**



SEM of plant extracted by hydrodistillation is remaining.

GC Graph GC of extracted oil



GC of extracted oil
Antimicrobial study

Bacteria name	Zone of inhibition	
	Bottle brush MW	Bottle brush HD
S. aureus	1	0.7
E. coli.	0.9	0.6
B. subtilis	0	0

- Antibacterial activity of bottle brush leave oil is more with microwave(MW) extracted oil as compare to hydrodistilled(HD) oil
- There is no antibacterial activity for B. Subtilis in both the oils

Scheme No:2

Extraction of essential oil from black cardamom by microwave

In this I have extracted the essential oil from cardamom solvent free

I have done the optimization of the yield of extracted oil by considering the three major factor affecting yield using Design of experiments

Also compare of yield with convention hydro distillation is done

GC of sample is done

Factor studied for DOE

	Factor to be studied	Higher Level	Lower level
1	Time	100	75
2	Power of microwave	8	6
3	Temperature	105	100

Result Table

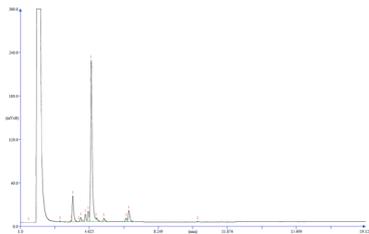
RUN	Exp no.	A	B	C	AB	AC	BC	ABC	Yield
1		-0.6	-0.6	-0.6	0.6	0.6	0.6	-0.6	0.6
2		0.53	-0.53	-0.53	-0.53	-0.53	0.53	0.53	0.53
3		-0.78	0.78	-0.78	-0.78	0.78	-0.78	0.78	0.78
4		0.67	0.67	-0.67	0.67	-0.67	-0.67	-0.67	0.67
5		-0.55	-0.55	0.55	0.55	-0.55	-0.55	0.55	0.55
6		0.54	-0.54	0.54	-0.54	0.54	-0.54	-0.54	0.54
7		-0.7	0.7	0.7	-0.7	-0.7	0.7	-0.7	0.7
8		0.67	0.67	0.67	0.67	0.67	0.67	0.67	0.67
	Σ	-0.22	0.6	-0.12	-0.06	0.14	-0.04	0.02	
	(Σ) ² /8	-0.0275	0.075	-0.015	-0.0075	0.0175	-0.005	0.0025	

Anova Analysis

Source	SS	MS	D. F.	M. S. S.	F calculation
A	-0.0275	0.0275	1	0.0275	3.38461538
B	0.075	0.075	1	0.075	9.23076923
C	-0.015	0.015	1	0.015	1.84615385
AB	-0.0075	0.0075	1	0.0075	
AC	0.0175	0.0175	1	0.0175	
BC	-0.005	0.005	1	0.005	
ABC	0.0025	0.0025	1	0.0025	
TOTAL		0.15	7		

- From Anova table F value for A, B, C factors we get
- From table of F critical we get is 7.708
- So only B (Microwave Power) is having more value i.e. 9.23 so factor B impact on yield
- Sum of square (s.s.) of the factor B is +0.075 so +ve value of factor B (Microwave Power) increases the yield of extraction of bottle brush leaves

GC



GC graph

Antimicrobial study

Bacteria name	Zone of inhibition	
	Microwave	Hydrodistillation
S. aureus	1.5	1.1
E. coli.	1.2	1
B. subtilis	1.6	1.1

- Antibacterial activity of Cardamom oil is more with microwave(MW) extracted oil as compare to hydrodistilled(HD) oil

Facility created under CoE-PI

- HP workstation

Project Title:

VALIDATION OF NEW HAND-PUMP DESIGN FOR WATER DISINFECTION: FIELD TRIALS

Project investigator:

Prof. A. B. Pandit

Current Scenario:

1. Ground water is the principal source of Potable water in our country and indispensable source of our life. It is very important that the drinking water is safe and potable.
2. The problem of groundwater quality is acute. The resulting degradation of water quality in water body creates an undesirable condition so that water cannot be used for intended beneficial uses including bathing, recreation and as a source of raw water supply.
3. According to Central Pollution Control Board, 90% of the water supplied in India to the town and cities are polluted, out of which only 1.6% gets treated.
4. The water which is not suitable for drinking purpose called non potable water and the use of such water for drinking purpose leads to illnesses, a major cause of death in many countries.

Introduction:

1. The hand pump can serve as an efficient tool for the disinfection of bore well water with specific modification in the geometry of the check valve.

2. This modification leads to the phenomena known as hydrodynamic cavitation, which can potentially disrupt the bacterial cells. This is an energy efficient mechanism than homogenization and acoustic cavitation and many other common chemical disinfection methods practiced regularly.
3. Hydrodynamic cavitation technology for hand pump has recently emerged for the application in water disinfection. This novel technology was validated by conducting the field trials/experiments and was found to have yielded >95% disinfection.
4. The people from rural areas across the world will have access to, the quality potable water and can be rescued from the number of water borne diseases. Hand pump cavitation will be a milestone in the emerging era of a novel and economical water disinfection systems.

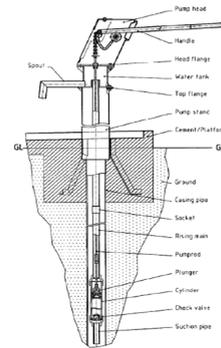


Image 1: India Mark II Hand Pump installed in a bore well

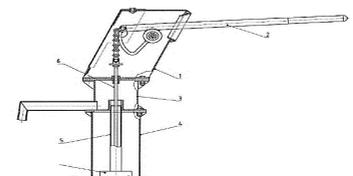
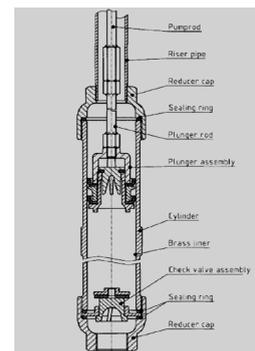


Image 2: Top Part Hand Pump



India Mark II Hand Pump

1. The India Mark II Hand Pump is suitable for lifting water from depths up to 50 m and can be installed in bore wells and dug wells.
2. The India Mark II Hand Pump was developed in India more than 6 years ago. In Indian villages, hand pumps are used by many people.
3. Sometimes they are used continuously for up to 18 hours a day.



Image 4: Cylinder parts of the India Mark II Pump

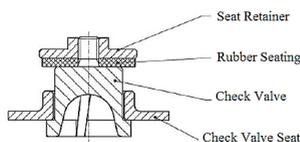


Image 5: Original Check Valve Assembly

Literature Survey:

I. Why Hand Pump???

- i. The generation of vapour cavities as well as size of the cavities will have increased during upward stroke of the hand pump due to the reduction in pressure (i.e. negative pressure). All the vapour cavities will collapse simultaneously due to huge back pressure generated during downward stroke, which includes hydrostatic head by the water column.
- ii. Many of the cavities will collapse automatically due to overgrowth; still population of cavities will be high.
- iii. Probably, asymmetric collapse is more possible by the reason simulated large lifespan of cavities. Thus mechanical as well as chemical effects could be intensified in the hand pump cavitation.
- iv. This is positive outcome of discrete back pressure

system (i.e. hand pump) rather than continuous one (i.e. existing hydrodynamic cavitation system). While, in continuous back pressure system, the cavity size will be less as well as shorten the lifespan. The symmetric collapse is more possible due to small cavity size to the pump-pipe diameter ratio. Hence it may have limited effects as well as the action of cavity will be limited in existing/conventional cavitation devices (i.e. nozzle, venturi, orifice etc.).

- v. The actual disintegration of microorganism occurs due to two mechanisms 1) Intensive collapse of the cavity in the vicinity and 2) The high velocity of micro jets generated during asymmetric collapse of the cavities.
- vi. A small injury is enough to render the bacteria, fungi and viruses, unviable. There are various cavitation devices developed to treat the water.
- vii. Although the use of hydrodynamic cavitation in hand pumps is a new proposed practice which is economically inexpensive and can be extensively implemented.

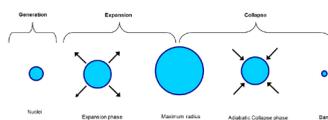


Image 6: Mechanism of Cavitation

Generation of back pressure is the key fact for the disinfection

or destruction of the bacteria, viruses and fungi. But back pressure should not affect the growth of the cavities. Because, if it does not get an opportunity to grow adequately, resulting in smaller size cavities which requires more back pressure to collapse impulsively as well as their effect will be limited. (e.g. Small balloon requires more pressure from opposite sides to burst than large one). As the back pressure increases, the expansion slashes down. The minimum expansion of cavities should be more than 2 times of the initial size for cavitation to occur significantly. The earlier collapse of the cavities can occur due to back pressure hence the collapse probability of the cavity is more in the bulk rather than the cylinder wall surface, protecting the latter from erosion. Therefore symmetric collapse will be often more in case of higher back pressure. Whereas, low bulk pressure introduces higher expansion as well as increase in the lifespan of the cavity leading to its collapse in the bulk. Hence cavity can travel up to the solid wall surface and can collapse which will be asymmetric in nature. Mass transfer rates are negligible for pressures greater than 3 atm, thus only mechanical effects are possible in the case of ultrasound system. Hence lower pressure is more preferable to enhance the chemical effects in the case of acoustic cavitation. The hand pump cavitation is balanced for the physical and chemical effects due to large pressure drop generated during upward and downward stroke of

the hand pump.

II. Selection Criteria for specific modification to the system:

With higher opening of the valve, the probability of generation of vapour cavities are less due to low fluid velocity or high relative pressure at the opening of valve. There may be generation of only small gaseous cavities due to degassing, which is limited in action as well as quantity. In addition, hydrostatic pressure could be higher, hence it affects the cavitation growth, consequently resulting into low disinfection. While in lower opening of valve, cavitation growth is more due to additional water evaporation.

This may have four consequences:

1. Buoyancy force act on the over growing cavities which pulls them towards upward surface of water and nullifies the effect, secondly
2. There may be chances of formation cavity clusters/ cloud because as the size of the cavities increases, interaction between them increases and they form cluster which can dampen the individual cavitation events,
3. The cavities may collapse automatically in the bulk before downward stroke (i.e. before experiencing the back pressure) of piston or plunger yoke due to their over growth resulting in less intensive collapse which

is not sufficient to kill the bacteria.

4. Very high velocities will decrease the number of formation of cavities at the opening of lower check valve.

Finally, Cavities should grow sufficiently large and which can collapse simultaneously after experiencing the action of back pressure applied by the piston during the downward stroke. Simultaneous collapse will generate highly intensive shock waves as well as micro jets during cavities asymmetric collapse resulting in more disinfection.

Brief Work Plan

Aim of the project was to deliver disinfected water (or water with pathogens within the prescribed limits or to reduce the number of pathogens which are responsible for causing the water borne diseases) by a modified hand pump design. To achieve this, it is necessary to understand the underlying principles of pumping, cavitation, disinfection due to cavitation, mechanism and hydrodynamics of the hand pump. In the past years, following work has been carried out.

1. Initially, standardization of CFU counting method has been done.
2. Design of the India Mark II Hand Pump was studied in detail (Manchalwar, Pandit et al)
3. After extensive numerical simulations, it was predicted that 40% opening generates

maximum cavitation effect that causes maximum pathogen destruction.

4. Different floats and devices were fabricated according to design and were tested first in the pilot setup and then in field in rural locations.

Experimentation:

I. Working Mechanism of hand pump

- i. The mechanism of hand pump is based on the working principle of a positive displacement pump, negative pressure being the potential behind lifting of water from well to the surface level.
- ii. The cylinder assembly of hand pump consists of cast iron cylinder body, with two caps, and the operating mechanism like plunger yoke body with follower, spacer-upper check valve assembly along with check valve assembly moves up inside cylinder.
- iii. At this point, the lower check valve is closed, thus allowing the water from the well to flow inside cylinder.
- iv. On the return stroke when the handle is moved up, the plunger assembly starts coming down inside the cylinder.
- v. The check valve at the bottom will close thereby forcing the water through the follower upwards opening the upper valve.
- vi. Thus when the plunger

moves up and down in the cylinder; the water is displaced and finds its way to the water tank.

II. Field Procedure

- i. The experimental place was selected in the rural areas where people are prone to water borne diseases due to unavailability of drinking water treatment facility.
- ii. Initially, sample was taken for existing system in 50 ml sample bottle.
- iii. The hand pump was then disassembled using assembling instruments and modified valve was fixed.
- iv. In the beginning, few strokes were operated to remove

rust which was mixed in the bore well water during assembling.

- v. The water was sampled after the removal of rust by visual inspection. Next valve was fixed and same process was repeated for remaining modified valves.
- vi. The experimental evidence is shown in Image 2.
- vii. All samples were kept in chilled sampling bag which is internally coated with thin Teflon sheet to avoid heat losses. Those samples were also surrounded with cool ice packs to maintain low temperature to prevent the growth of the bacteria.
- viii. The analysis of all

sample were done in laboratory within 24 hours of the sample collection.

- ix. Original/parent valve was considered as 100% flow opening valve and based on same 20, 30, 40 and 50% flow opening valves were used during experimentation.

Experimental Observations:

1. Temperature at field location varied from 29 0C to 34 0C
2. Capacity of single pipe: 3 litre
3. Water Bucket Capacity (Avg.): 13 litre (to calculate the water discharge quantity)

System	Strokes/min	Volume of water pumped (lit.)	Theoretical output	Stroke Efficiency
Original	40	11.14	15.827	70.38%
	50	13.68	19.783	69.14%
	60	17.6	23.74	23.73%
Modification	40	9.096	15.827	57.47%
	50	13	19.783	65.71%
	60	19.16	23.74	80.70%

Table 1. Study of Stroke efficiency

Sample Analysis of Institute of Chemical Technology laboratory

The plate count method means diluting bacteria with a diluents solution (e.g. sterile water) until the bacteria are dilute enough to count accurately when spread on a plate. The assumption is that each viable bacterial cell

will develop into a single colony. Bacterial cell numbers need to be reduced by dilution, to be able to count them accurately.

To avoid the contamination and growth of bacteria in the water brought to the lab, the sample bottles were stored at 4° in refrigerators.

Also, TOC analysis was done which gives the concentration of Organic and inorganic carbon present in water. TOC

analysis gives the ultimate idea of reduction in dissolved carbon after disinfection.

The materials needed to perform a plate count are:

- i. 0.9% (w/v) Sodium Chloride solution as diluent, test tubes plugged with cotton, petri dishes and the spreader were used.
- ii. Autoclave, Plate Count Agar, 70% ethanol, Laminar Cabinet, Variable

- 1ml Micro-pipette along with micro-tips and the Incubator were used.
- iii. Two Bunsen burners in a Laminar cabinet and Plate Count Agar, Thermometer, pH meter, Buffer capsules and Fridge were used. 50%, 40%, 30% and 20% opening valves fabricated from workshop.

Procedure to perform a plate count is as follows:

- pH meter was calibrated with the help of Buffer capsules and pH of each sample was measured.
- Viable cell count (spread plate) method was used to calculate the Colonies Forming Unit (CFU) of the potable bore well water.
- Sterilization of all equipment's in the autoclave at 121°C, 15 bar over 15 min were done.
- Plate Count Agar was used as

- media to grow the bacteria.
- Laminar flow chamber is washed with 70% ethanol and UV was switched on for 20 min for the cabinet disinfection.
 - Then all the sterilized samples were kept in laminar flow chamber.
 - The variable 1ml Micro-pipette along with micro-tips was used for the serial dilutions
 - Triplet of Four serial dilutions of each sample were made in between 2 flamed burners i.e. 10, 100, 1000 and 10000 times dilution with Distilled Water.
 - Aliquots were spread, using a sterile bacterial spreader (0.1 ml) of each dilution onto 4 agar plates.
 - Incubated the plates for 24 hr at 37°C as previously mentioned.

- Counting the number of bacterial colonies that appear on each of the plates.
- Those CFU are accounted having colonies in between 30 -300 per petri plate for each sample.
- One blank petri-plate filled with agar (media) was also kept during the analysis to account for contamination during CFU analysis and it showed completely empty (no CFU)

To compute the estimated total plate count on the surface we used the following formula:

$$\text{Total plate count} = N \cdot d$$

where, N = number of colonies counted on plate; d = dilution factor.

Example: N= 56 colonies; d = 1000;

Total plate count = 56000 CFU in 0.1 ml
= 560000 bacteria per ml

Sr. No.	Sample Type	Performance Date	Dilutions (times)				CFU**/0.1 ml of sample
			10	100	1000	10000	
1	Tap	30-04-2015	2	1	0	0	600
	Water						
2	Tap	18-05-2015	5	4	0	C*	2700

*C= Contamination or uncountable, **CFU = Colonies Forming Unit

Table 2: Results of standardization of Plate count (CFU) method

From above results, 100 times dilution is sufficient for the estimation of bacteria (CFU) from tap water collected from Institute of Chemical Technology, Mumbai, hence there is no need of further dilution for tap water.

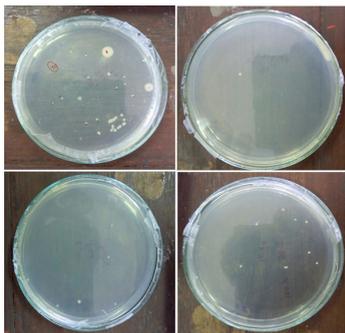


Image 7: CFU/ml Count observed after 48 hours of Incubation

From the growth rate of bacteria and incubation temperature, we can predict that, the bacteria are mesophilic. The higher growth of bacteria occurs at 37 °C, which is related to the mesophilic category while the growth of thermophilic occurs above 45 °C and the psychrophilic grows below 30 °C. The temperature of experimental/trial places supports the prediction. Newly **yellowish** bacterial strain is observed during repeated analysis with the white colonies during the counting

Replica system in the institute premises Trial in ICT Premises



Image 8: Setup site in ICT Premises



Image 9: India Mark II Hand Pump



Image 10: Water Tank with E.coli Culture

Field Trials:



Image 11: Prof A.B. Pandit addressing a brief talk to residents regarding the project details



Image 12: Field Trial Location: A/P Nibavde-415301, Taluka: Aatpadi. Dist.: Sangli, Maharashtra

Bore well Depth: 100 ft.



Image 13: Field Trial Location: A/P Nibavde-415301, Taluka: Aatpadi. Dist.: Sangli,

Maharashtra
Bore well Depth: 40 ft.



Image 15: Field Trial Location: A/P Bhadas Bk-412108, Taluka: Mulshi. Dist.: Pune, Maharashtra



Image 16: Field Trial Location: A/P Khudus- 413113, Tal. Malshiras, Dist. Solapur, Maharashtra (Bore well Depth: 60 ft.)



Image 17: Field Trial Location: A/P Zanjewadi- 413113, Tal. Malshiras, Dist. Solapur, Maharashtra (Bore well Depth: 90 ft.)

Collecting water after installation of modified valve with calibrated strokes



Image 14: Water received immediately after installation (showing mud, rust, etc.)

Water received after some time

RESULTS OF PILOT SETUP IN ICT PREMISES (05/07/2016):

Sample	Strokes per min	CFU/ml	Percent disinfection
Original	-	113000	0
Modification 1	40	19050	83.142
	50	1450	98.717
	60	3475	96.925
Modification 6	40	11000	90.265
	50	10100	91.062
	60	7500	93.363

Table 3: No. of strokes vs % Disinfection for pilot setup

Sample	10	100	1000	10000	100000	1000000	1E+07	1E+08	CFU/ml	Percentage Reduction
Original	C*	C*	0	1	15	0	C*	0	320000	0
	82	13	0	32	0	C*	0	0		
	C*	4	1	17	C*	0	0	0		
40 strokes	46	C*	6	1	1	0	0	0	3333.33	98.96
	C*	4	1	0	C*	1	0	0		
	55	4	26	0	0	0	0	0		

Sample	10	100	1000	10000	100000	1000000	1E+07	1E+08	CFU/ml	Percentage Reduction
50 strokes	C*	C*	C*	C*	0	C*	0	0	10000	96.87
	58	3	0	C*	0	2	0	0		
	45	C*	C*	1	0	C*	C*	0		
60 strokes	193	6	0	0	6	C*	0	0	20000	93.75
	75	C*	1	4	1	0	0	0		
	C*	20	6	C*	C*	4	C*	0		

Table 4: A typical Plate Count Analysis (CFU/ 0.1 ml, after 24 hr)

Results from Microbiology Laboratory, Nashik, India:

Sr. No	Sample ID	Bio Number	Analysis time, hr	Probability	Selected Organism
1	ICT/15/3B	014010302000000	8.25	93%	Kocuria rhizophilia
2	ICT/15/3A	014010302000000	8.00	93%	Kocuria rhizophilia
3	ICT/15/5A	000010100000000	6.00	99%	Kocuria rosea
4	ICT/15/5C	0252511565453220	14.25	97%	Bacillus megaterium
5	ICT/15/5D	041032310000000	5.00	98%	Micrococcus luteus
6	ICT/15/5B	1070171515647220	14.25	-	*Bacillus subtilis/ amyloliquefaciens/ atrophaeus

Table 5: The bacterial identification of treated water

Note: The 6th bacteria (ICT/15/5B) come under Bacillus category having 3 subtypes. To confirm the exact category, the additional (Inulin) test is necessary.

Detail of 6th bacterial test:

1. Bacillus subtilis: INULIN(11), Cell Chains(84)
2. Bacillus amyloliquefaciens: Cell Chains(20)
3. Bacillus atrophaeus: INULIN(83), Cell Chains(23)

Bacterial Identification Automated Antibiotic susceptibility testing (VITEK-2) clearly showed that the opportunistic pathogens (Kocuria, Bacillus, Micrococcus, etc.) were successfully killed

Results from Enviro Care Lab, Mumbai, India (05/07/2016):

Sr. No.	Sample	Incubation	Dilutions	Set I	Set II
1	Raw Water Treatment)	37 ± 1 °C	Undiluted	TNTC	TNTC
			10-Jan	TNTC	TNTC
			10-Feb	TNTC	TNTC
			10-Mar	TNTC	TNTC
			10-Apr	TNTC	TNTC
			10-May	TNTC	TNTC
			10-Jun	TNTC	TNTC
			10-Jul	TNTC	TNTC
			10-Aug	350	370
Total Count				3.6 × 10 ⁹ CFU/ml	
2	Treated Water (After	37 ± 1 °C	Undiluted	TNTC	TNTC
			10 Jan	TNTC	TNTC
			10-Feb	TNTC	TNTC
			10-Mar	TNTC	TNTC
			10-Apr	TNTC	TNTC
			10-May	41	48
			10-Jun	4	9
Total Count				4.4 × 10 ⁹ CFU/ml	

Table 6: Total Bacterial count in Raw and Treated water samples

(*TNTC = Too Numerous to Count)

Calculation:

% Reduction (Efficacy) = $\frac{\text{Count in Raw water sample} - \text{Count in treated water sample}}{\text{Count in Raw water sample}} \times 100$

$$= \frac{3.6 \times 10^9 - 4.4 \times 10^6}{3.6 \times 10^9}$$

$$= 99.87 \%$$

Conclusions: The bacterial reduction is in the range of 99.87 %.

Results from the Field Trials:

Sr. No.	% Opening of Valve	pH	Thermal Conductivity K(μS/cm)	TOC(ppm)
1	100	9.01	630	0.3708
2	50	9.07	940	0.363
3	40	9.3	490	0.3873
4	30	9.42	730	0.0546
5	20	9.18	910	0.0686

Table 7: pH and Thermal Conductivity measurement for First Trial

Trial No.	Location of field trial	System	% Disinfection
1	Khudus, Dist.-Solapur	M2	80.08
		M3	79.96
		M4	94.39
		M5	91.49
2	Malshiras, Dist.-Solapur	M2	90.91
		M3	98.78
		M4	98.63
		M5	89.54
3	Pandharpur, Dist.- Solapur	M2	97.73
		M3	95.85
		M4	70.84
		M5	98.96
4	Nimbawade, Dist.-Sangli	M2	97.28
		M3	99.81
		M4	93.23
		M5	98.36

Table 9: Percentage Disinfection observed at different rural locations

M1, M2, M3, M4, M5 are different geometric modification experimented with

System	Strokes/min	CFU/ml	% Disinfection	TOC, ppm	V actual m/s	% Stroke efficiency
M1	40	450000	0 (Ref.)	132.7	0.31	86.45
	50	420000	6.67	121.06	0.45	99.63
	60	380000	15.56	122.64	0.53	97.16
M2	40	300000	33.34	131.16	0.62	99.37
	50	20000	95.56	136.5	0.76	96.98
	60	27000	94	130.98	0.92	97.16
M3	40	80000	82.23	122.64	0.71	96.06
	50	340000	24.45	129	0.86	93.27
	60	135000	70	123.76	1.04	94.29
M4	40	180000	60	128.84	0.87	98.04
	50	310000	31.12	129.88	1.1	99.1
	60	18000	96	129.16	1.22	91.42
M5	40	43000	90.45	143.44	1.03	92.08
	50	24000	94.67	136.88	1.23	87.71
	60	31000	93.12	117.02	1.45	86.34

Table 10: Variation of Different Parameters observed at different modifications M1, M2, M3, M4, M5 are different geometric modification experimented with

Samples	Strokes per min	Dilutions				CFU/ml	% Disinfection	Average % Disinfection
		10	100	1000	10000			
Original		C*	C*	113	2	113000	-	-
M7	50	C*	101	19	0	10100	91.0619	91.0619
		C*	103	10	1	10300	90.8850	
		C*	99	13	3	9900	91.2389	

Table 11. Percentage disinfection observed at a remote location A/P Bhadas Village, Taluka Mulshi, District Pune, Maharashtra, where M7 is a geometric modification experimented with. (23rd December 2016)

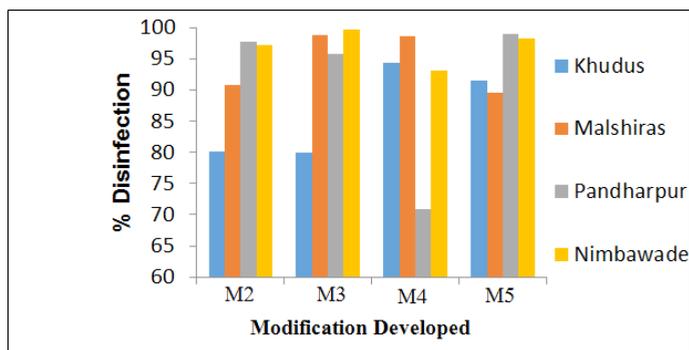


Image 18: Percentage Disinfection observed at different rural locations

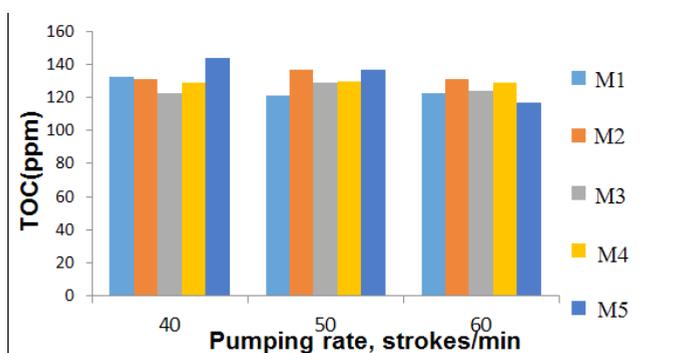


Image 19: Variation in TOC at different Modifications

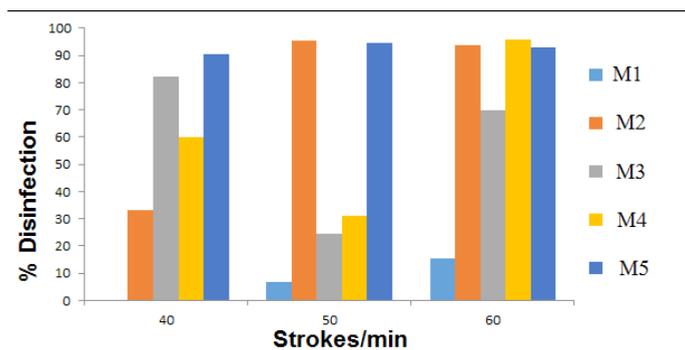


Image 20: No. of stokes Vs percent disinfection

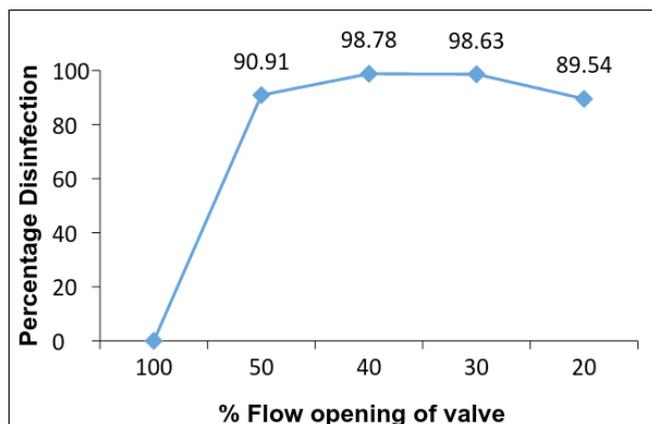


Image 21: Effect of check valve opening on percentage disinfection

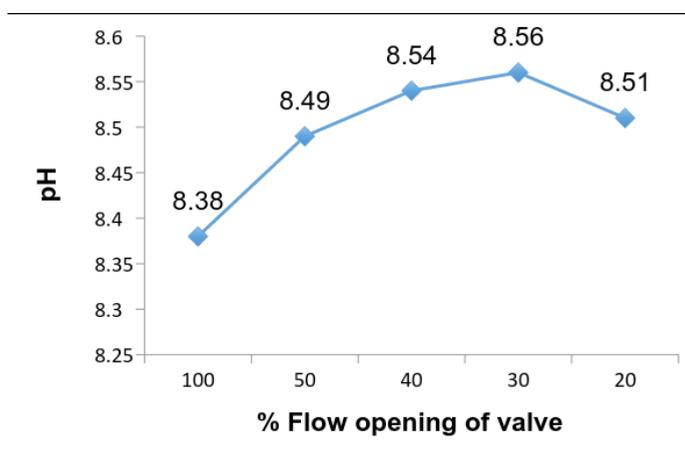


Image 22: Effect of check valve opening on pH

Concluding Remarks:

Cavitation is occurring in the modified device of the India Mark II hand pump cylinder. Present analysis is not sufficient to predict the effect of design parameters of the device on cavitation number. Also present analysis does not discuss the effect of nuclei concentration on cavitation number. So it is necessary to develop a CFD model combined with bubble dynamic model to predict the

effect of design parameters and nuclei concentration on cavitation number

The report indicates a successful implementation of the field trials using modified India Mark II Hand Pump. The optimised design operated in the field showed 93 to 98 % (two log reduction) disinfection, whereas laboratory controlled trials could achieve 99.84 % (3 log reduction) disinfection, making the water adequately potable.

Acknowledgement:

The researcher would like to acknowledge the funding organisation “TEQIP Process Intensification II” for carrying out this work. We would also like to thank all officials involved from accounts as well as from administration department for all the support. We would also like to thank chemical engineering office for all the cooperation offered to us during this course of period.

WORKSHOP ON “VALUE ADDITION TO HANDLOOM PRODUCTS”

Workshop on “VALUE ADDITION TO HANDLOOM PRODUCTS” was held from 14th -17th September, 2016, in Textile Department of ICT. The convener for the workshop was Dr Ravindra D Kale and Co Convener was Dr Amit Pratap. The purpose of this workshop was to educate and teach participants, skill of dyeing and printing with various techniques and using different tools in a way which they can implement in their home state. The target group were Handloom weavers and did not have any idea about textile colouration who outsource the dyeing and printing work. Since they were producing different products on the loom, the dyeing and printing has to be done with an artistic view to enhance the saleability of their products. To achieve this we roped in one expert Ms Ritika Jhunjunwala, Teacher at Sophiya college and her four students having experience in imparting such kind of training.

She and her team trained these people in simple and interesting manner. Different printing effects, design and patterns were created using by using tie and dye & block printing method. The participants were taken to one block printing unit at Kurla for half day where it is done on commercial scale to encourage them further. After the workshop was concluded, the participants were allowed to carry with them few printing blocks and colours & chemicals.



WORKSHOP ON PROCESS INTENSIFICATION

Process Intensification Workshop 2016, a two-day workshop was mutually organized by Institute of Chemical Technology (ICT) in association with Centre of Excellence Process Intensification II (COEPI) on 9th and 10th September 2016. The motto of the workshop was to bring research scholars across India on a common platform to share the emerging technologies and research progress on process intensification. The research areas covered were the novel techniques utilized for process intensification particularly

those concerned with themes such as equipment & plant miniaturization, alternative energy conversion & transport mechanisms, intensified hydrodynamics and intensified plant operation. The workshop comprised of around 130 students and researchers from various institutes across India. The workshop witnessed the presence of eminent personalities from academia in the area of process intensification. Keynote lectures were delivered by Prof. Sunil Bhagwat - Coordinator of COEPI TEQIP-II, Dr. Deepak

Palekar-Step India Pvt Ltd, Prof. V Moholkar - IIT Guwahati, Prof. P.K Ghosh- ICT, Mumbai, Prof. Juvekar- IIT Mumbai, Prof. V.G Gaikar- Vice Chancellor, BATU, Maharashtra and Prof. A.B Pandit - ICT, Mumbai. The participants were shown various equipments used in the area of process intensification like microwave extractor, ultrasonic bath, horn and continuous flow cell reactor and glass micro-reactor.

The workshop showed high level of engagement from all those present.



VARIOUS FACILITY CREATED UNDER COE-PI, TEQIP-II

Air Cooled Electrical Chiller: (Prof. S. S. Bhagwat)



Electric chiller is a machine that removes heat from a liquid via a vapor-compression. This liquid can then be circulated through a heat exchanger to cool equipment, or another process stream (such as air or process water). Electric Chillers are used extensively for large facility space cooling and in industrial process liquid cooling. Electric Chiller reduces energy usage without affecting comfort or production.

Experimental Setup for Micro hydro power Production (At Post: Kanher, Tal- Malshiras, Dist- Solapur) (Dr. D. V. Pinjari)



High Pressure Stirred Autoclave (Prof. S. S. Bhagwat)



High Pressure Stirring Autoclave is used to determine the Vapor liquid Equilibrium of solutions at specific temperature.

Design Expert Software V-10: (Dr. C. S. Mathpati)



It is a software as a tool for design of equipment. It improves the process by doing excellent design. It not only screen for vital factors, but also locate ideal process settings for top performance and discover optimal product formulations.

Hypermesh Software: (Dr. C. S. Mathpati)



Altair

HyperWorks

It is a software which is use

for discretization i.e. meshing of multiple geometries which is later applicable for CFD simulation.

Optical Microscope (1000X) with cadiod condenser (dark field Image) & Polarizer: (Prof. S. S. Bhagwat)



Optical microscopy is used extensively in microelectronics, nanophysics, biotechnology, pharmaceuticals research, mineralogy and microbiology. Optical Microscope is used to magnify biological samples. They can be used to view specimens in colour.

Membrane Set-up – I: (Prof. S. S. Bhagwat)



The ultrafiltration setup is a dead end filtration setup. Equipped with a provision for pressurising using Nitrogen,

using membranes of different sizes we can achieve filtration of different materials.

Laminar Air Flow: (Prof. V. K. Rathod)



It is purchased from labard instruchem Pvt. Ltd. It is an enclosed bench designed to prevent contamination of semiconductor wafers, biological samples, or any particle sensitive materials.

Air is drawn through HEPA filter and blown in a very smooth, laminar flow towards the user. The cabinet is usually

made of stainless steel with no gaps or joints

Air Compressor: (Dr. P. D. Vaidya)



This equipment is widely used to provide a constant supply of air wherein the electric motor power is converted to potential energy stored in pressurized air. We have the oil-free compressor designed to deliver better quality air.

Computing Workstation: (Prof. N. Sekar)



Rotovac: (Prof. V. K. Rathod)



Rotovac was purchased from scientific sales syndicate. It is a device used in chemical laboratories for the efficient and gentle removal of solvents from samples by evaporation under reduced pressure.

AWARDS:

Won best presentation award at “CoE, TEQIP-II - Conclave for Research Excellence through Collaboration” organized by The Centre of Excellence (CoE), College of Technology, GB Pant University of Agriculture & Technology, Uttarakhand held on October 06-08, 2016.



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Team ICT wins Cash Prize of Rs. 10lakh in University Challenge of IIGP 2.0

A team of research students from ICT under guidance of Prof. A.B. Pandit won the university challenge of India

Innovation Growth Program 2.0 (IIGP 2.0). They were awarded with a cash prize of INR 10 lakh as a research grant, at the award ceremony held at Hotel Taj Mahal, New Delhi on 26th July 2017.

About the Program:

The India Innovation Growth Program 2.0 (IIGP 2.0) made up of Tata Trusts along with founding stakeholders - the Department of Science and Technology (DST) and global security and aerospace company Lockheed Martin, displays a renewed focus on innovations addressing socio-economic challenges. The newly enhanced program is joined by new partners, Massachusetts Institute of Technology, Indian Institute of Management at Ahmedabad and Indian Institute of Technology Bombay, in addition to the on-going support and partnership from FICCI and IUSSTF.

Over 350 applications for innovative ideas across social and industrial sectors were received for the University challenge of the program. From these applications, 30 teams were invited to present their innovative ideas in Innovators Tank Challenge at IIT Bombay on June 24th. Top 9 teams were selected from the innovators tank as winners of Phase A of the program. The winning teams will also receive mentoring from experts for the next one year along with a cash prize worth INR 10 lakh each. The phase B of the competition will take place next year.

Dignitaries attending the IIGP 2.0 award ceremony:

The award ceremony organized by FICCI was attended by following dignitaries:

- **Dr. Harsh Vardhan**, Hon'ble Union Minister for Department of Science and Technology, Government of India
- **Mr. Y.S. Chowdary**, Hon'ble Minister of State for Department of Science and Technology, Government of India
- **H.E. Ms. MaryKay Loss Carlson**, Chargé d'Affaires, U.S. Embassy
- **Padmashree Mr. V.R. Mehta**, Trustee, Tata Trusts
- **Dr. Dana (Keoki) Jackson**, Chief Technology Officer, Lockheed Martin Corporation
- **Dr. A. Didar Singh**, Secretary General, FICCI
- **Dr. Robie Samanta Roy**, Vice President, Technology and Innovation at Lockheed Martin Corporation
- **Mr. Kamesh Gupta**, Sr. Vice President - Strategic Collaborations and Programmes, Group Technology & Innovation Office, Tata Sons
- **Mr. Harkesh Mittal**, Adviser, Member Secretary National Science & Technology Entrepreneurship Development Board (NSTEDB), Department of Science and Technology, Government of India

- **Prof. Ashutosh Sharma**, Secretary, Department of Science and Technology, Government of India
- **Dr. Rajiv Tayal**, Executive Director, Indo-US Science and Technology Forum

Names and Details of Team Members:

Mentor: Prof. A. B. Pandit

Students:

Mr. Sarjerao Doltade, Ph.D. (Tech.), Chemical Engineering,

Mr. Gaurav Dastane, Ph.D. (Tech.), Chemical Engineering,

Mr. Mayur Ladole, Ph.D. (Tech.), Chemical Engineering,

Mr. Nilesh Jadhav, Ph.D. (Sci.), Chemical Engineering,

Details of Innovation presented by Team ICT:

A significant portion of the world population relies on ground water for their daily

needs. As there are no affordable purification techniques available for the poor people, ground water consumption poses as a big health concern for them. It can be an easy gateway for spreading water borne diseases.

Hand pumps are usually the preferred device used in the villages of India to access the ground water. When the ground water is pumped to the surface using a hand pump, it passes through a check valve. When the water flows across the check valve, its pressure drops locally due to the smaller cross-sectional area available for the flow. This check valve can be effectively modified so that pressure reduces close to the vapor pressure of water in order to generate cavitation. Cavities generated in such a way will collapse downstream once the pressure recovers to a higher value. When the cavities collapse, they generate high amount of energy in the form of

local temperature and pressure. There is also a liquid jet formed due to collapse of cavity. This process is proven to be useful in microbial disinfection of water. This idea can also be extended to add multiple such cavitating devices in series inside the hand pump to ensure maximum disinfection.

This unique and novel technology is already validated by conducting field trials and is found to have yielded more than 95% disinfection with the modified check valve. Further disinfection is possible with addition of another cavitating device in series. This technology can potentially help people from rural areas across the world to access quality potable water and reduce the spread of water borne diseases. Hand pump cavitation can prove to be a milestone in emerging era of novel and economical water disinfection systems.

TEAM ICT BEING PRESENTED WITH THE CHEQUE

