SONOPANT DANDEKAR ARTS, V.S. APTE COMMERCE AND M.H. MEHTA SCIENCE COLLEGE, PALGHAR

Department of Chemistry

PROJECT REPORT

M.Sc. Organic Chemistry

Academic Year 2022-2023

Prepared by
Department of Chemistry
Sonopant Dandekar Arts, V.S. Apte Commerce and M.H. Mehta Science College, Palghar

INDEX

Sr. No.	Content
1	Notice for Project Submission
2	Curriculum where course (subject where project work/ field work is required)
3	List Learners with Project titles (Sem-VI)
4	Sample Projects

Sonopant Dandekar Shikshan Mandali's
Sonopant Dandekar Arts,
V. S. Apte Commerce &
M. H. Mehta Science College, Palghar
Estb.: 14 August 1968
Dr. Kiran Save, Principal

Kharekuran Road, Palghar (W), Tal. & Dist. Palghar,
Maharashtra - 401 404, INDIA
Tel. : +91 - 2525 - 252163
Principal : +91 - 2525 - 252317
Email : sdsmcollege@yahoo.com
Web. : www.sdsmcollege.com

Ref No.:

Date : 12/06/2023

Notice

Department of Chemistry

This is to inform you that all the **Master of Science (Organic Chemistry)** students are required to submit the hard copy of your final project report by **19th June 2023.** All submissions should be made to the **Chemistry Department** during office hours from 09.30 am to 02.00 pm. Ensure your report is properly computer typed.

Head of the Department
Department of Chemistry

S.D.S.M. COLLEGE PALGHAR DIST PALGHAR

Dr. Kiran J. Save
Principal

PRINCIPAL
Sonopant Dandekar Arts College,
V.S. Apte Commerce College &
M.H. Mehta Science College
PALGHAR (W.R.)
Dist. Palghar, Pin-401404

AC – 14/06/2018

Item No. 4.71

UNIVERSITY OF MUMBAI

Program : M.Sc.

(Choice Based Credit System)

Course : M.Sc. Organic Chemistry

Part – I

Syllabus for Semester III & IV

(To be implemented from the Academic year 2018-2019)

M.Sc. Organic Chemistry

Semester – III
Course Code: PSCHO301
Paper - I (**Theoretical organic chemistry-I**)

Unit 1	**Organic reaction mechanisms**	**[15L]**
1.1	Organic reactive intermediates, methods of generation, structure, stability and important reactions involving carbocations, nitrenes, carbenes, arynes and ketenes**.**	**[5L]**
1.2	Neighbouring group participation**:** Mechanism and effects of anchimeric assistance, NGP by unshared/ lone pair electrons, π-electrons, aromatic rings, σ-bonds with special reference to norbornyl and **bicyclo[2.2.2]octyl cation** systems (formation of non-classical carbocation)	**[3L]**
1.3	Role of FMOs in organic reactivity**:** Reactions involving hard and soft electrophiles and nucleophiles, ambident nucleophiles, ambident electrophiles, the α effect.	**[2L]**
1.4	Pericyclic reactions: Classification of pericyclic reactions; thermal and photochemical reactions. Three approaches: Evidence for the concertedness of bond making and breaking Symmetry-Allowed and Symmetry-Forbidden Reactions – • The Woodward-Hoffmann Rules-Class by Class • The generalised Woodward-Hoffmann Rule Explanations for Woodward-Hoffmann Rules • The Aromatic Transition structures [Huckel and Mobius] • Frontier Orbitals • Correlation Diagrams, FMO and PMO approach Molecular orbital symmetry, Frontier orbital of ethylene, 1,3 butadiene, 1,3,5 hexatriene and allyl system.	**[5L]**
Unit 2	**Pericyclic reactions**	**[15L]**
2.1	Cycloaddition reactions: Supra and antra facial additions, 4n and 4n+2 systems, 2+2 additions of ketenes. Diels-Alder reactions, 1, 3-Dipolar cycloaddition and cheletropic reactions, ene reaction, retro-Diels-Alder reaction, regioselectivity, periselectivity, torquoselectivity, site selectivity and effect of substituents in Diels-Alder reactions. **Other Cycloaddition Reactions-** [4+6] Cycloadditions, Ketene Cycloaddition, Allene Cycloadditions, Carbene Cycloaddition, Epoxidation and Related Cycloadditions. Other Pericyclic reactions: Sigmatropic Rearrangements, Electrocyclic Reactions, Alder 'Ene' Reactions.	**[7L]**
2.2	Electrocyclic reactions: Conrotatory and disrotatary motions, 4nπ and (4n+2)π electron and allyl systems.	**[3L]**
2.3	Sigmatropic rearrangements: H-shifts and C-shifts, supra and antarafacial migrations, retention and inversion of configurations. Cope (including oxy-Cope and aza-Cope) and Claisen rearrangements. Formation of Vitamin D from 7-dehydrocholesterol, synthesis of citral using pericyclic reaction, conversion of Endiandric acid E to Endiandric acid A.	**[5L]**

Unit 3:	**Stereochemistry-I**	**[15L]**
3.1	Classification of point groups based on symmetry elements with examples (nonmathematical treatment).	**[2L]**
3.2	Conformational analysis of medium rings: Eight **to** ten membered rings and their unusual properties**,** I-strain, transannular reactions.	**[3L]**
3.3	Stereochemistry of fused ring and bridged ring compounds: decalins, hydrindanes, **perhydroanthracenes**, steroids, and Bredt's rule.	**[5L]**
3.4	**Anancomeric systems,** Effect of conformation on reactivity of cyclohexane derivatives in the following reactions (including mechanism): electrophilic addition, elimination, molecular rearrangements, reduction of cyclohexanones **(with $LiAlH_4$, selectride and MPV reduction)** and oxidation of cyclohexanols**.**	**[5L]**

Unit 4	**Photochemistry**	**[15L]**
4.1	Principles of photochemistry: quantum yield, electronic states and transitions, selection rules, modes of dissipation of energy (Jablonski diagram), electronic energy transfer: photosensitization and quenching process.	**[3L]**
4.2	Photochemistry of carbonyl compounds:$\pi \rightarrow \pi^*$, $n \rightarrow \pi^*$ transitions, Norrish- I and Norrish-II cleavages, Paterno-Buchi reaction. Photoreduction, calculation of quantum yield, photochemistry of enones, photochemical rearrangements of α, β-unsaturated ketones and cyclohexadienones. Photo Fries rearrangement, Barton reaction.	**[8L]**
4.3	Photochemistry of olefins: cis-trans isomerizations, dimerizations, hydrogen abstraction, addition and Di- π- methane rearrangement including aza-di- π -methane. Photochemical Cross-Coupling of Alkenes, Photodimerisation of alkenes.	**[2L]**
4.4	Photochemistry of arenes**:** 1, 2- , 1, 3- and 1, 4- additions. Photocycloadditions of aromatic Rings.	**[1L]**
4.5	Singlet oxygen and photo-oxygenation reactions. Photochemically induced Radical Reactions. Chemiluminescence.	**[1L]**

REFERENCES:

1 March's Advanced Organic Chemistry, Jerry March, sixth edition, 2007, John Wiley and sons.
2 A guide to mechanism in Organic Chemistry, 6th edition, 2009, Peter Sykes, Pearson education, New Delhi.
3 Advanced Organic Chemistry: Reaction Mechanisms, R. Bruckner, Academic Press (2002).
4 Mechanism and theory in Organic Chemistry, T. H. Lowry and K. C. Richardson, Harper and Row.
5 Organic Reaction Mechanism, 4th edition, V. K. Ahluvalia, R. K. Parashar, Narosa Publication.
6 Reaction Mechanism in Organic Chemistry, S.M. Mukherji, S.P. Singh, Macmillan Publishers, India.
7 Organic Chemistry, Part A and B, Fifth edition,2007, Francis A.

B.S.Furniss, A. J.Hannaford, P. W. G. Smith, A. R. Tatchell, Pearson Education.
11. Laboratory Manual of Organic Chemistry, Fifth edition, R K Bansal, New Age Publishers.
12. Organic structures from spectra, L. D. Field, S. Sternhell, John R. Kalman, Wiley, 4th ed., 2011.

1. The candidate is expected to submit a journal and project certified by the Head of the Department /institution at the time of the practical examination.
2. A candidate will not be allowed to appear for the practical examination unless he/she produces a certified journal or a certificate from the Head of the institution/department stating that the journal is lost and the candidate has performed the required number of experiments satisfactorily. The list of the experiments performed by the candidate should be attached with such certificate.
3. Use of non-programmable calculator is allowed both at the theory and the practical examination.

Semester – IV
Course Code: PSCHO401
Paper - I (**Theoretical organic chemistry-II**)

Unit 1:	**Physical organic chemistry**	**[15L]**
1.1	Structural effects and reactivity**:** Linear free energy relationship (LFER) in determination of organic reaction mechanism, The Hammett equation, substituent constants, theories of substituent effects, interpretation of σ-values, reaction constants ρ, Yukawa-Tsuno equation**.**	**[7L]**
1.2	Uses of Hammett equation, deviations from Hammett equation. Dual parameter correlations, Inductive substituent constants. The Taft model, σ_I and σ_R scales, steric parameters Es and β. Solvent effects, Okamoto-Brown equation, Swain-Scott equation, Edward and Ritchie correlations, Grunwald-Winstein equation, Dimroth's E_T parameter, Solvatochromism Z-scale, Spectroscopic Correlations, Thermodynamic Implications.	**[8L]**
Unit 2	**Supramolecular chemistry**	**[15L]**
2.1	Principles of molecular associations and organizations as exemplified in biological macromolecules like nucleic acids, proteins and enzymes.	**[3L]**
2.2	Synthetic molecular receptors: receptors with molecular cleft, molecular tweezers, receptors with multiple hydrogen sites.	**[3L]**
2.3	Structures and properties of crown ethers, cryptands, cyclophanes, calixarenes, rotaxanes and cyclodextrins. Synthesis of crown ethers, cryptands and calixarenes.	**[5L]**
2.4	Molecular recognition and catalysis, molecular self-assembly.Supramolecular Polymers, Gelsand Fibres.	**[4L]**
Unit 3	**Stereochemistry- II**	**[15L]**

3.1	Racemisation and resolution of racemates including conglomerates: Mechanism of racemisation, methods of resolution: mechanical, chemical, kinetic and equilibrium asymmetric transformation and through inclusion compounds.	**[3L]**
3.2	Determination of enantiomer and diastereomer composition: enzymatic method, chromatographic methods. Methods based on NMR spectroscopy: use of chiral derivatising agents (CDA), chiral solvating agents (CSA) and Lanthanide shift reagents (LSR).	**[3L]**
3.3	Correlative method for configurational assignment: chemical, optical rotation, and NMR spectroscopy.	**[4L]**
3.4	Molecular dissymmetry and chiroptical properties: Linearly and circularly polarized light. Circular birefringence and circular dichroism. ORD and CD curves. Cotton effect and its applications. The octant rule and the axial α–haloketone rule with applications.	**[5L]**
Unit 4:	**Asymmetric synthesis**	**[15L]**
4.1	Principles of asymmetric synthesis: Introduction, the chiral pool in Nature, methods of asymmetric induction – substrate, reagent and catalyst controlled reactions.	**[3L]**
4.2	Synthesis of L-DOPA [Knowles's Mosanto process]. Asymmetric reactions with mechanism: Aldol and related reactions, Cram's rule, Felkin-Anh model, Sharpless enantioselective epoxidation, hydroxylation, aminohydroxylation, Diels-Alder reaction, reduction of prochiral carbonyl compounds and olefins.	**[9L]**
4.3	Use of chiral auxiliaries in diastereoselective reductions, asymmetric amplification. Use of chiral BINOLs, BINAPs and chiral oxazolines asymmetric transformations.	**[3L]**

<u>REFERENCES:</u>

1 March's Advanced Organic Chemistry, Jerry March, sixth edition, 2007, John Wiley and sons.
2 A guide to mechanism in Organic Chemistry, 6th edition, 2009, Peter Sykes, Pearson education, New Delhi.
3 Advanced Organic Chemistry: Reaction Mechanisms, R. Bruckner, Academic Press (2002).
4 Mechanism and theory in Organic Chemistry, T. H. Lowry and K. C. Richardson, Harper and Row.
5 Organic Reaction Mechanism, 4th edition, V. K. Ahluvalia, R. K. Parashar, Narosa Publication.
6 Reaction Mechanism in Organic Chemistry, S.M. Mukherji, S.P. Singh, Macmillan Publishers, India.
7 Organic Chemistry, Part A and B, Fifth edition,2007, Francis A. Carey and Richard J. Sundberg, Springer.
8 Carbenes, Nitrenes and Arynes. Von T. L. Gilchrist, C. W. Rees. Th. Nelson and Sons Ltd., London 1969.
9 Organic reactive intermediates, Samuel P. MacManus, Academic Press.

SCIENTIFIC PAPERS

Reporting practical and project work, Writing literature surveys and reviews, organizing a poster display, giving an oral presentation.

Writing Scientific Papers:

Justification for scientific contributions, bibliography, description of methods, conclusions, the need for illustration, style, publications of scientific work, writing ethics, avoiding plagiarism.

Unit IV: CHEMICAL SAFETY & ETHICAL HANDLING OF CHEMICALS [15L]

Safe working procedure and protective environment, protective apparel, emergency procedure, first aid, laboratory ventilation, safe storage and use of hazardous chemicals, procedure for working with substances that pose hazards, flammable or explosive hazards, procedures for working with gases at pressures above or below atmospheric pressur, safe storage and disposal of waste chemicals, recovery, recycling and reuse of laboratory chemicals, procedure for laboratory disposal of explosives, identification, verification and segregation of laboratory waste, disposal of chemicals in the sanitary sewer system, incineration and transportation of hazardous chemicals.

REFERENCES:

1. Dean, J. R., Jones, A. M., Holmes, D., Reed, R., Weyers, J., & Jones, A., (2011), *Practical skills in Chemistry,* 2nd Ed., Prentice Hall, Harlow.
2. Hibbert, D. B. & Gooding, J. J. (2006) *Data Analysis for Chemistry* Oxford University Press.
3. Topping, J., (1984) *Errors of Observation and their Treatment* 4th Ed., Chapman Hill, London.
4. Harris, D. C. (2007) *Quantative Chemical Analysis* 6th Ed., Freeman Chapters 3-5
5. Levie, R. De. (2001) *How to use Excel in Analytical Chemistryand in general scientific data analysis* Cambridge Universty Press.
6. Chemical Safety matters – IUPAC-IPCS, (1992) Cambridge University Press.
7. OSU Safety manual 1.01

Semester IV: Practicals
Course code: PSCHO4P1

Two steps preparations

1. Acetophenone → Acetophenone phenyl hydrazine → 2-phenyl

7. Macro-scale and Micro-scale Organic Experiments, K. L. Williamson, D. C. Heath.
8. Systematic Qualitative Organic Analysis, H. Middleton, Adward Arnold.
9. Handbook of Organic Analysis- Qualitative and Quantitative, H. Clark, Adward Arnold.
10. Vogel's Textbook of Practical Organic Chemistry, Fifth edition,2008, B.S.Furniss, A. J.Hannaford, P. W. G. Smith, A. R. Tatchell, Pearson Education.
11. Laboratory Manual of Organic Chemistry, Fifth edition, R K Bansal, New Age Publishers.
12. Organic structures from spectra, L. D. Field, S. Sternhell, John R. Kalman, Wiley, 4th ed., 2011.

1. The candidate is expected to submit a journal and project certified by the Head of the Department /institution at the time of the practical examination.
2. A candidate will not be allowed to appear for the practical examination unless he/she produces a certified journal or a certificate from the Head of the institution/department stating that the journal is lost and the candidate has performed the required number of experiments satisfactorily. The list of the experiments performed by the candidate should be attached with such certificate.
3. Use of non-programmable calculator is allowed both at the theory and the practical examination.

Sonopant Dandekar Arts, V.S. Apte Commerece and M.H. Mehta Science College, Palgahr

Class : MSc Part II Organic Chemistry

Academic Year : 2022-23

Research Projects

Sr. No.	Roll No.	Name of Students	Project Name	signature
1	601	Akshada Damu Gond	Test for Carbohydrates, Protein and Fats in Foodstuffs.	Akshada
2	602	Swapnali Sanjay Dodiya	Synthesis of 4-Nitro-Nbenzylidene-3-nitroaniline.	Swapnil
3	603	Asmita Mahadev Sawant	Green approach to sythesis dihydropyrimidone via Cytisus Scoparius	Asmita
4	604	Rohit Rajendra Suru	Preparation of oximes of aromatic aldehyde and ketone	Rsuru
5	605	Bipin Jagadish Pandye	Green synthesis of Schiff Base by using Natural Acid Catalysts.	BPandye
6	606	Gopan Gokul Nair	One Pot Synthesis of Aspirin Without Using Acetic Anhydride	Gopan
7	607	Krushik Sachin Save	Synthesis and Characterisation of Schiff Base of Salicyladehyde with Nickel Metal Complex	Krushik
8	608	Sumit Manoj Gupta	One Pot Synthesis of 1,3,4- Thiadiazole	Sumit
9	609	Prajwal Bhaurao Tandel	Preparation of tertiary butyl chloride SN1 reaction and preparation of ethyl bromide SN2 reaction	Prajwal
10	610	Akshay Milind Dalvi	Preparation of Azo dyes from aniline.	Akshay
11	611	Aditi anant Naik	Test for Carbohydrates, Protein and Fats in Foodstuffs.	Aditi
12	612	Bhakti Shyam Sankhe	Synthesis of azo dye from acacia cathechu and its application on silk fibres.	Bhakti

Sonopant Dandekar Arts, V.S. Apte Commerece and M.H. Mehta Science College, Palgahr

13	613	Gauri Narendra Dhanmeher	Antimicrobial activity of natural deep eutectic solvent of eugenol and benzyl triethyl Ammonium Chloride	Ghavathe.
14	614	Aniket Rajendra Patil	Synthesis and biological properties of eugenol-cetylpyidium chloride based Deep Eutectic Solvent	Ghavathe.
15	615	Aditya Vakesh Patil	Depolymerization of Waste PU Foam with DEA Catalyst.	A.V.patil.
16	616	Amol Ananta Punjari	Synthesis and Characterisation of Schiff Base of Salicyladehyde and Ethylene diamine with Magnesium metal complex.	A.A.punjari.
17	617	Manasi Suresh Jadhav	Test For Carbohydrates, Fats and Proteins.	Jadhav
18	618	Govinda Sandipan Gavhane	Preparation of phenol and chlorobenzene from aniline	G.S.Gavhane
19	619	Aniket Ramesh Mali	Synthesis and antibacterial evaluation of heterocyclic compound containing amino group	A.Mali
20	620	Amisha Ajit Machi	Synthesis of azo dye from acacia cathechu and its application on silk fibres.	Amachi
21	621	Aniket Tukaram Savant	Glycolysis of waste Polyster Polyurethane Foam by Chemical Recycling method	ATsavant
22	622	Vishva Purushottam Ahir	Synthesis of azo dye from acacia cathechu and its application on silk fibres.	[illegible]
23	623	Bhagyashree Ramesh Dongare	Degradation process for waste Polyurethane Foam with DEA Catalyst.	[illegible]
24	624	Nikhil Ravindra Chakravorti	Synthesis of azo dye from acacia cathechu and its application on silk fibres.	[illegible]
25	625	Priyanka Mohan Patil	Synthesis and Antibacterial evaluation of Heterocyclic Compound containing Amino moiety.	Patil

26	626	Rabinakhatu Zakir Hussen	Ecofriendly approach to designed Fluorenone by using Tarmarind Pulp Extract.	Rabinakhatu
27	627	Komal Sushil Sharma	Recovery of polyol by Chemical Recycling of Waste Polyurethane Foam.	[illegible]
28	628	Jyoti Mahanand Pathak	One pot synthesis of cinnamon acid	J Pathak
29	629	Umangi Jaybharat Kodya	Synthesis and Antibacterial evaluation of Heterocyclic Compound containing Amino moiety.	Umangi
30	630	Sakshi Manohar Bari	Test for Carbohydrates, Protein and Fats in Foodstuffs.	Sakshi
31	631	Rakshita Deepal Chaudhari	Preparation & spectrophotometric method for the determination of aspirin.	R. D. Chaudhari
32	632	Shweta Namdev Patil	Analysis of Foof quality and Food Adulterants from Different Department by Qualitative Analysis for food safety.	[illegible]
33	633	Sahili Sudhakar Shivgan	Antimicrobial activity of natural Deep Eutectic solvent of Eugenol and cetrimide	Sahil
34	634	Shubham Ravindra Yati		S Yati
35	635	Vishal Ashok Giri	Separation and characterisation of Citral from lemon grass oil	Vishal
36	636	Bhavesh Dilip Borase	Synthesis and Characterisation of Schiff Base of Salicyladehyde with Magnesium metal complex.	Bhavesh
37	637	Babita vijay More	Synthesis of benzimidazole by using Borassus Flabillifer.	Babita

38	638	Mayuri Mahesh Raul	A green approach to synthesis triazole via Cytisus Scoparius.	MRaul
39	639	Pankesh Mohan Shingada	Synthesis of Benzimidazole using Cytisus Scoparius.	Pshingada
40	640	Shrushti Rajesh Dhanu	Synthesis and Characterisation of Schiff Base of Salicyladehyde with Nickel Metal Complex	SRdhanu
41	641	Puja Shashikant Gaikwad	Synthesis and Characterisation of Schiff Base of Salicyladehyde and Ethylene diamine with Nickel Metal Complex	Pgaikwad
42	642	Anjali Laxman Patil	Borassus Flabillifer as an efficient bio-catalyst for synthesis of triazole.	APatil

4138453

ANTI-MICROBIAL ACTIVITY OF NATURAL DEEP EUTECTIC SOLVENT (DES) OF EUGENOL AND CETRIMIDE

A PROJECT REPORT SUBMITTED TO THE

DEPARTMENT OF CHEMISTRY

SONOPANT DANDEKAR COLLEGE, PALGHAR

IN PARTIAL FULFILLMENT OF THE DEGREE

OF

MASTER OF SCIENCE IN ORGANIC CHEMISTRY

SUBMITTED BY

MISS. SAHILI SUDHAKAR SHIVGAN

UNDER THE SUPERVISION OF

DR. DILIP YADAV

DEPARTMENT OF CHEMISTRY,

SONOPANT DANDEKAR ARTS,V.S.APTE COMMERCE AND

M.H MEHTA SCIENCE COLLEGE ,PALGHAR

UNIVERCITY OF MUMBAI 2023-2024

Certificate

This is to certify that **Miss. Sahili Sudhakar Shivgan** has successfully completed her project on **'Antimicrobial Activity of Natural Deep Eutectic Solvent of Eugenol and Cetrimide'** towards the partial fulfilment of the degree of Master of Science in Organic Chemistry under University of Mumbai, Mumbai.

Date: 19/06/2023

Place:Dandekar College, Palghar

Dr.Dilip K. Yadav

(Supervisor)

Dr. Suhas Janwadkar

(Head of Chemistry Department)

CERTIFICATE

This is to certify that **Miss. Sahili Sudhakar Shivgan** has successfully completed his project on **ANTI – MICROBIAL ACTIVITY OF NATURAL DEEP EUTECTIC SOLVENT (DES) OF EUGENOL AND CETRIMIDE.** towards partial fulfillment of the **Degree Of Masters In Organic Chemistry** under **University Of Mumbai**

DATE :-

PLACE :-

DR. SUHAS P. JAWADKAR

HEAD DEPARTMENT OF CHEMISTRY

DECLARATION

I hereby declare that this project entitled **ANTI MICROBIAL ACTIVITY OF NATURAL DEEP EUTECTIC SOLVENT (DES) OF EUGENOL AND CETRIMIDE.**

Is original work and is being submitted in particular fulfillment for award of degree, master of university of Mumbai . This project has not been submitted earliear to this university or any other affiliated colleges of this university.

Sahili S. shivgan

ACKNOWLEDGEMENT

I express my profound gratitude to Dr. Kiran save sir ,the principal of the SDSM college for giving me this opportunity and providing me efficient chemical lab for the completion of my project

I express my profound gratitude to Dr.Suhas p.janwadkar,H.O.D of chemistry department for his valuable guidance and support during the course

I extend my sincere thanks to my project guide Dr.Dilip Yadav who has been supporting and encouraging throughout.

I would thanks to lab assistant for providing all the help and being kind while carry out the experiment.

Last but not least I want to thanks my family and friends for their support.

Sahili S. Shivgan

Student

M.Sc – II (organic chemistry)

Roll no - 633

Index

CHAPTER - 1
INTRODUCTION

INTRODUCTION

1.1 Solvent

Solvent generally in liquid form, are used to dissolve,dilute,suspended any substances or extract other materials. Solvent are essential to most regions of science, the use of solvent in chemistry is enormous .Solvent are used in oil & paints , thinning pigments ,dissolving drugs & use to carry out reactions.

Some solvents being harmful to environment, toxic in nature , volatile , flammable this are major drawbacks of conventional solvent therefore the Conventional solvents were replaced by ionic liquids &now ionic liquids are replaced by Deep Eutectic Solvent.

1.2 Solvent classification

Solvent can be broadly classified into two categories

Polar solvent - it is type of solvent that has large partial charges or dipole moments

Non polar solvent – non polar solvent possess little or no dipolar character

1.3 Deep eutectic solvent

DES is green solvent discovered by Abbott in 2001,has emerged as an alternative for harmful & costly ionic liquids & organic solvent. DES is mixture of hydrogen bond acceptor (HBA) &hydrogen bond donor (HBD) in definite molar ratio.

DES consist of components that are held together by hydrogen bond rather than ionic bond. DES is mixture of two or more components that are typically solid at room temperature but when combined at particular molar ratio changes into liquid at room temperature.

DES are extensively used in organic reactions ,limited research work has been does on medical applications of DES.DES is organic solvent prepared by naturally occurring material .it is eco friendly, biodegradable, non toxic, low cost, non flammable,& readily available .

1.4 Types of deep eutectic solvent

1. Quaternary ammonium salt + metal salt hydrate
2. Quaternary ammonium salt +metal salt
3. Quaternary ammonium salt + hydrogen bond donor
4. Metal salt + hydrogen bond donor

Classification Of DES Based On Water Solubility:-

Depend upon water solubility there are two type of deep eutectic solvent

Hydrophilic DES (Water Soluble)

Hydrophobic DES (Water Insoluble)

DES have several advantages over traditional ionic liquid such as the ease of preparation and easy availability from relative inexpensive components.

The production of deep eutectic solvent involves the simple mixing of two components , generally with moderate heating. This maintains a comparatively low production cost with respect to conventional ionic liquids and permits large scale application.

CHAPTER - 2

REVIEW OF LITERATURE

Deep eutectic solvents (DES) and their applications

Emma L Smith, Andrew P Abbott, Karl S Ryder

Deep eutectic solvents (DES) are now widely acknowledged as a new class of ionic liquid (IL) analogues because they share many characteristics and properties with ILS The terms DES and IL have been used interchangeably in the literature though it is necessary to point out that these are actually two different types of solvent. Dess are systems formed from a eutectic mixture of Lewis or Brønsted acids and bases which can contain a variety of anionic and/or cationic species; in contrast, ils are formed from systems composed primarily of one type of discrete anion and cation. It is illustrated here that although the physical properties of dess are similar to other ils, their chemical properties suggest application areas which are significantly different. The research into ionic liquids (ILQ) has escalated in the last two decades, ever since the potential for new chemical technologies was realized. In the period covered by this review, approximately 6000 journal articles have been published on the topic. Compared to classical ILS the research into DES is comparatively in its infancy, with the first paper on the subject only published in 2001

A Natural Deep Eutectic Solvent Formulated to Stabilize β-Lactam Antibiotics

Belén Olivares, Fabián Martínez, ...Paola R. Campodonico Show authors .

β-lactam antibiotics, such as penicillin share a very unstable chemical structure. In water-based solutions, such as those used for clinical applications, the β-lactam ring is readily opened due to a nucleophilic or electrophilic attack, leading to the loss of antimicrobial activity. Since the achievement and maintenance of optimum therapeutic levels of β-lactam antibiotics is critical for the resolution of many infectious clinical situations, and to avoid antibiotic resistance generation, the design of new non-aqueous dosage forms is urgent. Recently, natural deep eutectic solvents (NADES) have emerged as alternative non-toxic and non-aqueous solvents for different biomedical applications. In this work, we formulated and characterized a NADES composed by betaine and urea (BU). Using this solvent, we evaluated the stability of clavulanic acid (CLV) and imipenem (IMP) and characterized their antimicrobial activities calculating the minimal inhibitory concentration.

In vivo and in vitro toxicity profile of tetrabutylammonium bromide and alcohol-based deep eutectic solvents

Shamaila Inayat, Sajid Rashid Ahmad, ...Qurban Ali Show authors

Scientific Reports volume 13, Article number: 1777 (2023) Cite this

Deep eutectic solvents (DESs) have emerged as new promising solvents in the field of "green chemistry," which possess a broad range of potential applications. However, the ecotoxicological profile of these solvents is still poorly known. In this study, ammonium-based deep eutectic solutions with glycerol (2:2), ethylene glycol (1:2), and diethylene glycol (1:2) as hydrogen bond donors in 1:2 proportion were evaluated for their interaction with various biological systems, including gram-positive and negative bacteria, fungi, fish, and human fibroblast cell lines. The DES synthesis was confirmed by Fourier transform infrared spectroscopy analysis, which analyses the interactions between DES precursors for their synthesis. The antimicrobial activity of tetrabutylammonium bromide: ethylene glycol was the most potent, while tetrabutylammonium bromide: diethylene glycol had a higher LC50 against C. carpio fish. Tetrabutylammonium

CHAPTER - 3
MATERIAL AND METHODOLOGY

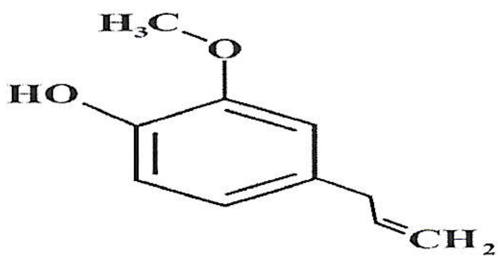
Des are extensively used in organic reactions, micro extraction techniques and metal processing method .the bioactivity of des is not yet explored, but literature has already reported cytotoxicity of ammonium and choline chloride – based DES for several cancer cell lines

Our study is based on DES prepared from components of Cetrimide which act as hydrogen bond acceptor and Eugenol (extracted from clove oil) act as a hydrogen bond donor

Cetrimide and Eugenol mixed together in definite molar ratio to form DES

3.1 CHEMICAL :-

1. **EUGENOL :-** Molecular Formula – $C_{10}H_{12}O_2$



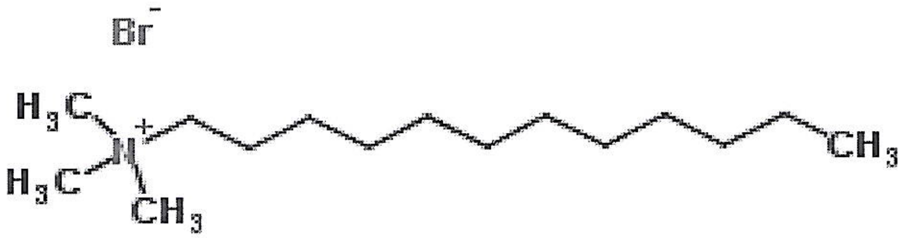
In vitro, Eugenol has been shown to have antibacterial, Antifungal antioxidant and antineoplastic activity
Eugenol is an allyl chain- substituted Guaiacol, member of Allyl benzene class of chemical compound.
Eugenol is naturally occurring phenolic molecule found in several plants such as cinnamon, clove and by leaves.

Uses of Eugenol :-

Eugenol useful for treatment of skin infection & inflammatory disorders.
Eugenol is used as a flavor or aroma ingredient.
It is use as wood preservative.
Eugenol when combine with zinc oxide has been used in dentistry for many years as root canal sealing

2) Cetrimide :- Molecular Formula - $C_{19}H_{42}BrN$

Molecular Weight - 365.45 g/mol



Cetrimide belongs to a class of drug Antiseptic Primarily used to treat wounds, cuts, minor, burns& protect against infection

Cetrimide is available as soap, lotion & cream form Cetrimide also known as Alkyltrimethyl ammonium bromide.

Cetrimide is mixture of 3 quaternary ammonium salt

i) Tetradonium Bromide (TTAB)
ii) Cetrimonium Bromide (CTAB)
iii) Lautrimonium Bromide (LTAB)

Cetrimide is freely soluble in water and ethanol.

Uses of Cetrimide :-

Treats Wounds, Minor Cuts

Sunburns, Reduce Blemishes And Acne

Reduce Blemishes And Acne

Treat Dry And Chapped Skin

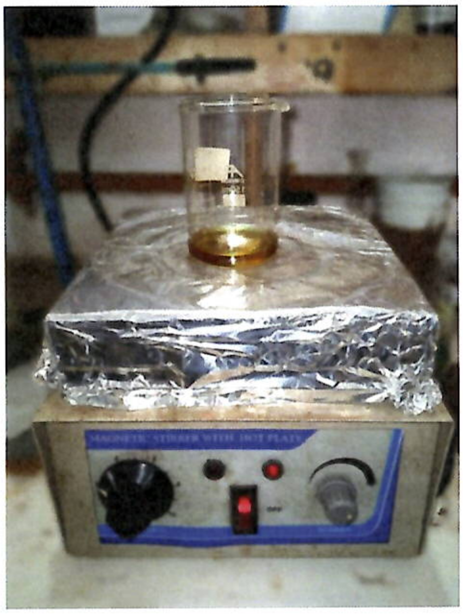
3.2 Equipment :-

very limitted material are use in preparation of deep eutectic solvent.

1) glasswares,

2) Hotplate with magnetic stirrer instrument –

Is use to conduct biological and chemical experiment by mixing two components.it is equally suitable for solids or liquids samples to obtain consistent liquid mixture.



HOT PLATE WITH MAGNETIC STIRRER INSTRUMENT

3.3 PROCEDURE OF MAKING DEEP EUTECTIC SOLVENT

The main concept of making DES is to form a hydrogen bonding between the two chemicals.so based on each chemical ratio stable liquid is formed.

Based on molar ratio example :-

molar ratio (1 : 1) take one mole of Eugenol and 1 mole of Cetrimide in glass beaker and apply heating by using magnetic stirrer instrument till liquid is properly mix together.

Different types ratio are tested and analysis given below

1) Molar ratio (1:1) = (Eugenol : Cetrimide)
   Highly viscous liquid is formed

2) Molar ratio (1:2) = (Eugenol : Cetrimide)
   Highly viscous liquid is formed

3) Molar ratio (1:3) = (Eugenol : Cetrimide)
   Viscous liquid is formed

Low viscous liquid is formed

Molar ratio (1 : 4) = (Eugenol : Cetrimide) is appropriate ratio for preparing sample and based on IR & NMR spectroscopy hydrogen bond formation is observed.

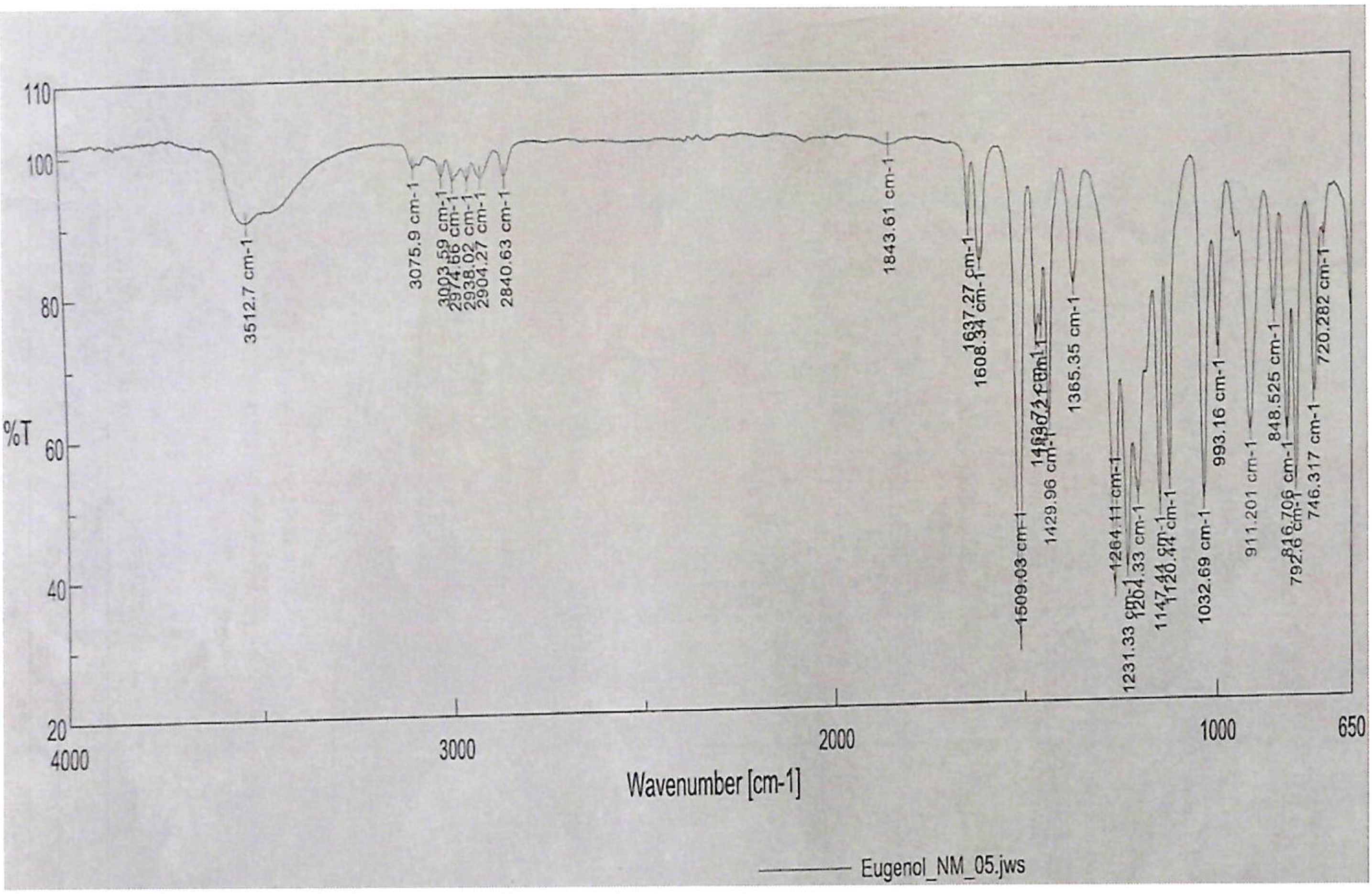
Present work describes a preparation method for Deep eutectic solvent of Cetrimide and Eugenol. The method is based on H- bond formation by (OH^-) group of Eugenol.

CHAPTER - 4
RESULT AND DISCUSSION

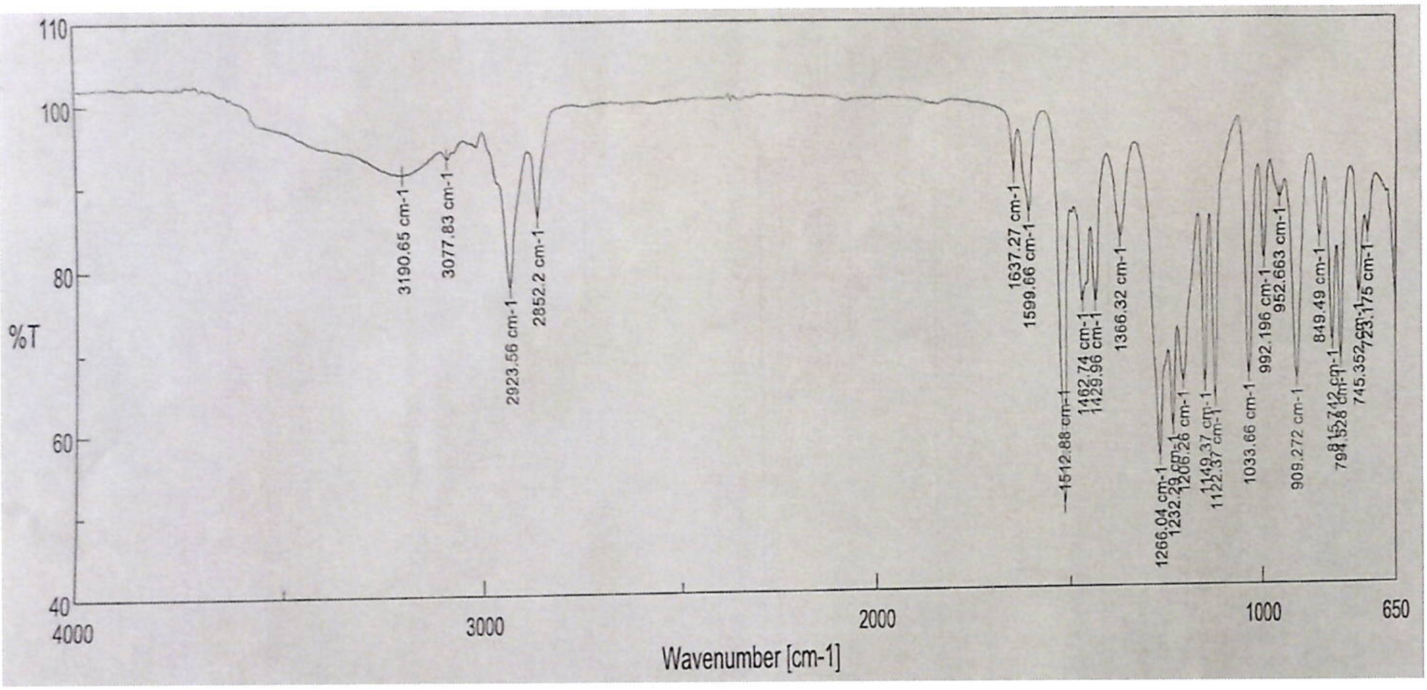
4.1 SPECTRAL STUDIES :-

IR Spectra of Eugenol shows broad band at 3518.13cm^{-1} corresponding to stretching of the free [-OH] group to the molecule. Which was absent in IR Spectra of DES indicates presence of Hydrogen-Bonding. So we can conclude that there is a formation of required DES.

IR spectra of Eugenol :-

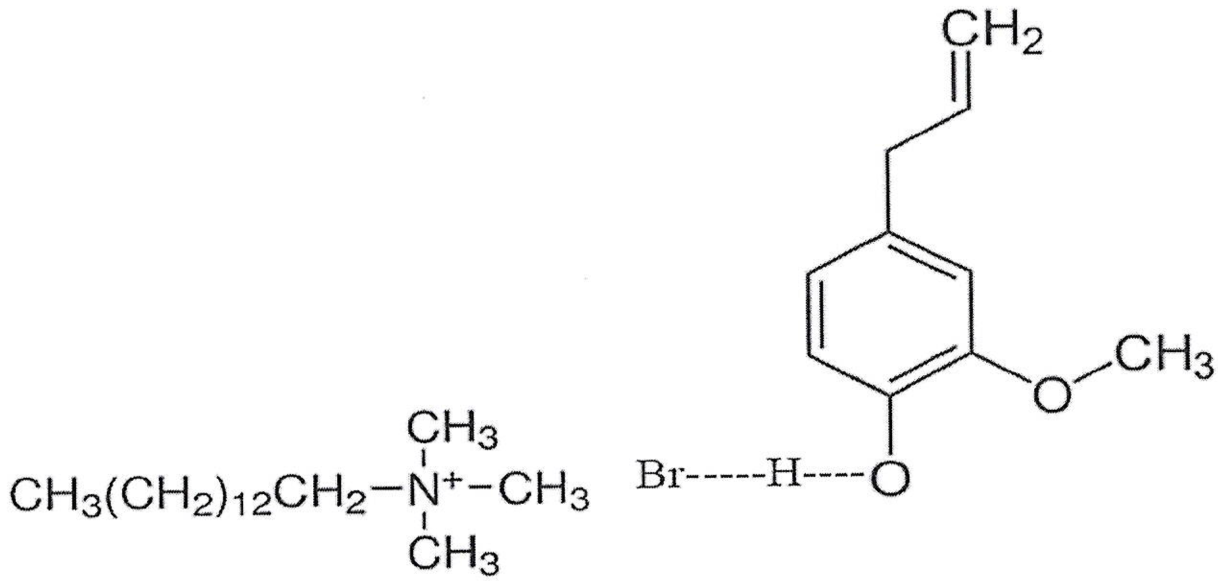


IR spectra of Deep eutectic solvent :-



Sr. No.	IR Spectra of Eugenol	IR Spectra of DES	Conclusion	Remark
1	**Broad Band 3512.7cm^{-1}**	**No Broad Band at 3500-3600**	**Indicates formation of H-bond between**	**Present only in Eugenol spectra**
2	3075.9 cm^{-1}	3077.83 cm^{-1}	Due to C-C present	Present in both
3	2840.63 cm^{-1}	2852.2 cm^{-1}	Presence of $-OCH_3$ group	Present in both
4	1637 cm^{-1}	1637 cm^{-1}	Presence of C=C	Present in both

Structure of Deep Eutectic Solvent : -



4.2 DENSITY STUDY

Density is an important concept because it's allow us to determine what substances will float and what substance will sink when placed in a liquid. Density is determine by using anton paar instrument.

Density of DES = 1.060 g/cm-3

Anton paar instrument

4.3 Physical properties : -

Boiling point of Eugenol :- 254^{0}C

Melting point of Cetrimide :- 248^{0}C

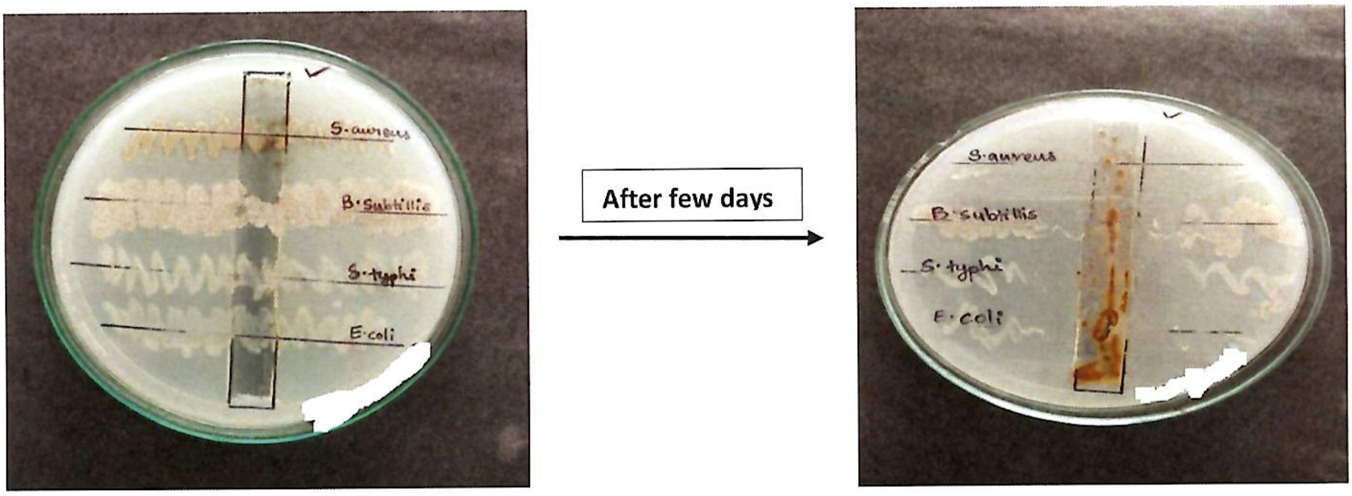
Boiling point of Deep eutectic solvent :- 180^{0}C

Freezing point of deep eutectic solvent :- -1^{0}C

4.4 Biological activities :-

Our Prepared DES shows Anti-microbial activity.

Biological activities of deep eutectic solvent is determine by using ditch pate method .this method chosen to test the anti-bacterial activity of compounds.it is preliminary method to screen the antimicrobial potential of compound .



Actual pictures : Department of Biotechnology

CHAPTER - 5
CONCLUSION

Deep eutectic solvents (DES) are a new generation of solvents that can offset the major drawbacks of common ILs, namely high toxicity, no biodegradability, complex synthesis requiring purification, and high cost of the starting materials

The similarity in physical properties between DESs and ILs suggests that they belong in the same class of liquid which is distinct from molecular liquids; yet, the disparity in chemical properties between the liquids means that to date DESs have found very different application fields to ILs.

The aim for this method used to introduce simple, reliable and inexpensive method for synthesis of DESs by using Cetrimide and Eugenol. Desired DES is prepared by mixing and heating molar quantities of Eugenol and one of the component of Cetrimide, which is confirmed by physical properties and IR spectra.

Experimental analysis shows that DESs of Cetrimide & Eugenol show the antimicrobial properties. DES is green material hence there is no harm to environment.

References

- Abbott, A.P.; Boothby, D.; Capper, G.; Davies, D.L.; Rasheed, R.K. *Deep Eutectic Solvents Formed between Choline Chloride and Carboxylic Acids: Versatile Alternatives to Ionic Liquids,* J. Am. Chem. Soc., 2004, 126, 9142–9147.

- Smith E.L.; Abbott, A.P.; *Deep Eutectic Solvents (DESs) and their applications*, Chem. Rev. ()014), 114, 21, 11060–11082

- Shamaila Inayat, Sajid Rashid Ahmad, ...Qurban Ali Show authors Scientific Reports volume 13, Article number: 1777 (2023)

- Belén Olivares, Fabián Martínez, ...Paola R. Campodonico Show authors Scientific Reports volume 8, Article number: 14900 (2018)

4138421

ANTI-MICROBIAL ACTIVITY OF NATURAL DEEP EUTECTIC SOLVENT (DES) OF EUGENOL AND BENZYL TRIETHYL AMMONIUM CHLORIDE

A PROJECT REPORT SUBMITTED TO THE

DEPARTMENT OF CHEMISTRY SONOPANT DANDEKAR COLLEGE, PALGHAR

IN PARTIAL FULFILLMENT OF THE DEGREE

OF

MASTER OF SCIENCE IN ORGANIC CHEMISTRY

SUBMITTED BY

MISS. GAURI NARENDRA DHANMEHER

UNDER THE SUPERVISION OF

DR. DILIP YADAV

DEPARTMENT OF CHEMISTRY,

SONOPANT DANDEKAR ARTS,V.S.APTE COMMERCE AND M.H MEHTA SCIENCE COLLEGE ,PALGHAR

UNIVERCITY OF MUMBAI 2023-2024

Certificate

This is to certify that **Miss. GAURI NARENDRA DHANMEHER** has successfully completed her project on **'Antimicrobial Activity of Natural Deep Eutectic Solvent of Eugenol and Benyzl Triethyl Ammonium Chloride'** towards the partial fulfilment of the degree of Master of Science in Organic Chemistry under University of Mumbai, Mumbai.

Date: 19/06/2023

Place:Dandekar College, Palghar

Dr. Dilip K. Yadav

(Supervisor)

Dr. Suhas Janwadkar

(Head of Chemistry Department)

24/06/23

S.D.S.M COLLEGE PALGHAR, DIST PALGHAR

DECLARATION

I hereby declare that this project entitled **ANTI MICROBIAL ACTIVITY OF NATURAL DEEP EUTECTIC SOLVENT (DES) OF EUGENOL AND BENZYL TRIETHYL AMMONIUM CHLORIDE.**

Is original work and is being submitted in particular fulfillment for award of degree, master of university of Mumbai . This project has not been submitted earliear to this university or any other affiliated colleges of this university.

Gauri.N.Dhanmeher

ACKNOWLEDGEMENT

I express my profound gratitude to the principal Dr.Kiran Save Sir for providing the fundings and Prof.DR. Suhas Janwadkar Sir, H.O.D of chemistry department for his valuable guidance and support during our course.

I extended my sincere thank you to our project guide Dr Dilip Yadav, who has been supporting and encouraging throughout.

I would like to thank Hardik Dada the lab Assistants for providing all the help while carrying out the experiment.

Last but not least I want to thank you my friends for their support.

Gauri N. Dhanmeher

Student

Sonopant Dandekar Arts,

V.S. Apte Commerce and

M.H. Mehta Science college,

Palghar

INDEX

CHAPTER 1:

INTRODUCTION

INTRODUCTION

1.1 Solvent

Solvent generally in liquid form, are used to dissolve,dilute,suspended any substances or extract other materials. Solvent are essential to most regions of science, the use of solvent in chemistry is enormous .Solvent are used in oil & paints , thinning pigments ,dissolving drugs & use to carry out reactions.

Some solvents being harmful to environment, toxic in nature , volatile , flammable this are major drawbacks of conventional solvent therefore the Conventional solvents were replaced by ionic liquids &now ionic liquids are replaced by Deep Eutectic Solvent.

1.2 Solvent classification

Solvent can be broadly classified into two categories

Polar solvent - it is type of solvent that has large partial charges or dipole moments

Non polar solvent – non polar solvent possess little or no dipolar character

1.3 Deep eutectic solvent

DES is green solvent discovered by Abbott in 2001,has emerged as an alternative for harmful & costly ionic liquids & organic solvent. DES is mixture of hydrogen bond acceptor (HBA) &hydrogen bond donor (HBD) in definite molar ratio.

DES consist of components that are held together by hydrogen bond rather than ionic bond. DES is mixture of two or more components that are typically solid at room temperature but when combined at particular molar ratio changes into liquid at room temperature.

DES are extensively used in organic reactions , limited research work has been does on medical applications of DES.DES is organic solvent prepared by naturally occurring material .it is eco friendly, biodegradable, non toxic, low cost, non flammable,& readily available .

1.4 DESs are classified into four types based on composition:

1) Type I – (Quaternary ammonium salt + metal chloride)

eutectics include a wide range of chlorometallate ionic solvents which were widely studied in the 1980s, such as imidazolium chloroaluminates which are based on mixtures of $AlCl_3$ + 1-Ethyl-3-methylimidazolium chloride.

2) Type II – (Quaternary ammonium salt + metal chloride hydrate)

eutectics are identical to Type I eutectic in composition yet include the hydrated from of the metal halide. Type III eutectics consist of hydrogen bond acceptors such as quaternary ammonium salts (eg. choline chloride) and hydrogen bond donors (e.g urea, ethylene glycol) and include the class of metal-free deep eutectic solvents

3) Type III – (Quaternary ammonium salt + hydrogen bond donor)

eutectics have been successfully used in metal processing applications such as electrodeposition, electropolishing, and metal extraction.

Type IV eutectics are similar to type III yet replace the quaternary ammonium salt hydrogen bond acceptor with a metal halide hydrogen bond acceptor while still using an organic hydrogen bond donor such as urea.

4) Type IV – (Metal chloride hydrate + hydrogen bond donor)

eutectics are of interest for electrodeposition as they produce cationic metal complexes, ensuring that the double layer close to the electrode surface has a high metal ion concentration.

Classification Of DES Based On Water Solubility:-

Depend upon water solubility there are two type of deep eutectic solvent

Hydrophilic DES (Water Soluble)

Hydrophobic DES (Water Insoluble)

DES have several advantages over traditional ionic liquid such as the ease of preparation and easy availability from relative inexpensive components.

The production of deep eutectic solvent involves the simple mixing of two components , generally with moderate heating. This maintains a comparatively low production cost with respect to conventional ionic liquids and permits large scale application.

Chapter 2 : - Review of literature

REVIEW AND LITERATURE

Our study is based on DES prepared from Cetylpyridinium chloride which acts as HBA and Eugenol (extracted from clove oil) as HBD. They are mixed together in definite Molar ratio to form DES. It is a hydrophobic DES. Their physical properties such as density, viscosity, boiling point, etc. are determined. Anti-microbial activities are to be studied.

Study undertaken by Ana Bjelić, Brigita Hočevar, et.al involves use of deep eutectic solvents (DESs) envisaged as the most advanced novel polar liquids that are entirely made of natural, molecular compounds that are capable of an association *via* hydrogen bonding interactions. DES has quickly emerged in various application functions thanks to a formulations' simple preparation. This research covers DESs for lignocellulosic component isolation, applications as (co)catalysts and their functionality range in biocatalysis. This method was to represent that there are infinite possibility of DESs to used.

Study undertaken by Maria Forsyth, Patrick C. Howlett, Anthony E. Somers Given a future prospects, surrounding the use of ionic liquids in the engineering of interphases to control charge transport thereby leading to improved performance of high-energy density batteries. In the search for cost-effective solutions, a relatively new class of ionic liquids, termed deep eutectic solvents, have also been explored as potential media for controlling surface films on reactive metals. The deep eutectic solvents class of ionic liquid materials offers many possible combinations of chemistry that can be targeted to produce desired properties.

The method developed by Joana M.Silva, EduardoSilva, Rui L.Reis, et.al that (DES) is used to explore these unmet medical needs. DES systems based on saturated fatty acids, namely, capric acid (CA), myristic acid (MA), lauric acid (LA) and stearic acid (SA) were produced and fully characterized at a physicochemical level. The thermal characterization results indicate a depression of the melting point in DES form when compared with the starting compounds to near-physiological levels, The systems revealed significant antimicrobial activity against the tested Gram-positive bacteria and *C. albicans*, with the CA:LA system showing the greatest overall inhibitory/bactericidal activity.

The aims of this study developed by Kristine OpsvikWikene, Håkon ValenRukke, to investigate the effect of dilution on the acid-containing NADES network, their antimicrobial activity on different planktonic microorganisms, and their influence on phototoxicity when used as solvents for a photosensitiser. In investigations of phototoxicity, the microorganisms were exposed to the photosensitiser meso-tetra(*p*-hydroxyphenyl)porphine (THPP; 1 nM) dissolved in diluted NADES combined with blue light (27 J/cm^2).
The eutectic network appeared to remain upon dilution ≤ 1:200.

Study undertaken by Tsvetinka Grozdanova, Boryana Trusheva, Kalina Alipieva, is to study four selected NADES were applied for the extraction of two medicinal plants: *Sideritis scardica*, and *Plantago major* as an alternative to water-alcohol mixtures, and the antimicrobial and genotoxic potential of the extracts were studied. The extraction efficiency was evaluated by measuring the extracted total phenolics, and total flavonoids. Best extraction results for total phenolics for the studied plants were obtained with choline chloride-glucose 5:2 plus 30% water; but surprisingly these extracts were inactive against all tested microorganisms. Low genotoxicity and cytotoxicity were observed for all four NADES and the extracts with antimicrobial activity.

Chapter 3 :-

Material and methodology

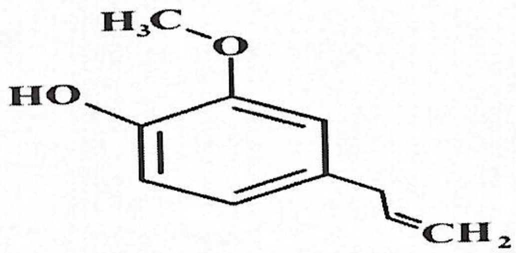
Des are extensively used in organic reactions, micro extraction techniques and metal processing method .the bioactivity of des is not yet explored, but literature has already reported cytotoxicity of ammonium and choline chloride – based DES for several cancer cell lines

Our study is based on DES prepared from components of Cetrimide which act as hydrogen bond acceptor and Eugenol (extracted from clove oil) act as a hydrogen bond donor

Alkyl dimethyl Benzyl Ammonium Chloride and Eugenol mixed together in definite molar ratio to form DES

CHEMICAL :-

1. EUGENOL :- Molecular Formula – $C_{10}H_{12}O$



In vitro, Eugenol has been shown to have antibacterial, Antifungal antioxidant and antineoplastic activity
Eugenol is an allyl chain- substituted Guaiacol, member of Allyl benzene class of chemical compound.
Eugenol is naturally occurring phenolic molecule found in several plants such as cinnamon, clove and by leaves.

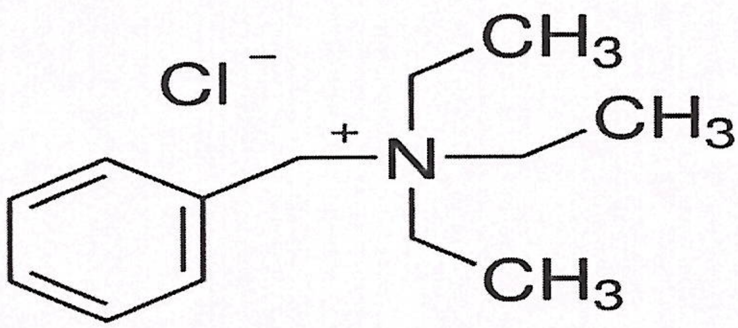
Uses of Eugenol :-

Eugenol useful for treatment of skin infection & inflammatory disorders.
Eugenol is used as a flavor or aroma ingredient.
It is use as wood preservative.
Eugenol when combine with zinc oxide has been used in dentistry for many years as root canal sealing

2) Benzyltriethyl Ammonium Chloride :-

Molecular Formula - $C_6H_5\ CH_2N(Cl)(\ C_2H_5)_3$

Molecular Weight - 227.77 g/mol



Benzyltriethyl Ammonium Chloride belongs to a class of drug Antiseptic Primarily used to treat wounds, cuts, minor, burns& protect against infection.

Benzyltriethyl Ammonium Chloride is available as synthesis of amphetamine class drugs.

Benzyltriethyl Ammonium Chloride are quaternary ammonium salt

Cationic surfactant.

Benzyltriethyl Ammonium Chloride is used as a laboratory chemical & phase transfer catalyst

Benzyltriethyl Ammonium Chloride is available as soap, shampoo.

Uses of Benzyltriethyl Ammonium Chloride:-

Treats Wounds, Minor Cuts

It is used as laboratory chemical & phase transfer catalyst

Sunburns, Reduce Blemishes And Acne

Reduce Blemishes And Acne

Treat Dry And Chapped Skin

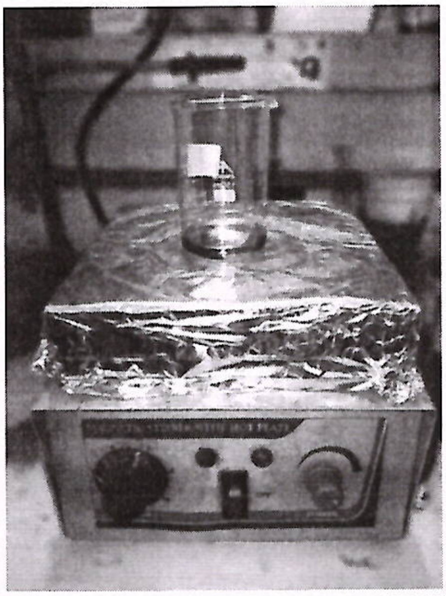
Topical antiseptic and surfactant

Equipment :-

very limitted material are use in preparation of deep eutectic solvent. 1) glasswares,

2) Hotplate with magnetic stirrer instrument –

Is use to conduct biological and chemical experiment by mixing two components.it is equally suitable for solids or liquids samples to obtain consistent liquid mixture.



HOT PLATE WITH MAGNETIC STIRRER INSTRUMENT

PROCEDURE OF MAKING DEEP EUTECTIC SOLVENT

The main concept of making DES is to form a hydrogen bonding between the two chemicals.so based on each chemical ratio stable liquid is formed.

Based on molar ratio example :-

molar ratio (1 : 1) take one mole of Eugenol and 1 mole of Benzyltriethyl Ammonium Chloride in glass beaker and apply heating by using magnetic stirrer instrument till liquid is properly mix together.

Different types ratio are tested and analysis given below

1) Molar ratio (1:1) = (Eugenol : Benzyltriethyl Ammonium Chloride)
   Highly viscous liquid is formed

2) Molar ratio (1:2) = (Eugenol : Benzyltriethyl Ammonium Chloride)
   Low viscous liquid is formed

Molar ratio (1 : 2) = (Eugenol : Benzyltriethyl Ammonium Chloride) is appropriate ratio for preparing sample and based on IR & NMR spectroscopy hydrogen bond formation is observed.

Present work describes a preparation method for Deep eutectic solvent of Benzyltriethyl Ammonium Chloride and Eugenol. The method is based on H- bond formation by (OH$^-$) group of Eugenol.

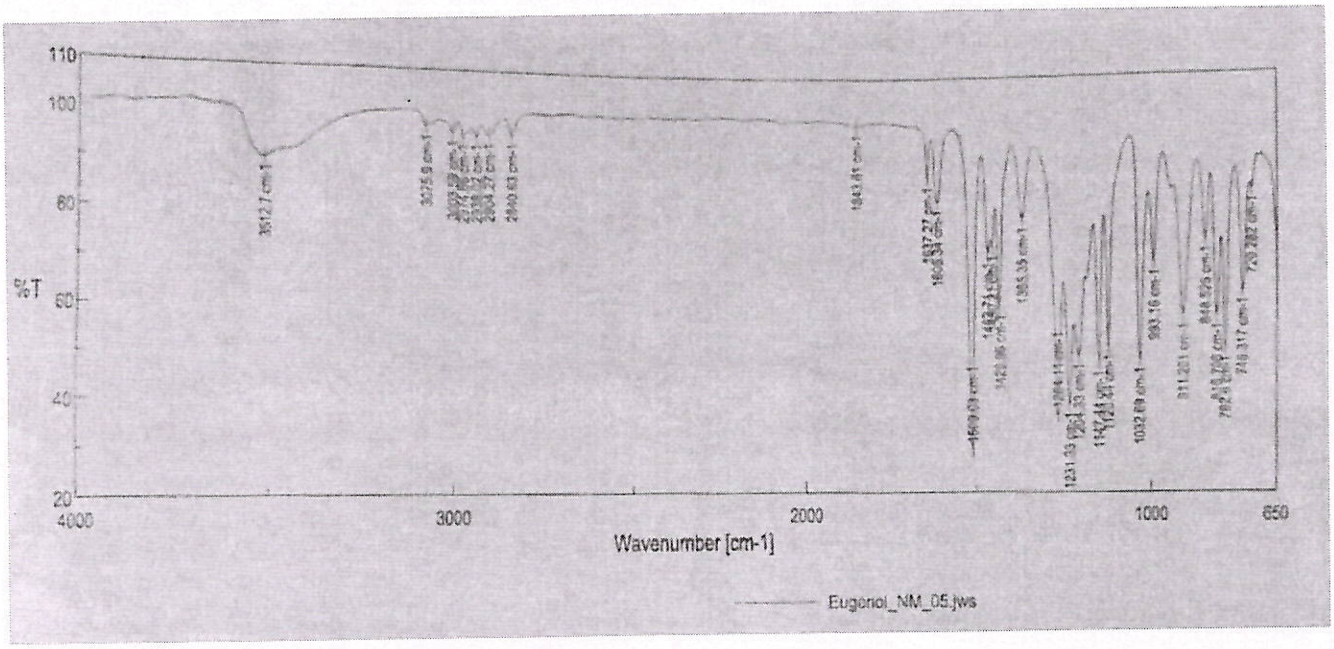
Chapter 4 :-

Result and discussion

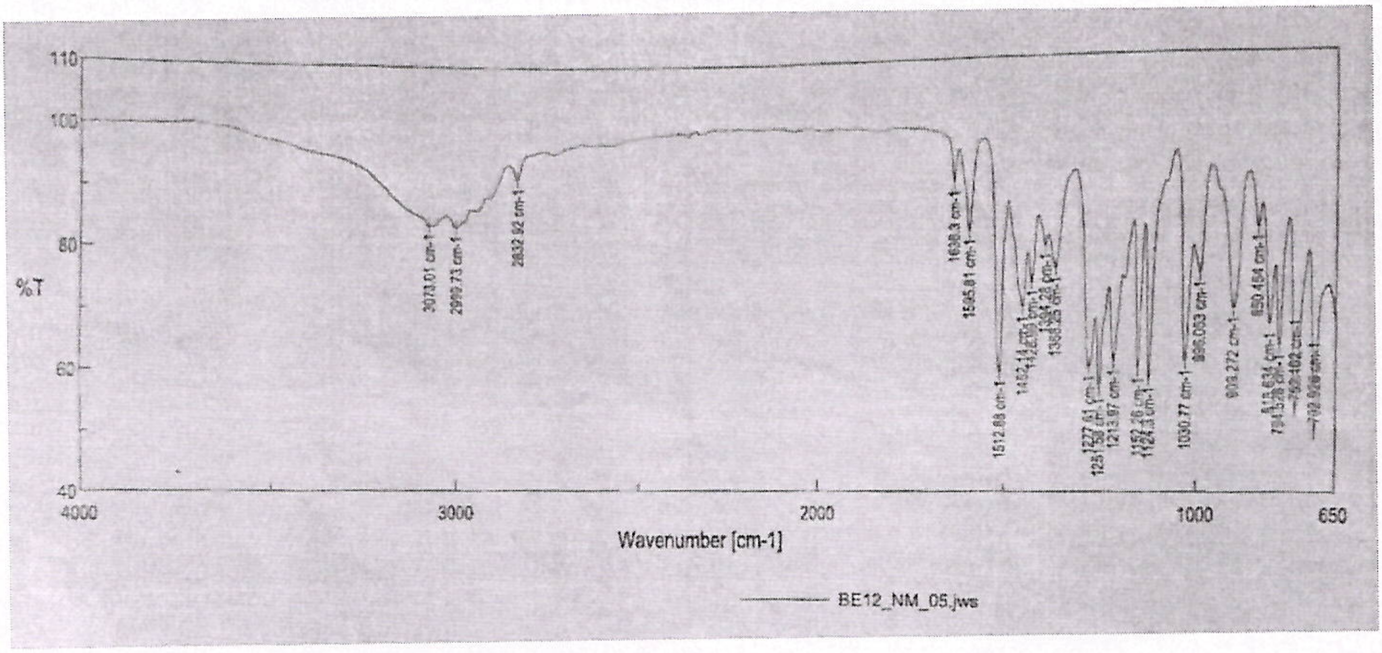
1) SPECTRAL STUDIES :-

IR Spectra of Eugenol shows broad band at 3518.13cm^{-1} corresponding to stretching of the free [-OH] group to the molecule. Which was absent in IR Spectra of DES indicates presence of Hydrogen-Bonding. So we can conclude that there is a formation of required DES.

IR spectra of Eugenol :-



IR spectra of Deep eutectic solvent :-

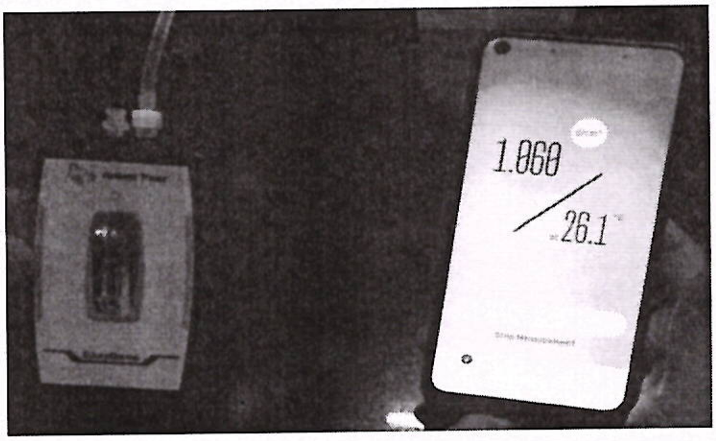


Sr. No.	IR Spectra of Eugenol	IR Spectra of DES	Conclusion	Remark
1	**Broad Band 3512.7cm^{-1}**	**No Broad Band at 3500-3600**	**Indicates formation of H-bond between**	**Present only in Eugenol spectra**
2	3075.9 cm^{-1}	3077.83 cm^{-1}	Due to C-C present	Present in both
3	2840.63 cm^{-1}	2852.2 cm^{-1}	Presence of $-OCH_3$ group	Present in both
4	1637 cm^{-1}	1637 cm^{-1}	Presence of C=C	Present in both

2) DENSITY STUDY

Density is an important concept because it's allow us to determine what substances will float and what substance will sink when placed in a liquid. Density is determine by using anton paar instrument.

Density of DES = 1.060/gcm-3



Anton paar instrument

3) Physical properties : -

Boiling point of Eugenol :- 254^0C

Melting point of Benzyltriethyl Ammonium Chloride :- 178^0C

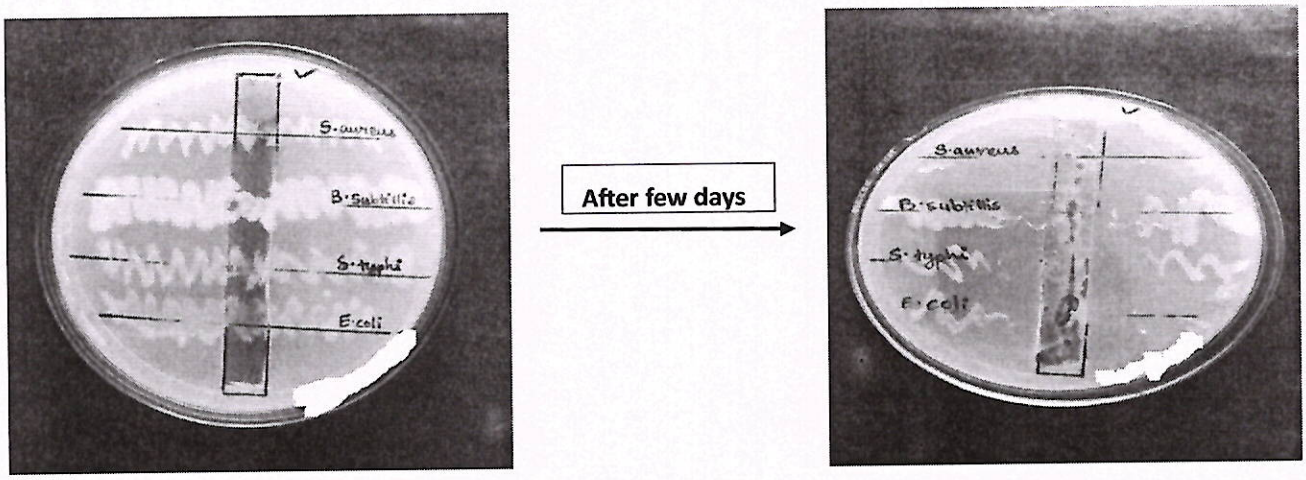
Boiling point of Deep eutectic solvent :- 180^0C

Freezing point of deep eutectic solvent :- -1^0C

4) Biological activities :-

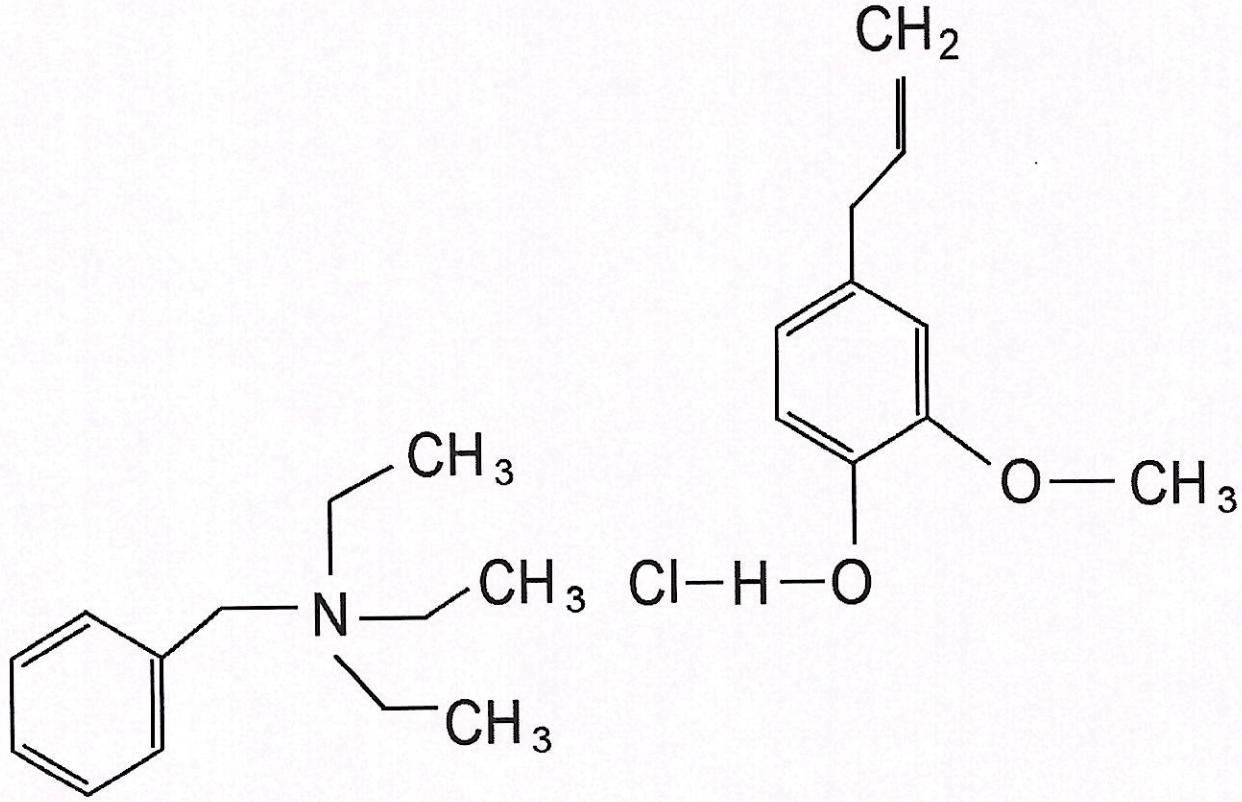
Our Prepared DES shows Anti-microbial activity.

Biological activities of deep eutectic solvent is determine by using ditch pate method .this method chosen to test the anti-bacterial activity of compounds.it is preliminary method to screen the antimicrobial potential of compound .



Actual pictures : Department of Biotechnology

Structure of deep eutectic solvent : -



CHAPTER 05 : -

CONCLUSION

Deep eutectic solvents (DES) are a new generation of solvents that can offset the major drawbacks of common ILs, namely high toxicity, no biodegradability, complex synthesis requiring purification, and high cost of the starting materials

The similarity in physical properties between DESs and ILs suggests that they belong in the same class of liquid which is distinct from molecular liquids; yet, the disparity in chemical properties between the liquids means that to date DESs have found very different application fields to ILs.

The aim for this method used to introduce simple, reliable and inexpensive method for synthesis of DESs by using Benzyltriethyl Ammonium Chloride and Eugenol. Desired DES is prepared by mixing and heating molar quantities of Eugenol and one of the component of Benzyltriethyl Ammonium Chloride, which is confirmed by physical properties and IR spectra.

Experimental analysis shows that DESs of Benzyltriethyl Ammonium Chloride & Eugenol show the antimicrobial properties. DES is green material hence there is no harm to environment.

References

- Abbott, A.P.; Boothby, D.; Capper, G.; Davies, D.L.; Rasheed, R.K. *Deep Eutectic Solvents Formed between Choline Chloride and Carboxylic Acids: Versatile Alternatives to Ionic Liquids,* J. Am. Chem. Soc., 2004, 126, 9142–9147.

- Smith E.L.; Abbott, A.P.; *Deep Eutectic Solvents (DESs) and their applications*, Chem. Rev. ()014), 114, 21, 11060–11082

- Jahromi Z.A; Assadi Y.; Hosseini M.R.M.; Jamali M.R.; *Dispersive liquid–liquid microextraction combined with graphite furnace atomic absorption spectrometry Ultra trace determination of cadmium in water samples*, Anal. Chim. Acta, (2007) 585, 305.

- *Silva J.M; Silva E; REIS R, L; Duarte A. R. c; A closer look in the antimicrobial properties of deep eutectic solvents based on fatty acids, Sustianable Chemistry and Pharmacy , (2019) 14, 100192.*

- *Hayyan M; Looi c. Y.; Hayyan, A.; Wong W. F.; Hashim, M. A.; In vitro and in vivo toxicity profiling of ammonium-based deep autectic solvents. OLoS ONE, (2015) 10(2), e0117934.*

- *Bjelic, A., Hocevar, B., Grilc, M., Novak, U., E Likozar, B. (2022). A review of sustainable lignocellulose biorefining applying (natural) deep eutectic solvents (DESs) for separations, catalysis and enzymatic biotransformation processes. Reviews in chemical Engineering, 38(3). 243-272.*

- *Forsyth, M., Howlett, P, C., Somers, A, E., MacFarlane, D. R., E Basile, A. (2017). Interphase engineering of reactive metal surfaces using ionic liquids and deep eutectic solvents-from corrosion control to next-generation batteries Npj Materials Degradation, 1(1). 1-6.*

413844

"SYNTHESIS & BIOLOGICAL PROPERTIES OF EUGENOL-CETYLPYRIDINIUM CHLORIDE BASED DEEP EUTECTIC SOLVENT"

A PROJECT REPORT SUBMITTED TO THE

DEPARTMENT OF CHEMISTRY

SONOPANT DANDEKAR COLLEGE, PALGHAR

IN PARTIAL FUIIFILMENT OF THE DEGREE

OF

MASTER OF SCIENCE IN ORGANIC CHEMISTRY

SUBMITTED BY

ANIKET RAJENDRA PATIL

UNDER THE SUPERVISION OF

DR. DILIP YADAV

DEPARTMENT OF CHEMISTRY.

SONOPANT DANDEKAR ARTS, V. S. APTE COMMERCE AND M. H. METHA SCIENCE COLLEGE, PALGHAR.

UNIVERSITY OF MUMBAI

2022 – 2023

Sonopant Dandekar Shikshan Mandali's
SONOPANT DANDEKAR ARTS, V. S. APTE COMMERCE AND M. H. MEHTA SCIENCE COLLEGE
Tal. Palghar, Dist. Palghar, Pin - 401 404.
Code.: (02525) 252163, Prin : 252317 • Resi. : 252316
website : www.sdsmcollege.com • Email:sdsmcollege@yahoo.com

Ref. No. : Date : ___________

Certificate

This is to certify that **Mr. Aniket Rajendra patil** has successfully completed his project on **'Synthesis & biological properties of eugenol-cetylpyridium chloride based Deep Eutectic Solvent'** towards the partial fulfilment of the degree of Master of Science in Organic Chemistry under University of Mumbai, Mumbai.

Date: 19/06/2023

Place:Dandekar College, Palghar

Dr. Dilip K. Yadav
(Supervisor)

Dr. Suhas Janwadkar
(Head of Chemistry Department)

Patil 24/06/23

S.D.S.M. COLLEGE PALGHAR DIST PALGHAR

DECLARATION

I Hereby declare that this project entitled **"SYNTHESIS & BIOLOGICAL PROPERTIES OF EUGENOL-CETYLPYRIDINIUM CHLORIDE BASED DEEP EUTECTIC SOLVENT"** Is original work and is being submitted in particular fulfilment for award of degree, Master of Science of University of Mumbai. This project has not been submitted earlier to this university or any other affiliated colleges of this university.

ANIKET RAJENDRA PATIL

Student

ACKNOWLEDGEMENT

I express my gratitude to Dr. Kiran Save ,The Principal of the SDSM College for giving me the opportunity and providing me efficient chemical lab for the completion of my project.

I express my profound gratitude to Dr. Suhas P. Janwadkar, H.O.D of the chemistry department for his valuable guidance and support during the course.

I extend my sincere thank to my project guide Dr Dilip yadav who has been encouraging and supportive throughout.

I would like to thank our lab assistants and non-teaching staff for providing all the necessary help with the apparatus regarding the experiments.

Last but not least I would like to thank my friends and family for their support.

ANIKET RAJENDRA PATIL

Student

Sonopant Dandekar arts,

V. S. Metha Commerce and

M. S. Metha science college

Palghar.

INDEX

CHAPTER 01:

INTRODUCTUON

INTRODUCTION

The use of solvents in chemistry is enormous. It is used in oils and paints, thinning pigments, dissolving drugs, also used to carry out reactions. Even though it has large applications, there are many side effects. Being harmful to the environment, toxic in nature, volatile, flammable are the major drawbacks of conventional solvents. Many improvements have been done in recent years regarding properties of the solvents. Conventional solvents were replaced by Ionic Liquids and now Ionic Liquids are replaced by DES.

DES which is a green solvent discovered by Abbott, et.al. 2001.has emerged as an alternative for harmful & costly Ionic liquids and organic solvents. It is the mixture of Hydrogen Bond Acceptor (HBA) and Hydrogen Bond Donor (HBD) in definite molar ratio. It has many of the same properties as Ionic Liquids (ILs). The difference is that DESs consist of components that are held together by hydrogen bonds rather than ionic bonds . Two solids or liquids can be mixed together in definite molar ratio to form Deep Eutectic Solvents. There is no need to purify them as molar ratio is fixed. These are termed as deep eutectic solvents due to the depression in their freezing point. They are bio-degradable, eco-friendly and cost effective. There are two types of deep eutectic solvents hydrophobic (water insoluble) and hydrophilic (water soluble).

DES are extensively used in organic reactions, micro extraction techniques and metal processing methods . Limited research work has been done on medical applications of DES. Although, some research papers are available on anti-microbial, cytotoxic properties of DES .

The bioactivity of DES is not yet explored, but, literature has already reported cytotoxicity of ammonium- and choline chloride-based DES for several cancer cell lines.The parent components of deep eutectic solvents engage in a complex hydrogen bonding network which results in significant freezing point depression as compared to the parent compounds. The extent of freezing point depression observed in DESs is well illustrated by a mixture of choline chloride and urea in a 1:2 mole ratio. Choline chloride and urea are both solids at room temperature with melting points of 302°C (decomposition point) and 133°C respectively, yet the combination of the two in a 1:2 molar ratio forms a liquid with a freezing point of 12°C.

DESs are classified into four types based on composition:

1) Type I – (Quaternary ammonium salt + metal chloride)

eutectics include a wide range of chlorometallate ionic solvents which were widely studied in the 1980s, such as imidazolium chloroaluminates which are based on mixtures of $AlCl_3$ + 1-Ethyl-3-methylimidazolium chloride.

2) Type II – (Quaternary ammonium salt + metal chloride hydrate)

eutectics are identical to Type I eutectic in composition yet include the hydrated from of the metal halide. Type III eutectics consist of hydrogen bond acceptors such as quaternary ammonium salts (eg. choline chloride) and hydrogen bond donors (e.g urea, ethylene glycol) and include the class of metal-free deep eutectic solvents

3) Type III – (Quaternary ammonium salt + hydrogen bond donor)

eutectics have been successfully used in metal processing applications such as electrodeposition, electropolishing, and metal extraction.

Type IV eutectics are similar to type III yet replace the quaternary ammonium salt hydrogen bond acceptor with a metal halide hydrogen bond acceptor while still using an organic hydrogen bond donor such as urea.

4) Type IV – (Metal chloride hydrate + hydrogen bond donor)

eutectics are of interest for electrodeposition as they produce cationic metal complexes, ensuring that the double layer close to the electrode surface has a high metal ion concentration.

PROPERTIES OF DESs:

1) DESs And Ils Have Different Chemical Properties But Have Same Physical Properties

2) Low Vapor Pressure

3) Non-flammable

4) Chemically Inert

5) High Density Thus High Viscosity

6) Less Expensive

7) High Thermal Stability And Solubility

DESs have several advantages over traditional ILs such as their ease of preparation, and easy availability from relatively inexpensive components (the components themselves are toxicologically well-characterized, so they can be easily shipped for large scale processing).

The production of DESs involves the simple mixing of the two components, generally with moderate heating. This maintains a comparatively low production cost with respect to conventional ILs (such as imidazolium based liquids) and permits large scale applications.

While the individual components of DESs tend to be individually well toxicologically characterized, there is very little information about the toxicological properties of the eutectic solvents themselves, and this needs to be further investigated by the scientific community.

Objectives

1) To review literature on DES and therapeutic uses of Eugenol and Cetylpyridinium Chloride.
2) To prepare Deep Eutectic Solvents based on Eugenol and Cetylpyridinium chloride in different praportions.
3) Confirm the hydrogen bond formation between eugenol and Cetylpyridinium chloride using IR spectra.
4) To find out physical properties of prepared DES
5) To study the biological activities of prepared DES.

CHAPTER 02:

REVIEW AND LITERATURE

REVIEW AND LITERATURE

Our study is based on DES prepared from Cetylpyridinium chloride which acts as HBA and Eugenol (extracted from clove oil) as HBD. They are mixed together in definite Molar ratio to form DES. It is a hydrophobic DES. Their physical properties such as density, viscosity, boiling point, etc. are determined. Anti-microbial activities are to be studied.

Study undertaken by Ana Bjelić, Brigita Hočevar, et.al involves use of deep eutectic solvents (DESs) envisaged as the most advanced novel polar liquids that are entirely made of natural, molecular compounds that are capable of an association *via* hydrogen bonding interactions. DES has quickly emerged in various application functions thanks to a formulations' simple preparation. This research covers DESs for lignocellulosic component isolation, applications as (co)catalysts and their functionality range in biocatalysis. This method was to represent that there are infinite possibility of DESs to used.

Study undertaken by Maria Forsyth, Patrick C. Howlett, Anthony E. Somers Given a future prospects, surrounding the use of ionic liquids in the engineering of interphases to control charge transport thereby leading to improved performance of high-energy density batteries. In the search for cost-effective solutions, a relatively new class of ionic liquids, termed deep eutectic solvents, have also been explored as potential media for controlling surface films on reactive metals. The deep eutectic solvents class of ionic liquid materials offers many possible combinations of chemistry that can be targeted to produce desired properties.

The method developed by Joana M.Silva, EduardoSilva, Rui L.Reis, et.al that (DES) is used to explore these unmet medical needs. DES systems based on saturated fatty acids, namely, capric acid (CA), myristic acid (MA), lauric acid (LA) and stearic acid (SA) were produced and fully characterized at a physicochemical level. The thermal characterization results indicate a depression of the melting point in DES form when compared with the starting compounds to near-physiological levels, The systems revealed significant antimicrobial activity against the tested Gram-positive bacteria and *C. albicans*, with the CA:LA system showing the greatest overall inhibitory/bactericidal activity.

The aims of this study developed by Kristine OpsvikWikene, Håkon ValenRukke, to investigate the effect of dilution on the acid-containing NADES network, their antimicrobial activity on different planktonic microorganisms, and their influence on phototoxicity when used as solvents for a photosensitiser. In investigations of phototoxicity, the microorganisms were exposed to the photosensitiser meso-tetra(*p*-hydroxyphenyl)porphine (THPP; 1 nM) dissolved in diluted NADES combined with blue light (27 J/cm^2). The eutectic network appeared to remain upon dilution ≤ 1:200.

Study undertaken by Tsvetinka Grozdanova, Boryana Trusheva, Kalina Alipieva, is to study four selected NADES were applied for the extraction of two medicinal plants: *Sideritis scardica*, and *Plantago major* as an alternative to water-alcohol mixtures, and the antimicrobial and genotoxic potential of the extracts were studied. The extraction efficiency was evaluated by measuring the extracted total phenolics, and total flavonoids. Best extraction results for total phenolics for the studied plants were obtained with choline chloride-glucose 5:2 plus 30% water; but surprisingly these extracts were inactive against all tested microorganisms. Low genotoxicity and cytotoxicity were observed for all four NADES and the extracts with antimicrobial activity.

CHAPTER 03 :

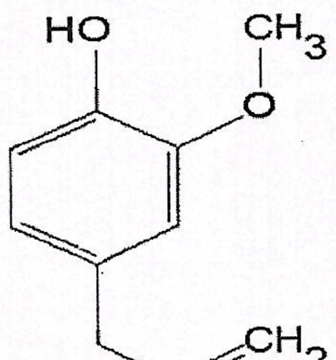
MATERIAL AND METHODOLOGY

MATERIAL AND METHODOLOGY

In this research project preparation of DES is proposed using Eugenol and Cetylpyridinium chloride. Eugenol and Cetylpyridinium chloride are mixed together in definite molar ratio. The mixture is heated with constant stirring till a transparent viscous liquid is formed. Purification step is not needed. Following steps are involved in research methodology

CHEMICAL:

1) EUGENOL



Chemical structure of eugenol ($C_{10}H_{12}O_2$)

Eugenol (C10H12O2 or CH3C6H3) is a volatile phenolic constituent of clove essential oil obtained from Eugenia caryophyllata buds and leaves, mainly harvested in Indonesia, India and Madagascar.

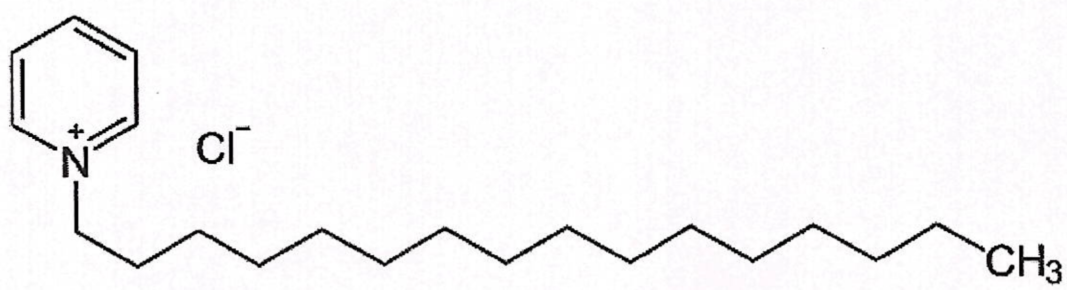
The name supposedly is derived from the scientific name for clove E. caryophyllata tree which has large leaves and flower buds which turn to red colour when they are ready for collection.

1,2 Eugenol is the main extracted constituent (70-90%) of cloves and is responsible for clove aroma.1 Eugenol, a phenylpropanoid, is pale yellow oil with a spicy aroma with the molecular weight of 164.2 g/mol.

This molecule is a weak acid which is soluble in organic solvents and specially extracted from clove oil, nutmeg, cinnamon, basil and bay leaf.

The oil derived from the flower buds of clove mainly consists of eugenol (60-GENOL90%), eugenyl acetate, caryophyllene and other substances, whereas oil derived from the leaves of the clove tree consists of eugenol (82-88%) and very little eugenyl acetate, and other minor constituents.

2) Cetylpyridinium chloride



Cetylpyridinium chloride (CPC) is a cationic quaternary ammonium compound It is an antiseptic that kills bacteria and other microorganisms. It has been shown to be effective in preventing dental plaque and reducing gingivitis.It has also been used as an ingredient in certain pesticides. Though one study seems to indicate cetylpyridinium chloride does not cause brown tooth stains

Properties of Cetylpyridinium chloride

Chemical formula: C21H38ClN

Molar mass: 339.99 g·mol−1

Appearance: Solid

Melting point: 77 °C (171 °F; 350 K)

Equipment :-

very limitted material are use in preparation of deep eutectic solvent.

1) glasswares,

2) Hotplate with magnetic stirrer instrument –Is use to conduct biological and chemical experiment by mixing two components.it is equally suitable for solids or liquids samples to obtain consistent liquid mixture.

HOT PLATE WITH MAGNECTIC STIRRER INTRUMENT

PROCEDURE OF MAKING DES

The main concept of making DES is to form a Hydrogen bonding between the two chemicals. So based on each chemical ratio stable liquid is formed.

Based on Molar ratio example :- molar ratio (1 :1) take 1mole of Cetylpyridinium chloride in 1mole of Eugenol in beaker and apply heat by placing on the 4magnetic stirrer till all the solid particle dissolve properl

Molar ratio

[1:3] = [Cetyl pyridinium chloride :Eugenol]

Molar Ratio [1 : 3]= [Cetylpyridinium chloride: Eugenol] is appropriate ratio for preparing DES sample. And based on IR Spectroscopy Hydrogen bond formation is observed.

The present work describes a preparation method for DEEP EUTECTIC SOLVENT Cetyl pyridinium chloride and Eugenol. The method is based on H-bonding formation by (-OH) Group of eugenol to the positive terminal Nitrogen atom of Cetyl pyridinium chloride

CHAPTER 04

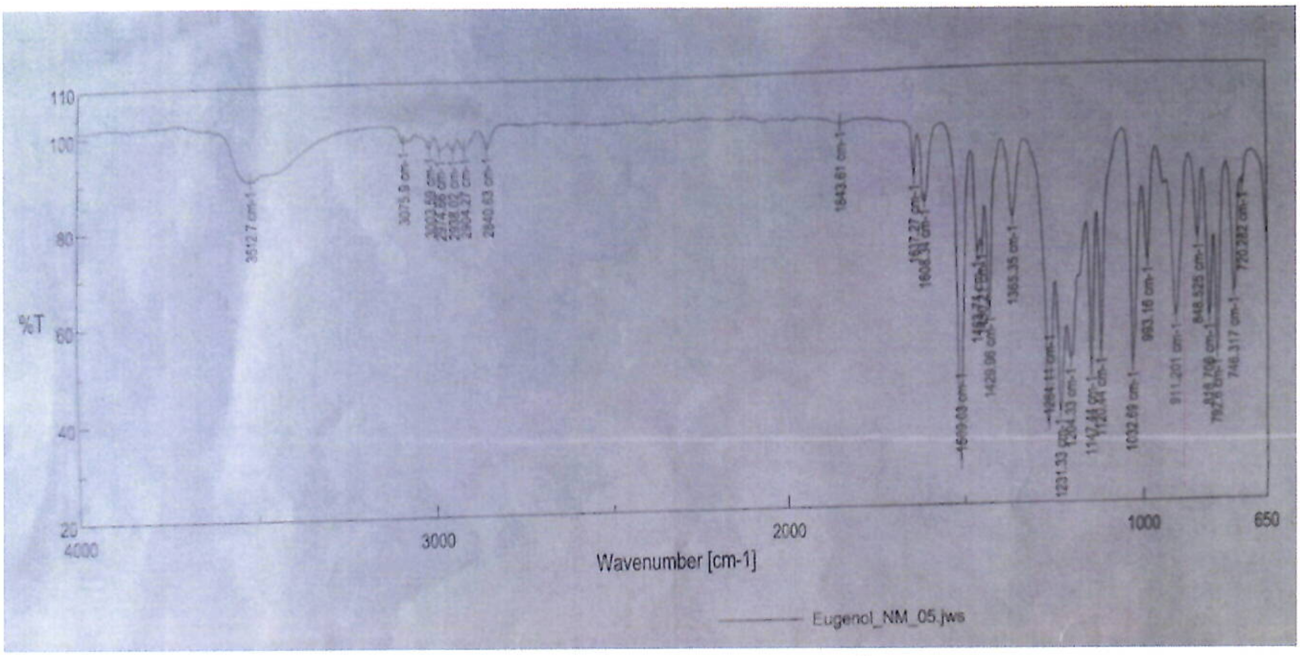
RESULT AND DISCUSSION

1) SPECTRAL STUDIES:

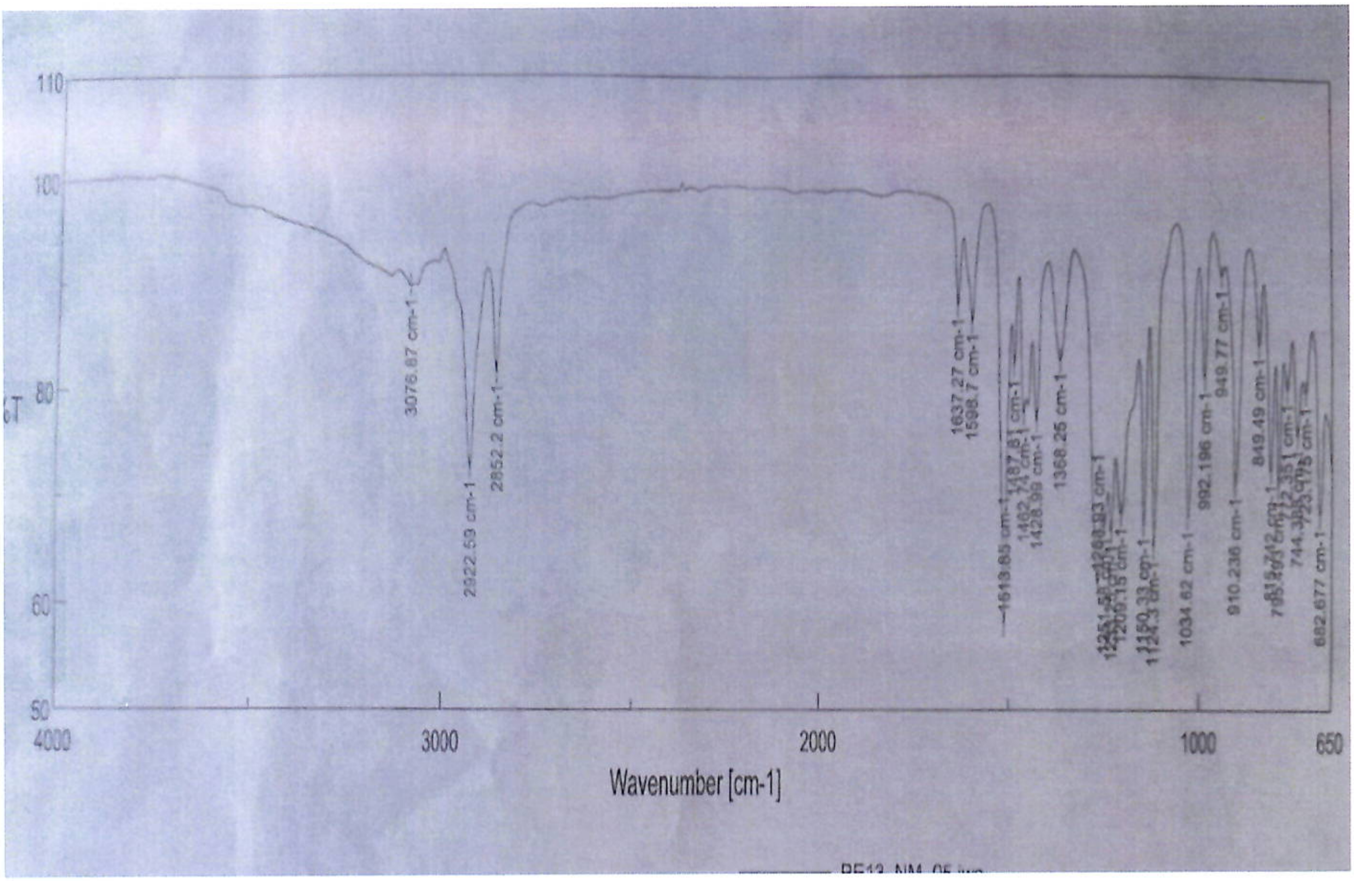
The formation of a eutectic solvent occurs due to hydrogen bonding of the precursors, together with intermolecular interactions involving Van der Walls and Electrostatic forces.

IR Spectra of Eugenol shows broad band at $3518.13 cm^{-1}$ corresponding to stretching of the free [-OH] group to the molecule. Which was absent in IR Spectra of DESs indicates presence of Hydrogen-Bonding. So we can conclude that there is a formation of required DESs.

Ir spectra of Eugenol :



Ir spectra of Cetylpyridinium chloride:



Sr. No.	IR Spectra of Eugenol	IR Spectra of DES	Conclusion	Remark
1	**Broad Band 3518.13 cm^{-1}**	**No Broad Band at 3500-3600**	**Indicates formation of H-bond between**	**Present only in Eugenol spectra**
2	3076 cm^{-1}	3075.52 cm^{-1}	Due to C-C present	Present in both
3	1267 cm^{-1}	1252 cm^{-1}	Presence of $-OCH_3$ group	Present in both
4	1637 cm^{-1}	1637 cm^{-1}	Presence of C=C	Present in both
5	Bands at 700 to 500 cm^{-1}	Bands at 700 to 500 cm^{-1}	Due to presence of substituted benzene ring.	Present in both

2) DENSITY STUDY

Density is an important concept because it's allow us to determine what substances will float and what substance will sink when placed in a liquid.

We determined density by using anton paar instrument

Density of prepared DESs =1.019 g/ cm^3

ANTON PAAR INSTRUMENT

3) Physical properties

1) Melting point of Cetylpyridinium chloride = 84°c

2) Boiling point of Eugenl =254°c

3)boiling points of prepared DESs =200°c

CHAPTER 05:

CONCLUSION

CONCLUSION

Deep eutectic solvents (DES) are a new generation of solvents that can offset the major drawbacks of common ILs, namely high toxicity, no biodegradability, complex synthesis requiring purification, and high cost of the starting materials

The similarity in physical properties between DESs and ILs suggests that they belong in the same class of liquid which is distinct from molecular liquids; yet, the disparity in chemical properties between the liquids means that to date DESs have found very different application fields to ILs.

The aim for this method used to introduce simple, reliable and inexpensive method for synthesis of DESs by using Cetylpyridinium chloride and Eugenol.

CHAPTER 06 :

REFRENCES

REFRENCES

1) Abbott, A.P.; Boothby, D.; Capper, G.; Davies, D.L.; Rasheed, R.K. *Deep Eutectic Solvents Formed between Choline Chloride and Carboxylic Acids: Versatile Alternatives to Ionic Liquids,* J. Am. Chem. Soc., 2004, 126, 9142–9147.

2) Smith E.L.; Abbott, A.P.; *Deep Eutectic Solvents (DESs) and their applications*, Chem. Rev. ()014), 114, 21, 11060–11082

3) Jahromi Z.A; Assadi Y.; Hosseini M.R.M.; Jamali M.R.; *Dispersive liquid–liquid microextraction combined with graphite furnace atomic absorption spectrometry Ultra trace determination of cadmium in water samples*, Anal. Chim. Acta, (2007) 585, 305.

4) Silva J. M.; Silva E.; Reis R. L.; Duarte A. R. C.; *A closer look in the antimicrobial properties of deep eutectic solvents based on fatty acids*, Sustainable Chemistry and Pharmacy, (2019) 14, 100192.

5) Hayyan M.; Looi C. Y.; Hayyan, A.; Wong W. F.; Hashim, M. A.; *In vitro and in vivo toxicity profiling of ammonium-based deep eutectic solvents.* PLoS ONE, (2015) 10(2), e0117934.

6) Bjelić, A., Hočevar, B., Grilc, M., Novak, U., & Likozar, B. (2022). A review of sustainable lignocellulose biorefining applying (natural) deep eutectic solvents (DESs) for separations, catalysis and enzymatic

biotransformation processes. *Reviews in Chemical Engineering*, *38*(3), 243-272.

7) Forsyth, M., Howlett, P. C., Somers, A. E., MacFarlane, D. R., & Basile, A. (2017). Interphase engineering of reactive metal surfaces using ionic liquids and deep eutectic solvents—from corrosion control to next-generation batteries. *npj Materials Degradation*, *1*(1), 1-6.