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Research and Reviews in Chemical Science Volume II

Editors:
Dr. Neeraj Mohan Gupta
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PREFACE

Welcome to "Research and Reviews in Chemical Science"! In this comprehensive volume, we delve into the fascinating world of chemical science, exploring its latest advancements, groundbreaking discoveries, and promising avenues for future research.

Chemical science lies at the heart of countless innovations that shape our modern world, from novel materials and pharmaceuticals to sustainable energy solutions and environmental remediation. This book serves as a platform for scholars, researchers, and enthusiasts to explore the multifaceted landscape of chemical science, covering a diverse array of topics ranging from theoretical principles to practical applications.

As editors, it is our privilege to present a collection of meticulously curated articles authored by experts and thought leaders from around the globe. Each contribution offers unique insights, methodologies, and perspectives, enriching our understanding of the complex interplay of molecules and matter.

Furthermore, this volume aims to foster interdisciplinary dialogue and collaboration by bridging the gap between fundamental research and real-world applications. By showcasing the latest developments and emerging trends in chemical science, we hope to inspire new ideas, spark innovative solutions, and propel the field forward into uncharted territories.

We extend our sincere gratitude to all the authors whose dedication and expertise have made this publication possible. Additionally, we express our appreciation to the reviewers and editorial team for their invaluable contributions in ensuring the quality and rigor of the content.

We invite readers from all backgrounds to embark on a journey of exploration and discovery through the pages of "Research and Reviews in Chemical Science." May this book serve as a beacon of knowledge and inspiration for generations to come, as we continue to unravel the mysteries of the molecular world and harness its transformative potential for the betterment of society.

Editors

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THE REMARKABLE ROLE OF MICA AS A SOURCE OF POTASSIUM

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Abstract:

Mica, a family of silicate minerals, has remarkable role as a source of potassium, an essential element for plant growth and numerous industrial applications. The geological origins, extraction methods and applications of mica-derived potassium. Mica predominantly consisting of muscovite, biotite and phlogopite, is formed within the Earth's crust through intricate geological processes. These minerals often find their origins in igneous and metamorphic rocks. The geological origins of mica thus serve as the foundation of its role as a source of potassium. Potassium is a critical nutrient for plant growth and is one of the primary elements in the NPK ratio, which is used to indicate the essential nutrients needed for plant nutrition. The connection between mica and potassium becomes evident when examining the potassium content within mica minerals. Muscovite and phlogopite, two common mica varieties, contain significant amounts of potassium ions (K^+). The extraction of potassium from mica involves a combination of physical and chemical processes designed to liberate potassium ions from the mica structure and convert them into usable forms. Once potassium is obtained, it can be further processed into various forms, such as potassium sulphate (K_2SO_4) or potassium chloride (KCl), depending on the intended application. The applications of mica-derived potassium are both diverse and essential. In agriculture, potassium is a vital nutrient for plants, influencing root development, disease resistance and fruit quality. Mica-derived potassium is a valuable component of fertilizers, enhancing soil fertility and increasing crop yields.

Keywords: Mica, Potassium, Geological Origins, Extraction Methods, Agricultural Fertilizers

Introduction:

In the Earth's geological heritage, there are minerals that harbour secrets, waiting to be unveiled, offering profound insights into sustainable agriculture, industrial progress and innovative technology. Mica, a family of silicate minerals renowned for its versatile applications, holds such a secret a remarkable, yet often underestimated, role as a source of potassium. As the world grapples with the imperative need for sustainable agriculture and energy storage solutions, mica emerges as a silent contributor, poised to address these pressing challenges. The multifaceted world of mica, exploring its geological origins, extraction methodologies and the diverse applications of potassium derived from this unassuming mineral. Through this exploration, we will illuminate the intricate relationship between mica and the geological processes of our planet, unveiling the profound impact mica can have on agriculture, industry and emerging technologies.

Mica, predominantly consisting of muscovite, biotite, and phlogopite, offers a unique insight into the Earth's geological history and the captivating ways in which minerals interact with the natural world. Born within the Earth's crust through the crucible of igneous and metamorphic processes, mica's origins lay intertwined with those of other minerals, particularly potassium-rich feldspars. This geological connection serves as the foundation of mica's compelling role as a source of potassium. Potassium, one of the primary elements within the essential NPK (Nitrogen, Phosphorus, Potassium) ratio for plant nutrition, is the cornerstone of our exploration. It plays a pivotal role in plant growth, influencing root development, disease resistance and the quality of fruits and vegetables. The fascinating aspect of mica's relationship with potassium comes to the forefront when we delve into the potassium content locked within mica minerals. Muscovite and phlogopite, two common mica varieties, house significant amounts of potassium ions (K^+), marking the beginning of our journey into understanding how mica can serve as a natural source of potassium, enriching soil and nourishing crops.

The extraction of potassium from mica entails a meticulously designed blend of physical and chemical processes. It commences with the comminution of mica rocks, breaking them down to a manageable size for subsequent processing. Flotation and separation techniques are then employed to isolate pure mica, ensuring a pristine

source of potassium. Leaching processes take centre stage in liberating potassium ions from mica. These processes often necessitate the use of chemical agents such as sulfuric acid, which facilitate the release of potassium ions. Additionally, ion exchange resins play a important role by capturing potassium ions and releasing them through elution. The potassium obtained from these processes can be further refined into various forms, such as potassium sulphate (K_2SO_4) or potassium chloride (KCl), catering to the diverse needs of agriculture, industry and emerging technologies.

The applications of mica-derived potassium encompass a spectrum of essential domains. In agriculture, potassium emerges as a vital nutrient, influencing not only the productivity but the quality of crops. Mica-derived potassium becomes an invaluable component in fertilizers, promoting soil fertility and elevating crop yields. Beyond agriculture, potassium plays a pivotal role in diverse industrial processes, spanning the production of glass, ceramics and detergents. Mica-derived potassium stands as a crucial raw material, supporting industrial growth. Mica's significance extends even further. Mica-rich deposits often occur in proximity to potash deposits, which are vital sources of potassium chloride (KCl). The mining industry extracts potassium chloride from these deposits, further underlining mica's role in the global potassium supply chain. The potassium-ion batteries, innovative energy storage solutions have the potential to revolutionize the way we store and utilize energy, presenting a sustainable alternative to conventional lithium-ion batteries. In this context, potassium sourced from mica may play a transformative role in the development of potassium-ion battery technology, reshaping the energy storage landscape and accelerating our transition towards sustainable energy solutions.

Geological origins

The formation of mica is a geological process that reveals the mineral's intricate connection to heat and pressure within the Earth's crust. Predominantly discovered in igneous and metamorphic rocks, mica's geological origins are subjected to the dynamic forces at work deep within the Earth. Mica, with its various varieties including muscovite, biotite and phlogopite, owes its genesis to the profound transformations occurring in the Earth's crust. This geological journey, with its relevance to mica as a potassium source, is foundational to understanding the mineral's role.

The formation of mica begins with the crystallization of silicate minerals under extreme heat and pressure conditions. These minerals are often associated with magmatic processes, where molten rock or magma, cools and solidifies to form igneous rocks. The mineral feldspar plays a pivotal role. Feldspar, rich in potassium, is a frequent companion to mica within these geological settings. The interplay between mica and feldspar is a crucial factor that establishes mica as a repository of potassium. As the molten rock cools and solidifies, the silicate minerals within it undergo complex chemical reactions and rearrangements, leading to the formation of mica crystals. The intricate lattice structure of mica, characterized by its sheet-like layers, is a direct consequence of the geological processes that gave birth to it. These layers, composed of aluminium, oxygen, silicon and potassium ions, are key to mica's role as a potassium source. These associations between mica and potassium-rich minerals such as feldspar underscore the mineral's potential to serve as a natural reservoir of potassium. The geological origins of mica, rooted in igneous and metamorphic processes, thus lay the groundwork for understanding how this unassuming mineral emerges as a significant source of potassium.

Potassium content in mica

Mica minerals contain substantial amounts of potassium ions (K^+), making them valuable reservoirs of this essential nutrient. Muscovite and phlogopite, the two most common types of mica, exhibit varying potassium content depending on their specific mineral composition and geological origins. Studies have demonstrated that mica typically contains potassium concentrations ranging from several hundred to several thousand parts per million (ppm) (Rai, 2018). The potassium content within mica becomes accessible to plants through weathering and decomposition processes. As mica minerals undergo weathering, facilitated by physical, chemical and biological factors, potassium ions are gradually released into the surrounding soil environment. This natural release mechanism ensures a continuous supply of potassium, contributing to soil fertility and plant nutrition (Guo *et al*., 2019).

The potassium released from decomposing mica minerals enriches the soil with this vital nutrient, enhancing its fertility and supporting robust plant growth. The availability of potassium from mica supplementation promotes improved root development, increased resistance to environmental stresses and enhanced crop

yields (Zhou *et al*., 2020). Furthermore, the gradual release of potassium from mica aligns with the long-term nutrient requirements of plants, offering sustained benefits to soil health and agricultural productivity.

Extraction and processing of potassium from mica

The extraction of potassium from mica involves a series of processes aimed at liberating potassium ions from the mineral structure and converting them into usable forms for various applications.

1. Crushing and grinding

The initial step in the extraction process involves the crushing and grinding of mica rocks to reduce their particle size. This process enhances the surface area of the mica material, facilitating subsequent processing stages. Mechanical crushers and grinding mills are commonly used equipment for this purpose (Wang *et al*., 2019).

2. Flotation and separation

Following crushing and grinding, mica particles are separated from associated minerals through flotation and various separation techniques. Flotation relies on the differences in surface properties between mica and other minerals to achieve selective separation. Techniques such as froth flotation and magnetic separation are employed to isolate pure mica from the ore matrix (Xu *et al*., 2020).

3. Leaching and ion exchange

Leaching processes are employed to release potassium ions from mica minerals. Chemical agents such as sulfuric acid are often used to facilitate this release by breaking down the mineral structure. The leaching solution containing potassium ions is then subjected to ion exchange processes. Ion exchange resins selectively capture potassium ions, which can be later eluted using suitable eluents (Wang *et al*., 2018).

4. Precipitation and crystallization

Potassium ions obtained from leaching are typically precipitated and crystallized to obtain potassium salts suitable for various applications. Common potassium salts include potassium sulfate (K_2SO_4) and potassium chloride (KCl). Precipitation is achieved by adding suitable reagents to the leaching solution under controlled

conditions, followed by crystallization to obtain pure potassium salts (Chen *et al*., 2021).

Applications of mica-derived potassium

Potassium derived from mica holds significant potential across diverse industries due to its role as a vital nutrient and its presence in various industrial processes.

1. Agriculture

Potassium is essential for promoting healthy plant growth and development. Mica-derived potassium serves as a valuable component in fertilizers, contributing to soil fertility and enhancing crop yields. By providing plants with adequate potassium, derived from mica sources, agricultural productivity can be improved, ensuring sustainable food production (Yao *et al*., 2020).

2. Industrial processes

Potassium is a critical element in various industrial processes, including the production of glass, ceramics and detergents. Mica-derived potassium serves as a raw material in these processes, offering a sustainable and readily available source of this essential element. Its inclusion in industrial applications highlights the versatility of mica-derived potassium in meeting diverse manufacturing needs (Yang *et al*., 2019).

3. Potash mining

Mica-rich deposits often coexist with potash deposits, particularly potassium chloride (KCl). The mining industry extracts potassium chloride from these deposits, utilizing mica-rich sources as a valuable resource. Potassium chloride obtained from mica-rich ores serves as a crucial source of potassium for fertilizers, supporting global agricultural demand and enhancing soil nutrient levels (Zhang *et al*., 2018).

4. Energy storage

Potassium-ion batteries are gaining attention as a promising alternative to lithium-ion batteries due to the abundance of potassium resources. Mica-derived potassium may play a significant role in the development of potassium-ion battery technology, offering a sustainable and environmentally friendly energy storage solution. Research efforts focused on utilizing mica-derived potassium in energy storage devices hold promise for advancing renewable energy technologies (Xie *et al*., 2021).

Conclusion:

Mica's role as a natural source of potassium is crucial and multifaceted. Geologically, mica forms through the cooling of molten rock, incorporating potassium from minerals like feldspar. This geological process results in mica deposits rich in potassium. Extraction and processing of mica unlock its potassium content, essential for various applications. In agriculture, potassium derived from mica is vital for fertilizers, promoting plant growth, stress resistance and disease prevention. Its use aids in sustaining crop yields to meet the demands of a growing global population. Furthermore, potassium from mica serves diverse industrial purposes, including glass, ceramics and cosmetics manufacturing, due to its catalytic and pH-regulating properties. Potassium compounds from mica are integral to energy storage systems, powering electronic devices, electric vehicles and renewable energy infrastructure. Understanding the relationship between mica and potassium highlights its potential to address global challenges sustainably. Exploration of mica deposits offers insights into Earth's geological processes, informing resource management and scientific research. By harnessing potassium from mica, we can meet societal needs while respecting environmental sustainability.

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SYNTHESIS OF VARIOUS [1,3] OXAZINE COMPOUNDS USING SILICOTUNGSTIC ACID UNDER MICROWAVE

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Introduction:

Heterocyclic chemistry comprises at least half of all organic chemistry research worldwide. The large numbers of biologically active molecules that contain the oxazine nucleus has play important roles in the drug discovery process and exhibit various biological activities.[1-2] Investigation of the 1,3-oxazine heterocycles has shown that they possess varied biological properties such as analgesic, anticonvulsant, antitubercular, antibacterial and anticancer activity.[3-6] Particular attention has been paid to these compounds since the discovery of the non-nucleoside reverse transcriptase inhibitor trifluoromethyl-1,3-oxazine-2-one, which shows high activity against a variety of HIV-1 mutant strains.[7] In addition, naphthoxazine derivatives have exhibited therapeutic potential for the treatment of Parkinson's disease.[8-9]

The synthesis of 2,3-dihydro- 1*H*-naphtho[1,2-e]-, 3,4-dihydro-2H-naphtho[2,1-e][1,3]oxazines involves one-pot condensation cyclization reaction of naphthols with formaldehyde and primary amines. Various methods have been reported in the literature which includes BF_3–SiO_2,[10] thiamine hydrochloride (VB_1),[11] Ammonium metavanadate,[12] ionic liquid[13] and alum.[14]

Solid-state syntheses have recently received much attention. These processes have many advantages such as high efficiency and selectivity, easy separation, purification and mild reaction conditions.[15] They are not only environmentally benign, but also economically beneficial because toxic wastes can be minimized or eliminated. The grinding mode for the solid-state reactions has earlier been employed for Grignard reaction,[16] reformatsky reaction,[17] aldol condensation,[18] Dieckmann condensation,[19] Knoevenagel condensation, [20] reduction[21] and other reactions.[22]

As per our interest to develop better protocols for the synthesis of biologically active heterocyclic molecules, we would like to report the synthesis of a series of new 3,4-dihydro-3-substituted-2*H*-naphtho[2,1-*e*][1,3]oxazine derivatives using 2-naphthol, formalin and various anilines as substrates in presence of silicotungstic acid under Microwave. To the best of our knowledge there is no report on the one-pot synthesis of 3, 4-dihydro-3-substituted-2*H*-naphtho[2,1-*e*][1,3]oxazines using silicotungstic acid as a catalyst.

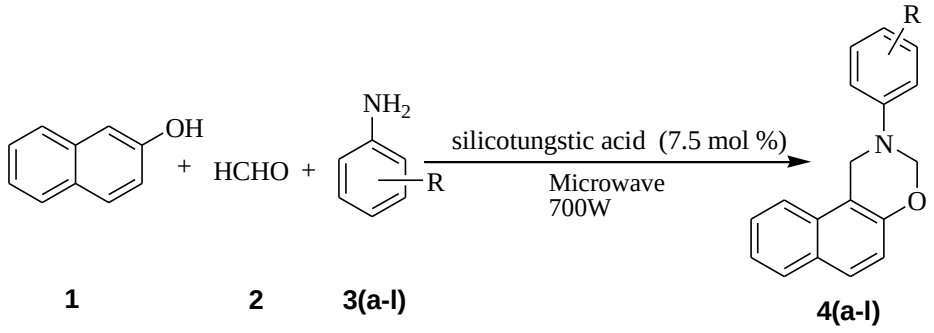
Experimental

Materials and Methods

All amines were obtained from freshly opened container and used without further purification. Melting points were determined in open capillary tubes in a paraffin bath. The progresses of the reactions were monitored by TLC (Thin Layer Chromatography). IR spectra were recorded on Perkin-Elmer FT spectrophotometer in KBr disc.^{1}H NMR spectra were recorded on 400 MHz FT NMR spectrometer in DMSO as a solvent and chemical shift values are recorded in units δ (ppm) relative to TMS as an internal standard.

General procedure

A mixture of 2-naphthol (1 mmol) formalin (2 mmol), aromatic amine (1 mmol), silicotungstic acid (7.5 mol%) and 7ml 90% aquas ethanol in 50 ml RBF was subjected to microwave irradiation for appropriate time in 700 W microwave oven for 6-7 min (successive irradiation of 30–40 sec with cooling intervals of time.) as indicated by TLC. After cooling, the reaction mixture was poured on crushed ice. The obtained crude solid product was filtered, dried and crystallized from ethanol.



Scheme 1: synthesis of 2,3-Dihydro-2-Phenyl-1H-Naphtho-[1,2-e] [1,3] Oxazine

Scheme I

Physical & Analytical data

Table 1: Effect of catalyst concentration [a]

Entry	Concentration (mol %)	Yield (%)[b]
1	2.5	56
2	5	78
3	7.5	91
4	10	91

[a]Reaction condition: **1** (1 mmol), **2** (2 mmol), **3a** (1 mmol), silicotungstic acid (7,5mol%) under microwave. [b]Isolated yield

Table2: Synthesis of 2, 3-Dihydro-2-Phenyl-1*H*-Naphtho-[1, 2-E] [1,3] Oxazin using silicotungstic acid [a]

Entry	Ar-NH_2	Product	Time (min)	Yield[b] (%)	M. P ^0C
1	C_6H_5-	4a	6	91	46-48
2	2-Me-C_6H_4	4b	8	89	58-60
3	2-NO_2-C_6H_4	4c	10	87	109-110
4	3-Me-C_6H_4	4d	9	90	70-72
5	3-OMe-C_6H_4	4e	6	91	76-78
6	3-NO_2-C_6H_4	4f	9	88	132-134
7	4-Me-C_6H_4	4g	7	91	87-89
8	4-OMe-C_6H_4	4h	5	93	78-80
9	4-NO_2-C_6H_4	4i	8	84	168-170
10	4-Br-C_6H_4	4j	8	87	114-116
11	4-F-C_6H_4	4k	9	86	136-138
12	4-OEt-C_6H_4	4l	6	90	69-71

[a]Reaction condition: **1** (1 mmol), **2** (2 mmol), **3a** (1 mmol), silicotungstic acid (7.5mol%) at room temperature. [b]Isolated yield

Spectral data

Spectroscopic data of synthesized some principal compounds

2,3-dihydro-2-(4-nitrophenyl)-1*H*-naphtho[1,2-e][1,3]oxazine (4i): ^{1}H NMR (DMSO) δ :5.05 (s, 2H, N-CH_2), 5.56 (s, 2H, O-CH_2-N), 7.01-7.97 (m, 10H, Ar-H).

HRMS m/z : 307.02 (M^+).

Results and Discussion:

Herein, we wish to report the synthesis of 3, 4-dihydro-3-substituted-2*H*-naphtho[2,1-e][1,3]oxazine derivatives promoted by silicotungstic acid as a catalyst (Scheme I). We have considered the reaction of 2-naphthol (1 mmol), formalin (2 mmol) and 4 methyl aniline (1 mmol) stirred at room temperature condition as the model reaction.

To determine the appropriate concentration of the catalyst silicotungstic acid, it has been investigated the model reaction first without catalyst and very less product is obtained (i.e. trace) at different concentrations of catalyst like 2.5, 5, 7.5 and 10 % the product formed in 56,78, 91 and 91 % yields, respectively (Table 1). This indicates that 7.5 mol% of silicotungstic acid is sufficient for the best result by considering the reaction time and yield of product.

To study the concentration of catalyst loading for model reaction, the procedure was optimized using different molar concentrations of silicotungstic acid under Microwave condition. High yield of product 4d was observed using 7.5mol% of catalyst. From these results, it was evident that, the concentration of catalyst plays a crucial role to improve the result to greater extent. It was also observed that, there is no greater change in yields of product greater than7.5 mol% of catalyst.

To generalize this methodology, we subjected a series of other amine having electron-donating as well as electron withdrawing substituent to obtain the corresponding [1,3] Oxazine derivatives under the optimized reaction conditions. As Table 2 shows yields are good to excellent in most cases.

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STABILITY INDICATING RP- UPLC METHOD FOR LAMIVUDINE AND TENOFOVIR DISOPROXIL FUMARATE DETERMINATION FROM PHARMACEUTICAL DOSAGE FORMULATION

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Abstract:

In present study, a successful attempt has been made to develop RP-UPLC method for the simultaneous determination of Lamivudine and Tenofovir disoproxil fumarate from combined pharmaceutical drug formulation. Chromatographic separation of Lamivudine and Tenofovir disoproxil fumarate was achieved with gradient elution on Waters Acquity UPLC BEH C18; 150 mm length x 2.1 mm ID, 1.7 µm particle size with mobile phase A- Buffer pH 2.0 in Water and mobile phase B- Methanol at a wavelength 260 nm. The method was validated in the terms of its linearity, accuracy, precision, robustness, ruggedness, LOD and LOQ. Linearity of the method was found to be in the concentration range of 150-450 µg/mL for both Lamivudine and Tenofovir disoproxil fumarate with correlation coefficient greater than 0.999 for both the analytes. The total eluting time for the both components is less than six minutes. Proposed method was found to be simple, precise, novel, rapid and accurate and can be successfully applied for routine quality control analysis and simultaneous determination of Lamivudine and Tenofovir disoproxil fumarate in combined pharmaceutical drug formulations [1-7].

Keywords: RP-UPLC, Lamivudine, Tenofovir Disoproxil Fumarate, Pharmaceutical Drug Formulations and Validation

Abbreviations:

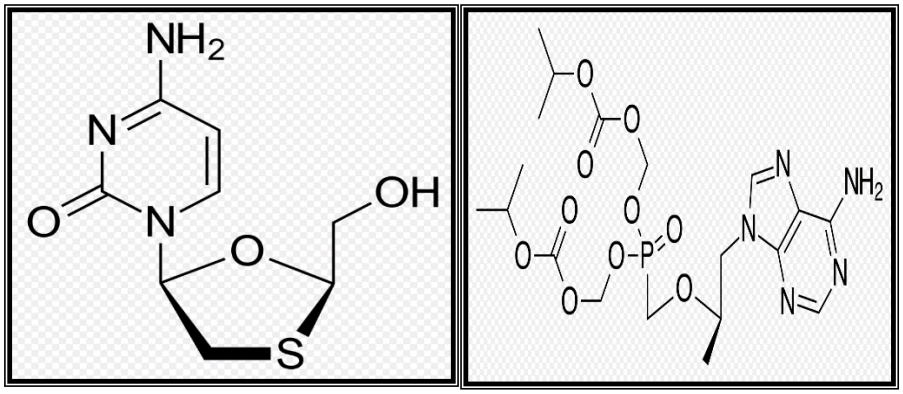
LAM- Lamivudine

TDF- Tenofovir Disoproxil Fumarate

Introduction:

In the topical countries like India, the major problems of health arise due to improper lifestyle, unhealthy environmental conditions, unhygienic and substandard food. Infections caused by the microorganisms like, fungi, protozoa, virus are the most common. In many cases, drugs with two active ingredients are prescribed to the patients to have an added advantage. Lamivudine is an antiviral medicine that prevents human immunodeficiency virus (HIV) or hepatitis B virus from multiplying in the body. Lamivudine is a nucleoside analogue and reverse transcriptase inhibitor used in the therapy of human immunodeficiency virus (HIV) and hepatitis B virus (HBV) infection to disrupt viral DNA synthesis. Tenofovir disoproxil fumarate, is a pro-drug, fumaric acid salt form of tenofovir, a nucleoside reverse transcriptase inhibitor analog of adenosine Tenofovir disoproxil fumarate is prescribed to treat HIV and chronic hepatitis B virus (HBV) in adults[8].

Structure:



Lamivudine **Tenofovir disoproxil fumarate**

Uplc method development:

Optimization of chromatographic condition:

Before developing any Chromatographic method, one must review the nature of the sample and goals of the separation. The sample related information that needs to be known prior to UPLC method development is: Sample solubility and number of components present, Nature of the sample, Chemical structures (functionality of the components), Molecular weight of components and Concentration range of the components in the sample of the interest.

The various parameters that were considered in the development process are:

Mode of separation, Selection of stationary phase, Selection of mobile phase, Selection of detection method (Detector used), Method validation

Mode of separation:

In the present research work, a reverse phase mode of the separation was employed taking in to account the polar nature of Lamivudine and Tenofovir disoproxil fumarate, their solubility in methanol and water. Therefore, a Reverse Phase mode of separation was chosen for simultaneous determination of Lamivudine and Tenofovir disoproxil fumarate using UPLC.

Selection of stationary phase/ Chromatographic column:

The column is the heart of UPLC separation process. The availability of stable, high-performance column is essential in developing a rugged, reproducible UPLC method.

The column is selected depending on the nature of the solute and the information about the sample. The number of theoretical plates (N) is an important characteristic of a column.

N- Defines the ability of the column to produce sharp, narrow peaks for achieving good resolution. In the method development, peak shape is equally important. Columns that provide symmetrical peaks are always preferred. In the present research work, Acquity UPLC BEH C18; 150 X 2.1 mm, 1.7 µm (Make- Waters) was selected for the analysis.

Selection of mobile phase:

In Liquid chromatography, the solute retention is governed by partition coefficient of the solute, which depends on the interactions of the solute with the stationary and the mobile phase. For a given stationary phase, the partition coefficient of a solute will depend upon the mobile phase. The nature and the composition of which has to be judiciously selected in order to get an appropriate and required solute retention.

Solvent polarity is the key word in chromatographic separations, since the polarity of mobile phase decides the retention time. Polar mobile phases give rise to high solute retention in normal phase and low solute retention in reverse phase liquid chromatography. The choice of the mobile phase for a given separation constitutes a very important stage in producing a good separation in UPLC. Methanol and acetonitrile are the most popular solvents in UPLC, both are water miscible, have comparatively low viscosity, low surface tension and readily available in pure form hence they mostly constitute the mobile phase. In the present research work, the best

resolution was obtained with gradient elution using the Mobile Phases i.e. Mobile phase A- Buffer pH 2.0 and Mobile phase B- Methanol.

Material and Methods:

Chemicals and reagents:

Standard Cefixime and Cloxacillin were obtained from local pharmaceutical company with claimed purity above 99.0%. All the solutions were prepared in double distilled water. All the necessary reagents used i.e. water and methanol (HPLC grade). Mobile phase was filtered using 0.45µm syringe filter made by Millipore whereas; Whatman's filter paper No.41 (purchased from local market) was used in the preparation of sample solution

Apparatus and chromatographic conditions:

Instruments:

UPLC:

Waters Acquity UPLC H-class is an Ultra-performance liquid chromatographic system with a quaternary, high-pressure mixing pump inline vacuum degassing and PDA Detector with Chromeleon software.

Chromatographic Mode	Gradient
Column	Waters Acquity UPLC BEH C18; 150 mm length x 2.1 mm ID, 1.7 µm particle size
Wavelength	260 nm
Column oven temperature	45 °C
Autosampler temperature	25 °C
Injection Volume	5.0 µl
Flow rate	0.5 ml/min
Buffer pH 2.0	Weigh and dissolve 2.16 g of 1-octane sulphonic acid sodium salt in 1000 ml of purified water. Add 1 ml of Triethylamine and mix well. Adjust pH to 2.0 with orthophosphoric acid.
Mobile Phase	Mobile phase A- Buffer pH 2.0 Mobile phase B- Methanol
Diluent	Water: Methanol (50:50 v/v)

Solution preparation:

Preparation of 300 µg/mL and 300 µg/mL solution of standard for Lamivudine and Tenofovir disoproxil fumarate [LAM + TDF]

Weighed accurately 15 mg of Lamivudine standard and 15 mg Tenofovir disoproxil fumarate standard transfer it into a 50 ml standard flask, added 35 ml of diluent and sonicate to dissolve. Allowed it to cool at room temperature, mixed well and made up to the volume with diluent to obtain 300 µg/mL of Lamivudine and 300 µg/mL of Tenofovir disoproxil fumarate. This solution was used as working concentration of Lamivudine and Tenofovir disoproxil fumarate and used as 'Standard'.

Preparation of sample solution for Lamivudine and Tenofovir disoproxil fumarate [LAM + TDF]

Commercial brand containing of Lamivudine and Tenofovir disoproxil fumarate in combination was procured. Each brand contained a label claim of 300 mg of Lamivudine and 300 mg of Tenofovir disoproxil fumarate per tablet.

Ten tablets were weighed and powdered for the analysis. The powder (about 912 mg) equivalent to 300 mg of Lamivudine and 300 mg of Tenofovir disoproxil fumarate was accurately weighed, transferred into 100 ml standard flask; added 70 ml of diluent and sonicate to dissolve. Allowed it to cool at room temperature, mixed well and the mixture was sonicated for 30 mins, finally volume of the solution was made up to 100 mL with diluent (Stock solution). The solution was filtered through 0.45 µm membrane filter paper and 2 mL of stock solution was diluted to 20 mL with the diluent to obtain a solution containing 300 µg/mL of Lamivudine and 300 µg/mL of Tenofovir disoproxil fumarate. This solution was used as working concentration of Lamivudine and Tenofovir disoproxil fumarate and used as 'Sample'.

The validation parameters studied for the simultaneous determination Lamivudine and Tenofovir disoproxil fumarate are as mentioned below:

Analytical method validation: [9-10]

System suitability:

System suitability test is used to verify that the system has adequate reproducibility for the analysis to be carried out. It also verifies the proper functioning of the operating system. The test was carried out by injecting 5 µL of the

standard solution containing 300µg/mL of LAM and 300µg/mL of TDF i.e. [at their working concentration] into stabilized chromatographic system, under optimized chromatographic conditions (Table 1).

Specificity:

Specificity is the ability of the method to assert the presence of the analyte unequivocally in the presence of other components that are present. To show that the other constituents present in the sample formulation do not interfere with the retention times of Lamivudine and Tenofovir disoproxil fumarate. The peaks corresponding to LAM and TDF in the sample solution were identified by comparing with the resulting chromatograms of the sample, with that of standard Lamivudine and Tenofovir disoproxil fumarate (Table 1).

Limit of Detection [LOD] and Limit of Quantification [LOQ]:

Limit of Detection [LOD] is the lowest concentration of the analyte that can be detected under the operational conditions of the method. Limit of Quantification [LOQ] is defined as lowest concentration of the analyte that can be determined with acceptable precision and accuracy, under the operational conditions of the method. Standard deviation of responses (σ) and slope (S) was used to establish LOD (LOD = σ/S X 3.3) and LOQ (LOQ = σ/S X 10), respectively. LOD and LOQ for Lamivudine were 36.5 µg/mL and 110.6 µg/mL for Tenofovir disoproxil fumarate were found to be 19.2 µg/mL and 58.1 µg/mL respectively is given in Table 1

Linearity and range:

The linearity for Lamivudine and Tenofovir disoproxil fumarate was observed simultaneously by addition of standard solution. The linear working range for LAM was found between 150 to 450 µg/mL and for TDF it was found between 150 to 450 µg/mL The calibration curves were constructed with concentration (C) against peak area. The slope, intercept, regression equation and correlation coefficient for the LAM and TDF was obtained is given in Table 1 and Figure 1-3.

Intraday and interday precision:

The intra-day and inter-day precision was used to study the variability of the method. It was checked by recording the chromatograms of sample solutions of LAM and TDF at working level i.e. 100% both at intra-day (six times within 24 hour) and inter-day (six times during 3 days intervals) to check the precision. The mean % RSD

for intra-day and inter-day precision was found to be less than 1.0% for both CFX and CLX. Result of intra and inter day precision studies are given in Table 1.

Assay

The developed chromatographic method was used for simultaneous determination of Lamivudine and Tenofovir disoproxil fumarate from commercial brand of formulation. The sample solutions were analysed by the developed method described above. Chromatograms were recorded under the optimum experimental conditions. Resulting peak area of Lamivudine and Tenofovir disoproxil were measured and the amount of Lamivudine and Tenofovir disoproxil fumarate calculated using already constructed calibration graph. Result of assay studies are given in Table 2.

Calculation formula for determination of % Assay content is detailed below;

$$\% \text{ Assay} = \frac{\text{Peak Area of Sample X Weight of Std X Sample Dil. Factor X Potency of Std}}{\text{Avg. Peak Area of Std X Std Dil. Factor X Weight of Sample X 100 X Avg. Weight X Label claim}} \text{ X } 100$$

Robustness:

The robustness of the method was examined by the consistency of peak height and peak shape with the deliberately small changes in the experimental parameter. It is a measure of its capacity to retain unaffected by small, but deliberate variations in method parameters and provides an indication of its reliability during normal usage. Robustness of the method was performed by intentionally modifying the chromatographic conditions such as composition of mobile phase, change in flow rate and change in oven temperature. The chromatographic parameters of each analyte such as retention time, tailing factor, resolution and theoretical plates were measured at each changed condition. In the robustness study, the influence of small, deliberate variations of the analytical parameters on retention time of the drugs was examined. The following two factors were selected for change: Change in the pH of buffer for mobile phase A by ± 0.2 of the original flow in the proposed analytical method i.e., from pH 2.0 to 1.8 and 2.2. Change in column oven temperature by ± 2°C of original Temperature, i.e., change in oven temperature from 45 °C to 43°C and 45 °C to 47°C.

One factor at the time was changed to estimate the effect. The working concentration solution of both the drugs was applied onto the column. A number of replicate analyses (n = 3) were conducted for evaluation of each change of factors. It was observed that there were no marked changes in the chromatograms, which demonstrated that the RP-UPLC method developed is robust.

Accuracy (Recovery):

The recovery was used to evaluate the accuracy of the method. Accuracy of the method was determined using the method of varying weight of sample for sample preparation. A weight of sample was varied at different concentrations of preanalyzed sample solutions and analyzed by proposed method. The percentage recovery was determined at different levels i.e. from 50% to 150% level. The results of recovery analysis for Lamivudine and Tenofovir disoproxil fumarate are shown in Table 3.

Result and Duscussion:

In the present work conditions were optimized for development and validation of a simple and accurate HPLC method for simultaneous quantification of Lamivudine and Tenofovir disoproxil fumarate in combined pharmaceutical drug formulation. Method development was right from optimization of the condition and parameters i.e., selection of system, column, mobile phase, different composition of mobile phases have been tried. During optimizing the method, Methanol and Acetonitrile were choices as organic solvents. The cost of acetonitrile favored to choose methanol as solvent for further studies. The chromatographic conditions were optimized by using 1-octane sulphonic acid sodium salt, triethylamine and orthophosphoric acid as a buffer for mobile phase preparation. After a series of screening experiments, it was concluded that gradient elution using buffer pH 2.0 and methanol gave better peak shapes and resolution, finally mobile phase A- Buffer pH 2.0 and mobile phase B- Methanol is the most appropriate composition because both the components were eluted with good resolution and good peak shape. Under the described experimental conditions, sharp peaks that belong to LAM and TEN were obtained with gradient elution at retention time of 1.3 min and 4.6 min respectively (Figure 1). The developed chromatographic method was validated using ICH guidelines. A new chromatographic method has been developed and subsequently validated for the

simultaneous quantification of Lamivudine and Tenofovir disoproxil fumarate from a combined drug formulation. The advantages of this method for analytical purposes lie in the rapid determination, its cost effectiveness, easy preparation of the sample, good reproducibility.

Table 1: Method validation parameters for the determination of Lamivudine and Tenofovir disoproxil fumarate

Parameters	**Values**	
	Lamivudine	**Tenofovir disoproxil fumarate**
System suitability Theoretical Plates- Tailing Factor-	 More than 10835 1.0	 More than 73396 1.0
Linearity range (µg/mL)	150 to 450 µg/mL	150 to 450 µg/mL
Slope (m) [a)]	6979.3	6350.3
Intercept(c) [a)]	52820	54108
Correlation coefficient (R^2)	0.9999	1.0000
LOD (µg/mL)	36.5 µg/mL	19.2 µg/mL
LOQ (µg/mL)	110.6 µg/mL	58.1 µg/mL
Intraday precision (n=6)	0.2%	0.1%
Interday precision (n=6)	0.2%	0.2%
Assay	98.8% to 99.0%	102.6% to 103.0%
Recovery	99.1% to 101.5%	98.1% to 102.0%

Sample details

Brand Name: TEMOLAM (HETRO HEALTH CARE LTD)

Batch No.: 2011512

Active ingredients: Lamivudine -300 mg and Tenofovir disoproxil fumarate -300 mg

Excipients: q. s.

Colour: Lake indigo carmine and Titanium dioxide IP

Table 2: Result of Assay studies of Lamivudine and Tenofovir disoproxil fumarate

Brand Name	**TEMOLAM (HETRO HEALTH CARE LTD)**	
	Lamivudine	**Tenofovir disoproxil fumarate**
Labeled claim (mg)	300 mg	300 mg
Drug found in (mg)	301.5 mg	299.4 mg
% RSD (n=6)	0.3	0.3
% Assay	100.5 %	99.8 %

Table 3: Results of Recovery studies of Lamivudine and Tenofovir disoproxil fumarate

Analyte	**Level**	**RSD (%) (n = 6)**	**Recovery (%)**	
			Min.	**Max.**
Lamivudine	50%	0.2	101.1	101.5
	100%	0.1	100.2	100.4
	150%	0.0	99.1	99.1
Range			**99.1**	**101.5**
Tenofovir disoproxil fumarate	50%	0.4	101.1	102.0
	100%	0.2	99.2	99.6
	150%	0.1	98.1	98.3
		Range	**98.1**	**102.0**

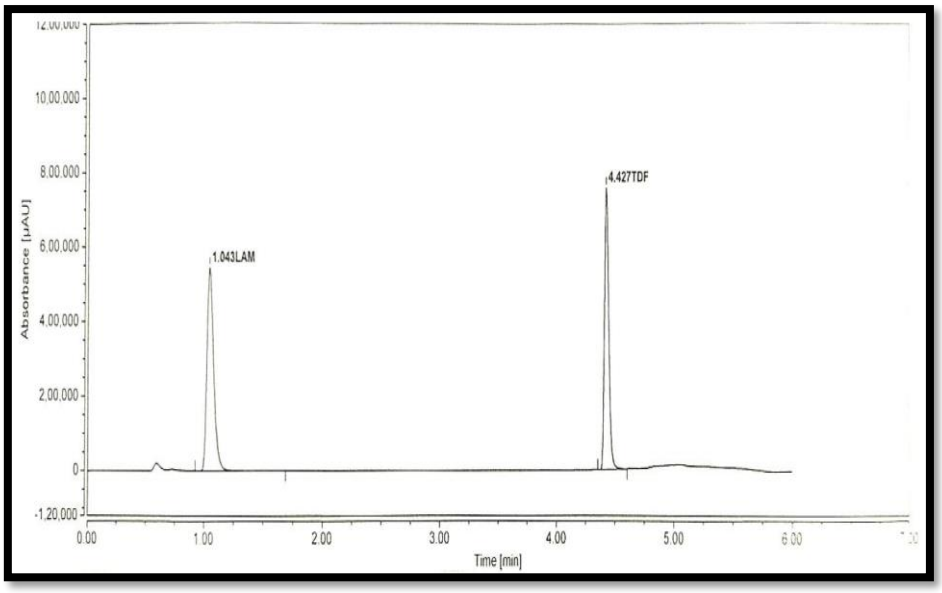


Figure 1: UPLC Chromatogram for Standard Lamivudine and Tenofovir disoproxil fumarate respectively

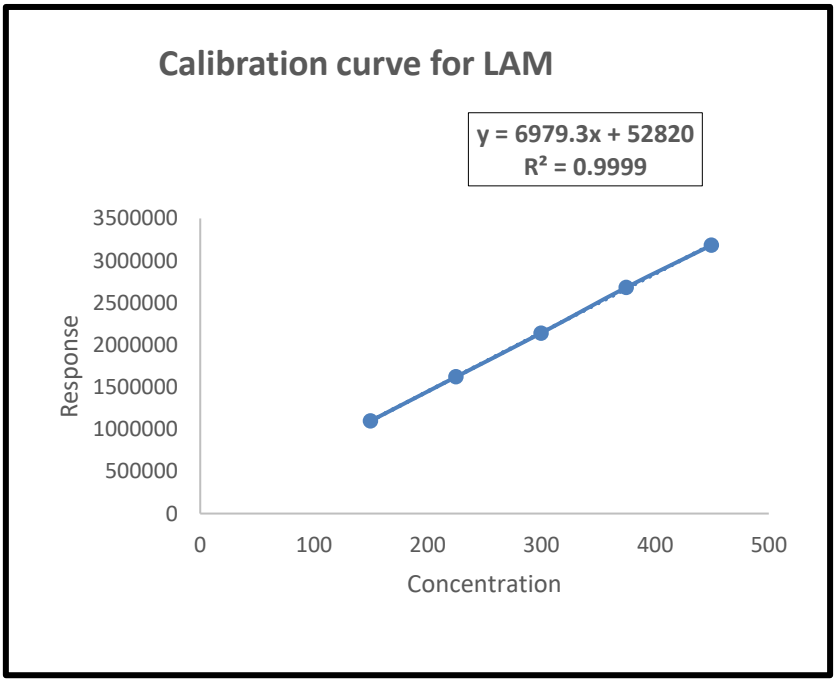


Figure 2: Linear working range for LAM

Y-axis – Peak Area

X-axis- Concentration of Drug in µg/m

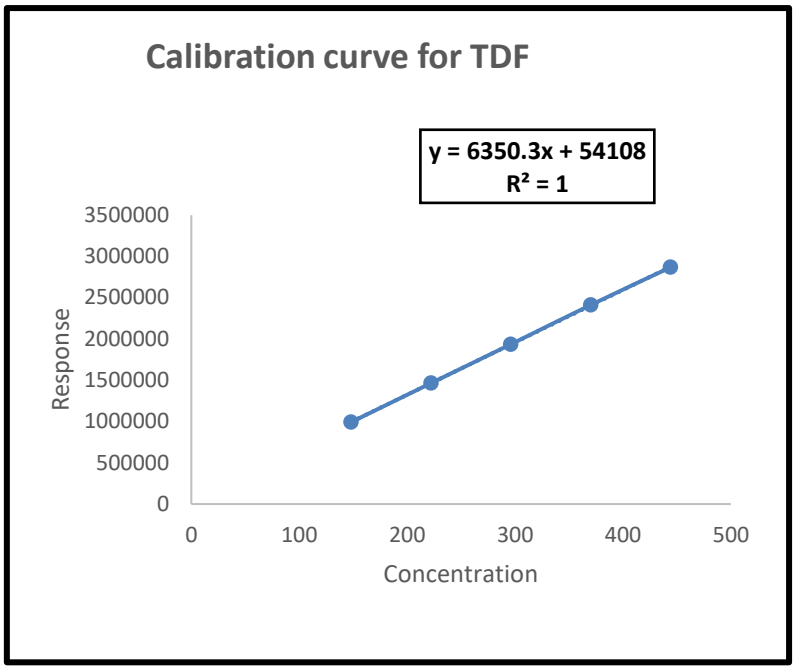


Figure 3: Linear working range for LAM

Conclusion:

In addition to above mentioned points, the proposed method is found to be more simple, economic, accurate and practical. Thus, presented method can be recommended for simultaneous determination of Lamivudine and Tenofovir disoproxil fumarate in routine quality control analysis in combined drug formulations.

Acknowledgement:

Authors thank the Department of Chemistry St. Xavier's College, Mumbai for providing us all the necessary instrumentation facilities and their technical assistance.

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PERCEPTIONS INTO SYNTHESIS AND STRUCTURAL PROPERTIES OF $ZnFe_2O_4$ VIA SOL-GEL, SOL-GEL AUTO-COMBUSTION, CO-PRECIPITATION AND HYDROTHERMAL METHODS

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Abstract:

In this study, $ZnFe_2O_4$ nanoparticles were successfully produced through the utilization of sol-gel, sol-gel autocombustion, co-precipitation, and hydrothermal techniques. The research delved into the synthesis and structural characteristics of the synthesized nanoparticles employing a variety of methodologies such as X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FTIR), field emission scanning electron microscopy (FESEM), energy dispersive X-ray analysis (EDX), among others. Analysis of XRD data indicated the presence of a cubic structure (spinel phase) with Fd-3m space group in the samples. Calculations based on Debye-Scherrer's equation revealed average crystal sizes of 15.85, 13.84 nm, 24.14, and 18.14 nm for the $ZnFe_2O_4$ crystals using different techniques. The FTIR data unveiled vibrational modes representing the surface functionality. Furthermore, the SEM analysis results displayed detailed microstructural morphology, showcasing non-uniform grain shapes. Elemental compositions were identified through the EDX data. This investigation contributes valuable insights into the relationship between synthesis methodologies and the resulting properties of $ZnFe_2O_4$, presenting a thorough comprehension of how these factors can be adjusted for specific technological applications.

Keywords: $ZnFe_2O_4$, WH Plot, FESEM, EDX

Introduction:

Ferrites (MFe_2O_4) with M = Zn, Cu, Ni, Co etc. are essentially magnetic materials composed of ferric oxide ions, resulting from the reaction of metal oxides to

form a magnetic substance. Recently, spinel magnetic ferrites NPs have garnered considerable interest across various fields like ceramics, catalytic materials, semiconductors, sensors, among others. These ferrites offer many benefits, including suitability at higher frequencies, enhanced heat and corrosion resistance, and cost-effectiveness. They exhibit intriguing magnetic, electrical, and optical properties, such as high corrosion resistance, electrical resistivity, low eddy current, moderate saturation magnetization, and a wide range of coercivity. Thus, ferrites are ideal for a wide range of technological applications, finding uses in transformers, electric motors, microwave machineries, microchip technology, instrumentation, computers, power adaptation, magnetic recording, catalysis, and bioengineering [1-12].

$ZnFe_2O_4$ nanoparticles (NPs) are currently a focal point in research owing to their expanding technological uses. The groundwork was laid by Hilpert and furthered by Forestier, initiating the synthesis of various ferrites and opening up a promising avenue for future investigation. $ZnFe_2O_4$ nanoparticles, belonging to the Fd-3m. space group, representing a spinel ferrite example utilized as catalysts, sensors, and photo-catalysts, among other applications. $ZnFe_2O_4$ is a mixed metal oxide possessing a spinel structure that presents a diverse range of applications thanks to its distinct blend of magnetic, electrical, and structural properties. Its ferrimagnetic nature with moderate saturation magnetization and high Curie temperature makes it suitable for magnetic sensors and data storage devices. The semiconducting attributes of $ZnFe_2O_4$ allow for its use as a catalyst and photocatalyst in chemical reactions, water splitting, and the decomposition of organic pollutants. In the realm of electronics and spintronics, it contributes to devices like magnetic field sensors and memory storage. The frequency-dependent dielectric and magnetic losses of $ZnFe_2O_4$ are advantageous in microwave devices such as filters and resonators. Furthermore, $ZnFe_2O_4$ NPs show promise in biomedical applications for targeted drug delivery and magnetic hyperthermia, and can be explored for energy storage in batteries and supercapacitors. The ability to synthesize $ZnFe_2O_4$ with control over particle size and shape further boosts its versatility across these applications [13-31].

Experimental section:

1. Sol-gel method

10 mM of $Zn(NO_3)_2.6H_2O$ and 20 mM of $Fe(NO_3)_3.9H_2O$ were combined with the right amount of ethanol (40 mL), then mixed with 30 mM of Citric acid (CA). The molar ratio of Zn^{+2}: Fe^{+3}: CA was determined to be 1: 2: 3. The reaction blend was agitated at 500-600 rpm for 1-3 hours at temperatures between 70-90°C, resulting in a wet gel-type solution. This gel was subsequently crushed using a mortar to produce finer powders. The resultant powdered sample was then subjected to heat in a furnace at 600°C for further processing. The wet gel was dried in an oven at 120°C for 24 hours to form a dry gel [1,4-5]. Nanoparticles were isolated, and Figure 1 illustrates the synthesis process of $ZnFe_2O_4$ using the sol-gel technique.

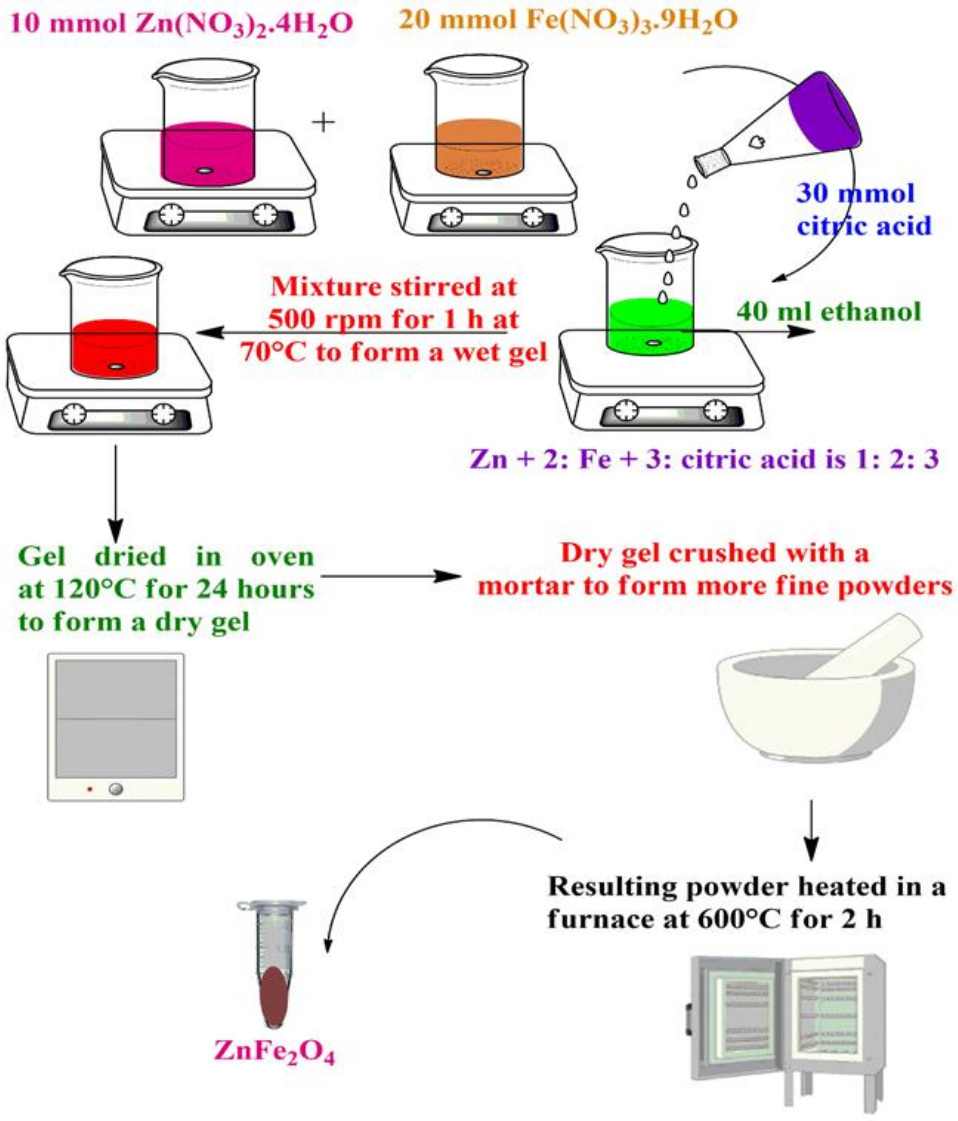

Fig. 1: Scheme for Synthesis of $ZnFe_2O_4$ by Sol-gel Method

2. Sol-gel autocombustion method

The sol-gel auto-combustion method for synthesizing $ZnFe_2O_4$ involves combining sol-gel chemistry with a combustion process. Initially, a precursor solution is prepared using zinc nitrate and iron nitrate, along with a chelating agent like citric acid. Adjusting pH triggers gelation, forming a gel-like network. Heating the gel to around 300°C leads to auto combustion, where the organic components burn off, and the gel combusts, leaving behind $ZnFe_2O_4$ powder. The powder is then calcined at 600°C to improve crystallinity and phase purity. This method offers precise control over stoichiometry and yields nanoscale particles. Fig. 2 shows synthesis diagram of $ZnFe_2O_4$ by sol-gel auto combustion method.

3. Co-precipitation method

The coprecipitation method for synthesizing $ZnFe_2O_4$ involves precipitating the metal ions from a solution to form a precursor. In this process, aqueous solutions containing zinc nitrate and iron nitrate, are mixed in a stoichiometric ratio of 1:2. A base i.e. NaOH, is added to the solution to adjust the pH and induce precipitation of a mixed metal hydroxide. The resulting precipitate is washed and dried to remove impurities, then calcined at ~ 600°C to promote the formation of $ZnFe_2O_4$ and improve crystallinity. This method is advantageous for its simplicity and efficiency with good control over particle size and uniform composition. Fig. 3 shows synthesis diagrams of $ZnFe_2O_4$ by co-precipitation method.

4. Hydrothermal method

The hydrothermal method for synthesizing $ZnFe_2O_4$ involves the reaction of zinc and iron precursors in a high-pressure, high-temperature aqueous environment to produce the desired compound. In this process, an aqueous solution containing zinc nitrate and iron nitrate, is prepared in a stoichiometric ratio of 1:2. The solution is placed in a sealed autoclave, and pH is adjusted using ammonium hydroxide to promote the formation of a precursor. The autoclave is then heated here at 250°C and maintained under high pressure for 12.5 hours. This process facilitates nucleation and growth with controlled size and morphology. After cooling, the product is collected, washed, and dried. This method offers advantages such as high purity, uniform particle distribution, and the ability to tailor particle size and shape. Fig. 4 shows synthesis diagrams of $ZnFe_2O_4$ by hydrothermal method.

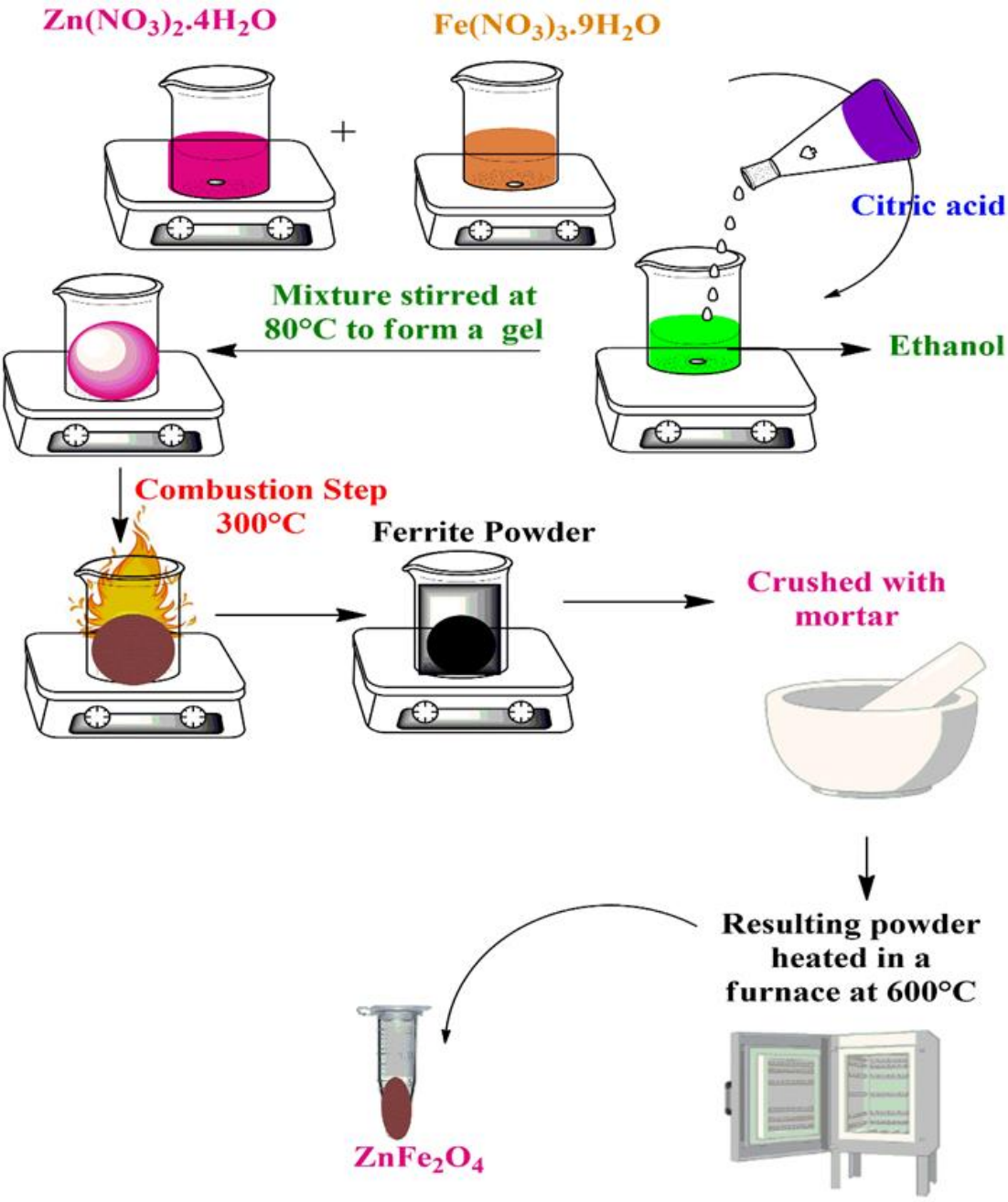

Fig. 2: Scheme for Synthesis of $ZnFe_2O_4$ by Sol-gel Autocombustion Method

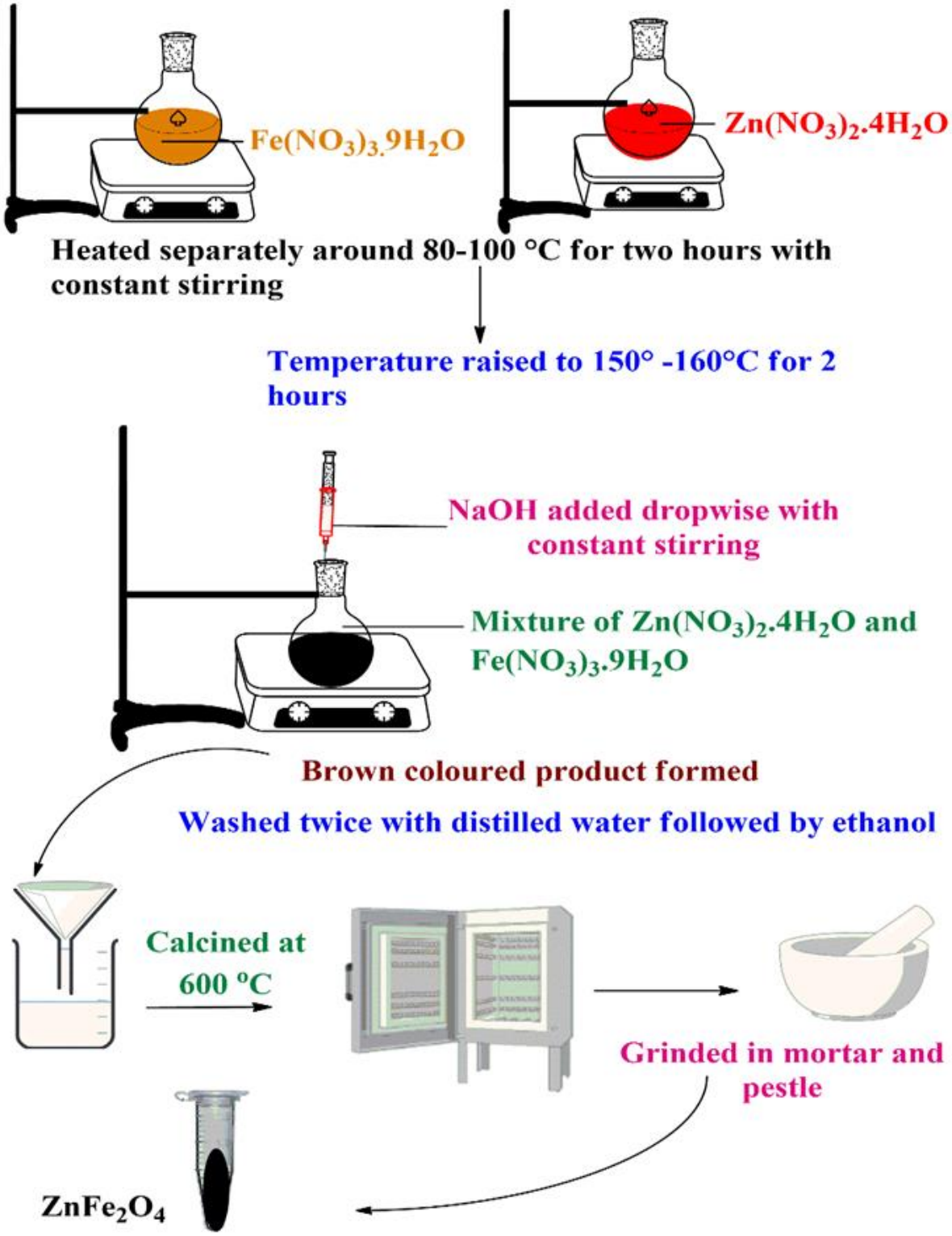

Fig. 3: Scheme for Synthesis of $ZnFe_2O_4$ by Co-precipitation Method

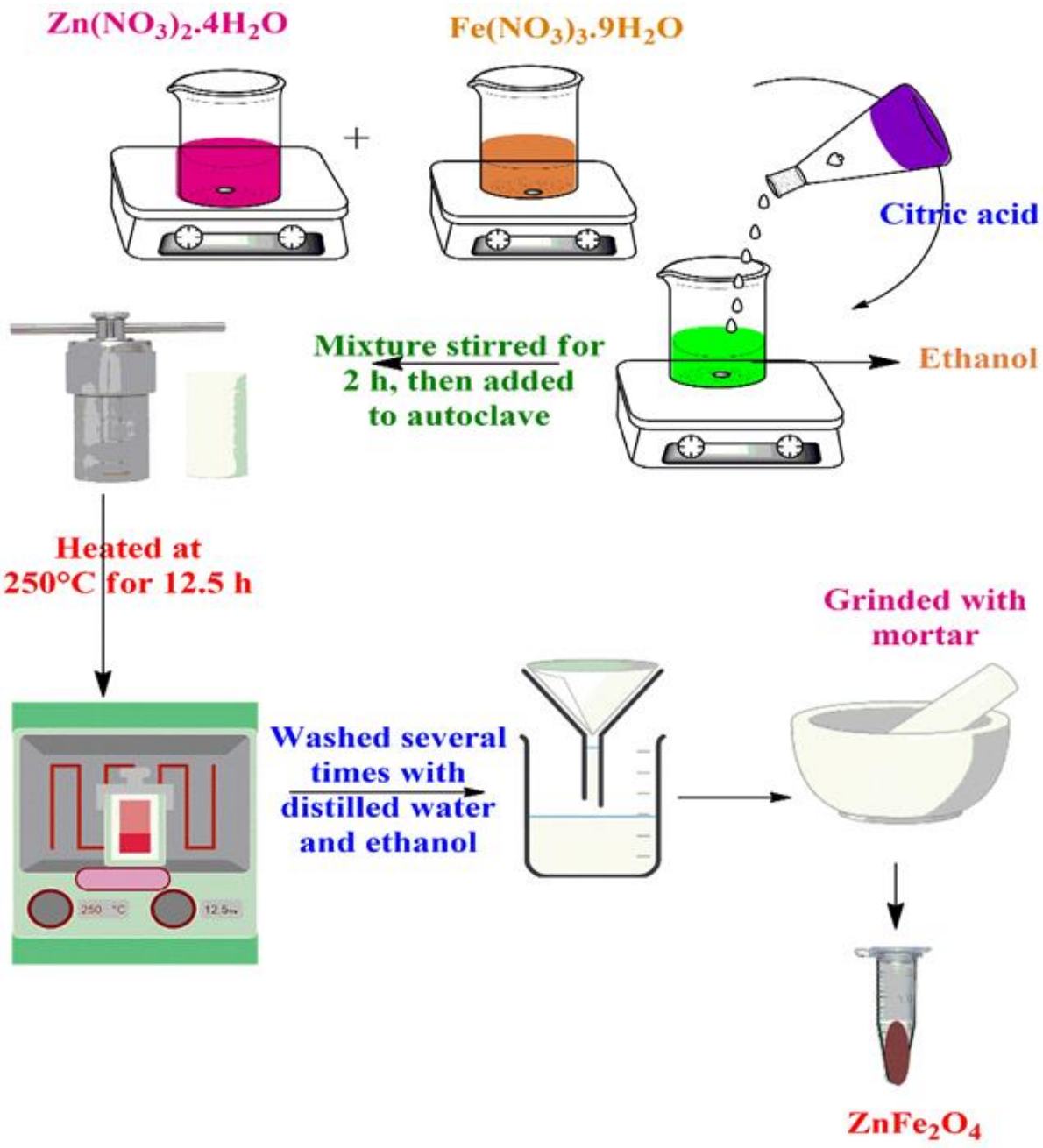

Fig. 4: Scheme for Synthesis of $ZnFe_2O_4$ by Hydrothermal Method

5. Characterization used

To investigate the phase transition of $ZnFe_2O_4$ samples, we employed an X-ray diffractometer. FTIR spectra showed the chemical makeup and surface functioning of the NPs. Samples have been created in KBr for FTIR spectrum in the 400-4000 cm^{-1} range, using an FT-IR spectrometer. The microstructural morphologies have been investigated with field emission scanning electron microscopy (FESEM) apparatus.

The elemental compositions were determined by energy dispersive X-ray analysis (EDX), linked to FESEM.

Result and Discussions

1. Structural analysis

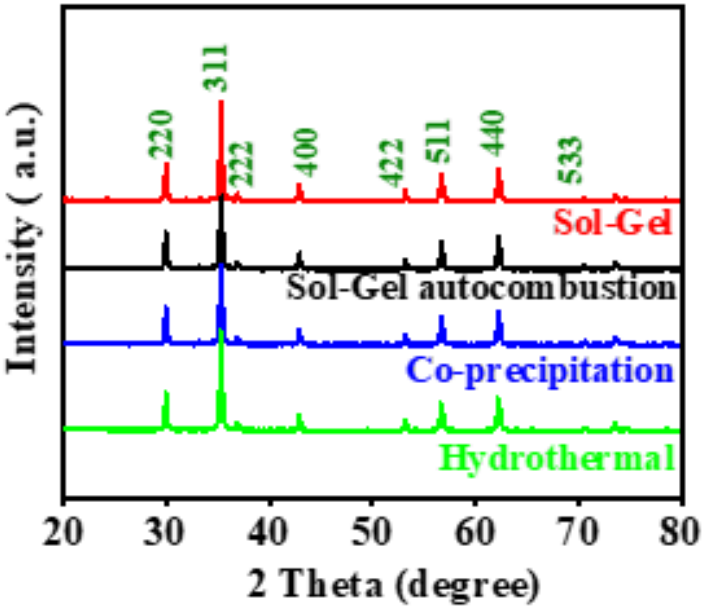

Fig. 5: Comparison of XRD of $ZnFe_2O_4$ synthesized by all 4 methods

An XRD examination was done to assess phase identification, purity, and crystalline structure, as shown in Figure 5. XRD investigation reveals that the diffraction pattern fully matches the anticipated cubic spinel structure of ferrites NP, which has been identified as $ZnFe_2O_4$ (JCPDS no. 82-1049). The diffraction pattern for the complete spinel ferrite nanoparticle sample reveals its crystalline structure [1,4-5]. The Scherrer equation, represented in equation (1):

$$\tau = K\lambda / \beta \cos\theta \qquad [1]$$

where τ is the mean size of ordered domains, K is a dimensionless shape factor (0.94), λ is the X-ray wavelength (1.54A^o), β is the line broadening at FWHM (0.44), θ is Bragg angle (17.65). Using Debye-Scherer's equation (eq. 1), average sizes of $ZnFe_2O_4$ synthesized by sol-gel, sol-gel autocombustion, co-precipitation and hydrothermal methods were determined to be 15.85, 13.84 nm, 24.14 and 18.14 nm respectively. Moreover, there was good agreement between the notable peaks of $ZnFe_2O_4$ synthesized by all these methods.

2. Williamson- Hall (WH) Plot

The widening seen in XRD peaks is a result of smaller crystallite size (L) and micro-strain (Ɛ). Therefore, it is evident that the crystallite size calculated using the Debye Scherrer equation may not always be accurate. To address this issue, a well-known method called the W-H plot, introduced by Williamson and Hall, is employed:

$$\beta \, Cos \, \theta = k\lambda/L + 4\varepsilon \, Sin \, \theta \qquad [2]$$

where symbols hold their usual meanings. The W-H plot for $ZnFe_2O_4$ samples is illustrated in Fig. 6(a) to (d) showing a straight line in each case. The slope 'm' directly provides the crystallite size value, while the intercept 'c' helps determine the crystallite size. The obtained values of L and Ɛ are 17.35 nm and 1.46 x 10^{-3}, 15.95 nm and 2.04 x 10^{-3}, 28.95 nm and 1.94 x 10^{-3}, 21.08 nm and 1.51 x 10^{-3} respectively for all 4 methods. The crystallite size values determined using the W-H plot tend to be higher than those from the Debye Scherrer equation due to a strained crystal structure, surface defects, and micro-strain contributions [1-7].

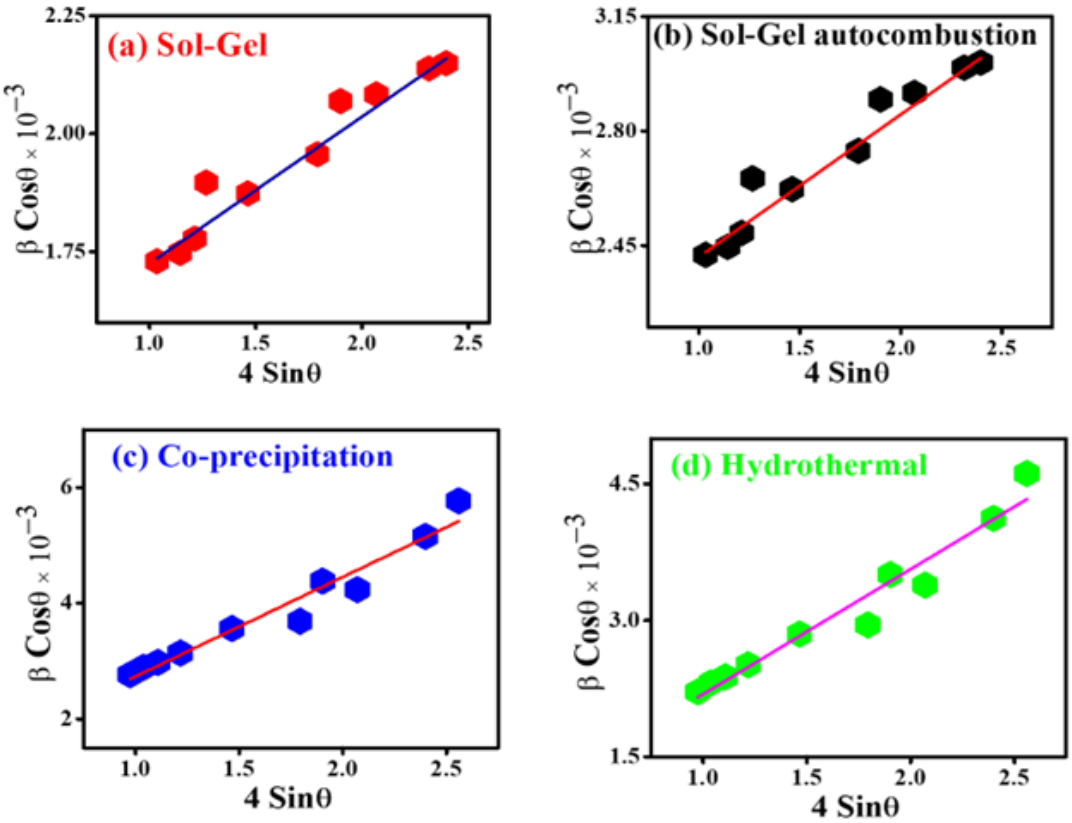

Fig. 6: Williamson-Hall plot of samples $ZnFe_2O_4$ synthesized by (a) sol-gel, (b) sol-gel auto-combustion, (c) co-precipitation and (d) hydrothermal methods

3. FTIR analysis

The FTIR spectra of $ZnFe_2O_4$ prepared using various methods like sol-gel, sol-gel autocombustion, co-precipitation, and hydrothermal methods are illustrated in Fig. 7. In these spectra, characteristic peaks are visible around 450 cm^{-1} and 550 cm^{-1}, representing vibrations of metal oxygen bonds like Zn-O and Fe-O. Specifically, the peak at 550 cm^{-1} corresponds to Fe–O bonds. Spinel ferrites generally exhibit two absorption bands below 600 cm^{-1}, with the sharp peak at 562 cm^{-1} indicating Fe-O bond stretching vibration and the peak at 1641 cm^{-1} signifying -OH bending vibration in the FTIR spectrum of $ZnFe_2O_4$. The frequency bands range from 580 to 600 cm-1 for high-frequency and 400 to 436 cm^{-1} for low-frequency, reflecting vibrations of tetrahedral (T_d) and octahedral (O_h) components in spinel ferrites. Additionally, the peak at 3400 cm^{-1} is linked to O-H stretching of surface-adsorbed water, while the formation of a peak around 1600 cm^{-1} is attributed to H-OH bending in water molecules [1,4-5].

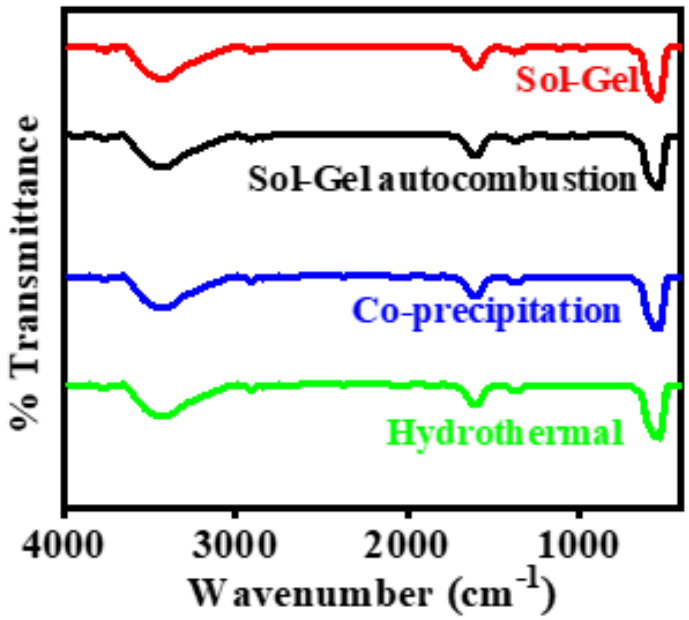

Fig. 7: FTIR spectra of $ZnFe_2O_4$ synthesized by all 4 methods

4. Microstructural analysis of FESEM data

To observe particle sizes, particle morphologies, surface properties, and microstructures of $ZnFe_2O_4$, FESEM images were taken as shown in Fig. 8 (a) to (d). As a result of electrostatic forces, magnetic attraction, and Van der Waal's

interactions, both specimens clumped. It was observed that $ZnFe_2O_4$ particles demonstrated a non-uniform distribution of grains. The majority of the particles exhibited irregular shapes, including triangular, spherical, and cylindrical. The grain size followed the descending order of co-precipitation> Hydrothermal> Sol-gel > Sol-gel autocombustion methods, as evidenced by the histogram curves as well; possibly due to a higher tendency to cluster [5-6]. Morphological investigation reveals the existence of tiny spaces in the chemically synthesized substances caused by sample porosity or void percentage, as well as agglomeration. Porosity was calculated with ImageJ software and determined to be 11.38%, 15.37%, 5.83%, and 12.86%, respectively. The frequency distribution histogram graph of ZnFe2O4 samples showed a Gaussian distribution, as illustrated in Fig. 9 generated with ImageJ program. The typical grain sizes of $ZnFe_2O$ respectively using sol-gel, sol-gel auto-combustion, co-precipitation, and hydrothermal processes are about 18, 9, 38, and 23 nm.

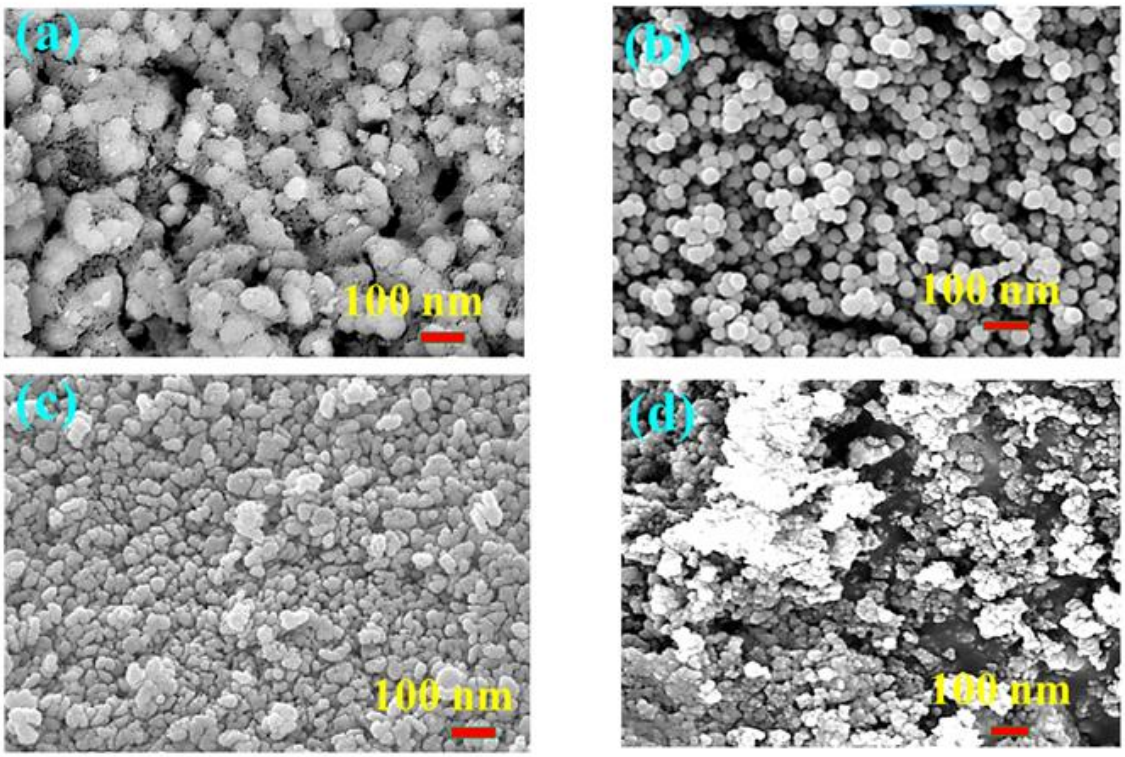

Fig. 8: FESEM images of $ZnFe_2O_4$ via (a) sol-gel, (b) sol-gel auto-combustion, (c) co-precipitation and (d) hydrothermal methods

5. Energy-Dispersive X-ray spectroscopy (EDX)

Energy-dispersive X-ray spectroscopy (EDX) is used to analyze the elemental composition of $ZnFe_2O_4$ synthesized by different methods such as sol-gel, autocombustion, coprecipitation, and hydrothermal is illustrated in Fig. 10 (a) to (d) respectively. Regardless of the method, the spectra show the presence of zinc (Zn), iron (Fe), and oxygen (O), reflecting the expected stoichiometry of $ZnFe_2O_4$, ideally at a molar ratio of 1:2:4. Deviations from this ratio due to impurities or uneven element distribution is hardly seen. While the sol-gel and hydrothermal methods generally yield well-crystallized $ZnFe_2O_4$ with controlled composition, autocombustion introduces slight impurities from combustion by-products, and coprecipitation varying if not tightly controlled [5-6]. Table 1 summarizes the structural parameters of $ZnFe_2O_4$ synthesized by all 4 methods.

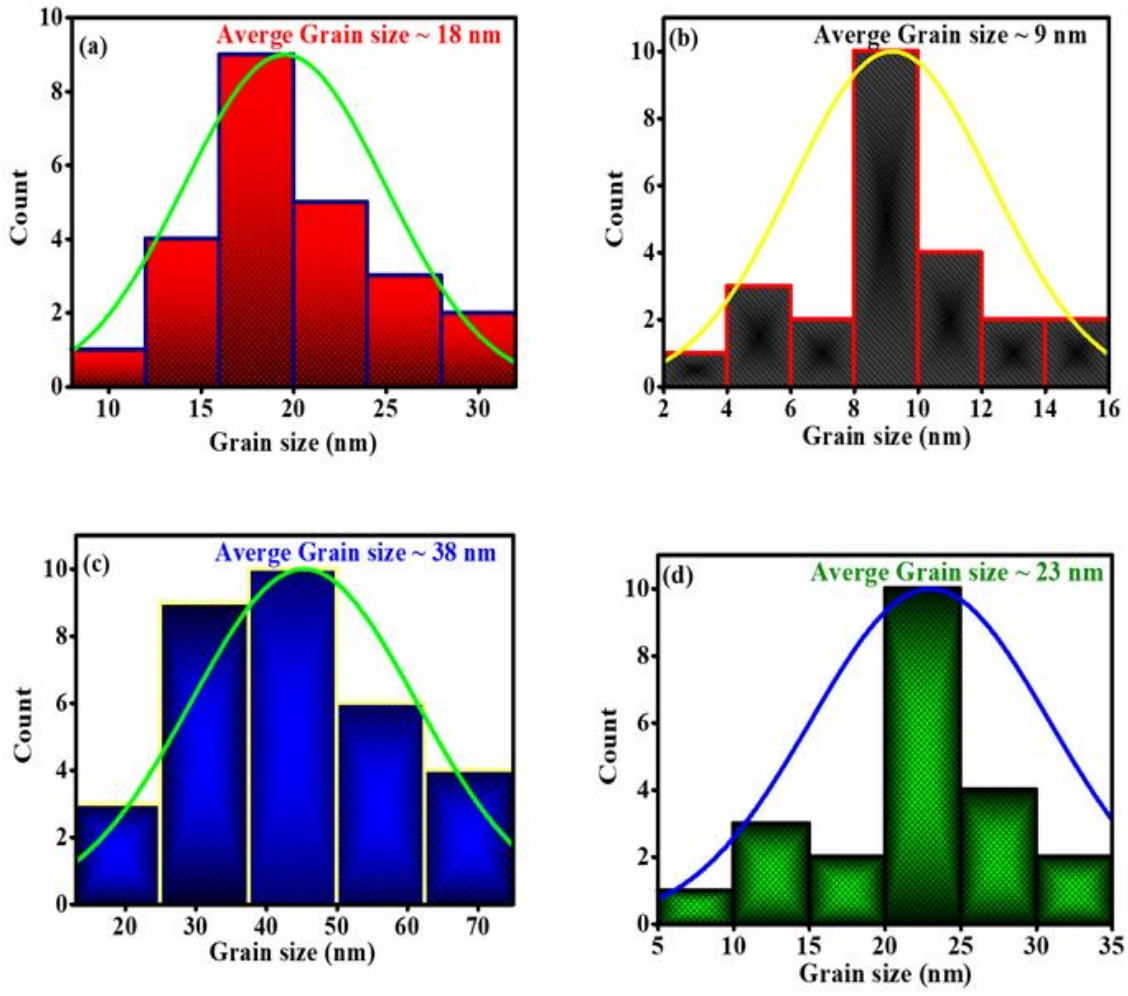

Fig. 9: Histogram for average grain size for $ZnFe_2O_4$ via ((a) sol-gel, (b) sol-gel auto-combustion, (c) co-precipitation and (d) hydrothermal methods

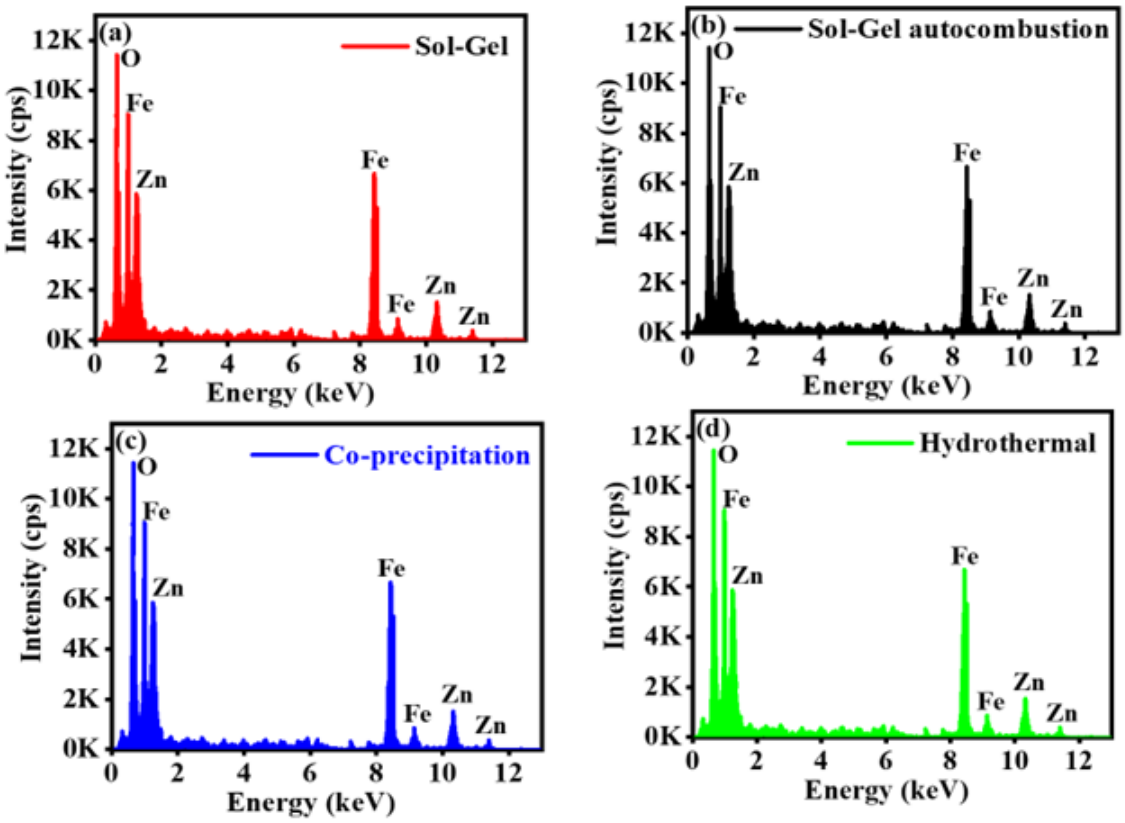

Fig. 10: EDX spectra for average grain size for $ZnFe_2O_4$ via (a) sol-gel, (b) sol-gel auto-combustion, (c) co-precipitation and (d) hydrothermal methods

Table 1: Structural parameters of $ZnFe_2O_4$ synthesized by sol-gel, sol-gel autocombustion, co-precipitation and hydrothermal methods

Sample	Crystallite size (nm) By Scherer's Eq.	Crystalline size (nm) By WH Eq.	Strain (x 10^{-3})	Grain Size (nm)
$ZnFe_2O_4$ (Sol-gel)	15.85	17.35	1.46	18
$ZnFe_2O_4$ ((Sol-gel autocombustion)	13.84	15.95	2.04	9
$ZnFe_2O_4$ (Co-precipitation)	24.14	28.95	1.94	38
$ZnFe_2O_4$ (Hydrothermal)	18.14	21.08	1.51	23

Conclusion:

Spinel $ZnFe_2O_4$ functionalized nanoparticles were effectively produced using zinc nitrate and iron nitrate as precursors in procedures such as sol-gel, sol-gel auto combustion, co-precipitation, and hydrothermal. These methods might also be used to create a variety of different ferrite nanoparticles important to nanotechnology. The detailed processes for synthesizing, functionalization, and characterization reveal the successful generation of spinel ferrites, which have the potential to be a useful tool in the future. The present task is to investigate the potential uses of samples prepared for efficient utilization in the following decade.

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A COMPREHENSIVE REVIEW ON DRUGS AND ENVIRONMENTAL ASPECTS

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Abstract:

This review offers an ample examination of the connections between pharmaceuticals and the environmental implications of the sustainable development goals, climate change, and environmental sustainability. It underscores both direct and indirect associations, emphasizing the considerable impact of pharmaceuticals on the local environment. Furthermore, it assesses the latest enacted policies that have direct and indirect effects on environmental conservation and ecosystems. Pharmaceuticals enter the environment through both their use and disposal, with incorrect disposal also household unused medicine being a major source of environmental pollution and public health risks. Similarly, emerging global standards will be employed to systematically incorporate environmental protection into the planning and monitoring of alternative development endeavors. The insights and conclusions drawn from this analysis are presented herein.

Keywords: Pharmaceutical Waste, Eco-Friendly Drug Discovery, Household Waste, Consumer Behavior, Management of Pharmaceutical Waste.

Introduction:

The term 'drug' originates from the French word 'Drogue' which refers to a dry herb and is defined as a substance utilized in the prevention, diagnosis, and treatment of diseases in humans or other animals. Presently, drugs are derived from various sources: chemical synthesis (50%), higher flowering plants (25%), microorganisms (12%), minerals (7%), and animals (6%). This demonstrates that the

majority of drugs come from chemical synthesis, microorganisms, and minerals. Drugs can be prepared via natural, semi-synthetic, or synthetic routes. In this regard, the pharmaceutical industry plays a crucial role in promoting human and animal health. During the synthesis of synthetic drugs, numerous chemicals are utilized as raw materials, including organic solvents, reagents, catalysts, and intermediates. Similarly, the methods of preparation vary from one drug to another.

The environmental impact of pharmaceuticals becomes a concern when waste generated from drug preparations and their residues affects soil, water, and air quality, as well as indirectly impacting animals and the food chain (Yannik *et al.,* 2013). Manufacturing synthetic drugs typically occurs in remote pharmaceutical company locations or industrial areas, leading to the disposal of toxic drug-related waste in forests, rivers, or sewage systems (Wilkinson *et al.,* 2022). The discharge of such waste into wastewater can have particularly severe consequences in regions or communities with inadequate or non-existent wastewater treatment facilities. Additionally, pharmaceutical pollution can arise when residues are excreted after drug consumption or when unused or expired medications are improperly discarded. The amount of household medication turning into waste is significant and steadily increasing due to consumption trends. Thousands of molecules with unknown properties regarding their usefulness and toxicity undergo various stages of analysis and clinical studies. It is uncertain whether their acute and chronic toxicity, teratogenicity, and other effects have been studied before their disposal, and all these molecules can ultimately find their way into water bodies, eventually reaching humans through plants and animals.

1. Pharmaceutical industry

As we are aware, pharmaceutical intermediates and products offer numerous beneficial characteristics, but they also possess properties that can contribute to significant environmental pollution (Kadam *et al.,* 2016; Mohammed *et al.,* 2021). In accordance with government policies and standards, wastewater generated by pharmaceutical industries must undergo treatment before being discharged into water bodies, or if feasible, reused for other purposes (Larsson., 2014). Various parameters related to water quality standards, such as temperature, pH, turbidity, conductivity, total hardness, total suspended solids (TSS), heavy metals, chemical

oxygen demand (COD), and biological oxygen demand (BOD), need to comply with specified threshold limits. The degree of pollution caused by industrial effluents can be accessed through simple analysis methods to determine COD and BOD levels. Quantitative chemical analysis of water samples is conducted using analytical techniques such as classical wet chemistry, along with sophisticated instruments for identifying trace metals and organic compounds.

It is mandatory for every pharmaceutical company to have its own effluent treatment plant (ETP) for wastewater treatment, and as per government regulations, each company must display pollution-indicating parameters. To oversee compliance, government bodies such as the Central Pollution Control Board and State Pollution Control Boards are established. However, despite these measures, numerous companies discharge untreated wastewater into remote areas or rivers, exacerbating the environmental situation. Recent studies indicate that aquatic life, including fish and other animals, may exhibit abnormalities due to wastewater effluents.

Similarly, incomplete drug metabolisms are discharged into municipal sewage through urine and feces, remaining untreated due to the lack of advanced treatment techniques. The accumulation of complex pharmaceuticals in tissues can lead to acute and chronic damage, resulting in behavioural changes and reproductive harm.

India is a leading producer of active pharmaceutical ingredients (APIs) and drugs globally. According to regulations from the “United States Federal Environmental Protection Agency” (FEPA), drugs classified as hazardous are designated as "P-listed" for acute toxicity, "U-listed" for toxicity, and "D-listed" for chemicals exhibiting corrosivity, ignitability, reactivity, and other properties. Presently, in India, there are some regulations established by the Bureau of Indian Standards (BIS) to manage the level of pharmaceutical-contaminated wastewater or drinking water. The Indian Ministry of Environment has categorized pharmaceutical manufacturing as a "red category" activity due to the hazardous waste it generates. Additionally, biomedical waste (BMW) generated from hospitals is a significant source of waste and should be segregated into labelled bags, collected, and properly disposed. Indian industries, to mitigate pollution, are subject to various rules and acts, such as...

Environmental Protection Act-1986,

Drugs and Cosmetic Act-1940,

The Water (Prevention and control of pollution) Act-1974,

New Drugs, Medical Devices and Cosmetics Bill, 2022.

The importance of sustainable drug discovery and manufacturing to mitigate environmental impacts is becoming increasingly evident. Sustainability involves meeting present needs without compromising the needs of future generations and respecting ecological boundaries. This requires ethical and environmentally conscious practices. The pharmaceutical and biotech sectors are gradually transitioning towards such practices to reduce environmental strain. However, this transition is complex and multifaceted, lacking a simple checklist of solutions. Instead, ongoing initiatives aim to improve conditions and mitigate existing negative impacts.

Today, several pharmaceutical companies are adopting Good Manufacturing Practices (GMP) in their production to reduce environmental pollution. Similarly, they review the manufacturing process to avoid releasing antibodies into the environment. Hence, to minimize environmental hazards, it requires…

Sustainable and eco-friendly drug discovery

Sustainability is a pressing concern across various industries, including healthcare and clinical research. Hospitals and clinical facilities generate significant waste and carbon emissions, while medical research depletes non-renewable resources at an alarming rate, posing enduring environmental risks. Sustainable drug discovery and development represent crucial steps towards reducing the environmental footprint of the healthcare and clinical industries (Chaturvedi *et al.,* 2017).

Yet, the shift towards sustainable clinical research practices cannot be solely driven by biotech and pharmaceutical sponsors. Contract research organizations (CROs) must also embrace sustainability to minimize environmental impact throughout the drug discovery and development processes. It's increasingly apparent that the healthcare and clinical research sectors cannot sustain their current practices without causing irreversible harm to the planet. Therefore, doctors and scientists must address immediate concerns while also prioritizing long-term environmental preservation.

Evelien Wynendaele, *et al*. presented 10 sustainability principles in Drug Discovery, aimed at guiding the drug discovery process. These principles serve as a framework for scientists and healthcare practitioners to evaluate their existing research and development methodologies, encouraging them to adopt more environmentally friendly practices.

- **Ecological and environmental impact:**

This concept, also known as "benign by design," emphasizes minimizing the environmental impact throughout the entire drug development process, spanning from discovery and development to distribution and clinical utilization. Sustainable practices include responsibly sourcing materials for new drugs, utilizing tissue culture technologies instead of potentially endangered plants, and repurposing waste from one process as materials for another.

- **Medical Needs:**

The principle of addressing medical needs prioritizes meeting unmet or neglected therapeutic needs over the profitability of pharmaceuticals. Scientists can achieve this by repurposing existing clinical data and medical solutions to target these underserved areas, thereby fostering a more sustainable drug discovery process.

- **Green chemistry:**

Green chemistry involves employing sustainable chemical processes to synthesize and analyze compounds, thereby minimizing waste, carbon emissions, and the consumption of non-renewable resources. Techniques such as computer-aided drug design can help identify synthetic pathways that reduce resource usage and waste generation.

- **Artificial Intelligence (AI) and big data:**

Utilizing AI and big data in drug discovery and development enhances efficiency by automating various phases of the process. By accurately predicting parameters and results for in vivo testing, scientists can reduce costs, resource consumption, and the reliance on animal and human subjects in the early phases.

- **Root causes:**

Focusing on addressing the root cause of illnesses rather than solely managing symptoms can lead to a more sustainable healthcare model. Prevention of diseases

reduces the need for medications and equipment, emphasizing a shift towards preventative measures over reactionary treatments.

- **Risk and decision models:**

AI and computational modeling can aid in reducing the risks associated with drug discovery by identifying optimal pathways and mechanisms of action. By assessing sustainability beforehand, scientists can make informed decisions to minimize environmental impact.

- **Biomarkers and bioinformatics:**

Utilizing biomarkers and bioinformatics enables more precise medication, reducing ineffective or unnecessary prescriptions. This approach enhances accuracy in diagnosis and treatment, promoting cost-effectiveness and sustainability in medication usage.

- **Cost-effectiveness:**

Ensuring equitable pricing and transparency in the pharmaceutical industry promotes cost-effectiveness and social justice in healthcare. Companies should prioritize socially-acceptable costs over profitability, fostering equitable access to medications.

- **Lean discovery process:**

Implementing a lean discovery process involves utilizing fast, efficient, and value-adding methods in drug discovery. By optimizing experimental designs and focusing on essential avenues, researchers can minimize resource consumption and maximize breakthrough potential.

- **Responsible research and innovation:**

Pharmaceutical and biotech companies, along with academic institutions, have a responsibility to conduct research that benefits the public and minimizes unnecessary resource usage. Prioritizing socially beneficial research initiatives over purely profitable endeavors ensures responsible and sustainable innovation in healthcare.

Lastly, transitioning drug development processes to more sustainable and environmentally friendly methods, both in discovery and testing phases, could help minimize the industry's environmental impact.

Healthcare industry and sustainable healthcare

While medical services are indispensable for preserving and prolonging human life, it's imperative to acknowledge that the healthcare industry's activities can pose ecological threats. The healthcare sector contributes to carbon emissions through its day-to-day operations, including energy usage, heating, cooling, and supply chain activities. Additionally, significant amounts of pharmaceuticals, worth billions of dollars, are discarded due to inadequate packaging. Hospitals generate substantial waste annually, particularly in the form of single-use plastics such as syringes, drug packaging, surgical equipment, and personal protective kit.

Although the necessity of these items and equipment is undeniable for patient care, ongoing research reveals the long-term environmental ramifications, underscoring the necessity for implementing more sustainable medical and clinical practices. Globally, the healthcare sector accounts for approx 5% of greenhouse gas emissions. Hospitals and clinical production facilities have the potential to significantly reduce their carbon footprints, which could greatly reduce the costs associated with global health.

Furthermore, climate change, manifested through increasingly frequent and severe weather events, can lead to a rise in water and food borne diseases, mental health issues, and medical emergencies, thereby posing a threat to public health. Over time, deteriorating air quality and compromised food safety in urban areas may exacerbate health risks. Environmental degradation can severely hinder the healthcare industry's capacity to deliver safe and effective care worldwide. Factors such as hospital evacuations, power outages, shortages of medical supplies, and other disruptions can further compromise the quality of care provided. Decreasing the carbon footprint of the medical sector could result in significant enhancements to overall human health, as well as notable social and economic advantages. Adopting more eco-friendly practices, such as reducing single-use plastics, would not only lead to less plastic production and waste but could also pave the way for more cost-effective alternatives.

The COVID-19 pandemic has resulted in a surge in the consumption of certain over-the-counter (OTC) medications, exacerbating the issue of self-medication. There's been an increase in the use of self-medication for respiratory symptoms, even

in attempts to prevent COVID-19. Antibiotics, a category of pharmaceuticals, are being excessively consumed, posing a growing concern. These drugs are utilized for treating human and animal diseases, promoting growth, and as prophylactics. The elevated consumption of antibiotics leads to their increased presence in the environment, potentially impacting organisms' survival, reproduction, metabolism, and population, and altering ecosystem dynamics such as biomass production and biodiversity. Moreover, antibiotic overuse contributes significantly to antibiotic resistance. Some antibiotics degrade easily, like penicillin, while others persist longer, such as fluoroquinolones and tetracyclines, remaining in the environment for extended periods and accumulating in higher concentrations.

Pharmaceuticals enter the environment through both their use and disposal, with incorrect disposal being a major source of environmental pollution and public health risks (Rogowska *et al.,* 2022; Nakiganda *et al.,* 2023). Discarded medicines flushed down toilets and sinks infiltrate sewage systems, possibly leaking into freshwater systems. Reducing the release of pharmaceutical residues into sewage systems can be achieved by curbing excessive drug consumption, particularly of OTCs, or by improving wastewater treatment methods. Additionally, improper disposal of unused medications can be minimized through take-back schemes and public awareness campaigns.

However, there is currently no global strategy for limiting the production and disposal of pharmaceutical waste. Improper handling of unused medications by individuals poses a significant challenge. Wastewater is a primary source of pharmaceutical pollution in the Baltic Sea, and existing treatment plants are not equipped to remove micro-pollutants, necessitating costly and lengthy modernization processes. Proper disposal of medications by Baltic Sea region residents is crucial to reducing pharmaceutical pollution in the sea. Enhancing unnecessary drug collection systems and raising public awareness about their purpose and environmental impact are vital steps towards reducing pharmaceutical pollution.

In light of the above, this study aims to highlight the significant problem of improper handling of unused or expired drugs by society, which affects waste management systems and the environment. The publication also discusses actions taken in various countries to mitigate the impact of pharmaceutical waste on the

environment. Household medications can become waste due to various reasons, such as non-adherence, early recovery, therapy changes, or prescription and purchasing errors. Estimates suggest that household medication waste ranges from 3% to as high as 50%.

The increase in chronic health conditions, the availability of low-cost generic treatments, and shifts in clinical practices have resulted in a surge in pharmaceutical prescriptions and usage. Consequently, the volume of unused medications being discarded is also rising, underscoring the growing importance of managing them in an environmentally responsible manner.

Improper disposal of expired or unused medication is prevalent and leads to substantial environmental pollution and public health hazards. Pharmaceutical substances discarded in regular household waste can seep into the environment, posing risks if not properly collected and treated. This improper disposal has three main implications: firstly, certain pharmaceuticals can harm ecosystems by causing increased mortality among aquatic species and inducing changes in their physiology, behavior, or reproductive patterns. Additionally, the release of antibiotics can spur the development of antimicrobial-resistant bacteria and mutations in animals. Secondly, there's a potential public health risk of accidental or deliberate misuse and poisoning if unused medication is retrieved from public or private waste bins. Thirdly, the disposal of unused pharmaceuticals represents squandered healthcare resources and economic losses.

Policy measures focusing on prevention and the collection of unused medication, along with improved consumer education, can help mitigate pharmaceutical household waste. Prevention through enhanced disease prevention strategies, personalized medicine, or better packaging sizing can reduce pharmaceutical waste. Establishing markets for and redistributing close-to-expiry medicines can also enhance supply-demand matching and curtail waste. However, eliminating unused medicines entirely is challenging due to factors like patients' changing treatment regimens or failure to adhere to prescribed medications.

The collection and disposal of unavoidable pharmaceutical waste should be tailored to each country's context and specific challenges. Separate collection is recommended where there's a risk of pharmaceuticals leaching or being misused

when disposed of in mixed waste. Extended producer responsibility schemes, particularly those with full national coverage and collection points at pharmacies, have proven effective in organizing environmentally sound separate collection and treatment. Alternative approaches, such as publicly funded take-back schemes, can also be useful but may not fully adhere to the polluter pays principle.

Moreover, limited consumer awareness regarding proper disposal methods and drug take-back programs undermines their effectiveness in many countries. Governments should develop or mandate producer responsibility organizations to conduct targeted communication campaigns to raise citizen awareness about proper disposal methods and the availability of drug take-back programs. A focus should be placed on educating people about the proper disposal of liquids, ointments, and creams, which are often discarded improperly. Non-governmental organizations (NGOs) also play a crucial role in raising awareness among the public about the disposal of unused medication, and governments should organize awareness campaigns in public spaces. Additionally, governments should mandate that pharmaceutical companies include appropriate disposal instructions on product containers or packaging materials.

The assessment of environmental and health hazards varies across nations. Some countries perceive these risks as significant and therefore establish distinct systems for collecting unused medication (Begum *et al.,* 2021; Erik *et al.,* 2023). Conversely, other nations have concluded that the potential environmental and health impacts are insufficient to warrant the separate collection of unused drugs. In certain countries, such as the Netherlands, Italy, and the United States, policies have been adopted to collect and redistribute unused or nearly expired medication to low-income individuals and those in need. In Australia, some programs are solely government-funded, while others receive financial support from the pharmaceutical industry or pharmacies. Additionally, voluntary collection events are organized in certain countries. In India, it is imperative to prioritize the implementation of such policies to mitigate pollution stemming from unused pharmaceuticals.

Some of the common methods to be implemented for the proper disposal of waste

Incineration

If the final disposal of separately collected unused medicines/biomedical waste is ideally done at a high temperature (more than 1000 °C), incineration is a better way for the destruction or removal of the substances of concern.

All these efforts aim for environmentally sound management of waste medicine for a better tomorrow.

Pollution control

Solid dust is an accumulation of the pollutants produced during the incineration process. The flue gases that exit the incinerator must be monitored in the chimney since they have specified restrictions.

Monitoring

Incinerators must adhere to specific standard operating procedures, and they are closely monitored by the state and central pollution control authorities. If any parameter exceeds the allowed limitations, they may act in accordance with the rules and apply environmental fines.

Liquid waste

All pharmaceutical industries need to process effluents as per norms to have a zero liquid discharge system, 100% of the liquid waste has to be recycled or evaporated and can be recovered for reuse.

Plastic recycling

According to the Pharmaceutical Waste Management Rules of 2016, Extended Producer Responsibility (EPR) is required for the pharmaceutical business to properly process and dispose of its waste. The plastic used in the pharmaceutical sector should be fully recyclable.

Suggestions for effectively managing pharmaceutical household waste:

Policies concerning pharmaceutical waste management should adopt a lifecycle perspective, integrating strategies that target the sources, users, and management of waste. These initiatives should involve all relevant stakeholders and employ a

combination of voluntary measures, economic incentives, and regulatory frameworks.

- Prioritise preventing unused or expired medicines.
- Utilise marketplaces and redistribution platforms for near-expiry medicines to optimise supply and demand, reduce waste, and save money.
- Customise unused medication collection and place collection units in public areas.
- During residential waste collection, the Municipal Corporation should implement a policy to collect unused pharmaceuticals.
- Pharmacists should establish collection units for unwanted pharmaceuticals and offer bonus points for proper disposal.
- The Counseling of patients should be done during hospitalization and on prescription by doctors.
- An awareness campaign should be organized for the effective implementation of the collection of unused household medicines.
- Regular reviews by government authorities can ensure the effective management of pharmaceutical household waste.

Conclusion:

Drugs and Environmental aspects play a crucial role in shaping public health and sustainability, as their protection, use, and disposal can have significant impacts on ecosystems, natural resources, and human well-being. Furthermore, the complex interactions between pharmaceuticals and the environment require careful consideration in order to mitigate any potential adverse effects on both our health and the planet. Therefore, it is imperative for stakeholders from both the pharmaceutical industry and environmental sector to collaborate in developing sustainable practices that prioritize the health of individuals and the environment alike.

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AN OVERVIEW IN RECENT ADVANCES IN GREEN CHEMISTRY

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Abstract:

Sustainable catalysis plays a pivotal role in advancing green chemistry principles by enabling efficient and environmentally friendly chemical transformations. This abstract provides an overview of sustainable catalysis, highlighting its importance, key principles, and applications. Sustainable catalysis involves the design and development of catalysts and reaction methodologies that minimize waste generation, energy consumption, and reliance on hazardous or scarce materials. Key principles include the use of renewable feedstocks, biodegradable catalysts, and benign reaction conditions. Applications of sustainable catalysis span various industries, including pharmaceuticals, fine chemicals, materials science, and energy. This abstract emphasizes the importance of sustainable catalysis in addressing global challenges related to sustainability, environmental protection, and resource conservation.

Keywords: Sustainable Catalysis, Green Chemistry, Catalyst Design, Renewable Feedstocks, Environmental Sustainability, Chemical Transformations.

Introduction:

Recent advances in green chemistry have propelled the field towards more sustainable and environmentally friendly practices. One notable advancement is the development of catalytic processes that utilize Earth-abundant metals or biocatalysts, reducing the reliance on scarce or toxic catalysts (Beller *et al.*, 2019). These catalysts enable selective and efficient transformations while minimizing waste and energy

consumption. Additionally, the exploration of novel reaction media, such as ionic liquids and supercritical fluids, has facilitated greener synthesis routes by eliminating the need for traditional volatile organic solvents (Jessop *et al.*, 2005). Furthermore, the integration of renewable feedstocks, such as biomass-derived building blocks, into chemical synthesis has garnered significant attention, offering a sustainable alternative to petrochemical-derived starting materials (Huber *et al.*, 2006). Another notable advancement is the emergence of sustainable materials, including biodegradable polymers, recyclable plastics, and renewable-based coatings, which contribute to reducing environmental impact across various industries (Auras *et al.*, 2010). Overall, these recent advancements signify a paradigm shift towards greener and more sustainable approaches in chemical synthesis and materials science, marking significant progress towards a more environmentally conscious future.

Introduction to green chemistry

Green chemistry, also known as sustainable chemistry, is a field that seeks to design chemical products and processes that reduce or eliminate the use and generation of hazardous substances. It is rooted in the principles of environmental sustainability, aiming to minimize the environmental impact of chemical processes while maximizing efficiency and safety. With growing concerns about environmental degradation, resource depletion, and climate change, green chemistry has emerged as a critical discipline for addressing these challenges and promoting a more sustainable future.

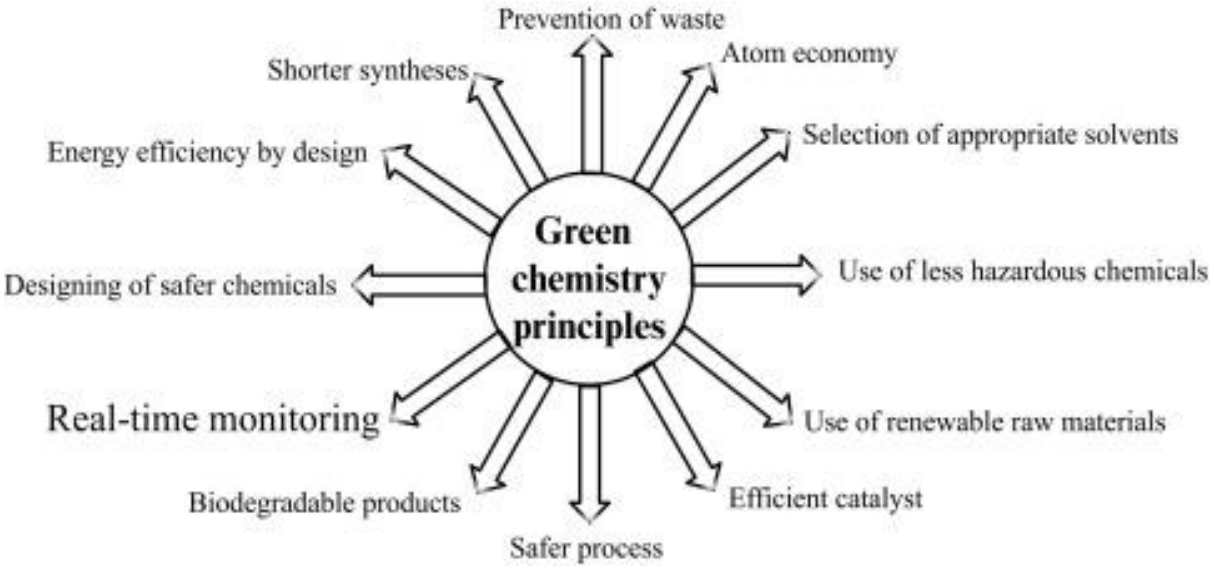

Fig. 1: Applications of Green Chemistry

1. Importance of green chemistry

The importance of green chemistry lies in its potential to revolutionize traditional chemical practices and mitigate their adverse effects on the environment and human health. Conventional chemical processes often rely on toxic solvents, generate hazardous waste, and consume large amounts of energy and resources. In contrast, green chemistry emphasizes the development of cleaner and more efficient alternatives that minimize waste generation, reduce energy consumption, and utilize renewable resources.

2. Principles of green chemistry

The principles of green chemistry, as outlined by Paul Anastas and John Warner, provide a framework for guiding the development of environmentally benign chemical processes. These principles include minimizing waste, designing safer chemicals and products, maximizing atom economy, and using renewable feedstocks (Anastas & Warner, 1998). By adhering to these principles, chemists can create more sustainable pathways for the synthesis of chemicals and materials.

3. Applications of green chemistry

Green chemistry principles are applicable across a wide range of industries, including pharmaceuticals, agrochemicals, materials science, and manufacturing. In the pharmaceutical sector, for example, green chemistry approaches can lead to the development of safer and more sustainable drug synthesis routes, reducing the environmental impact of pharmaceutical production (Constable *et al.*, 2007). Similarly, in the field of materials science, green chemistry principles can guide the design and synthesis of biodegradable polymers, renewable plastics, and eco-friendly coatings.

4. Challenges and opportunities

Despite the significant progress made in green chemistry research and applications, challenges remain in implementing sustainable practices on a global scale. These challenges include technological barriers, economic constraints, regulatory hurdles, and the need for interdisciplinary collaboration (Clark & Macquarrie, 2016). However, these challenges also present opportunities for

innovation, collaboration, and the development of new technologies that can drive the transition towards a more sustainable chemical industry.

Sustainable catalysis:

Catalysis plays a pivotal role in modern chemistry by facilitating chemical transformations with higher efficiency and selectivity. Sustainable catalysis aims to minimize environmental impact and resource consumption while maximizing the utilization of renewable feedstocks and energy sources. This overview explores various aspects of sustainable catalysis, including key principles, recent advances, and future prospects.

1. Principles of sustainable catalysis

Sustainable catalysis is guided by principles such as atom economy, selectivity, and the use of non-toxic and renewable catalysts (Anastas & Warner, 1998). By focusing on these principles, chemists aim to develop catalytic processes that minimize waste generation, energy consumption, and reliance on scarce or toxic materials.

2. Biocatalysis

Biocatalysis involves the use of enzymes or whole cells as catalysts for chemical reactions. Enzymes are highly selective and efficient catalysts that operate under mild conditions, making them ideal candidates for sustainable catalysis (Faber, 2011). Recent advances in biocatalysis include the engineering of enzymes for improved stability, selectivity, and substrate scope.

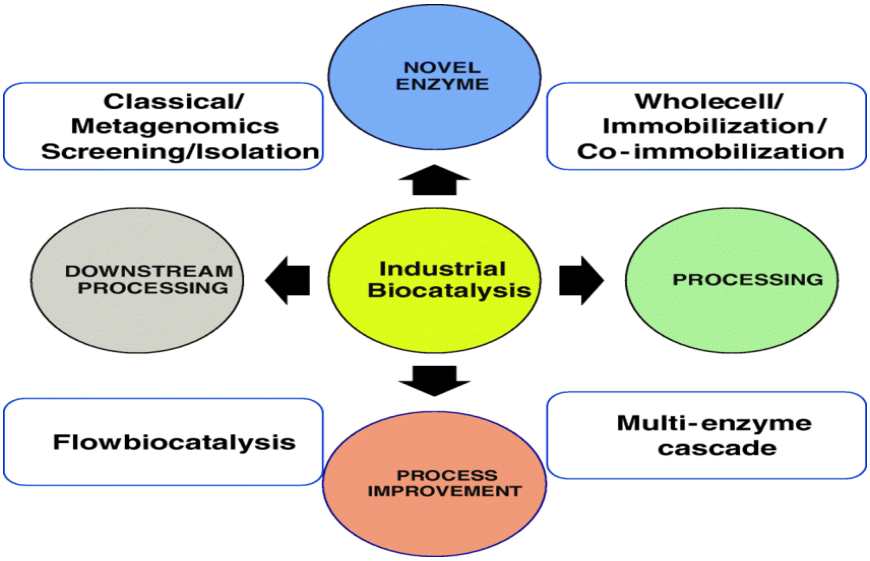

Fig. 2: Flow Diagram for Biocatalysis process

3. Heterogeneous catalysis

Heterogeneous catalysis involves the use of solid catalysts that are immobilized on a support material. These catalysts offer several advantages, including ease of separation, recyclability, and reduced environmental impact (Thomas *et al.*, 2005). Recent developments in heterogeneous catalysis include the design of nanoparticle-based catalysts with enhanced activity and selectivity.

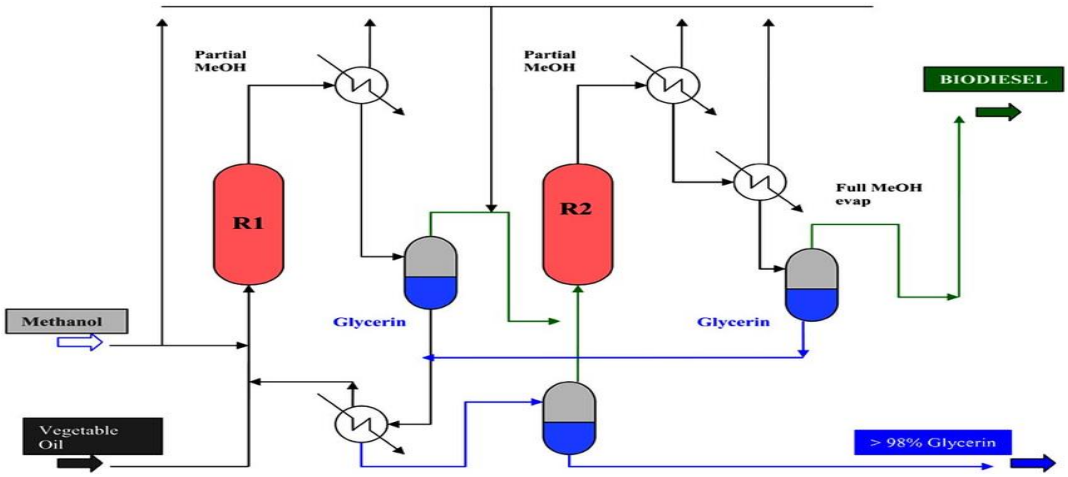

Fig. 3: Process of Heterogenous Catalysis

4. Homogeneous catalysis

Homogeneous catalysis involves soluble catalysts that are present in the same phase as the reactants. While homogeneous catalysis offers high selectivity and activity, challenges such as catalyst recovery and waste generation need to be addressed for sustainable applications (Cornils & Herrmann, 2003). Recent advances in homogeneous catalysis include the development of ligand design strategies for improved catalyst stability and selectivity.

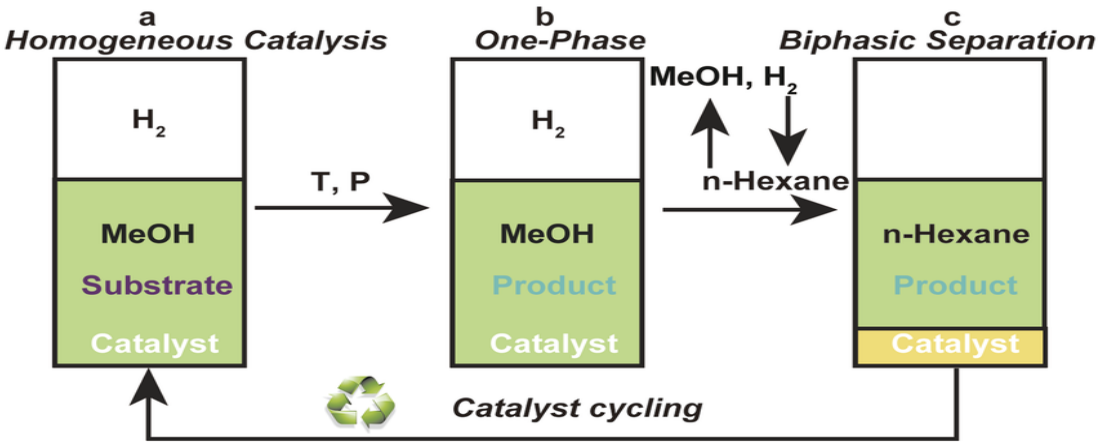

Fig. 4: Recovery by Homogenous Catalysis process

5. Renewable feedstocks in catalysis

The utilization of renewable feedstocks, such as biomass-derived compounds, presents opportunities for sustainable catalysis (Huber *et al.*, 2006). By integrating renewable feedstocks into catalytic processes, chemists can reduce reliance on fossil resources and contribute to the development of a bio-based economy.

6. Flow chemistry

Flow chemistry involves performing chemical reactions in continuous-flow systems, offering advantages such as improved reaction control, safety, and scalability (Baxendale *et al.*, 2006). Flow chemistry has emerged as a powerful tool for sustainable catalysis, enabling rapid optimization of reaction conditions and minimizing waste generation.

7. Photocatalysis

Photocatalysis utilizes light to initiate chemical reactions, offering environmentally benign pathways for synthesis (Xiang *et al.*, 2016). Photocatalysts can harness solar energy for driving chemical transformations, making them attractive for sustainable catalysis applications.

8. Chiral catalysis

Chiral catalysis involves the use of chiral catalysts to control the stereochemistry of reactions, enabling the synthesis of enantiomerically pure compounds (Jacobsen *et al.*, 1999). Sustainable chiral catalysis methods have been developed, including organocatalysis and biocatalysis, which offer environmentally friendly alternatives to traditional metal-based catalysts.

9. Metal-free catalysis

Metal-free catalysis has gained attention for its potential to avoid issues associated with metal contamination and toxicity (Narayan *et al.*, 2017). Recent advances in metal-free catalysis include the development of organic catalysts and photoredox catalysis for sustainable synthesis routes.

In conclusion, sustainable catalysis encompasses a diverse array of approaches aimed at reducing environmental impact, conserving resources, and promoting economic viability. By integrating principles of green chemistry into catalytic

processes, chemists can contribute to a more sustainable and environmentally conscious future.

Solvent free reactions:

Solvent-free reactions, also known as neat or dry reactions, are chemical transformations conducted without the use of solvents. These reactions offer several advantages, including reduced environmental impact, improved reaction kinetics, and simplified product isolation. This section provides an overview of solvent-free reactions, including their principles, applications, and recent advances.

1. Principles of solvent-free reactions

Solvent-free reactions are based on the principle of mixing reactants in the absence of any solvent or diluent. By eliminating the need for solvents, these reactions minimize waste generation, reduce energy consumption associated with solvent evaporation, and eliminate solvent-related environmental hazards (Tanaka *et al.*, 2000). Solvent-free conditions also promote higher reaction concentrations, leading to improved reaction kinetics and higher yields.

2. Techniques for solvent-free reactions

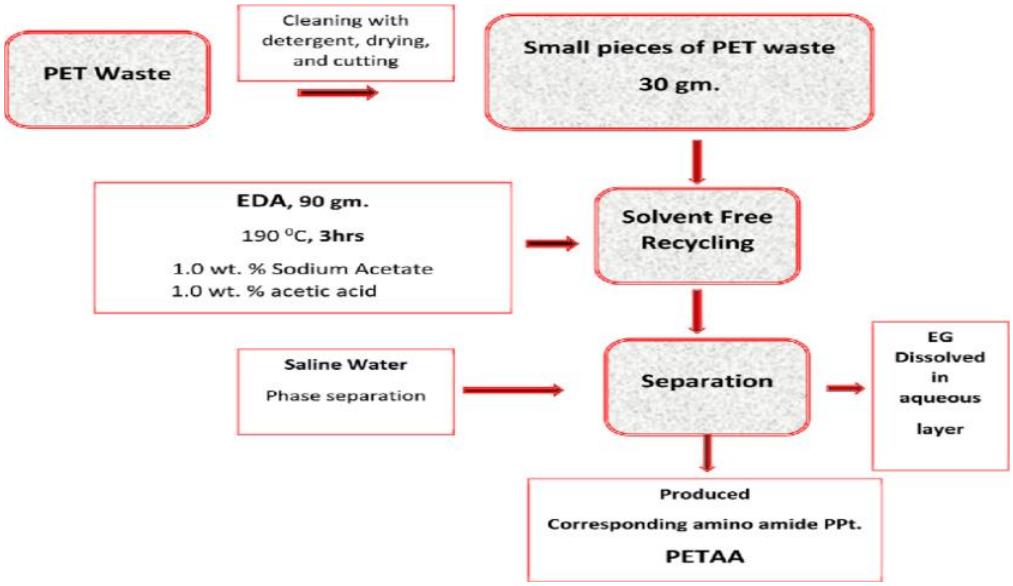

Fig. 5: Technique for Solvent free Reaction

Various techniques have been developed to facilitate solvent-free reactions, including ball milling, microwave irradiation, and solid-state synthesis. Ball milling involves grinding reactants together with grinding balls in a ball mill, promoting intimate mixing and reaction (James *et al.*, 2012). Microwave irradiation provides

rapid and uniform heating of reaction mixtures, enabling faster reaction rates and shorter reaction times (Kappe, 2004). Solid-state synthesis involves mixing reactants in the solid state, often by grinding or pressing, to induce chemical reactions without the need for solvents (Boldyreva, 2006).

3. Applications of solvent-free reactions

Solvent-free reactions find applications in various fields, including organic synthesis, materials science, and pharmaceuticals. In organic synthesis, solvent-free conditions have been employed for the preparation of natural products, pharmaceutical intermediates, and fine chemicals (Tanaka *et al.*, 2000). Solvent-free methods are also used in the preparation of functional materials such as polymers, nanoparticles, and metal-organic frameworks (MOFs) (Aida *et al.*, 2012). Furthermore, solvent-free reactions have been applied in pharmaceuticals for the synthesis of drug candidates and active pharmaceutical ingredients (APIs), offering advantages such as improved purity and reduced environmental impact (Tanaka *et al.*, 2000).

4. Recent advances in solvent-free reactions

Recent advancements in solvent-free reactions include the development of new catalysts, reaction methodologies, and scalable processes. For example, mechanochemical synthesis has emerged as a powerful tool for conducting solvent-free reactions, enabling the synthesis of complex molecules under mild conditions (James *et al.*, 2012). Additionally, the use of alternative reaction media, such as ionic liquids and supercritical fluids, has expanded the scope of solvent-free reactions and improved their efficiency (Jessop *et al.*, 2005).

In summary, solvent-free reactions offer a greener and more sustainable approach to chemical synthesis, with applications spanning organic synthesis, materials science, and pharmaceuticals. Recent advances in solvent-free methodologies and techniques continue to expand the scope and applicability of these environmentally friendly reactions.

Sustainable materials:

Sustainable materials are essential components of a circular and environmentally conscious economy. These materials are designed, produced, used, and disposed of in a way that minimizes environmental impact, conserves resources, and promotes

social equity. This section provides an overview of sustainable materials, including their characteristics, applications, and recent developments.

1. Characteristics of sustainable materials

Sustainable materials exhibit several key characteristics that differentiate them from conventional materials. These include:

- **Renewable or recycled sources:** Sustainable materials are often derived from renewable resources such as biomass, or they are produced from recycled materials to reduce reliance on finite resources and minimize waste (Auras *et al.*, 2010).
- **Low environmental impact:** Sustainable materials are manufactured using processes that minimize energy consumption, greenhouse gas emissions, and other environmental pollutants throughout their lifecycle (McDonough & Braungart, 2002).
- **Biodegradability or recyclability:** Sustainable materials are designed to be either biodegradable, allowing them to break down naturally at the end of their useful life, or recyclable, enabling them to be reused or repurposed in new products (Farahani *et al.*, 2020).

2. Applications of sustainable materials

Sustainable materials find applications across various industries, including construction, packaging, transportation, and consumer goods. In construction, sustainable materials such as recycled steel, bamboo, and engineered wood are used to reduce the environmental footprint of buildings and infrastructure (Kibert *et al.*, 2016). In packaging, biodegradable polymers and compostable materials offer alternatives to traditional plastics, reducing pollution and waste in the environment (Auras *et al.*, 2010). Sustainable materials are also used in automotive and aerospace industries to improve fuel efficiency, reduce emissions, and enhance the recyclability of vehicles (Büchs *et al.*, 2015).

3. Recent Developments in sustainable materials

Recent developments in sustainable materials focus on enhancing their performance, durability, and scalability while maintaining their eco-friendly attributes. Researchers are exploring novel bio-based polymers, such as polylactic acid (PLA) and polyhydroxyalkanoates (PHAs), for applications in packaging, textiles,

and biomedical devices (Auras *et al.*, 2010). Advanced manufacturing techniques, including 3D printing and additive manufacturing, are being used to create intricate structures and components from sustainable materials, enabling customization and resource efficiency (Domingo-Espin *et al.*, 2021). Additionally, the development of sustainable materials with self-healing, antibacterial, and UV-resistant properties is opening up new opportunities for their use in various sectors (Huang *et al.*, 2017).

In conclusion, sustainable materials play a crucial role in advancing the transition towards a more sustainable and circular economy. With ongoing research and innovation, sustainable materials offer promising solutions to address environmental challenges and promote a more resource-efficient and resilient future.

Renewable feedstocks:

Renewable feedstocks are raw materials derived from sustainable and replenishable sources, such as plants, algae, agricultural residues, and waste biomass. These feedstocks are essential for the production of bio-based materials, chemicals, and fuels, offering a renewable alternative to fossil resources. This section provides an overview of renewable feedstocks, their advantages, applications, and recent developments.

1. Advantages of renewable feedstocks

Renewable feedstocks offer several advantages over traditional fossil-based resources:

- **Sustainability:** Renewable feedstocks are derived from sources that can be replenished within a reasonable timeframe, reducing reliance on finite fossil resources and mitigating environmental impact (Huber *et al.*, 2006).
- **Carbon neutrality:** Many renewable feedstocks, such as biomass, absorb carbon dioxide (CO_2) during growth, making them carbon-neutral or even carbon-negative when used in place of fossil fuels (Balat, 2011).
- **Diversification of supply:** Utilizing renewable feedstocks diversifies the supply chain, reducing dependence on geopolitically unstable regions and volatile commodity markets (Balat, 2011).

2. Applications of renewable feedstocks

Renewable feedstocks find applications across various industries, including:

- **Biofuels:** Renewable feedstocks such as corn, sugarcane, and lignocellulosic biomass are used to produce biofuels such as ethanol, biodiesel, and bio-jet fuel, providing a sustainable alternative to petroleum-based fuels (Balat, 2011).
- **Bioplastics:** Biomass-derived feedstocks are used to produce biodegradable and compostable bioplastics, reducing plastic pollution and dependency on fossil-based plastics (Auras *et al.*, 2010).
- **Fine chemicals:** Renewable feedstocks serve as starting materials for the synthesis of specialty chemicals, pharmaceuticals, and personal care products, enabling the development of greener and more sustainable chemical processes (Sheldon & Woodley, 2018).

3. Recent developments in renewable feedstocks

Recent developments in renewable feedstocks focus on improving their conversion efficiency, scalability, and economic viability. Advanced bio-refining technologies, such as enzymatic hydrolysis, fermentation, and thermochemical conversion, enable the conversion of lignocellulosic biomass into biofuels and bio-based chemicals (Huber *et al.*, 2006). Biotechnological approaches, including metabolic engineering and synthetic biology, are used to enhance the productivity and sustainability of renewable feedstock-based processes (Stephanopoulos, 2007).

Renewable feedstocks play a crucial role in transitioning towards a more sustainable and bio-based economy. With ongoing research and innovation, renewable feedstocks offer promising solutions to address energy security, climate change, and resource scarcity, paving the way for a greener and more sustainable future.

Case studies in green synthesis

Green synthesis encompasses a variety of methodologies and principles aimed at reducing the environmental impact of chemical processes while maintaining efficiency and yield. This section presents case studies highlighting successful applications of green synthesis principles in various industries.

1. Pharmaceutical industry: Synthesis of artemisinin

Artemisinin, a key component in the treatment of malaria, was traditionally extracted from the sweet wormwood plant. However, the extraction process was inefficient and environmentally burdensome. In a groundbreaking achievement, researchers developed a green synthesis pathway for artemisinin from inexpensive and readily available starting materials (Yadav *et al.*, 2011). This innovative approach not only reduced the environmental footprint of artemisinin production but also ensured a stable and affordable supply of this life-saving medication.

2. Materials science: Synthesis of biodegradable polymers

The demand for biodegradable polymers as alternatives to traditional plastics has grown significantly due to environmental concerns. Researchers have developed green synthesis routes for various biodegradable polymers, such as polylactic acid (PLA), polyhydroxyalkanoates (PHAs), and cellulose-based materials (Auras *et al.*, 2010). These polymers are derived from renewable resources and can be produced using environmentally friendly processes, offering sustainable solutions for packaging, textiles, and biomedical applications.

3. Catalysis: Green synthesis of fine chemicals

Catalysis plays a crucial role in green synthesis by enabling efficient and selective transformations of starting materials. For example, researchers have developed green catalytic methods for the synthesis of pharmaceutical intermediates, agrochemicals, and specialty chemicals (Anastas & Warner, 1998). These catalytic processes often use Earth-abundant metals or biocatalysts and operate under mild conditions, minimizing waste and energy consumption.

4. Energy sector: Green synthesis of biofuels

The production of biofuels from renewable feedstocks offers a sustainable alternative to fossil fuels. Green synthesis routes for biofuels, such as biodiesel and bioethanol, involve enzymatic or microbial conversion of biomass into usable fuels (Balat, 2011). These processes utilize renewable feedstocks, such as corn, sugarcane, and lignocellulosic biomass, and produce lower greenhouse gas emissions compared to conventional fossil fuels.

These case studies demonstrate the diverse applications and benefits of green synthesis across industries. By adopting green synthesis principles, researchers and

industries can develop more sustainable processes and products, contributing to environmental protection and resource conservation. Continued innovation in green synthesis is essential for addressing global challenges such as climate change, pollution, and resource depletion.

Conclusion:

The future of green synthesis is characterized by innovation, collaboration, and a commitment to sustainability. By harnessing the power of catalysis, renewable energy, bio-based materials, nanotechnology, digitalization, and supportive policies, green synthesis has the potential to revolutionize the way chemicals and materials are produced, leading to a more sustainable and resilient society. Continued research, investment, and interdisciplinary cooperation will be crucial for realizing this vision of a greener and more sustainable future.

Future perspectives in green synthesis

Green synthesis holds tremendous potential for addressing pressing global challenges related to sustainability, environmental protection, and resource conservation. Looking ahead, several key trends and future perspectives can be identified in the field of green synthesis:

1. Advancements in catalysis:

Continued research and development in catalysis will drive the innovation of more efficient, selective, and sustainable catalytic processes. Exploration of new catalysts, such as bio-inspired catalysts and metal-organic frameworks, holds promise for expanding the scope of green synthesis reactions (Sheldon & Woodley, 2018).

2. Integration of renewable energy:

The integration of renewable energy sources, such as solar and wind power, into chemical processes will further reduce the carbon footprint of green synthesis. Renewable energy-driven processes, coupled with efficient catalysts and reaction methodologies, will enable the production of chemicals and materials with minimal environmental impact (Stephanopoulos, 2007).

3. Bio-based materials and circular economy:

The development of bio-based materials from renewable feedstocks will play a crucial role in transitioning towards a circular economy. Bio-based polymers,

composites, and coatings offer sustainable alternatives to conventional materials, contributing to resource efficiency and waste reduction (Auras *et al.*, 2010).

4. Green Nanotechnology:

Nanotechnology holds potential for revolutionizing green synthesis by enabling precise control over material properties and synthesis conditions. Green nanomaterials, such as nanocatalysts and nanostructured materials, offer opportunities for sustainable applications in catalysis, energy storage, and environmental remediation (Khan *et al.*, 2018).

5. Digitalization and machine learning:

The integration of digitalization, big data analytics, and machine learning algorithms into green synthesis research will accelerate materials discovery, process optimization, and predictive modeling. Computational tools can aid in the design of greener chemicals and processes by predicting reaction outcomes, optimizing reaction conditions, and reducing experimental waste (Gomez-Bombarelli *et al.*, 2018).

6. Regulatory and policy support:

Continued support from regulatory agencies and policymakers will be essential for fostering the adoption of green synthesis practices on a global scale. Incentives, regulations, and standards that promote sustainability, innovation, and eco-efficiency will drive industry-wide adoption of green synthesis principles (Anastas & Warner, 1998).

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SOME COMMON STAINS AND THEIR REMOVAL

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Introduction:

Every single person is a consumer of textiles. We all love to wear clothes that are clean and fresh, so we need to know how they should be cared for clean. It is important to known the nature of the stain that is whether it is soluble or insoluble. Special techniques and skill are required for stain removal. Soap and detergents are cleansing agents, they dislodge the unwanted particles from cloth. Detergent generally contains sequestering(complexing) or chelating agent which soften the wash water. Sequestering agent: EDTA and NTA (Nitrilotriacetic acid)

1. Borax ($Na_2B_4O_7$)

2. Calcium hypochlorite [$Ca(OCl)_2$]

3. Sodium hypochlorite (NaOCl): Bleaching of cellulose fibre. Sodium hydrosulphite and sodium hypochlorite normally used for removing stain and dyes colours.

4. Sodium perborate ($NaBO_2$): Oxygen releasing agent, mild sodium perborate bleach is the best for silk and wool blended theses fibre. The oxygen bleaches are safer for all fabrics. Most dyes that are colourfast are not affected by oxygen bleaches.

5. Sodium chlorite ($NaClO_2$): Oxidising bleach suitable for cellulose and synthetic fibres.

6. H_2O_2: Per hydroxyl ion responsible for bleaching.

Fluorescent whitening agents are destroyed by chlorine bleach. Perborate based oxygen bleaches require high water temperature. The potassium mono persulphate is efficient at lower water temperature washing temperature of 65^0 C for 10 minutes or 71^0 C for 3 minutes.

$$2ClO_2 + Na_2O_2 \rightarrow 2\ NaClO_2 + O_2$$

Chloride peroxide + Sodium peroxide → Sodium chlorite

$Ca(OCl)_2 + CO_2 + H_2O \rightarrow CaCO_3 + 2HOCl$

Hypochlorous acid

$NaOCl \rightarrow 2NaCl + O_2$ at pH 4.6

$Na_2B_4O_7 + 2NaOH \rightarrow 4\ NaBO_2 + H_2O$

Borax + Caustic soda → sodium perborate

$H_2O_2 \rightarrow HO_2^- + H^+$ $2H_2O_2 + \text{Trace alkali} \rightarrow 2H_2O + O_2$

Per hydroxyl ion

Oxalic acid, white vinegar is used widely as an acid rinse in the laundry industry. The acetic acid in vinegar will destroy some bacteria and virus. Acetic acid is simple cleaner to remove dirt.

Plants derived cleaning enzymes like monnanase and pectinase as well as coconut oil fatty acid which acts as surfactant to lift dirt and oils. Enzymes used to break lipid molecules into smaller pieces. Enzyme soaps are stain removal.

Bleaches can be classified as either oxidising or reducing

(i) Oxidising bleaches: Sodium perborate, H_2O_2, Sodium percarbonate, Sodium hypochlorite, $KMnO_4$, etc.

(ii) Reducing bleaches: Sodium bisulphite, Sodium hydrosulphite, Titanium sulphate, Oxalic acid etc.

Phosphate e.g Sodium hexametaphosphates (contains phosphorus) are used in laundry detergent to soften the water.

Alkaline reagent: Sodium Carbonate (Washing soda), Sodium tetraborate (borax), Ammonium hydroxide (ammonia).

Acid reagents: Oxalic acid, Salt of lemon (Potassium Oxalate), Acetic acid, Oleic acid (Olein).

Solvents: Cleaning benzene or petrol, Carbon tetrachloride, Acetone, Methylated Spirits (alcohol), Turpentine

Absorbents: Common salt, bran, chalk and bread crumbs, etc. Talcum powder (Mg, SiO),

Frech chalk ($3Mgo.\ 4SiO_2.H_2O$) Fullers earth ($Al_2O_3.SiO_3.xH_2O$), Powdered magnesia (MgO)

Discussion:

The oxalic acid with H_2O_2 removes tannin base of writing ink. Acetone is an effective spotting agent for stains caused by cosmetics, nail polish and lipstick, paint and varnish and shoe polish. Turpentine acts as solvent for grease, varnishes, paint and printer's ink. The world's oldest method of bleaching is that of treating fabrics in the open air and sunlight.

Oleic acid (fatty acids) produced soap when mixed with an alkali. It is used for the spotting of grease and oil stains caused by machinery. Sodium hydrosulphite or sodium carbonate anhydrous are sometimes used to lighten the colour of a garment (Discoloration). Baking soda and white vinegar are homemade stain remover.

The liquid laundry detergent and few drops of ammonia removes tough stains. Rubbing a lemon slice over a stain is a highly effective remedy to remove many stains.

Stain removing chemicals:

(i) Vinegar – Acetic acid (10%)

(ii) Acetone

(iii) Alcohol – Isopropyl alcohol

(iv) Ammonia

(v) Amyl acetate

(vi) Coconut oil

(vii) Oxalic acid

(viii) Sodium thiosulphate

(ix) Chlorobenzene

(x) Carbon tetrachloride

(xi) Borax powder

(xii) Turpentine

(xiii) NaCl

(xiv) Glycerine

(xv) Talcum powder

Treatment of particular stains

Sr. No.	Stain	Reagent required	Method of application
1.	Ball point (Black and Blue)	Denatured spirit / Lab solvent	Rub lightly with lemon and table salt then soaked in lab solvent / denatured alcohol / Acetone
2.	Boot polish (Colour in wax)	Lab solvent denatured spirit	The wax is removed by solvent if colour will remain treat with denatured spirit.
	Shoe polish	Hot water and denatured alcohol	Work oil or grease into the stain to emulsify it and launder with hot water. Treat with denatured alcohol and launder in hot water.
3.	Beetle leaf (Paan)	CaO paste / Ca $(OH)_2$ lemon juice	The lime slurry paste (CaO in H_2O) on stain. Then wash with soap.
4.	Paan – Supari Gutkha	Curd	Stain rub lightly 2 to 3 times with curd and wash
5.	Blood	Milk Salt	Blood stain soak with milk or NaCl and then detergent wash
		Hot water and soap CCl_4	Stain treats with hot water and soap and then CCl_4 and Launder
6.	Chocolate / Ice-cream	Cold water + H_2O_2	Soak with cold water then stain wash with H_2O_2.
		Talcum powder	Fresh stain dry with Talcum powder and hot water wash
			Ice-cream stain soak with aqueous Borax powder and then wash with water.
			Apply petrol or CCl_4. Launder

			Ice-cream stain remove easily with ammonia.
7.	Fruit juice	NaCl, Glycerine, NH_3	Stain rub with NaCl and wash.
		Vinegar	Apply white vinegar lightly to the stain.
		Sodium hypochlorite	White fabric may be bleach with sodium hypochlorite and coloured fabric may be soaked in warm borax solution.
		NaCl and CCl_4	Stain rub with NaCl and then treat with CCl_4 and launder
8.	Grease / Cycle lubricant	Turpentine	Stain rub with lemon, launder with hot H_2O
		Niligiri oil	Stain rubs with Nilgiris Oil of launder with hot H_2O
	Glue, Gum	Glycerine	Few drops of glycerine in hot water launder it. Stain dissolving some cases few drops of acetic acid added.
		Lab solvent	Gum dissolved in lab solvent
9.	Henna (Mehendi)	Milk	Soak in milk for an hour, launder
10.	Milk	Talcum powder	Apply talcum powder / soap / flour paste, launder
11.	Chewing gum	Ice cold water and CCl_4	Apply ice to the stain. Allow to soak in ice cold water for a few minutes, launder
12.	Candle wax	Tissue paper and Ironing	Place stain between two tissue paper or blotting paper and press with warm iron
13.	Tea, Coffee	Borax powder NaCl + Glycerine	Apply borax powder paste and allow to dry and treat with

			glycerine, if necessary, launder
			Stain treats with soda water (soft drink) or baking soda
			Apply tooth paste or NaCl salt or vinegar or soap or detergent to stain and launder
14.	Colour	Kerosene	Apply kerosene or acetone or Colgate tooth paste and dry to stain, use aqueous hydrosulphide (acid) and launder
15.	Paint	Turpentine CCl_4 Paraffin	Apply turpentine or kerosene or denatured spirit to stain; when wet easily removed
16.	Ink	lime juice and salt	Apply lime juice and salt for 30 minute and launder
		Sour milk (cured)	Stain soak in curds for 30 minutes and launder
			Place in dilute oxalic acid for 30 minutes then rinse thoroughly in dilute borax solution, launder
			Ink stain removes with kerosene or Dettol **or** Treat with isopropyl alcohol
			Stain rubs with lemon and salt, launder
			Sponge the area around the stain with denatured alcohol
			Apply few drops of white vinegar and glycerine, dishwash

			detergent, stand for 30 minutes.
17.	Iodine	Ammonia solution, sodium thiosulphate (hypo solution)	Apply over then iodine mark and launder
		Starch paste	Apply starch paste to absorb stain and launder
18.	Lipstick	Ammonia	Sponge with 1 part ammonia and 2- part water. Rinse thoroughly (wool or silk can't be treated with ammonia)
			Soften the stain with glycerine and launder
			Apply methylated spirit, launder
		Hot water and soap	Stain wash with hot water then soap and launder
19.	Nail points / Polish / Varnish	Acetone / methylated spirit /sodium hydrosulphite	Apply acetone to stain, launder (Do not use on acetate rayon)
			Bleach with sodium hydrosulphite
			Apply Lemmon juice + Baking soda on stain
20.	Mud	Vinegar	Apply vinegar to mud stain and then water wash
			Use potato paste on mud stain of Jean pant
		Sodium carbonate	Soak the stain in alkaline bath (Na_2CO_3)
21.	Turmeric	Vinegar / lemon	Apply lemon to turmeric stain

		detergent	and detergent wash
			Place in cold water of NaCl
			Stain escapes in sunlight and coated with soap paste and water wash
	Turmeric Kumkum	Soap Hydrogen peroxide	Soak in hot soapy water / detergent and dry in the sun
			Apply few drops of hydrogen peroxide and dry in the sun
			Apply sodium bicarbonate and launder
22.	Edile oily stain Ghee stain	Talcum powder	Apply talcum powder to oily stain or Flour pastes or vinegar for 10 minutes then detergent wash
			Rub with lemon and NaCl, Soap Wash
		CCl_4	Rub with CCl_4 cotton.
23.	Egg stain	NaCl	Soak in cold NaCl water and launder
			Soak in enzyme detergent, launder
24.	Protein stain	Baking soda ($NaHCO_3$) Hydrogen peroxide (H_2O_2)	Apply Hydrogen peroxide and baking soda to stain
25.	Sauces	White vinegar	Apply white vinegar to the stain with eye dropper Flush the spot with cool water
26.	Corrosive Rust (Iron rust)	Lemon and Curd HF acid and oxalic acid.	Apply lemon and curd to corrosive spot and Launder
			Stain soak in 1 % oxalic acid for

			15 min and then launder
27.	Curry (Turmeric and oil)	Soap	Apply soap and bleach in sunlight when dry if stain has not disappeared, wet it and put it back in sunlight again.
	Chilli oil	Lemon juice and steam pressure	Stain removed by lemon containing acid with steam pressure and temperature (285^0 F) in vessel
28.	The Grease, Engine oil, Chilli oil	Slit clay and soap Paraffin	Stain rub with slit clay and wash with soap / detergent / alkali / turpentine / petrol
29.	Engine oil	oxalic acid and sodium bicarbonate	Stain rubs with aqueous oxalic acid then sodium bicarbonate and water wash
		CCl_4 and detergent	Stain rub with CCl_4 and detergent or soap wash
30.	Red wine	Lime paste	Apply lime paste and dry, launder
			Cover stain with salt. Pour hot water apply while vinegar with eye dropper and launder.
31.	Scorch	H_2O_2 / Sodium perborate / Potassium permanganate /sunlight and Soapy water	Bleach with hydrogen peroxide **or** with sodium perborate. **or** potassium permanganate, Launder
			Brush the surface, rewash article, sunlight bleach
			Hot iron spot on cloth removes with placing wet bread when dry, it washes

32.	Sweat stain perspiration	Oxalic acid and H_2O_2	Soak in 1% oxalic acid for 10 minutes. Rinse through with water and soak for 10 minutes in hydrogen peroxide.
	$NaHCO_3$	White vinegar and hot water	Apply ammonia to fresh stain or white vinegar to old stain Rinse with hot water
		Baking soda, H_2O_2 and water	Apply paste to baking soda, H_2O_2 and water and rub the mixture into the stain, rest at least an hour and wash
33.	Mildew	Potassium permanganate and sodium sulphite / oxalic acid	Soak stain in 1% $KMnO_4$ for 15 minutes. Rinse in cold water once. Dip in warm 1% solution of sodium bisulphate till the brown colour of the potassium permanganate disappears, Launder
		Javelle water (aq. solution of sodium hypochlorite)	Mildew is formed by the growth of fungus, white wool – on damp fabric it is removed by Javelle water
34.	Dye stain	Sodium hydrosulphite and H_2O_2	Treat stain with hot sodium hydrosulphite solution
			Bleach with H_2O_2 solution $\frac{1}{2}$ hour While silk – treat hydrosulphate first then H_2O_2
		Warm soapy solution alkali or acid	While cotton and linen – treat – dilute alkali or acid first then hydrosulphite solution for 5 – 10 minutes

35.	Food stain	Javelle water (NaOCl)	Treatment with sodium hypochloride (on cotton and linen)
36.	Lead pencil stains		Generally removed by normal washing
37.	Rouge	Petrol, Ammonia	Apply petrol to remove grease wash with hot soapy water and few drops of ammonia. Rinse thoroughly
38.	Make – up (mascara)	Denatured alcohol	Sponge with denatured alcohol or petrol or ammonia. Launder in hot water
39.	Mercurochrome	Denatured alcohol	Sponge with water and alcohol (equal parts) then work glycerine into the stain as long as colour bleeds, launder
		Detergent and Ammonia	Steep for one hour in detergent solution containing ammonia
		Vinegar / acetic acid and denatured spirit / alcohol	Rinse in water. Soak in water containing vinegar, rinse and dry. Apply denatured spirit / alcohol, rinse in water, Launder
40.	Medicine	Ethyl alcohol / surgical spirit	Steep in ethyl alcohol or surgical spirit, launder
41.	Ink (Black)	Lime juice / curds	Soak stain in curd overnight then wash out Bleach in hot solution of $KMnO_4$ and use oxalic acid solution to remove the brown stain.
	Red ink	Borax / Ammonia	Steep in borax solution (1 tea spoon borax in warm water)
			Steep in ammonia solution

	Marking ink	Iodine solution and sodium thiosulphate	Steep in iodine solution and follow by steeping in sodium thiosulphate solution.
42.	Perfumes	Methylated spirit Acetic acid	Perfumes consists of essential oils and alcohol. Remove oil by methylated spirit
43.	Sealing wax	Methylated spirit	Soften with methylated spirit and dissolved in warm dry - cleaning solvent
44.	Tar	Oil / Grease – solvent	Rub with oil or grease solvent
45.	Unknown stain	Sodium hypochlorite (NaoCl) Sodium hydrosulphite	Bleaching carried out a) i) Vegetable and rayon fibres – hypochlorite bleach ii) Animal fibres: - Hydrogen peroxide b) If this is not effective for all fibres: hydrosulphite bleach
46.	Urine stain On mattresses	Ethyl alcohol and chloroform	a) Treat as perspiration or apply ethyl alcohol and allow to evaporate. Then apply chloroform and allow to evaporate, Launder
			b) Sponge in area with a mixture of water and detergent. Rinse with a mixture of vinegar and water. Let dry if an Odour remains, sprinkle the area with baking soda and let stand for 24 hours and dry.
47.	Soot	Starch paste	Apply starch paste and launder

48.	Mustard	Perchloroethylene Vinegar and H_2O_2, add drop of NH_3	Stain treats with perchloroethylene and allow to dry. Then it soaks in water and vinegar. Further bleach for 10 minutes in H_2O_2 then adds drop of ammonia, launder
49.	Cough syrup	Dish washing detergent and white vinegar	Soak stain with detergent and vinegar for 15 – 30 minutes then rinse and launder
50.	Silk	Oxalic acid	Soak silk in dilute oxalic acid for 5 minutes and then wash
51.	Cotton and white colour cloth	Bleaching powder	Soaked in boiled bleaching powder aqueous solution then wash with clean water
		Turpentine	It is used for coloured cotton cloth
52.	Chadari (Baby clothes)	Bleaching powder	Chadar dipped in boiled bleaching powder (1 spoon) aqueous solution and then wash with clean water
53.	Wool cloth (Turkish towel)	Detergent	Wash with detergent white colour cloth
		Ritha, Acetic acid and glycerine	For coloured cloth use Rita powder for washing

The dyes used as laundry blues were ultramarine blue, Prussian blue, Aniline blue. We are peoples follows Wash and Ware system of clothes for good health.

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EXPLORING THE VERSATILE APPLICATIONS OF IONIC LIQUIDS: FROM ELECTROCHEMISTRY TO GREEN CHEMISTRY

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Introduction:

Brief overview of ionic liquids: Definition, properties, and significance:

Room temperature molten salts, commonly known as room temperature ionic liquids (RTILs), have garnered immense interest across diverse scientific disciplines due to their unique physicochemical properties. These properties, encompassing aspects such as size, shape, solvent miscibility, polarity and hydrophobicity are highly customizable by combining different cations and anions [1]. The vast number of potential combinations, estimated to exceed 10^6, has led to the development of a wide spectrum of ionic liquids (ILs) with applications covering material science, nanoscience, supercapacitors, electrochemical sensors, biocatalytic reactions, biosensors, biopreservation, protein solubilization, stabilization, and crystallization etc. [2-8]

The characterization of ILs typically involves their classification as fused salts with melting points below 100°C, while RTILs maintain their liquid state under ambient temperature and pressure conditions. Various synthesis methods for ILs are well-documented, including quarterisation reactions, metathesis of halide salts, and acid-base neutralization reactions. Notably, the preparation of the ionic liquid [Emim][BF_4] via metathesis highlighted diverse approaches in IL synthesis. The versatility of ILs, arising from their diverse cation and anion combinations, has earned them the designation of 'designer molecules.' This flexibility finds utility across various domains, from Organic Chemistry, Electrochemistry, Analytical Chemistry to Biochemistry [9]. The physical and chemical properties of ILs play a

pivotal role in their applications, with factors such as ion symmetry, alkyl chain length, and ion size asymmetry influencing properties like melting point, viscosity, density, and surface tension. Molecular dynamics simulations and experimental studies continue to unveil the intricate interplay of forces governing IL behaviour, contributing to their growing importance as environmentally friendly alternatives to traditional solvents [9-11].

Historical background and evolution of research in ionic liquids:

The history of ionic liquids traces back to significant milestones, such as Gabriel's discovery of ethanolammonium nitrate as the first protic ionic liquid in 1888 and Walden's synthesis of ethylammonium nitrate, the first room temperature ionic liquid, in 1914 [1]. This surge in interest led to extensive reviews by experts like Welton, providing valuable insights into the preparation, handling, solvent properties, and applications of RTILs [1].

Purpose and scope of the book chapter:

The chapter aims to comprehensively explore the various applications of ionic liquids (ILs) across scientific and industrial domains, with a focus on:

1. **Electrochemical applications:**

To investigate, how ionic liquids function as electrolytes in batteries, supercapacitors, fuel cells, and electrochemical sensors, highlighting their contributions to enhancing energy storage, efficiency, and device performance [12-16]

2. **Green chemistry applications:**

To know about the use of ionic liquids as eco-friendly solvents in catalysis, chemical synthesis, extraction, and separation processes. Estimate their environmental advantages, including lower volatility, recyclability, and reduced environmental impact as compared to conventional organic solvents [17,18].

3. **Physical and Chemical Properties:**

To learn about the physical and chemical properties of ionic liquids that useful for different applications. This includes their ionic conductivity, thermal stability, viscosity, Surface Tension, sound velocity, and broad electrochemical window which contribute to their versatility [19,20].

4. Research Developments:

To learn about the current research trends and improvements in the field of ionic liquids, covering various applications, innovative synthesis methods and emerging technologies that influence the unique properties of ILs [21].

5. Challenges and Future Directions:

To learn about the challenges in the synthesis of Ionic Liquids and about its Purity, cost-effectiveness, scalability. Discuss potential innovations and future directions aimed at harnessing the full potential of ionic liquids for advancing electrochemical processes, green chemistry practices, and industrial applications.

Fundamentals of Ionic Liquids:

a. Molecular structure of ionic liquids: Cations and anions

Ionic liquids (ILs) are a class of organic salts that remain in liquid form even at ambient temperatures. They are composed of organic cations such as imidazolium, pyridinium, pyrrolidinium, phosphonium, and ammonium, along with organic or inorganic anions. These anions may have side chains of alkyl groups or various functional groups and aromatic moieties, examples of which include sulfonate, carboxylate, phosphate, halides, and amino acids, trifluoromethanesulfonate and bis(trifluoromethyl)sulfonyl imide, thiocyanate, $[SCN]^-$, dicyanamide, $[DCA]^-$, tricyanomethanide $[TCM]^-$ and tetracyanoborate, $[TCB]^-$ [22]. The preparation of ionic liquids derived from amino acids involves utilizing their unique molecular structure, which contains both a carboxylic acid residue and an amino group in a single molecule. This allows amino acids to function as either anions or cations within the ionic liquid. Additionally, these functional groups can introduce specific functionalities into the resulting ionic liquids. [23-25] Most research on ionic liquids (ILs) has historically centered around monocationic-based ILs. However, there has been a recent shift towards studying dicationic-based ILs due to their distinct properties, including exceptional thermal stability and surface tension [26, 27]. In water, ionic liquid surfactants form specific self-assembled structures determined by their molecular makeup, such as micelles, reverse micelles, or vesicles. These surfactants, also called amphiphiles, may comprise surfactants, ILs, block copolymers, or lipids, containing both hydrophilic and hydrophobic parts. This dual nature allows them to organize into structured assemblies when in an aqueous environment.

b. Ionic interactions and their impact on physical properties:

Different parameters can significantly influence the structure and properties of ionic liquids (ILs). These include:

1. Nature of the Anion/Cation:

The choice of anion and cation can have a significant impact on the overall properties of the IL, such as its solubility, conductivity, and thermal stability. Ionic liquids (ILs) containing polar substituents like hydroxyl, alkoxy, nitrile, or ethoxy groups within their side chains tend to exhibit lower toxicity compared to ILs with long alkyl side chains. The presence of long chain substituents significantly increases the toxicity of ILs with various cations and accelerates their degradation in environmental conditions. Among different cations, pyridinium is generally less toxic than imidazolium cation. Anions such as bromides, dicyanamides, and ethylsulfates have been described as highly toxic. To reduce toxicity of Ionic liquids researchers are trying to synthesize ionic liquids.

2. Alkyl Chain Length:

Varying the length of alkyl chains in both the anion and cation can alter the viscosity, melting point, and polarity of the IL, affecting its suitability for different applications. Lethesh *et al.* [28] have synthesized hydroxyl-containing pyridinium-based ionic liquids (ILs) with varying alkyl side chains on the cation, paired with Br^- and $[Tf_2N]^-$ anions. These different alkyl side chains have a significant impact on the physicochemical and electrochemical properties of the ILs. The effect of chain length on the physical properties of ionic liquids is significant. Generally, longer alkyl chains in the cation of an ionic liquid lead to increased viscosity and higher melting points. This is due to the increased van der Waals forces and greater molecular interactions between longer alkyl chains.

Physical properties of ionic liquids:

The physical properties of pure Ionic Liquids (ILs) are pivotal in understanding various chemical phenomena, where solute and solvent nature greatly influence dissolution rates and chemical equilibria like dissociation, association, tautomerism, isomerism, and phase transfer reactions [29]. The widespread use of volatile organic solvents, often hazardous to health, has driven the search for safer alternatives such as ILs for chemical processes. ILs have shown promise as environmentally friendly

solvents due to properties like high polarity, negligible vapor pressure, high thermal stability, wide liquid range, and a broad electrochemical window [30].

The purity of ILs significantly impacts their physical properties. Even small contaminations of halides, water, or other solvents can dramatically alter these properties. Molecular solvents within ILs can decrease viscosity and density, whereas viscosity increases with chloride content. The melting point depends on the cation-anion combination, with lower symmetry, weak intermolecular interactions, and well-distributed charge favoring lower melting points [31]. Studies indicate that density decreases with increased bulkiness on the cation, i.e., longer alkyl chain lengths, potentially due to poorer crystal packing with bulkier cations. Replacing hydrogen with heavier elements like F, Cl, or Br also increases density. Viscosity changes are influenced by van der Waals forces and hydrogen bonding; longer alkyl chains generally decrease viscosity due to reduced strong interactions [32]. However, in imidazolium-based ILs, longer alkyl chains can increase viscosity due to stronger van der Waals interactions and suppressed hydrogen bonding. Research on surface tension and self-diffusion coefficients in ILs further illustrates their complex behavior. Surface tension decreases with ion size asymmetry, while self-diffusion coefficients can vary between cations and anions, with certain ILs showing higher diffusion coefficients for cations compared to anions over a range of temperatures [33-34]. The self-diffusion coefficients given by Tokuda *et al.* [35] for different ionic liquids having same anion decreases in the order of [emim][$(CF_3SO_2)_2N$]>[mmim][$(CF_3SO_2)_2N$]>[bmim][$(CF_3SO_2)_2N$]>[C_6mim][$(CF_3SO_2)_2$ N] > [C_8mim][$(CF_3SO_2)_2N$][].

Aggregation if Ionic Liquids in aqueous solution:

The detailed investigation of thermodynamic and phase equilibria in IL-water mixtures is crucial for developing extraction methods. The aggregation behavior of ILs in aqueous solutions is intriguing, as these salts can act as a unique class of surfactants with distinctive properties [36]. Studies on the aggregation behavior of 1-n-alkyl-3-methylimidazolium ([Cnmim]) based ILs in aqueous solutions reveal some degree of inhomogeneity due to the amphiphilic nature of the [Cnmim] cation, comprising both a polar imidazolium group and a nonpolar alkyl tail [37]. Small-chain [Cnmim] based ILs in aqueous solutions exhibit self-aggregation akin to short-chain

cationic surfactants. Research by Singh *et al.* delves into the influence of alkyl chain length of the cation or anion on the shape and size of IL aggregates in aqueous solutions using H1 NMR spectroscopy [38-40]. They propose that ILs form intramolecular hydrogen bonding between protons on the imidazolium cationic ring and counterions. Additionally, ILs in aqueous solutions form intermolecular hydrogen bonds with water, impacting chemical shifts in dilute solutions relative to post-micellar or pre-micellar regions [41,46].

The conformational changes induced by aggregation in different ILs depend on factors like aromatic ring, alkyl chain, counterions, and their interactions with water. The formation of aggregates hinges on the relative strengths of Columbic, H-bonding, hydrophobic, and van der Waals interactions, with the nature of ions in ILs playing a pivotal role. This affects micellization behavior, including critical micelle concentration (CMC) and aggregation number [46-51]. Methods such as tensiometry, conductometry, small angle neutron scattering, turbidity, and potentiometry are used to determine CMC in aqueous IL solutions. Electrical conductivity measurements, as utilized by Inoue *et al.* [52], provide insights into CMC and parameters like degree of counterion binding (β) and aggregation number. Studies on the effect of alkyl chain length on cation for $[C_n mim][Br]$ (n=12, 14, 16) reveal an increase in aggregation number with longer alkyl chains.

Applications in electrochemistry:

Ionic liquids (ILs) have numerous applications in electrochemistry due to their unique properties. Some key applications include:

a. Electrolytes in batteries:

ILs are used as electrolytes in various types of batteries, including lithium-ion batteries and supercapacitors. Their high ionic conductivity and thermal stability make them suitable for enhancing battery performance and safety. In electrochemical applications, the crucial properties are electroconductivity and ion conductivity, which are fundamental for the electrolyte solutions vital for energy devices [53-54]. For instance, the current limitation of cell voltage in electrochemical capacitors is attributed to the degradation of electrolytes using organic solvents. So, researchers are trying for the safer alternatives for the existing electrolytes.

Ionic liquids (ILs) have preserved significant interest in the context of lithium-ion batteries (LIBs) due to their unique properties that can address several challenges associated with traditional electrolytes.

(i) Safety and stability:

Commercialized lithium battery electrolytes typically contain lithium salts and organic solvents like vinyl carbonate, dimethyl carbonate, and methyl ethyl carbonate. Despite their widespread use, these solvents have drawbacks. Lithium metal reacts with carbonate solvents to form an unstable solid electrolyte interface (SEI), leading to dendrite growth and reduced coulombic efficiency (CE) and cycling performance. Dendrite growth can also cause short-circuiting and overheating, risking combustion of the organic electrolyte. Developing nonflammable electrolytes to inhibit dendrite growth is crucial. Additionally, high-voltage (>5 V) cathode materials are being developed, but current electrolytes are unstable at high anode potentials, necessitating suitable alternatives [55].

(ii) High Ionic Conductivity:

Ionic liquids exhibit high ionic conductivity, which is crucial for efficient ion transport within the battery. This high conductivity helps in achieving faster charging and discharging rates, leading to improved battery performance [56]. Some of the papers are available on the theoretical study of different combination of cations and anions and its properties based on the variation.

(iii) Wide Electrochemical Window:

Electrochemical windows (EW) represent a crucial characteristic to ascertain in solvents and electrolytes utilized in electrochemical applications. EW denotes the potential range and difference, calculated by subtracting the oxidation potential (anodic limit) from the reduction potential (cathodic limit) [57-58]. Zhang investigated the electrochemical windows (EW) of different ionic liquids (ILs) and traditional solvents using Au, glassy carbon (GC), and Pt working electrodes. They noted that for 1-n-butyl-3-methylimidazolium tetrafluoroborate [BMIm][BF_4], the reductive window's magnitude aligned with the sequence of electrode materials as Au ≈ GC > Pt, whereas the magnitude of the oxidation window followed the order Au > GC ≈ Pt [59]. Essentially, EW signifies the voltage span within which the substance being tested remains inert, neither oxidized nor reduced. ILs have a wide

electrochemical window, allowing them to operate at higher voltages without undergoing decomposition. This property is essential for the development of high-energy-density lithium-ion batteries [60].

(iv) Electrochemical Sensors:

ILs are utilized in electrochemical sensors for their ability to facilitate electron transfer and provide a stable environment for sensing reactions. They are used in sensors for detecting various analytes, such as ions, gases, and biomolecules. Ionic liquids offer a diverse range of inherent properties that are highly advantageous. By manipulating the combinations of anions and cations, specific properties of ILs can be tailored for particular applications. Novel electrochemical sensors utilizing different ionic liquids have been developed, where ILs serve as modifier materials for electrode modifications or function as electrolytes. Ionic liquid-based electrodes possess numerous desirable traits such as consistent stability, heightened sensitivity, notable catalytic capabilities, and enhanced conductivity. Lu *et al.* [61] explored the use of chitosan/1-butyl-3-methylimidazolium hexafluorophosphate ($BMIMPF_6$) as a composite material to immobilize various proteins and investigated the electrochemical performance of hemoglobin (Hb) on a glassy carbon electrode [62-65]. Due to their non-flammability, low volatility, and exceptional thermal and electrochemical stability, ILs are considered safe and suitable for constructing electrochemical sensors.

Industrial and technological applications

Ionic liquids (ILs) have emerged as revolutionary compounds with a wide range of applications across various industrial and technological sectors. Their unique properties, such as low volatility, high thermal stability, and tunable chemical characteristics, make them incredibly versatile and valuable in diverse applications.

Role of ionic liquids in industry: Lubricants, coatings, and process fluids.:

Ionic liquids (ILs) indeed possess exceptional lubrication properties attributed to their unique characteristics. Their low volatility, high thermal stability, and customizable chemical structures make them highly effective lubricants, especially in industries like aerospace, automotive, and manufacturing where traditional lubricants may struggle under extreme conditions[66]. This makes ILs a valuable choice for enhancing performance and durability in various industrial applications.

Ionic liquids (ILs) are indeed utilized as corrosion inhibitors and protective coatings in various industries [67]. Their capability to form stable films on metal surfaces plays a crucial role in preventing corrosion and prolonging the lifespan of equipment and structures. Additionally, IL-based coatings have the ability to repel fouling agents such as salts, minerals, and organic deposits. This not only reduces maintenance costs but also enhances the efficiency of marine and industrial equipment by maintaining clean and protected surfaces [68].

Emerging technologies utilizing ionic liquids: Nanomaterial synthesis, Biotechnology, and Energy Storage.

ILs offer precise control over nanoparticle synthesis, enabling the production of uniform and tailored nanoparticles with specific sizes, shapes, and surface properties. This capability is essential in electronics, catalysis, and biomedical devices. Additionally, ILs act as stabilizing agents for nanoparticles, preventing agglomeration and ensuring long-term stability, which is vital for maintaining the desired properties of nanomaterial [69].

ILs also exhibit biocompatibility and compatibility with biomolecules, making them valuable in biotechnological applications such as enzyme stabilization, protein folding studies, and biocatalysis. Furthermore, ILs are explored for drug delivery systems, offering advantages such as controlled release, enhanced solubility of poorly soluble drugs, and targeted delivery to specific tissues or cells.

Conclusion:

In conclusion, the chapter explores into the multifaceted world of ionic liquids (ILs), highlighting their crucial role across diverse scientific and industrial domains. From their origins and historical milestones to their modern-day applications, ILs have evolved into 'designer molecules' with a vast array of customizable properties. The chapter extensively covers their applications in electrochemistry, where ILs serves as electrolytes in batteries, supercapacitors, and electrochemical sensors, contributing to advancements in energy storage, efficiency, and device performance. Moreover, their utilization in green chemistry practices showcases ILs as eco-friendly solvents with lower volatility, recyclability, and reduced environmental impact compared to traditional solvents. The discussion on physical and chemical properties underscores the importance of factors like ion symmetry, alkyl chain length, and ion

interactions in shaping IL behavior and suitability for various applications. Research developments and emerging technologies demonstrate the on-going innovations in IL synthesis, characterization, and application, paving the way for future advancements. However, challenges such as synthesis purity, cost-effectiveness, and scalability remain areas of focus for further progress. Looking ahead, harnessing the full potential of ILs requires continued exploration, innovation, and collaboration across scientific disciplines, promising a bright future for these versatile and impactful molecules in shaping sustainable technologies and industrial practices.

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CLOUD POINT DETERMINATION OF ORANGE-OT DYE WITH NON-IONIC SURFACTANTS

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Introduction:

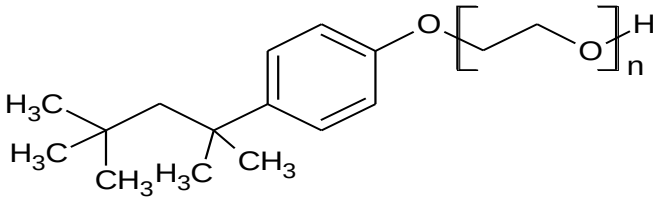
Surfactant is not only a technical term but also diminutive form of phrase SURFace ACTive AgeNT (SURFACTANT), means surfactant is contraction of the term surface active agent[2]. The surfactant present in lower concentration in a system has the property of adsorbing onto surfaces or interfaces of system and is capable of changing the surface or interfacial free energies. The boundary between any two immiscible phases is termed as interface while the term surface denotes interface where one phase is gas, usually air. The interfacial free energy is the minimum amount of work requires creating that interface. The first surfactant developed in Germany during First World War-I in order to overcome less availability of animal and vegetable fats. Those materials where short chain alkyl naphthalene sulphonate prepared from reaction of propyl or butyl alcohol with naphthalene followed by sulphonation. These products marginally useful as 'detergents' with good wetting characteristics. The polyoxyethylenated non-ionic surfactants (if an oxyethylene content is below near about 80%) in their aqueous solution becomes turbid on heating and the temperature at which turbidity appears called as cloud point and observed separation of the solution into two phases. This phase separation occurs within a narrow temperature range which is fairly constant for surfactant concentration below a few percent. The theories to explain phase separation have been proposed by Kraft and Wiglow and later by Murray and Hartley. The phase separation is reversible and on further coding the mixture to a temperature below cloud point these two phases merge with each other and once again forms a clear and transparent solution without turbidity.

The phase separation would be occurred due to sharp increase in aggregation number of micelles and decrease in intermicellar repulsion arises from the decreased hydration of the oxyethylene oxygen in the polyoxyethylene hydrophilic group with increase in temperature. As the temperature increases, micellar growth and increased

intermicellar attraction shows formation of bigger and large particles hence the solution becomes turbid. Thus, phase separation occurs due to difference in density of the micelle rich and micelle poor phase. An understanding of the clouding behavior in non-ionic surfactant solution is of practical and theoretical interest. The removal of hydration of water from hydrophilic mantle appears to be main reason for occurrence of CP, one method for removal of hydration water is the raising of temperature hence hydration forces give way to Vander Wall attraction, second method includes filling micellar hydrophilic region with additive hence number of water molecules per monomer get reduced. Many researchers have studied the molecular interactions in surfactants in the presence of added electrolytes. Thus, CP of non-ionic surfactants is very sensitive to external additives like electrolyte, nonelectrolyte and ionic surfactants. The effect of additives is mainly responsible for the change in cloud point values of surfactant. Therefore, several researchers provide excellent tool for investigating polymer-surfactant interactions by the measurement of ionic or non-ionic surfactants and polymer alone and mixture.

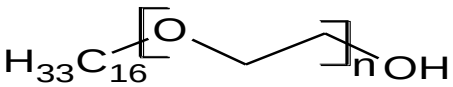
Material method:

The non-ionic surfactants Triton X-114 (MW 537) the products of Sigma-Aldrich (USA) and Brij-56 (MW 682) is product of E-Merck (Germany) were in this work were used as received. The structures of all the non-ionic surfactants are represented as;



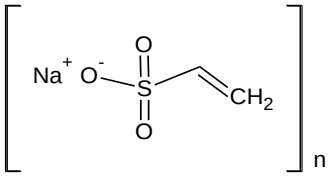
n = 7-8

Structure of Triton X-114

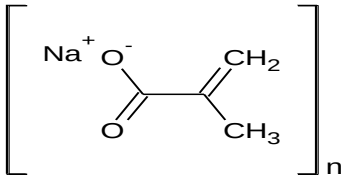


Structure of Brij-56

The water-soluble polymers used for the present study are polyvinylsulphonic acid 25% aqueous sodium salt solution (MW-5000) is product of Sigma-Aldrich (USA) and polymethacrylic acid 15% aqueous sodium salt solution (MW 8415337) is a product of National Chemicals Baroda (India). Both the polymers were used as received in this work. The structure of both water-soluble polymers is represented as;



PVSA



PMA

The GPC of PMA obtained from ICT; Mumbai as shown in Figure No. 1:

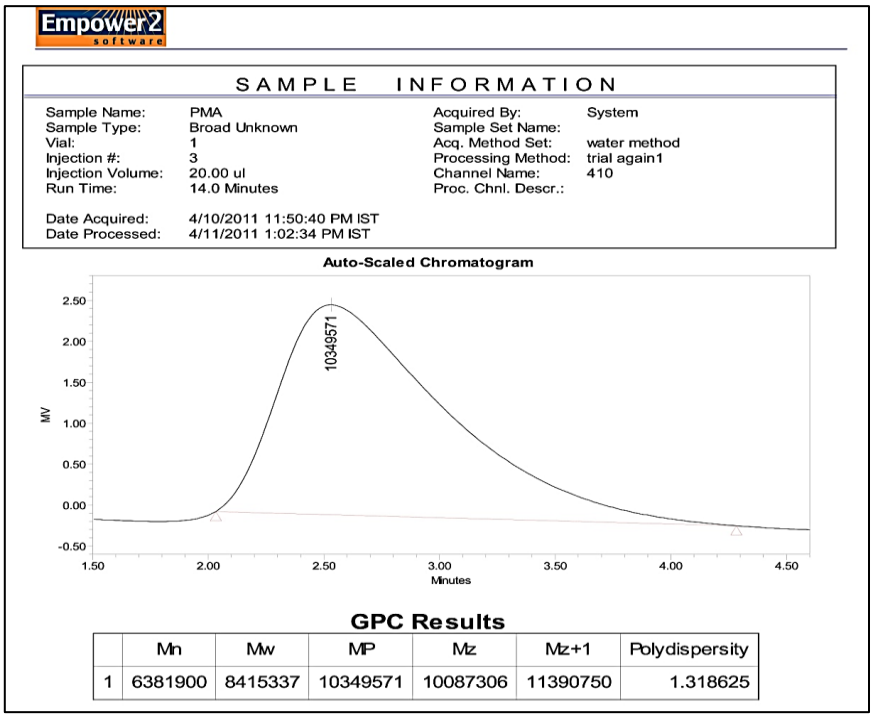


Figure 1: GPC of PMA

Preparation and characterization of orange-ot dye:

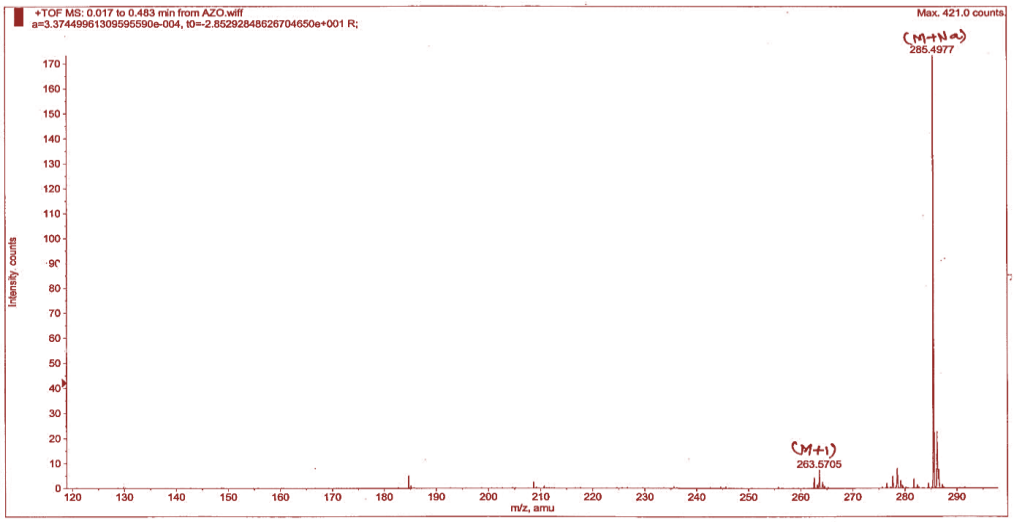


Figure 2: LC-MS of orange OT dye

Orange-OT dye (1-o-tolyl azo-2-naphthol) (MW-262.3) prepared from o-Toluidine and 2-naphthol undergoes diazotization followed by coupling reaction and

was purified twice by precipitating it from acetone with water and finally recrystallized from ethyl alcohol (MP is 124-126°C).

The orange-OT dye thus obtained characterized by its LC-MS (Figure No. 2) and IR Spectra (Figure No. 3). The LC-MS and IR of orange OT dye obtained from NCL, Pune.

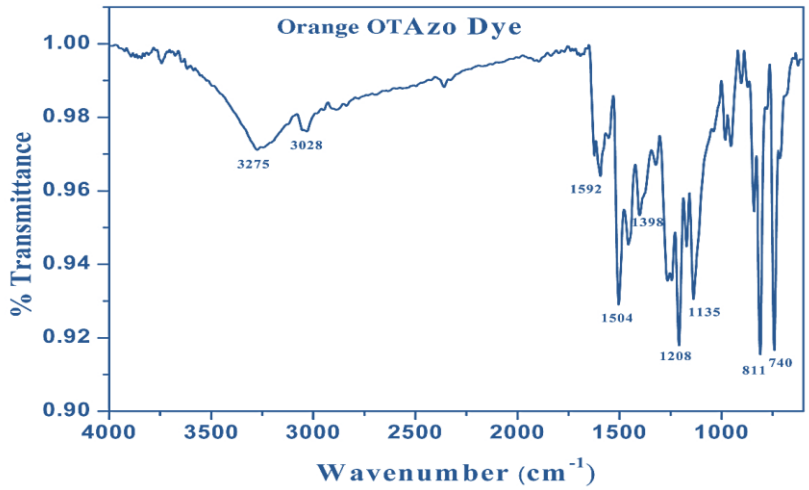
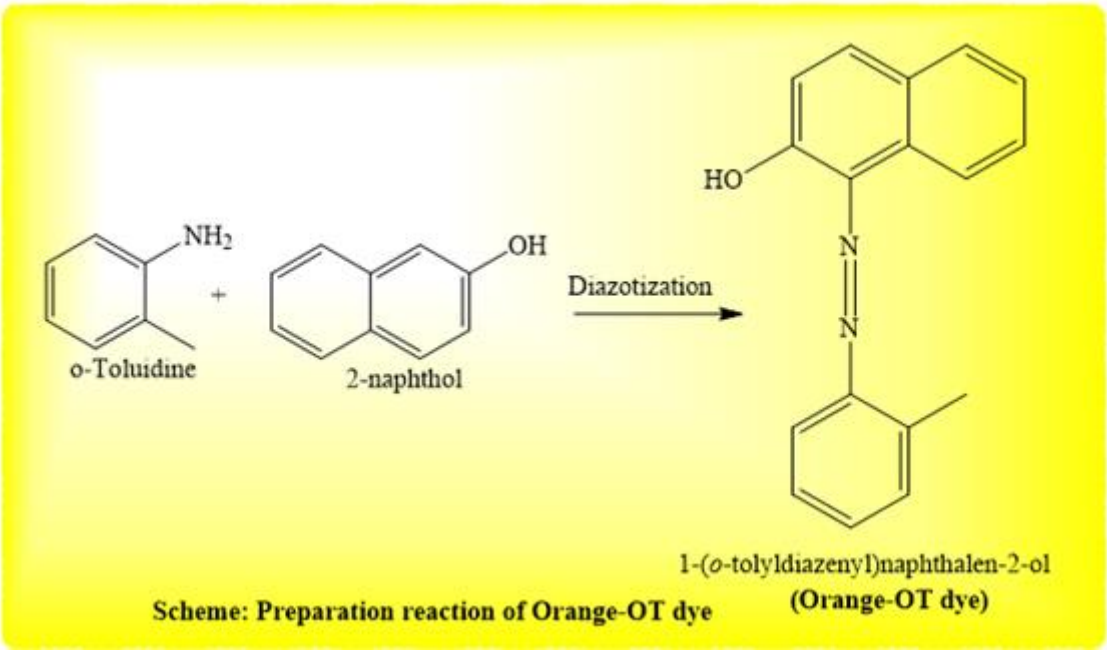


Figure 3: IR spectra of orange OT dye

The doubly distilled water obtained from Glass apparatus with specific conductance 2-4 μs cm^{-1} at 303K is used for the preparation of all the solution of various concentrations.

CLOUD POINT DETERMIATION:



Scheme: Preparation reaction of Orange-OT dye

The cloud point decrease with increasing length of hydrocarbon chain and decreasing length of oxyethylene chain of homologues series of non-ionic surfactants.

The effect of structural changes in the surfactant molecule on the cloud point shows that, at constant oxyethylene percent cloud point is decreased due to the following factors

(A) Decreased molecular weight of surfactant,

(b) Broader distribution of polyoxymethylene chain length,

(c) Branching of hydrophobic group,

(d) More central position of the polyoxymethylene hydrophilic group of surfactant molecule

(e) Replacement of the terminal hydroxyl group of the hydrophilic group by methoxyl and

(f) Replacement of the ether linkage between the hydrophobic and hydrophilic group by an ester linkage.

CP of pure non-ionic surfactants and CP of non-ionic–polymer mixed system:

Surfactants contain two distinct grouping in their structure. Strongly polar or charged group at one end of surfactant molecule is the "head group" which is hydrophilic in nature and long chain of alkyl or aryl group as the "tail" which is hydrophobic in nature. When surfactants are added to water at low concentration, they are dispersed as discrete molecules. However, at a particular concentration, surfactant molecules get associated to form aggregates or micelles, this concentration is known as critical micellar concentration (CMC) which is an important property of surfactant. Above CMC, the surfactant molecules exist as aggregates or micelles. The CMC of a surfactant was determined by several methods such as conductance, surface tension, dye solubilization / micellization, light scattering, diffusion, cloud point determination, ultrasonic velocity measurement etc. The non-ionic surfactants or with additives in aqueous solution cannot withstand at elevated temperatures and become perceptible even with naked eyes known as clouding and that point is referred as cloud point (CP). The cloud point is an important property of nonionic surfactants. Below CP a single phase of molecular solution or micellar solution exists and above CP, the solubility of surfactant in water is reduced and forms cloudy dispersion by forming giant molecular aggregates in the state of separate phase and the critical phenomenon in micellar solution and the micro-emulsions is increasingly becoming important and investigated by a number of workers. The water-soluble polymers also exhibit clouding behavior by similar mechanism, the phenomenon is reversible and CP stands for transition from water

soluble state to oil soluble state.

Result and Discussion:

Brij surfactants such as Brij-72, Brij-30, Brij-76, Brij-56, Brij-78 and Brij-35 are used in hygienic products, textile verabeitung, plant protection agent, colours and coatings, adhesives and other industrial applications. The main functions of Brij-56 are as O/W co-emulsifier, O/W emulsifier, wetting agent. The important applications of Brij-56 are in creams and lotions, conditioning, hair styling, hair treatment, colouring, facial make-up etc. Brij-56 not only acts as a dispersant but also affects the formation of the micelles due to its compatibility with anionic surfactant in forming micelles. The Brij-56 is used in the preparation of proton conducting tungstosilicate mesoporous materials. Brij-56 used as a structure-directing agent in the preparation of thin films of bicontinuous cubic mesostructured silica. Triton X-114 is used in separation of lipophilic and hydrophobic proteins. The principal use of Triton X-114 surfactant is in industrial and household detergent applications and as emulsifying agent. It is used almost in every type of liquid, paste and powdered cleaning compounds ranging from heavy-duty industrial products to gentle detergents for fine fabrics. Triton X -114 is also important ingredients of primary emulsifier mixtures used in the manufacture of emulsion polymers, stabilizers in latex polymers and emulsifiers for agricultural emulsion. concentrates, and the wettable powders. The cloud point of pure non-ionic surfactants Brij-56 at various concentrations in weight percentage are given in Table No. 1. The cloud points of the surfactant Brij-56 was found to be decreased with increased concentration of Brij-56 due to increase in micelle concentration and the micelle-micelle interaction causes phase separation.

Table 1: Cloud Points of Pure Brij-56 at different concentrations

[Brij-56] Weight (%)	Molarity x 10^{-2}	Mole fraction x 10^{-4}	CP	
			^{0}C	K
0.5	0.7331	1.3195	66.0	339.0
1	1.4663	2.6386	63.3	336.3
2	2.9326	5.2759	62.1	335.1
3	4.3988	7.9117	61.0	334.0
4	5.8651	10.5462	60.3	333.3
5	7.3314	13.1792	58.3	331.3

The linear plots of *lnXs* Vs (l/T x 10^{-3}) for non-ionic surfactants Brij-56 depicted in Figure 4.

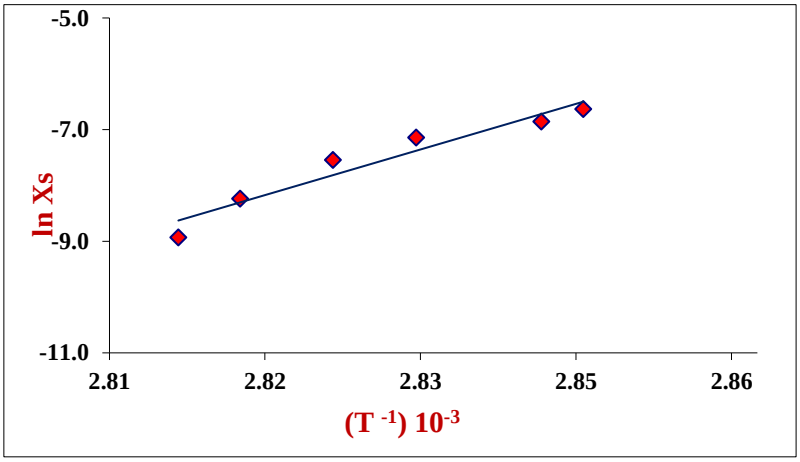


Figure 4: Plot of lnXs Vs (l/T x 10^{-3}) for Brij-56

Table 2: Cloud Points of Pure TritonX-114 at different concentrations.

[TX-114] Weight (%)	Molarity x 10^{-2}	Mole fraction x 10^{-3}	CP	
			^{0}C	K
1	1.8622	0.3351	24.9	297.9
2	3.7244	0.6699	25.1	298.1
3	5.5866	1.0046	25.4	298.4
4	7.4488	1.3390	25.6	298.6
5	9.3110	1.6732	26.1	299.1
6	11.1732	2.0072	27.2	300.2
7	13.0354	2.3409	28.1	301.1
8	14.8976	2.6744	28.3	301.3
9	16.7598	3.0077	29.4	302.4
10	18.6220	3.3408	30.3	303.3

The clouding of non-ionic surfactants and water-soluble polymers is an interesting phenomenon and is important for researchers and scientists deals with physical and chemical processes and at increased temperature condition the clouding species non-ionic surfactant and polymers undergoes de-solvation.

The influence of PMA on the non-ionic surfactants Brij-56, and Triton X-114 has been given in Table No. 3 and 4 respectively.

Table 3: Influence of PMA on CP of Brij-56

[PMA] Weight (%)	CP⁰ C					
	[Brij-56] Weight (%)					
	0.5	**1**	**2**	**3**	**4**	**5**
0.010	84.4	83.6	81.3	80.1	79.0	78.7
0.0250	83.8	82.7	81.4	79.6	78.8	78.6
0.05	82.4	81.6	80.1	79.2	78.3	77.9
0.075	81.2	80.8	79.9	78.0	77.8	76.6
0.1	80.9	80.5	79.6	77.7	77.4	76.2

Table 4: Influence of PMA on CP of TritonX-114

[PMA] Weight (%)	CP⁰ C									
	[TritonX-114] Weight (%)									
	1	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**
0.02	49.9	49.4	48.6	48.1	47.8	47.1	46.5	46.0	45.4	44.6
0.04	48.1	47.0	46.4	46.0	45.6	45.3	44.9	44.3	43.6	43.1
0.06	45.5	44.3	44.0	43.7	43.2	42.7	42.2	41.4	40.0	39.3
0.08	43.4	42.0	41.7	41.2	40.9	40.5	40.3	39.5	38.8	38.4
0.1	42.7	41.7	41.0	40.6	39.9	39.1	38.4	37.7	37.0	36.3

The dependence of CP on concentration of PMA is depicted in the non-ionic surfactants Brij-56, and Triton X-114 respectively.

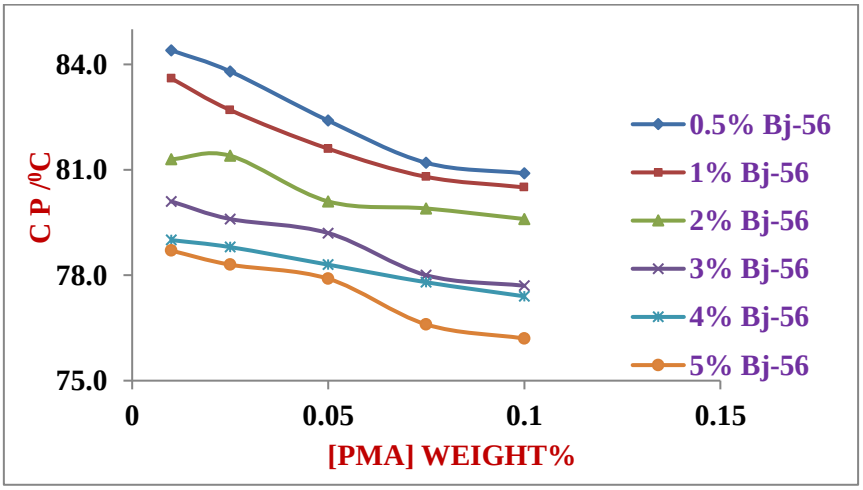


Figure 5: Influence of PMA on CP of Brij-56

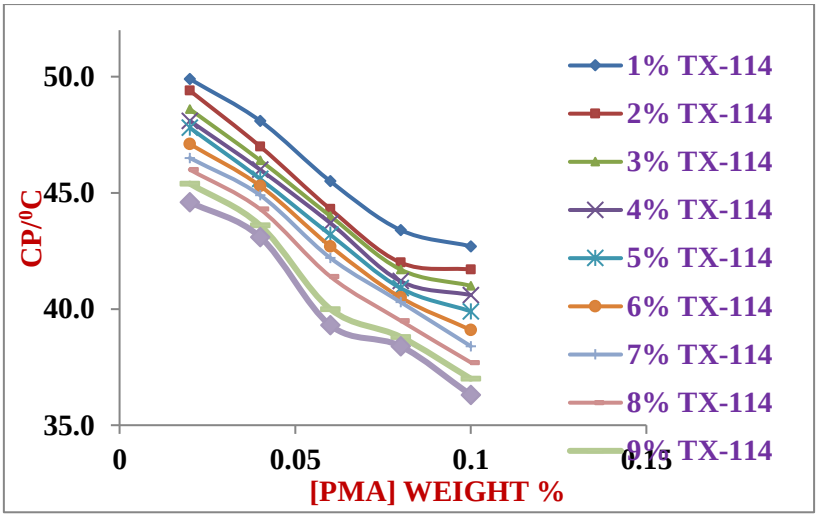


Figure 6: Influence of PMA on CP of TritonX-114

Conclusion:

The influence of water-soluble polymer PMA on the CP of Brij- 56 and TritonX-114 has been studied and the results on the influence of polymer on thermodynamic behavior of non-ionic surfactant are highlighted as follows;

The cloud point of non-ionic surfactant Brij-56 and TritonX-114 decreased considerably with increased concentration of water-soluble polymer PMA.

These results indicating that, for same surfactant concentration when concentration of polymer increased then in general the CP increase indicating the increase in micellar charge density, hence it is suggested that charge density on mixed micelle will determine the cloud point i.e. higher the charge density higher is the cloud point. The increase in concentration of polymer need not always increase the charge density because charge density will be depend upon micelle size i.e. micelle size might be change due to addition of polymer**,** Secondly polymer-surfactant complex is stronger due to solute-solvent interaction, some of the water molecules remain attached to this complex and hence higher temperature is required to break down this strong complex system and an incorporation of the polyelectrolyte PMA into non-ionic micelles introduce electrostatic repulsion between the micelles causes hindering the phase formation and raising the cloud point. The above results indicating that, the removal of water from surfactant by added polymer and helps the surfactant micelles to come closer with each other resulting the lowering of CP. The CP may also be decreased due to water-structure breaking additive provide water molecule for hydration of non-ionic surfactants.

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