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Best Wishes & Greetings



It gives me immense pleasure to extend my best wishes for the One-Day International Conference on **“Frontiers In Sustainable And Advanced Chemical Sciences”-(FSACS-2026)”** organized by the Department of Chemistry and Research Center, Waghire College of Arts, Commerce and Science, Saswad, affiliated to Savitribai Phule Pune University, Pune. Chemistry is central to building a sustainable future. The theme of this conference rightly addresses the urgent need for green technologies, energy-efficient processes, and advanced materials that balance scientific progress with environmental responsibility. I am confident that the deliberations and research papers presented here will contribute significantly to both academic excellence and societal development. I congratulate the Principal, faculty members, and organizing team for this commendable initiative and for publishing the Abstract Book with ISBN. My best wishes to all delegates, researchers, and students for a fruitful academic exchange. Wish you all the best. !

Hon. Sunetra Ajitdada Pawar
(Deputy Chief Minister, Maharashtra State)
(President, Pune District Education Association's, Pune)

Best Wishes & Greetings



It gives me immense pleasure to extend my heartiest congratulations and best wishes for the One-Day International Conference on “***Frontiers in Sustainable and Advanced Chemical Sciences – FSACS-2026***” organized by the Department of Chemistry and Research Center, Waghire College of Arts, Commerce and Science, Saswad, affiliated to Savitribai Phule Pune University, Pune. In today’s era of scientific advancement, sustainable chemical research is vital for addressing global challenges related to environment, energy, and human health. The theme of this conference is both timely and relevant, as it brings together researchers and academicians to deliberate on innovative solutions for a better future. I am confident that the exchange of ideas during this conference and the publication of the ISBN Abstract Book will serve as a valuable academic resource and inspire young researchers to pursue excellence in chemical sciences. I deeply appreciate the efforts of Principal Dr. Pandit N. Shelke, the Department of Chemistry, and the organizing committee for hosting this prestigious international conference. I wish the event great success and all participants a fruitful academic experience. Wish you all the best. !

A handwritten signature in blue ink, appearing to read 'Rajendra Ghadage'.

Vice President
Hon. Rajendra Ghadage
(Vice-President, Pune District Education Association’s)

Best Wishes & Greetings



It gives me immense pleasure to extend my best wishes for the One-Day International Conference on “**Frontiers in Sustainable and Advanced Chemical Sciences – FSACS-2026**” organized by the Department of Chemistry and Research Center, Waghire College of Arts, Commerce and Science, Saswad, affiliated to Savitribai Phule Pune University, Pune. Chemical sciences today stand at the crossroads of innovation and sustainability. The deliberations on green chemistry, advanced materials, and sustainable technologies during this conference will surely contribute to resolving pressing global challenges and promoting responsible scientific development. I wholeheartedly congratulate Principal Dr. Pandit N. Shelke, the faculty of the Chemistry Department, and the entire organizing team for this commendable academic initiative and for publishing the Abstract Book with ISBN. Such endeavors reflect the institution’s commitment to research, academic excellence, and societal progress. I am confident that this conference will provide a vibrant platform for scholars, researchers, and students to exchange knowledge and build lasting collaborations. I wish the event a grand success.



Hon. Sandip Kadam
Secretary, Pune District Education Association’s

Best Wishes from Principal



It gives me immense pleasure to extend my best wishes for the One-Day International Conference on **“Frontiers in Sustainable and Advanced Chemical Sciences – FSACS-2026”** organized by the Department of Chemistry and Research Center, Waghire College of Arts, Commerce and Science, Saswad, affiliated to Savitribai Phule Pune University, Pune. At Waghire College, we firmly believe that scientific inquiry must go hand in hand with social responsibility. The theme of this conference aptly emphasizes the role of chemistry in achieving sustainability through green technologies, advanced materials, and innovative research practices. It is truly a matter of pride that our institution is hosting this intellectual gathering of eminent scientists, academicians, and young researchers from across the globe. I sincerely appreciate the dedicated efforts of the Head, Department of Chemistry, the Research Center, and the entire organizing committee for conceptualizing this conference and publishing the ISBN Abstract Book. Such initiatives strengthen our commitment to quality education, research culture, and interdisciplinary collaboration. I am confident that the deliberations at FSACS-2026 will inspire new ideas, foster research partnerships, and contribute meaningfully to the scientific community. I extend a warm welcome to all delegates and wish the conference a grand success.

A handwritten signature in blue ink, appearing to read 'Pandit Shelke', with a large, stylized initial 'P' at the beginning.

Dr. Pandit Shelke
Principal

Waghire College of Arts, Commerce and Science, Saswad

Editorial Message!



It is with great pleasure and enthusiasm that I extend a warm welcome to all delegates, resource persons, and participants to the One-Day International Conference on “**Frontiers in Sustainable and Advanced Chemical Sciences – FSACS-2026**”, organized by the Department of Chemistry and Research Center, Waghire College of Arts, Commerce and Science, Saswad, affiliated to Savitribai Phule Pune University, Pune. The International Conference "Frontiers in Sustainable and Advanced Chemical Sciences" (FSACS-2026) brings together researchers, academicians, and industry experts to explore cutting-edge advancements in chemical sciences. The seminar focuses on sustainable practices, innovative technologies, and interdisciplinary research, aiming to address global challenges and shape the future of chemical sciences. the main objectives of the conference is to discuss innovative approaches for sustainable chemical processes and technologies, to encourage collaboration among chemists, engineers, biologists, and other experts, to showcase cutting-edge materials research and applications, to focus on chemical sciences solutions for environmental, energy, and health issues, to promote innovation and entrepreneurship: inspire participants to translate research into impactful applications and startups. I sincerely thank our Hon'ble Management, Principal Dr. Pandit N. Shelke, and the Research Advisory Committee for their constant support and guidance. I also appreciate the dedicated efforts of the organizing committee, reviewers, and student volunteers who have worked tirelessly to make this event possible. I am confident that FSACS-2026 will serve as an excellent platform for knowledge exchange, networking, and fostering collaborative research. The publication of the ISBN Abstract Book will further enrich the academic value of this conference. I wish all participants a rewarding and intellectually stimulating experience. May your deliberations lead to innovations for a sustainable and advanced scientific future. Thank you!

A handwritten signature in black ink, appearing to read 'Dr. Kamble'.

Dr. Narendra Ramdas Kamble
Co-ordinator (FSACS-2026)



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Original Article

An Efficient Photodegradation of Methylene Blue Using Ni²⁺ Substituted Nano Zinc Ferrites Synthesized by Oxalate Precursor Method

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Abstract

The oxalate precursor method was used to prepare nano Ni²⁺ substituted nano zinc ferrites. The oxalate precursor was synthesized and calcined for four hours at 600 °C. Two major bands were observed in the FT-IR spectra of calcinated Ni²⁺ substituted nano zinc ferrites, the higher frequency band (ν₁ 560–625 cm⁻¹) and the lower frequency band (ν₂ 378–498 cm⁻¹) confirming the spinel structure's typical features. The XRD patterns confirmed the formation of the single-phase cubic spinel structure, with no additional peaks that corresponded to any other phases. When Ni²⁺ was substituted, the lattice constant and X-ray density decreased. The SEM images show tiny particles with some degree of agglomeration. Nano Ni²⁺-substituted nano zinc spinel ferrites have been shown to exhibit good activity for methylene blue degradation. As the pH of the solution increased, the rate of methylene photodegradation increased. For the photodegradation of methylene blue, nano spinel Ni_{0.5}Zn_{0.5}Fe₂O₄ ferrite nanoparticles are an inexpensive, easily magnetically recoverable, simply produced, and effective photocatalyst.

Keywords: Zinc Ferrite; Methylene blue; Photodegradation; Oxalate precursor

Introduction

The extensive use of hazardous materials in various industrial areas elevates hazards to human health and the environment. The continuous release of untreated effluents from pulp and paper, leather tanning, textile processing and pharmaceutical manufacture causes damage to aquatic systems due to the variety of hazardous organic and inorganic contaminants [1]. Organic dyes are used extensively by the textile, printing and leather industries all over the world. These industries produce wastewater containing many artificial pigments and colorants. The decline in fresh water resources is a big environmental issue. This is because serious water pollution occurs which has an adverse effect on aquatic life, human health and the overall ecological balance. It has been reported that effluents from industrial organic dyes commonly used are one of the major causes of water pollution [2]. Recently, some of the innovative treatment techniques, such as ozonation, adsorption, biodegradation, chemical oxidation and photocatalysis, have been utilized for the removal of hazardous contaminants and highly poisonous organic dyes from polluted water. However, most of these methods have been of limited practical use, due to their high operating costs, technological complexity and large sludge formation. Another method is to use a heterogeneous photocatalyst, which is a popular way to treat stubborn industrial wastewater. Photocatalytic nanomaterials are capable as they can completely removes organic contaminants to CO₂ and H₂O without generating harmful byproducts [3]. Residual dyes in wastewaters from textile, paper and related industries are a major environmental problem, particularly in developing countries. Conventional treatment methods such as adsorption and chemical coagulation are mainly used to transfer the dyes from the aqueous phase to the solid phase, generating secondary waste which needs further management.

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Oxidative reactions, however, offer a more efficient way to degrade dyes and other organic pollutants in industrial effluents. It has been proved that semiconductor-mediated photocatalytic oxidation is effective in decomposing the dye pollutants [4]. At room temperature, methylene blue (MB) is a dark green, odourless crystalline solid that dissolves in water to create a vivid blue solution. Tetramethyl thionine chloride, or 3,7-bis (dimethyl amino) phenothiazine chloride, is its IUPAC name. Its molecular weight is $319.85 \text{ g mol}^{-1}$, and its formula is $\text{C}_{16}\text{H}_{18}\text{N}_3\text{SCL}$. These dyes degrade the visual quality of surface waters, drastically restrict light penetration in water bodies, and decrease photosynthesis in aquatic plants [5,6]. The textile industry often releases large amounts of methylene blue (MB) into natural water bodies, endangering microbial populations and human health [7]. MB is known to be hazardous to humans and shows considerable toxicity at high concentrations [8]. MB's poisonous, carcinogenic, and non-biodegradable characteristics cause serious effects on human health and the aquatic environment. Environmental scientists continue to have a great deal of difficulty in the treatment of contaminated water bodies by removing dyes, which calls for the creation of effective and long-lasting treatment methods. For wastewater treatment, different traditional practices have been used, such as coagulation, ozonation, aerobic and anaerobic biodegradation, electrochemical oxidation, precipitation, adsorption, and photocatalysis. Amongst these, photocatalytic degradation has become well-known as a reasonably priced and simple method since the reaction takes place immediately on the catalyst's surface when exposed to light [9]. Semiconductor nanoparticle-mediated multi-component photocatalysis has gathered significant interest for the degradation of organic dye contaminants during the last few decades. For treating wastewater tainted with dangerous organic chemicals, this strategy is thought to be economical, safe for the environment, and easy to use [10]. The inexpensive cost of the catalyst, combined with the usage of renewable solar energy, makes this approach extremely appealing when compared to traditional treatment methods [11]. An efficient and crucial method for eliminating methylene blue (MB) from industrial wastewater is photocatalytic oxidation of MB to CO_2 and H_2O [12]. Ferrite nanoparticles employed by several research groups as effective catalysts for the reduction of nitroaromatic chemicals and the photocatalytic degradation of harmful dyes. Specifically, the degradation of an orange azo dye has been studied using Zn-ferrite nanoparticles doped with Al^{3+} . When Al^{3+} was doped in the zinc ferrite

lattice, the photocatalytic efficiency was greatly increased; the composition with equimolar quantities of Al^{3+} and Fe^{3+} showed the maximum activity over the whole doping range [15]. According to Niyaz [16], copper-ferrite nanoparticles were used as the catalyst for photocatalytic ozonation of colours. With the general formula AB_2O_4 , where A and B are divalent and trivalent metal cations, respectively, and iron is often one of the constituent metals, many ferrites take on the spinel structure. An inverse spinel arrangement is produced in spinel ferrites when the oxygen ions (O^{2-}) form a face-centered cubic (FCC) lattice, with A cations occupying one-eighth of the tetrahedral interstices and B cations occupying half of the octahedral interstices [19]. The type of metal cations and distribution between the tetrahedral (A) and octahedral (B) sites have an extensive effect on the magnetic and electrical characteristics of ferrites. Therefore, understanding cation distribution is crucial to comprehending spinel ferrites' magnetic behaviour. For example, manganese ferrite typically demonstrates a mixed inverse spinel structure, with the degree of inversion being highly dependent on the preparation route. The properties (such as electrical and magnetic) of ferrites can be considerably altered by the addition of non-magnetic ions [20–22]. Nickel's unique electrical and magnetic properties make it a very intriguing dopant. The crystal structure, magnetic characteristics, and electrical conductivity of zinc ferrite are changed when Ni^{2+} is substituted, making nickel-doped zinc ferrites appropriate for uses like electrocatalysis, microwave absorption, and magnetic data storage [23]. Because of the particular occupancy of Ni^{2+} and Fe^{3+} cations at the tetrahedral and octahedral positions, zinc ferrite takes on an inverse spinel structure and displays ferrimagnetism. Significant changes in the material's electrical conductivity and magnetic properties result from substitution with Ni^{2+} ions, which modifies the cation distribution [24]. Among ferrite materials, zinc ferrite (ZnFe_2O_4) has been systematically investigated due to its exceptional chemical stability, high electrical resistivity, and advantageous magnetic properties. It might be used in several applications, such as magnetic data storage, sensing technologies, transformer cores, microwave components, and catalytic processes in chemical reactions, thanks to its cubic spinel crystal structure and ferrimagnetic characteristic [25]. For the manufacture of nanoferrites, the oxalate precursor pathway is acknowledged as an effective and cost-effective method. In this process, metal ions combine with oxalic acid to create corresponding metal oxalate intermediates. These intermediates then undergo

thermal decomposition (calcination) at high temperatures, typically between 600 and 1000 °C, to form the required ferrite phase [32]. Ferrites with high crystallinity and low impurity content can be produced using the oxalate precursor method, which is crucial for applications requiring outstanding material performance. Furthermore, the process is simple, affordable, and scalable, which makes it appropriate for the large-scale production of superior nanoferrites.

2. Material and Methods:

2.1 Materials and Chemicals –

Ni²⁺-substituted nanocrystalline zinc ferrites with compositions ZnFe₂O₄, Ni_{0.25}Zn_{0.75}Fe₂O₄, and Ni_{0.5}Zn_{0.5}Fe₂O₄ were synthesized using the oxalate precursor route. Analytical grade reagents employed in the synthesis included zinc (II) sulfate hexahydrate (ZnSO₄·6H₂O), nickel (II) sulfate pentahydrate (NiSO₄·5H₂O), ferric sulfate hydrate (Fe₂(SO₄)₃·7H₂O), oxalic acid dihydrate (H₂C₂O₄·2H₂O), along with deionized water as the solvent.

2.2 Synthesis of Ni²⁺ substituted Nano Zinc Ferrite

The oxalate precursor method was used to synthesize Ni²⁺-substituted nanocrystalline zinc ferrites with compositions of ZnFe₂O₄, Ni_{0.25}Zn_{0.75}Fe₂O₄, and Ni_{0.5}Zn_{0.5}Fe₂O₄. AR grade metal sulphates and oxalic acid were used without further purification. Initially stoichiometric amounts of corresponding metal sulphates were dissolved in deionized water with constant stirring at 60 °C. All metal ions completely precipitated as metal oxalates by adding saturated oxalic acid with stirring. The precipitate was washed with deionized water after 30 minutes of aging till removal of sulphate ions (tested by barium chloride). Then the precipitate was filtered, washed and dried at room temperature. The final Ni²⁺-substituted spinel zinc-ferrite obtained by calcinating dried metal oxalate precursor for four hours at 600 °C.

2.3 Characterization –

AFT-IR spectrophotometer was employed to analyse the oxalate precursors and the calcined Ni²⁺-substituted nano zinc ferrites. Using a Phillips-3710 diffractometer with Cu-Kα radiation (λ = 1.5405 Å), the structural characteristics of the Ni²⁺-substituted

nano-zinc ferrites were examined using X-ray diffraction (XRD). Using a JEOL JSM-5600N scanning electron microscope, the microstructure of the Ni²⁺-substituted nano zinc ferrites was investigated.

2.4 General Procedure for Photodegradation of Methylene Blue:

Methylene blue (MB) was used as a model cationic dye in this study to assess the photocatalytic degradation capability of the produced Ni_{0.5}Zn_{0.5}Fe₂O₄ ferrite nanoparticles. Methylene blue, chemically known as 3,7-bis(dimethylamino)-phenothiazine-5-in chloride (tetramethyl thionine), molecular weight of 319.85 g/mol. By breaking down methylene blue (MB) at five different pH levels while exposed to natural sunshine, the impact of pH on photocatalytic activity was examined. Each experiment used 100 mL of MB solution (10 mg/L) to scatter 10 mg of Ni_{0.5}Zn_{0.5}Fe₂O₄ ferrite nanoparticles. As needed, diluted H₂SO₄ or NaOH was used to alter the solution's original pH. The suspension was agitated for 30 minutes in the dark to achieve adsorption–desorption equilibrium. The combination was then exposed to natural sunshine to start photocatalytic breakdown. 3 mL aliquots were centrifuged and subjected to UV-Vis spectroscopic analysis every 30 minutes. The degradation was monitored by tracking the absorption peak at 664 nm.

Results And Discussion:

3.1 FT - IR analysis of Precursor –

The FT-IR spectrum of the synthesized oxalate precursor [ZnFe₂(C₂O₄)₃·2H₂O] recorded in the range of 400–4000 cm⁻¹ (Figure 1). The symmetric and asymmetric stretching vibrations of coordinated water molecules (OH₂) are represented by two bands detected at 3396 cm⁻¹ and 3322 cm⁻¹, respectively. The C=O stretching vibration is responsible for a high absorption band at 1665 cm⁻¹. The closely spaced peaks at 1392 cm⁻¹ and 1338 cm⁻¹ identify the symmetric and asymmetric C–O stretching vibrations, respectively. The O–C–O deformation vibration is the source of the band at 845 cm⁻¹. Furthermore, Fe–O and Zn–O stretching vibrations bands observed at 478 cm⁻¹ and 521 cm⁻¹, respectively.

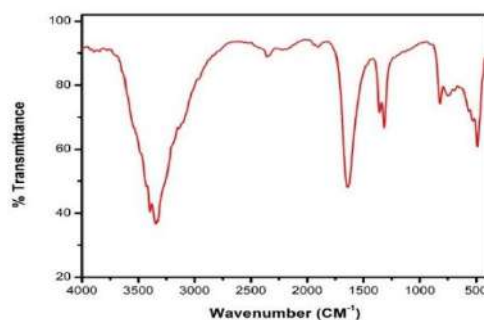


Figure 1: Typical IR spectrum of Oxalate Precursor $[ZnFe_2(C_2O_4)_3 \cdot 2H_2O]$

3.2 FT - IR analysis of Ferrite –

The FT-IR spectra of calcined Ni^{2+} -substituted nano zinc ferrites were obtained between 400 cm^{-1} and 800 cm^{-1} (Figure 2). The stretching vibrations of tetrahedral complexes are associated with the higher wavenumber band (ν_1) at 562 cm^{-1} , while octahedral complexes are associated with the lower wavenumber band (ν_2) at 425 cm^{-1} . The ν_2 band in the Ni^{2+} -

substituted nano zinc ferrite originates from $Fe^{3+}-O^{2-}$ vibrations in the tetrahedral (A) sublattice, whereas the ν_2 band is linked to metal-oxygen vibrations at the octahedral (B) sites. The discrepancy in $Fe^{3+}-O^{2-}$ bond lengths between the tetrahedral and octahedral sites is responsible for the observed difference in the locations of ν_1 and ν_2 [32].

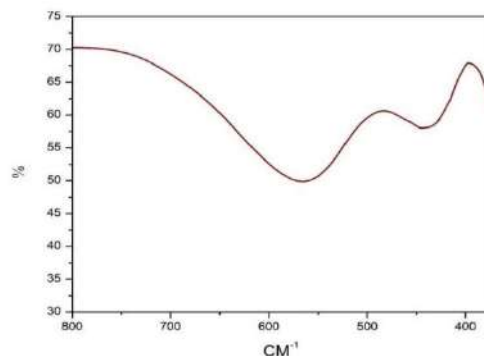


Figure 2: Typical IR spectrum of $ZnFe_2O_4$

3.2 XRD analysis –

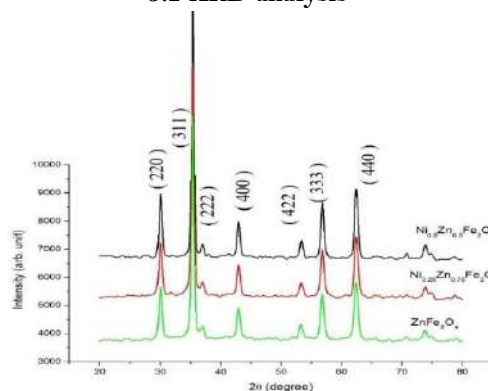


Figure 3: XRD patterns of calcined $ZnFe_2O_4$, $Ni_{0.25}Zn_{0.75}Fe_2O_4$, and $Ni_{0.5}Zn_{0.5}Fe_2O_4$

Figure 3 displays the X-ray diffraction (XRD) patterns of $ZnFe_2O_4$, $Ni_{0.25}Zn_{0.75}Fe_2O_4$, and $Ni_{0.5}Zn_{0.5}Fe_2O_4$ samples that were calcined at 600°C . The cubic spinel structure typical of Ni^{2+} -substituted zinc ferrites is steady with all observed diffraction peaks. The formation of a single-phase cubic-spinel structure with great phase purity is confirmed by the lack of any other peaks. The formation of the nickel-

zinc spinel ferrite structure is confirmed by the diffraction peaks corresponding to the (220), (311), (222), (400), (422), (333), and (440) planes. Using the Debye-Scherrer formula, the average crystallite size of the Ni^{2+} -substituted nano zinc ferrites was calculated from the higher intense (311) peak [8]. Table 1 displays the computed crystallite sizes. Using the relationship previously published [8], the lattice parameter (a) was determined from the XRD data:

$$a = d\sqrt{h^2 + k^2 + l^2} \quad (1)$$

where, d is the interplanar distance and a is the lattice constant. Table 1 displays the computed lattice parameters for the Ni²⁺-substituted nano zinc ferrites. The following formula was used to calculate the samples X-ray density [8]:

$$d_x = \frac{8M}{Na^3} \quad (2)$$

where M is the molecular weight, a is the lattice constant, N is Avogadro's number and 8 is the number of formula units per unit cell. Table 1 displays the X-ray density values that were obtained.

Table 1: Lattice constant (a), X-ray density (d_x) and crystallite size (D_{xrd}) of Ni²⁺ substituted Nano Zinc Ferrites

Sample	'a' (Å)	'd' (g/cm ³)	Particle size D _{xrd} (nm)
ZnFe ₂ O ₄	8.436	5.632	33.21
Ni _{0.25} Zn _{0.75} Fe ₂ O ₄	8.423	5.515	30.36
Ni _{0.5} Zn _{0.5} Fe ₂ O ₄	8.415	5.436	28.42

3.3 SEM analysis –

The SEM micrograph (Figure 4) shows that the Ni_{0.5}Zn_{0.5}Fe₂O₄ nanoparticles calcined at 600 °C, prepared by the oxalate precursor route, have a relatively uniform morphology consisting of spherical to cubic particles with a narrow size distribution. The particles are fine and there is a moderate level of aggregation.

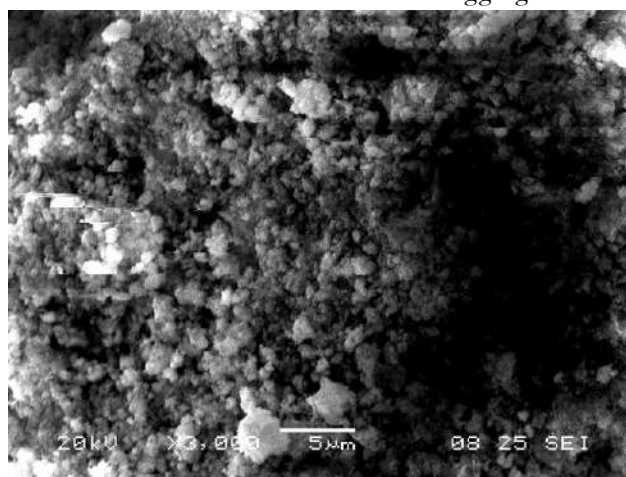


Figure 4: Typical SEM micrograph of calcined Ni_{0.5}Zn_{0.5}Fe₂O₄

3.4 Photodegradation of Methylene Blue using Ni_{0.5}Zn_{0.5}Fe₂O₄ nanoparticles spinel ferrite Photocatalyst:

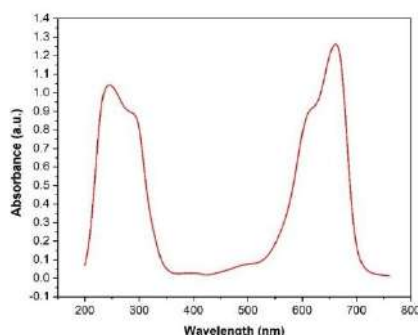


Figure 5: UV - visible spectrum of methylene blue

At 291 nm and 664 nm, methylene blue shows prominent absorption bands. When exposed to visible light, the distinctive band at 664 nm gradually decreases intensity of band as the irradiation period increases in the presence of the catalyst (Figure 5).

An efficient and sustainable method for removing organic dyes from industrial wastewater is the photocatalytic breakdown of methylene blue (MB) into CO₂ and H₂O. In this method, a Ni-Zn spinel ferrite remains unaltered while absorbing photons to

produce charge carriers that speed up the redox processes that cause dye mineralization. While electrons in the conduction band decrease dissolved oxygen, holes in the valence band oxidize adsorbed water molecules, producing superoxide anion radicals ($O_2^{\bullet-}$) and hydroxyl radicals ($HO^\bullet$). The

breakdown of organic contaminants is mostly caused by hydroxyl radicals, which have a strong oxidizing ability. The following equations illustrate how the photogenerated electrons propel subsequent redox processes by creating superoxide species.

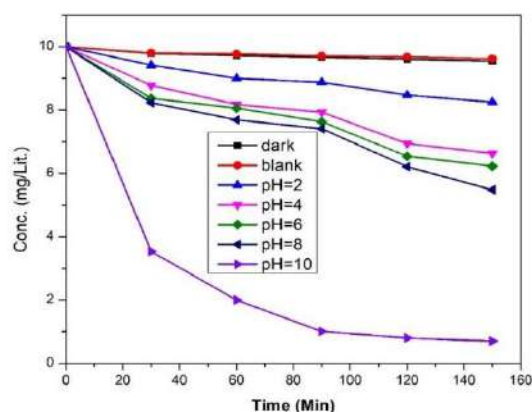
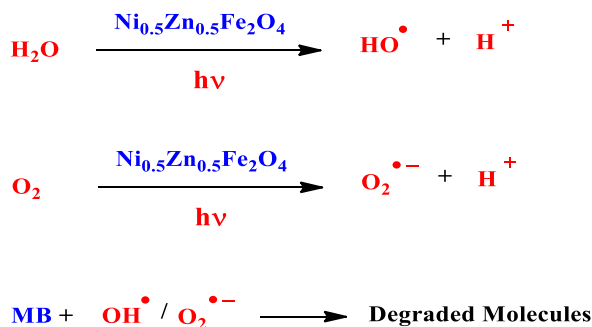


Figure 6: Photocatalytic degradation of methylene blue (Conc. Vs Time)

$Ni_{0.5}Zn_{0.5}Fe_2O_4$ ferrite nanoparticles were used to photocatalytically degrade methylene blue (MB) at various pH levels. UV-Vis's spectroscopy was used to track the change in MB absorbance when the $Ni_{0.5}Zn_{0.5}Fe_2O_4$ photocatalyst was present (Figure 6). The $Ni_{0.5}Zn_{0.5}Fe_2O_4$ photocatalyst efficiently starts the breakdown of methylene blue (MB) when exposed to

visible light. MB can be decomposed via two-electron reduction or oxidative degradation [33]. A significant deterioration of MB was attained after 150 minutes of exposure. In comparison to pH 2, 4, 6, and 8, the best degradation efficiency is reached at pH 10, according to the degradation versus time profiles (Figures 6, 7, and 8).

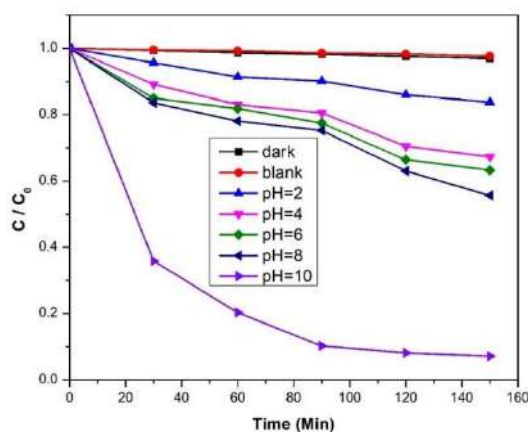


Figure 7: Photocatalytic degradation of methylene blue (C/C_0 Vs Time)

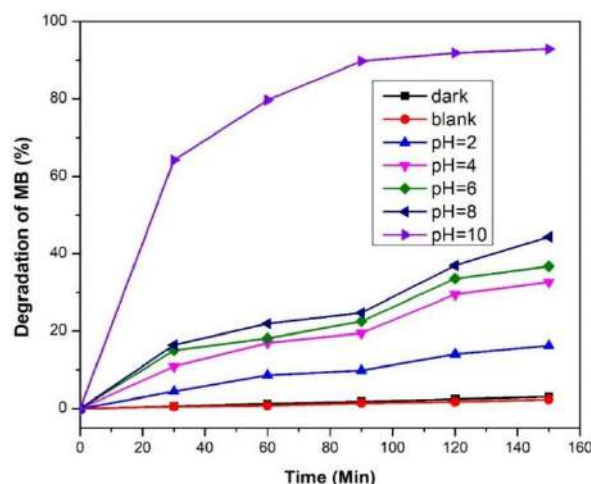


Figure 8: Photocatalytic degradation of methylene blue (% Degradation Vs Time)

The following equation was used to estimate dye degradation.

$$\text{Degradation \%} = \frac{C_0 - C_t}{C_0} \times 100$$

$$\text{Degradation \%} = \frac{A_0 - A_t}{A_0} \times 100$$

A_0 and C_0 represent the initial absorbance of the methylene blue dye solution, while A_t and C_t denote the absorbance measured after exposure to sunlight for time t .

Effect of Irradiation Time

The time of irradiation and adsorption-desorption equilibrium between the spinel ferrite photocatalyst and methylene blue (MB) are the important factors affecting the total photodegradation efficiency. Longer irradiation time results in a constant increase in MB dye degradation. The distinctive MB absorption peak at 664 nm gradually decreases with time and the colour of the solution becomes faint blue. The decrease in absorption is due to breakdown of the MB chromophore.

The photodegradation of methylene blue (MB) first increases progressively with irradiation time before it reaches its peak, indicating that equilibrium has been reached. The impact of irradiation duration on MB deterioration is depicted in Figures 6 and 7, which demonstrate a gradual decline in the intensity of the distinctive absorption peak at 664 nm. The decrease in MB concentration seen in Figure 8 further supports this tendency. Both light irradiation and the photocatalyst are required for efficient degradation, as demonstrated by control experiments carried in the absence of the photocatalyst (blank) and in the dark using $\text{Ni}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$ ferrite that showed no discernible change in MB concentration.

The pH Effect on Photodegradation of Methylene Blue:

The pH of the solution greatly influences both the routes involved in photocatalytic processes and the behaviour of dye molecules. It affects important chemical processes like the production of hydroxyl radicals, reduction reactions at the conduction band, and oxidation by photogenerated holes. Furthermore, pH heavily influences the photocatalyst's surface charge, controlling its interaction with dye species. Methylene blue (MB) is a cationic dye; therefore, situations where the photocatalyst surface has a larger negative charge are favourable for its adsorption.

As shown in Figures 6–8, the pH of the reaction medium has a substantial impact on the efficiency of methylene blue (MB) photodegradation. Alkaline circumstances cause the photocatalyst surface to become negatively charged, which encourages positively charged MB molecules to adsorb through electrostatic attraction. On the other hand, abundant H^+ ions in acidic environments compete with MB for active sites on the catalyst surface, which inhibits dye adsorption. The interaction between MB and reactive hydroxyl radicals ($\cdot\text{OH}$) is limited by the decreased adsorption, which lowers the efficacy of photocatalytic degradation. Methylene blue (MB) and hydroxyl species have much less competition for adsorption sites at higher pH levels. Hydroxyl ions are repelled by the photocatalyst's negatively charged surface, which keeps them mostly in the bulk solution. As a result, MB adsorption is enhanced in alkaline environments. Specifically, the $\text{Ni}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$ ferrite photocatalyst's negatively charged surface increases electrostatic interaction with the cationic MB molecules, increasing adsorption efficiency. The data shown in Table 1 and Figure 8 demonstrate that the rate of methylene blue photodegradation rises

with increasing pH of the solution when the $\text{Ni}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$ ferrite photocatalyst is present.

Table 1: Photodegradation of methylene blue after 150 min irradiation time.

pH	2	4	6	8	10
% Degradation	18.36	34.19	36.98	46.42	94.18

At pH 10, the $\text{Ni}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$ ferrite photocatalyst exhibits maximum photocatalytic performance, achieving 94.18% degradation of methylene blue.

Conclusion

The oxalate precursor method was successfully used to prepare nano-sized Ni^{2+} -substituted zinc ferrites. Two different absorption bands characteristic of a spinel structure were shown by FT-IR analysis of the calcined Ni^{2+} -doped samples: a lower-frequency band (ν_2) between 378 and 498 cm^{-1} and a higher-frequency band (ν_1) between 560 and 625 cm^{-1} . The development of a single-phase cubic spinel structure devoid of impurity peaks was confirmed by XRD data. It was shown that when the Ni^{2+} content increased, both lattice parameters and X-ray density steadily dropped. The Scherrer equation was used to determine the crystallite size, which observed between 28.42 and 33.21 nm. The tiny particles with moderate aggregation were formed, according to SEM pictures. Methylene blue's concentration did not significantly change in the absence of a photocatalyst or in the dark with $\text{Ni}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$ according to control studies, indicating minimal adsorption and self-degradation. In the presence of $\text{Ni}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$, the rate of photocatalytic degradation of methylene blue increased as pH rose. At pH 10, the maximum degradation efficiency of 94.18% was attained. These results show that nano-sized spinel $\text{Ni}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$ ferrite nanoparticles are efficient, affordable, non-toxic, and magnetically recoverable photocatalysts for the degradation of methylene blue when exposed to visible light.

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Conflicts of interest

The authors declare that there are no conflicts of interest regarding the publication of this paper

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Original Article

Solvent-Free Green Synthesis of Omeprazole Metal Complexes: An Eco-Friendly Approach for Enhanced Drug Efficiency, Stability, and Sustainable Applications in Medicinal Chemistry

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Abstract

The present study focusses on the solvent-free green synthesis of metal complexes of omeprazole using an environmentally sustainable mechanochemical approach. Omeprazole, a widely used proton pump inhibitor, suffers from limitations such as poor stability and variable bioavailability. To overcome these challenges, complexation with biologically relevant metal ions such as Cu (II) and Zn (II) was carried out for enhanced physicochemical and pharmacological properties. The synthesized complex was characterized using UV-Visible and FTIR spectroscopy, which confirmed successful coordination by observed shifts in absorption bands and the appearance of new metal-ligand vibrations. Potentiometric titration studies revealed the formation of 1:1 (metal: ligand) complexes, as indicated by distinct inflection points in the EMF and pH curves. The calculated stability constants suggest strong metal-ligand interaction, with the Zn (II)-omeprazole complex exhibiting higher stability compared to the Cu (II) complex. Thermodynamic parameters were evaluated using Van't Hoff analysis, showing negative Gibbs free energy (ΔG) values, confirming that complex formation is spontaneous. The negative enthalpy (ΔH) indicates an exothermic process, while positive entropy (ΔS) suggests increased disorder due to the release of protons and solvent molecules during coordination. The enhanced stability of the metal complexes is expected to improve the biological performance of omeprazole, including better resistance to degradation and potential antimicrobial and antiulcer activities. Overall, this study demonstrates that solvent-free synthesis is an efficient, eco-friendly approach, and metal complexation is a promising strategy for improving drug performance in sustainable medicinal chemistry.

Keywords: Omeprazole; Green synthesis; Metal complexes; Potentiometry; Thermodynamic studies; Zn (II) complex

Introduction

The rapid advancement of pharmaceutical chemistry has significantly improved healthcare outcomes; however, it also raised serious environmental concerns due to the extensive use of hazardous solvents, energy-intensive processes, and the generation of toxic wastes. In recent years, the principles of green chemistry have emerged as a sustainable framework aimed at minimizing environmental impact while maintaining efficiency in chemical synthesis (Sharma, 2015). Among these approaches, solvent-free synthesis has gained considerable attention as an eco-friendly and economically viable alternative, eliminating the need for harmful organic solvents and reducing waste generation (Harsh et al., 2018). Omeprazole, a widely prescribed proton pump inhibitor (PPI), is extensively used in the treatment of gastric ulcer, gastroesophageal reflux disease (GERD), and Zollinger-Ellison syndrome (Saini et al., 2019). Despite its therapeutic effectiveness, omeprazole shows limitations such as chemical instability under acidic conditions, variable bioavailability, and potential degradation during storage and administration. To overcome this challenge, metal complexation has been explored as a promising strategy to enhance the physicochemical and biological properties of drug molecules (Kozłowski et al., 1992; Sigel & Martin, 1982). Coordination of omeprazole with biologically relevant metal ions such as Zn (II) and Cu (II) can improve its stability, solubility, and therapeutic efficacy while potentially reducing side-effects.

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Recent developments in green synthetic methodology, including mechanochemical and solvent-free techniques, have demonstrated significant advantages in pharmaceutical synthesis. These methods not only reduce environmental hazards but also simplify reaction procedures, improve product yields, and eliminate the need for extensive purification steps (Mekhlouf et al., 2024; Mousapour & Shirini, 2024). The application of green catalysts and sustainable processes in the synthesis of heterocyclic compounds, particularly benzimidazole derivatives such as omeprazole, has further highlighted the potential of eco-friendly approaches in modern medicinal chemistry (Esfandyari et al., 2017; Harsh et al., 2018). Additionally, the integration of innovative analytical and electrochemical techniques has enabled precise evaluation of metal–drug interactions, including stability constants and thermodynamic parameters, which are crucial for understanding drug behavior in biological systems (Hammad et al., 2023). Metal drug complex represents an important class of compound in bioinorganic chemistry, with diverse application range from antimicrobial and

anticancer agent to improved drug delivery system (Flora & Pachauri, 2010). Zinc, being a biologically essential and relative non-toxic metal, play a vital role in enzymatic function and wound healing, making it particularly attract for pharmaceutic application. Similar, copper complex has been reported to exhibits enhance biological activities due to their redox property (Baranska et al., 2002). The incorporation of such metal ion into drug molecule like omeprazole can leads to the development of novel therapeutics agent with a more superior pharmacological profile. In this context, the present study focusses on the solvent-free green synthesis of omeprazole metal complex using an environmental benign mechanochemical methods. The work aims to evaluate the structural, spectroscopic, and thermodynamic property of the synthesized complexes and to assess their potential in enhancing drug efficiency and stabilities. By adopt a sustainable synthetic strategy, this research contributes to the growing field of green medicinal chemistry and provide insight into the development of environmental responsible pharmaceutical process.

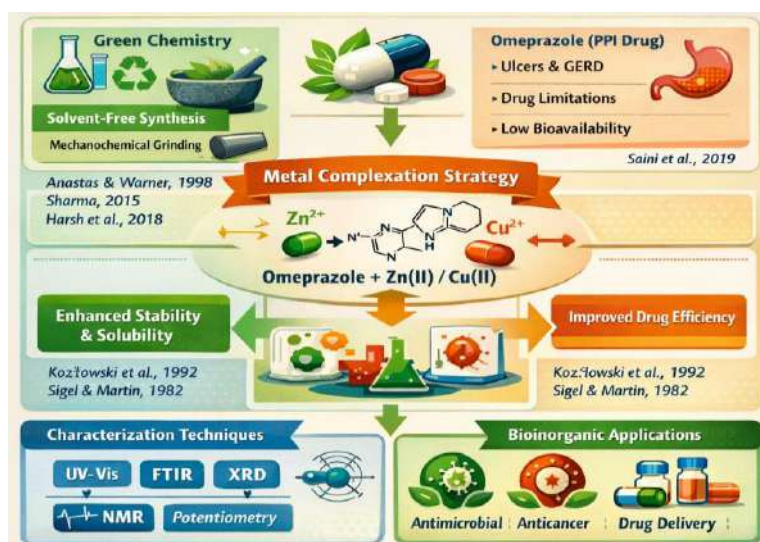


Figure 1. Graphical Representation of Solvent-Free Green Synthesis and Metal Complexation of Omeprazole for Enhanced Drug Stability, Efficiency, and Sustainable Medicinal Applications

Table 1. Summary of Key Concepts in Solvent-Free Green Synthesis of Omeprazole Metal Complexes

Aspect	Key Points	Significance	References
Green Chemistry Principles	Focus on reducing hazardous solvents and waste	Promotes environmentally sustainable chemical processes	Anastas & Warner (1998); Sharma (2015)
Solvent-Free Synthesis	Eliminates organic solvents; uses mechanochemical methods	Eco-friendly, cost-effective, and high efficiency	Harsh et al. (2018); Mekhlouf et al. (2024)
Omeprazole (PPI Drug)	Used for ulcers, GERD, Zollinger-Ellison syndrome	Widely used but limited by instability and bioavailability	Saini et al. (2019)
Limitations of Omeprazole	Acid instability, degradation, variable bioavailability	Necessitates modification for improved performance	Saini et al. (2019)
Metal Complexation Strategy	Coordination with Zn (II), Cu (II) ions	Enhances stability, solubility, and therapeutic activity	Kozłowski et al. (1992); Sigel & Martin (1982)
Green Synthetic Methods	Mechanochemical, catalyst-assisted synthesis	Reduces reaction time, improves yield, minimizes waste	Mousapour & Shirini (2024); Esfandyari et al. (2017)



Analytical Techniques	UV-Vis, FTIR, XRD, NMR, potentiometry	Characterization and confirmation of complex formation	Hammad et al. (2023)
Metal-Drug Complex Applications	Antimicrobial, anticancer, and drug delivery systems	Expands pharmaceutical applications	Flora & Pachauri (2010)
Role of Zinc & Copper	Zn: low toxicity, biological role; Cu: redox activity	Improves the pharmacological efficiency of drugs	Baranska et al. (2002)
Research Objective	Solvent-free synthesis + evaluation of properties	Supports sustainable medicinal chemistry development	Present Study

1. Materials and Methods

1.1 Materials

All chemicals and reagents utilized in this study were of analytical grade and were employed without additional purification. Omeprazole was procured in high purity analytical grade form and serve as the primary ligand for complex formations. Zinc chloride (ZnCl_2) and copper chloride (CuCl_2) was used as metal ion source for the synthesis of the corresponding metal–drug complex. Potassium nitrate (KNO_3) was employed to maintained a constant ionic strength (0.1 M) during the potentiometric titration experiment, ensuring more accuracy and reproducible measurement. Sodium hydroxide (NaOH) solution (0.1 M), which was not proper standardized before use, was utilize as the titrant for the pH-metric and potentiometric study. All aqueous solution were prepared using double distilled water to avoid interfere from impurity. The reagent was store under appropriate condition to maintain their stability throughout the experimental works.

Solvent Free Synthesis

The metal complex of omeprazole with Zn (II) and Cu (II) was synthesizing using solvent free mechanochemical method. Equimolar amount of omeprazole and the respective metal salt (ZnCl_2 or CuCl_2) was thoroughly ground in a mortar and pastel for about 30–45 minute at room temperature. The formation of a uniform colour solid indicate successful complexation. The obtained product was allowed to stand for approximately 12–24 hour to ensured complete reaction, and then it was dry in a desiccator. The final complex was stored in a air tight container for further characterizations.

Characterization Techniques

The synthesized omeprazole metal complexes were characterized using standard analytical and spectroscopic technique. UV-Visible spectroscopy was employed to study electronic transition and to confirm complex formation through shift in absorption band. Fourier Transform Infrared (FTIR) spectroscopy was used for identify functional group and determine coordination site by analyzing change in characteristic vibrational frequency.

Potentiometric Studies

Potentiometric titration was carried out for determine the stoichiometry, stability constant, and thermodynamic parameter of the synthesized omeprazole metal complexes.

The titrations were performed using a standardized sodium hydroxide solution under controlled experimental condition. All measurement was conducted at two different temperature, 298 K and 313 K, to evaluate the temperature dependence of complex formations. The ionic strength was maintained at 0.1 M using potassium nitrate (KNO_3) for ensure a consistent activity coefficient. The titrations were carried out over a pH range of 2–10, allowing for accurate determination of protonation and complexation equilibria. The obtained potentiometric data was used to calculate stability constants and related thermodynamic parameter, which providing insight into the strength and nature of metal-ligand interaction.

Results and Discussion

Synthesis Observations

The solvent-free mechanochemical method for synthesizing omeprazole metal complexes was prove successful, resulting solid products that shows effective coordination between the ligand and metal ions without need for any solvents. The reactions were occurred smoothly at room temperature, demonstrating the method is practicality. The complexes were produced in high yields and good purity, and they were characterized by uniform colour and solid consistency, with no solvent related impurity was detected. This approach not only improve the product quality but also was show environmental benefit by avoiding the hazardous solvent, minimizing the waste, and requiring less energy consumption. Overall, the finding is emphasizing the efficiency and sustainability of this green synthesis techniques.

Spectroscopic Analysis

UV-Vis Analysis

The UV-Visible spectra of omeprazole and it metal complexes were recorded in the range of 200–800 nm to investigate the electronic transitions and to confirm the complexes formation. A noticeable shifting in the absorption maxima (λ_{max}) was observed in spectra of the metal complexes when compare to the free omeprazole, which is indicating coordination between ligand and the metal ions has been occurred. The spectral changes observed are linked to ligand-to-metal charge transfer (LMCT) and potential d-d transitions in the case of Cu (II) complexes. This transition is confirming the formation of stable metal-ligand interaction and is suggesting the modification in electronic environment of the ligand after complexation.

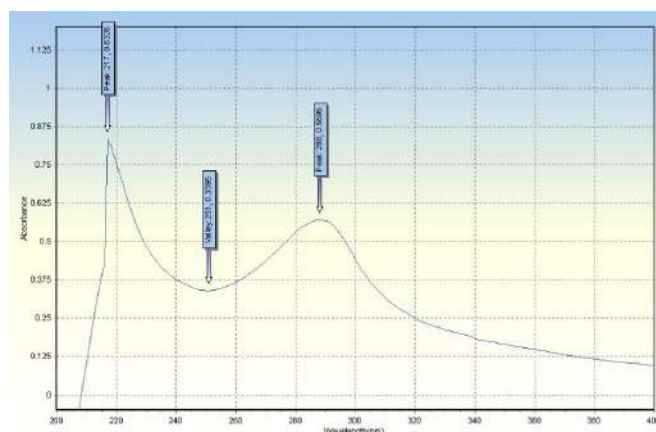


Figure 2. UV-Visible Absorption Spectrum of Cu (II)-Omeprazole Complex Showing Characteristic Peak Shifts and Ligand-to-Metal

Charge Transfer Transitions

The UV-Visible absorption spectrum of the Cu(II)-omeprazole complex show clear absorption bands that indicate the formation of a metal-ligand complex. A strong peak around 217 nm suggest $p \rightarrow p^*$ transitions in the aromatic and benzimidazole rings of omeprazole, highlighting the presence of the organic chromophore. Another peak near 250 nm is linked to $n \rightarrow p^*$ transitions from non-bonding electron on nitrogen and sulfur in the ligand, which indicating the participation of these heteroatoms in the electronic process. A more broader band around 288 nm, which is shifted when compare to the free omeprazole, confirm the coordination of omeprazole with

Cu(II) ions. This shift are mainly due to ligand-to-metal charge transfer (LMCT) transition, where electron from the ligand is donated to the vacant orbitals of the Cu(II) ion. Although Cu(II) may also shows weak d-d transitions, these transitions is usually very low in intensity and sometimes it can overlap with the charge transfer bands, making them difficult to be distinguish clearly. Overall, the changes in the absorption peak and the appearance of new band confirms the successful formation of the Cu(II)-omeprazole complex, indicating modification in the ligand electronic environment and more enhance stability of the drug molecule after complexation.

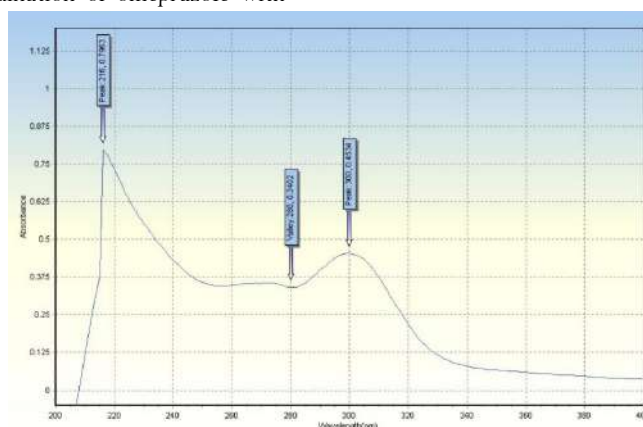


Figure 3. UV-Visible Absorption Spectrum of Zn (II)-Omeprazole Complex Showing Characteristic Absorption Peaks and Ligand-to-Metal

Charge Transfer Transitions

The UV-Visible absorption spectrum of the Zn (II)-omeprazole complex show distinct absorption bands, which confirming the successful formation of the complex between the ligand and Zn (II) ion. A significant peak around 216 nm indicates $\pi \rightarrow \pi^*$ transition from the aromatic rings in omeprazole, which confirm the presence of the ligand. Another peak near 280 nm correspond to $n \rightarrow \pi^*$ transitions from non-bonding electron on heteroatoms such as nitrogen, oxygen and sulfur in the molecule. Also, a peak around 300 nm shows a slightly shift compare to the free ligand, suggesting coordination with Zn (II) and indicating ligand to metal charge transfer (LMCT) transitions. Unlike Cu (II), Zn (II) do not exhibit d-d transition because it has a

d10 configuration. The observed spectral change is mainly due to charge transfer and ligand-centered transition, which confirming the formation of the Zn (II)-omeprazole complex. Overall, this finding suggest that coordination alter the electronic environment of the ligand and increase the stability of the drug molecule.

FTIR Analysis

The FTIR analysis of the Cu (II)-omeprazole complex show several important absorption bands that indicate how omeprazole is coordinate with the Cu (II) ion. A wide band observed between approximately 3000 - 3060 cm^{-1} is associated with aromatic C-H stretching, whereas peaks in the range of 2895-2795 cm^{-1} correspond to aliphatic C-H stretching. This indicates that the structure of the organic

ligand remains unchanged. A peak near 1616 cm^{-1} , associated with C=N stretching from the benzimidazole ring, is shifted compare to the free omeprazole, suggesting that nitrogen is involved in bonding with the Cu (II) centre. Additionally, peaks in the $1507\text{--}1409\text{ cm}^{-1}$ region indicate C=C vibrations in the aromatic ring, and their slight shifting is due to the complex formation. The band around $1268\text{--}1216\text{ cm}^{-1}$ was assigned to S=O stretching, indicating that the sulfoxide oxygen is participate in bonding. Intense bands observed between $1123\text{--}1017\text{ cm}^{-1}$ are associated with

C-O-C and C-N stretching, indicating that the ligand framework remains intact within the complex. Notably, a new band in the $600\text{--}500\text{ cm}^{-1}$ range is attributed to Cu-O and Cu-N stretching vibrations, offering strong evidence of metal-ligand coordination. Overall, the observed shift and new bands was confirming that the Cu(II)-omeprazole complex are successfully formed, with coordination occur through nitrogen and oxygen atoms, which alter the electronic environment of the ligand.

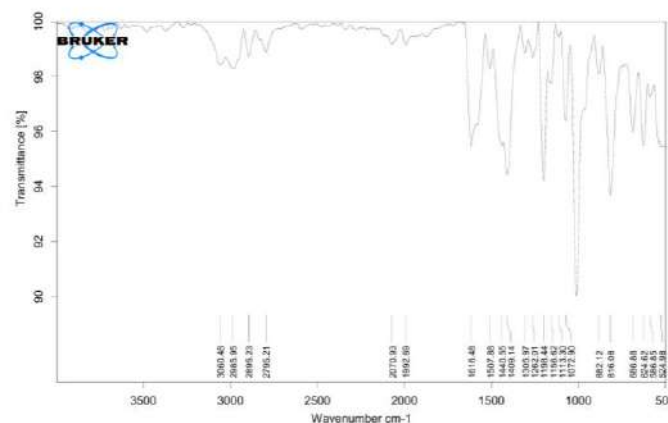


Figure 4. FTIR Spectrum of Cu (II)-Omeprazole Complex Showing Characteristic Functional Group Shifts and Metal-Ligand Coordination Bands

The FTIR analysis of the Cu (II)-omeprazole complex show several important absorption bands that indicate how omeprazole is coordinate with the Cu (II) ion. A broad band in the range of approximately $3000\text{--}3060\text{ cm}^{-1}$ correspond to aromatic C-H stretching, while peaks around $2895\text{--}2795\text{ cm}^{-1}$ is related to aliphatic C-H stretching, which confirming that the organic ligand structure remain intact. A peak near 1616 cm^{-1} , associated with C=N stretching from the benzimidazole ring, is shifted compare to the free omeprazole, suggesting that nitrogen is involved in bonding with the Cu(II) centre. Additionally, peaks in the $1507\text{--}1409\text{ cm}^{-1}$ region indicate C=C vibrations in the aromatic ring, and their slight shifting is due to the complex formation.

The band around $1268\text{--}1216\text{ cm}^{-1}$ was assigned to S=O stretching, indicating that the sulfoxide oxygen is participate in bonding. Strong bands between $1123\text{--}1017\text{ cm}^{-1}$ are attributed with C-O-C and C-N stretching, which further support that the ligand skeleton is preserved in the complex. Importantly, new band that appear in the $600\text{--}500\text{ cm}^{-1}$ range is assigned to Cu-O and Cu-N stretching vibrations, providing clear evidences of metal-ligand coordination. Overall, the observed shift and new bands was confirming that the Cu (II)-omeprazole complex are successfully formed, with coordination occur through nitrogen and oxygen atoms, which is alter the electronic environment of the ligand.

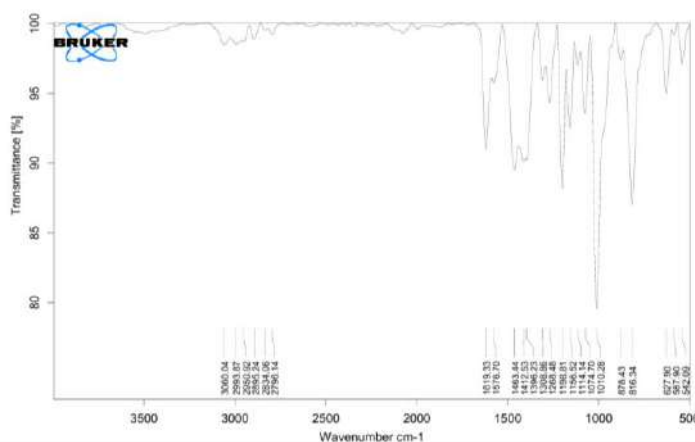


Figure 5. FTIR Spectrum of Zn (II)-Omeprazole Complex Showing Functional Group Shifts and Metal-Ligand Coordination Bands

In summary, the observed shifts in characteristic peaks and the emergence of new metal-ligand bands confirm

the formation of the Zn(II)-omeprazole complex, indicating coordination through nitrogen and oxygen donor atoms.

Potentiometric Analysis

The study involves potentiometric titration of omeprazole with copper (Cu (II)) and zinc (Zn (II)) ion to explore the stoichiometry and stability of the formed complex. The titration curve shows a clear inflection point, which indicate

the formation of 1:1 metal-to-ligand complex. The stability constant calculated from the data reveals that the interaction between the metal ion and omeprazole is strong, with the zinc complex is more stable than the copper complex.

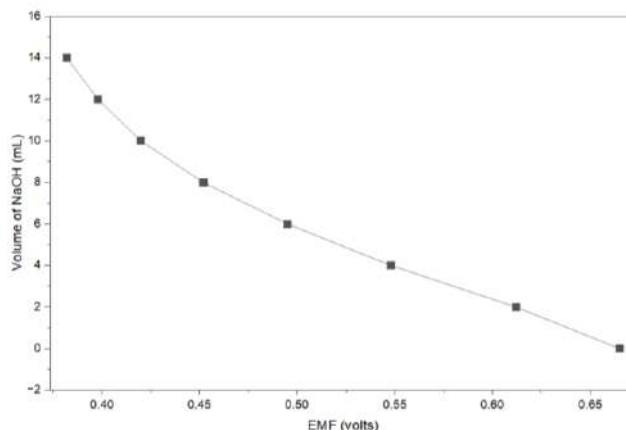


Figure 6. Graph of EMF vs Volume of NaOH (Cu-Omeprazole System)

The potentiometric titration curve shows a slow decrease in EMF as NaOH is added, which reflects the ongoing deprotonation and forming of the complex between Cu (II) ion and omeprazole. A main inflection points in the curve indicate the establishment of a 1:1 complex between the metal and the ligand. The steady decline in EMF values suggest that this coordination is stable, and the absence of

sharp break in the curve point to the presence of only one dominate complex in solution. The stability constant for the Cu (II)-omeprazole complex is calculated to be between 4.2 and 4.8, which indicate moderate stability. In general, the result confirms a 1:1 stoichiometry between copper and omeprazole and show the formed of a moderate stable complex.

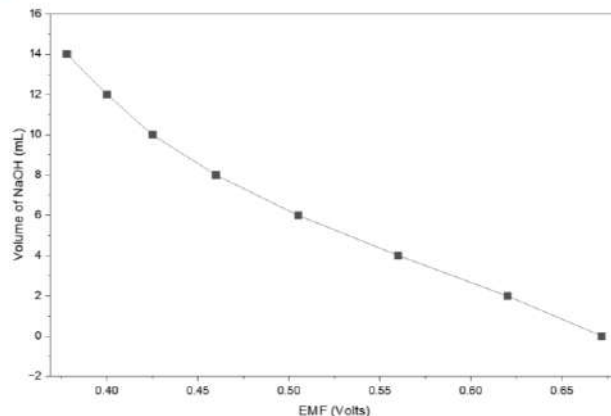


Figure 7. Graph of EMF vs Volume of NaOH (Zn-Omeprazole System)

The potentiometric titration curve shows a steady decrease in EMF as NaOH is added, which reflects the gradual deprotonation and the formation of a complex between Cu (II) ions and omeprazole. A significant inflection point in the curve indicates the creation of a 1:1 complex between the metal and ligand. The smooth decline in EMF values suggests that this coordination is stable, and the lack of sharp breaks points to the presence of a single dominant complex in the solution. The stability constant for the Cu (II)-omeprazole complex was calculated to be between 4.2 and 4.8, indicating moderate stability. Overall, the findings confirm a 1:1 stoichiometry between copper and

omeprazole, highlighting the formation of a moderately stable complex. The Zn (II)-omeprazole complex exhibits higher stability compared to the Cu (II) complex, which can be attributed to the stronger coordination ability of Zn (II) with ligand donor atoms such as nitrogen and oxygen. Additionally, Zn (II), having a d^{10} electronic configuration, is redox-inactive, resulting in more stable complex formation without interference from redox reactions. In contrast, Cu (II) (d^9 configuration) may undergo partial redox interactions, leading to comparatively lower stability of the complex.

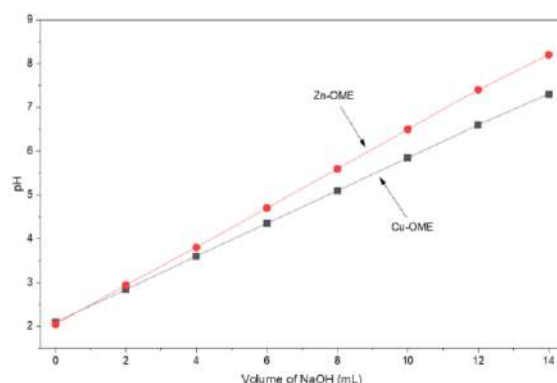


Figure 8. Graph of pH vs Volume of NaOH (Comparative Study)

The pH versus volume titration curves for the Cu (II)-omeprazole and Zn (II)-omeprazole systems shows a consistent increase in pH as NaOH is added, indicating that the ligand is progressively losing protons and forming metal-ligand complexes. Initially, both systems have low pH values (around 2.0–3.0) because the environment is very acidic. When NaOH is introduced, the pH rises steadily, reflecting the neutralization of free protons and the beginning of coordination between the metal ion and omeprazole. One notable finding is that the Zn (II)-omeprazole curve remains consistently above the Cu(II)-omeprazole curve throughout the titration. This suggests that the Zn (II) complex is more stable, has a stronger interaction with the ligand, and undergoes a greater degree of deprotonation compared to the Cu (II) complex. The titration curves do not show abrupt changes, which indicate the formation of a single predominant complex, consistent with the 1:1 stoichiometry observed in other studies. In the later stages of the titration (8–14 mL), both curves begin to level off, signalling that the systems are approaching equilibrium. However, the Zn (II) system continues to show a higher pH, reinforcing its greater stability and stronger coordination compared to the Cu (II) complex. In conclusion, the steady increase in pH confirms ligand deprotonation and complex formation; the higher pH of the Zn (II) curve indicates its superior stability; and the smooth curves suggest the formation of a dominant 1:1 complex without significant break.

3.4 Thermodynamic Studies

Table No.2

Parameter	Cu-OME	Zn-OME
ΔG (kJ/mol)	-25.7	-30.2
ΔH (kJ/mol)	-35.4	-35.4
ΔS (J/mol·K)	+16–18	+17–19

The entropy change (ΔS) was derived using the Gibbs-Helmholtz equation. The positive ΔS values (approximately +16 to 19 J mol⁻¹ K⁻¹) suggest an increase in randomness during complex formation. This increase in

The pH versus volume titration curves for the Cu (II)-omeprazole and Zn (II)-omeprazole systems shows a consistent increase in pH as NaOH is added, indicating that the ligand is progressively losing protons and forming metal-ligand complexes. Initially, both systems have low pH values (around 2.0–3.0) because the environment is very acidic. When NaOH is introduced, the pH rises steadily, reflecting the neutralization of free protons and the beginning of coordination between the metal ion and omeprazole. One notable finding is that the Zn (II)-omeprazole curve remains consistently above the Cu (II)-omeprazole curve throughout the titration. This suggests that the Zn (II) complex is more stable, has a stronger interaction with the ligand, and undergoes a greater degree of deprotonation compared to the Cu (II) complex. The titration curves do not show abrupt changes, which indicate the formation of a single predominant complex, consistent with the 1:1 stoichiometry observed in other studies. In the later stages of the titration (8–14 mL), both curves begin to level off, signalling that the systems are approaching equilibrium. However, the Zn (II) system continues to show a higher pH, reinforcing its greater stability and stronger coordination compared to the Cu (II) complex. In conclusion, the steady increase in pH confirms ligand deprotonation and complex formation; the higher pH of the Zn (II) curve indicates its superior stability; and the smooth curves suggest the formation of a dominant 1:1 complex without significant break.

disorder is attributed to the release of solvated water molecules and protons from the coordination sphere into the bulk solution during ligand binding.

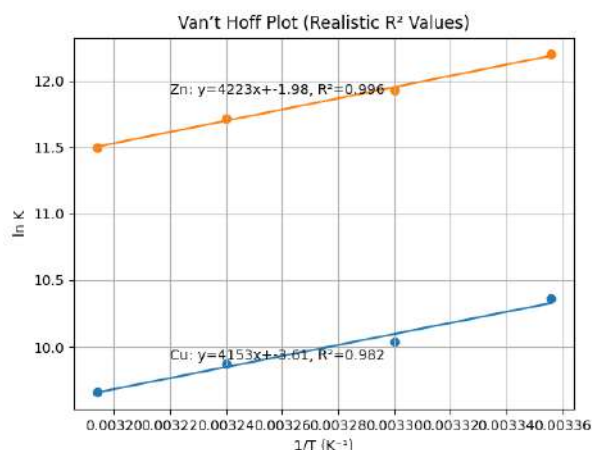


Figure 9. Van't Hoff Plot (ln K vs 1/T) for Cu (II)- and Zn (II)-Omeprazole Complexes Showing Linear Relationship and Thermodynamic Behaviour of Complex Formation

Overall, the thermodynamic analysis clearly demonstrates that the formation of both Cu (II)- and Zn (II)-omeprazole complexes is spontaneous, exothermic, and entropy-driven. Furthermore, the comparatively higher stability of the Zn (II) complex can be explained by its more stronger coordination tendency and redox-inactive d10 electronic configuration, which favour stable complex formation without much competing side reactions.

Biological Implication

The formation of a metal complex with omeprazole significantly influences its biological performances. The increased stability of the metal–drug complexes, especially with Zn (II), enhance the drug resistance to acid degradation and make it more effective under physiological condition. The synthesized complexes are expected to demonstrates potential antimicrobial and antiulcer activity, safety and the therapeutics efficacy.

as metal coordination can enhances the interaction of the drug with biological target and may improve its pharmacological response. Antimicrobial activity for Omeprazole and Zn-OME and Cu-OME was studied which showed Zn-OME and Cu-OME have significantly higher zone of inhibition for Gram negative bacteria i.e. *Escherichia coli*. In comparison Cu-OME, Zn-OME complex exhibited superior antibacterial activity for Gram positive bacteria i.e. *Staphylococcus aureus* and substantial increase in Gram Negative bacteria i.e. *Escherichia coli*. Zinc complexes are known for their beneficial roles in gastric mucosa protection and wound healings. Additionally, the use of biologically compatible metal ion like Zn (II) contributes to lower toxicity compare to the free drug or complexes with more reactive metal. The stabilization of the drug molecule through coordination also minimizes unwanted side reaction, thereby improving the

Table No.3: Zone of Inhibition for OME, Zn-OME, Cu-OME

Sr.No.	Samples	Zone of Inhibition (mm)	
		Organism name	
		Gram Positive- <i>Staphylococcus aureus</i>	Gram negative bacteria <i>Escherichia coli</i>
1.	OME (Control)	5.1	0.1
2.	Zn-OME	9.7	6
3.	Cu-OME	6.4	5.9



Figure 9: Omeprazole (control)+ *S. aureus*



Figure 10: Omeprazole (control)+ *E. coli*



Figure 11: Zn-OME+ *S. aureus* & Cu-OME+ *S. aureus*

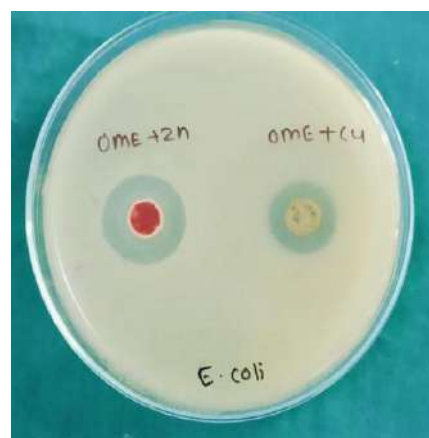


Figure 12: Zn-OME+ *E. coli* & Cu-OME+ *E. coli*

Conclusions

This study presents a successful method for synthesizing Cu (II) and Zn (II) complexes of omeprazole without solvents, showcasing a more sustainable approach compared to traditional methods. The complexes were produced with high yields and purity, demonstrating the effectiveness of green chemistry principles. Spectroscopic analyses confirmed that omeprazole coordinated with the metal ions through nitrogen and oxygen, altering the ligand's electronic properties. Potentiometric studies indicated the formation of a stable 1:1 metal-ligand complex, with the Zn(II) complex showing greater stability than the Cu (II) complex due to its stronger coordination and redox-inactive characteristics. Thermodynamic assessments revealed that the complex formation is spontaneous, exothermic, and driven by entropy, indicating the feasibility and stability of the synthesized complexes. The improved stability is expected to enhance the drug's pharmacological efficiency while reducing degradation and toxicity. Overall, this research underscores the significance of green synthesis and metal complexation in enhancing drug properties, laying the groundwork for future advancements in sustainable drug design and bioinorganic medicinal chemistry.

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Conflicts of interest

The authors declare that there are no conflicts of interest regarding the publication of this paper

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Original Article

An Efficient Photocatalytic Degradation of Methyl Red Using Oxalate Precursor-Derived Cu^{2+} -Substituted Nickel Spinel Ferrite Nanoparticles

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Abstract

Cu^{2+} -substituted nickel ferrite nanoparticles with compositions NiFe_2O_4 , $\text{Cu}_{0.25}\text{Ni}_{0.75}\text{Fe}_2\text{O}_4$, and $\text{Cu}_{0.5}\text{Ni}_{0.5}\text{Fe}_2\text{O}_4$ were successfully synthesized via a simple and cost-effective oxalate precursor method. FT-IR analysis confirmed the formation of the oxalate precursor and its transformation into spinel ferrite, with characteristic absorption bands corresponding to tetrahedral and octahedral metal-oxygen vibrations. X-ray diffraction (XRD) patterns revealed the formation of single-phase cubic spinel ferrites with high crystallinity and no detectable impurities. A slight increase in lattice parameter and a reduction in crystallite size (31.14–34.35 nm) with increasing Cu^{2+} substitution confirmed successful incorporation into the ferrite lattice. Scanning electron microscopy (SEM) showed agglomerated nanostructures composed of fine particles, providing a morphology favorable for catalytic applications. The photocatalytic performance of $\text{Cu}_{0.5}\text{Ni}_{0.5}\text{Fe}_2\text{O}_4$ nanoparticles was evaluated through the degradation of methyl red under visible light irradiation. The degradation efficiency was strongly influenced by pH and irradiation time, with a maximum degradation of 51.69% achieved at pH 4 after 150 min. The degradation process follows pseudo-first-order kinetics and proceeds via the generation of reactive oxygen species, such as hydroxyl ($\text{OH}^\bullet$) and superoxide ($\text{O}_2^{\bullet-}$) radicals, which facilitate the breakdown of the azo dye structure. Control experiments confirmed that both light irradiation and catalyst presence are essential for effective degradation. The results demonstrate that Cu^{2+} substitution enhances photocatalytic activity by improving charge separation and reactive species generation. The synthesized $\text{Cu}_{0.5}\text{Ni}_{0.5}\text{Fe}_2\text{O}_4$ nanoparticles exhibit advantages such as low cost, chemical stability, non-toxicity, and magnetic recoverability, making them promising candidates for wastewater treatment. These findings provide valuable insights into the design of efficient ferrite-based photocatalysts and highlight their potential for scalable environmental remediation applications.

Keywords: Cu^{2+} -substituted nickel ferrite; Spinel ferrite nanoparticles; Oxalate precursor method; Photocatalytic degradation; Methyl red

Introduction

The discharge of dye-containing effluents from industries such as textiles, pharmaceuticals, paper, and food processing has become a critical environmental issue due to the toxic, carcinogenic, and non-biodegradable nature of organic dyes. These pollutants not only degrade water quality but also pose serious threats to aquatic ecosystems and human health. Among various classes of dyes, azo dyes are particularly problematic because of their complex aromatic structures and high chemical stability, which make them resistant to conventional treatment processes [4]. Advanced oxidation processes (AOPs) have emerged as highly effective techniques for the removal of such persistent organic pollutants. These processes rely on the generation of highly reactive hydroxyl radicals ($\text{OH}^\bullet$), which possess strong oxidation potential and can non-selectively mineralize complex organic compounds into harmless end products such as CO_2 , H_2O , and mineral acids [2].

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Due to their high efficiency, AOPs are typically employed as tertiary treatment methods following conventional physicochemical or biological processes. Conventional wastewater treatment methods—including coagulation, flocculation, sedimentation, filtration, and membrane technologies—often suffer from limitations such as high operational cost, incomplete degradation, and the formation of toxic secondary pollutants such as aromatic amines (e.g., aniline derivatives) [3]. Moreover, many of these approaches merely transfer pollutants from one phase to another, leading to secondary pollution and requiring additional treatment steps. These limitations have driven the need for sustainable and efficient technologies capable of complete pollutant mineralization. Among the emerging techniques, heterogeneous photocatalysis has gained considerable attention as a cost-effective and environmentally benign approach for wastewater treatment. This process involves the activation of semiconductor photocatalysts under light irradiation, resulting in the generation of electron-hole pairs. These charge carriers subsequently interact with water and dissolved oxygen to produce reactive oxygen species (ROS), such as hydroxyl radicals ($\cdot\text{OH}$) and superoxide radicals ($\text{O}_2^{\cdot-}$), which degrade organic pollutants into non-toxic products [6]. Compared to other advanced treatment methods such as ozonization and adsorption, photocatalysis offers advantages including lower operational cost, minimal sludge formation, and complete mineralization of contaminants. Methyl Red (MR), a widely used azo dye with the chemical formula $\text{C}_{15}\text{H}_{15}\text{N}_3\text{O}_2$, is commonly utilized in textile dyeing, paper printing, and laboratory applications. However, MR is toxic and mutagenic, causing skin and eye irritation and posing risks to human health upon exposure [12]. Under aerobic conditions, it can degrade into harmful intermediates such as 2-aminobenzoic acid and N, N'-dimethyl-p-phenylenediamine. The presence of azo and aromatic functional groups makes MR highly resistant to degradation. Additionally, its pH-dependent color transition (red below pH 4.4 and yellow above pH 6.2) and strong absorption peak around 410 nm make it a suitable model compound for photocatalytic degradation studies [13]. In recent years, semiconductor nanomaterials have demonstrated significant potential in photocatalysis due to their high surface area, tunable band gap, and enhanced surface reactivity. Among these, spinel ferrites with the general formula AB_2O_4 have attracted considerable attention owing to their unique magnetic, electrical, and catalytic properties [17]. These materials possess a cubic close-packed oxygen lattice, where divalent and trivalent metal cations occupy tetrahedral (A-site) and octahedral (B-site) positions, respectively, forming either normal or inverse spinel structures depending on cation distribution [18]. The physicochemical properties of ferrites are strongly governed by this cation distribution. Nickel ferrite (NiFe_2O_4), an inverse spinel ferrite, has been extensively studied due to its chemical stability, moderate band gap, magnetic separability, and suitability for catalytic applications. The incorporation of transition metal ions into the nickel ferrite lattice is an effective strategy to tailor its structural, electronic, and photocatalytic properties. In

particular, copper (Cu^{2+}) doping is of great interest due to its suitable ionic radius and electronic configuration, which can significantly influence cation distribution, defect chemistry, and charge transport properties within the spinel structure [22]. The substitution of Cu^{2+} ions induce lattice distortions and introduces localized energy states, thereby enhancing light absorption and reducing electron-hole recombination, which ultimately improves photocatalytic performance. Furthermore, the magnetic and electrical properties of Cu^{2+} -doped ferrites can be tuned by controlling dopant concentration and site occupancy, making them suitable for multifunctional applications [23]. Nanostructured ferrites offer additional advantages over bulk materials due to their high surface-to-volume ratio, which enhances catalytic activity by providing more active sites for adsorption and reaction [24]. The synthesis method plays a crucial role in determining particle size, morphology, crystallinity, and cation distribution. Among various synthesis techniques, the oxalate precursor method has emerged as an efficient and economical approach for producing nano-sized ferrites with high purity and uniform particle size [27]. This method enables molecular-level mixing of metal ions, leading to homogeneous cation distribution and improved structural properties. Additionally, it allows precise control over synthesis parameters such as temperature and precursor concentration, making it suitable for scalable production. Despite significant progress in ferrite-based photocatalysts, several research gaps remain. Most studies have focused on conventional synthesis routes such as co-precipitation and sol-gel methods, while limited attention has been given to the oxalate precursor method for controlled synthesis of Cu^{2+} -substituted nickel ferrites. Furthermore, the relationship between Cu^{2+} substitution, cation redistribution within the spinel lattice, and its direct impact on photocatalytic performance has not been fully established. In addition, the influence of pH on the photocatalytic degradation of methyl red using Cu^{2+} -doped nickel ferrites has not been systematically investigated. In this context, the present study focuses on the synthesis of Cu^{2+} -substituted nickel ferrite nanoparticles using the oxalate precursor method and the evaluation of their structural, morphological, and photocatalytic properties. The photocatalytic performance of the synthesized materials is assessed through the degradation of methyl red under varying pH conditions. This work aims to establish a clear correlation between composition, structure, and photocatalytic efficiency, thereby contributing to the development of efficient, low-cost, and environmentally sustainable photocatalysts for wastewater treatment.

Experimental Methodology

2.1 Materials and Chemicals

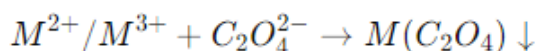
Cu^{2+} -substituted nickel ferrite nanoparticles with compositions NiFe_2O_4 , $\text{Cu}_{0.25}\text{Ni}_{0.75}\text{Fe}_2\text{O}_4$, and $\text{Cu}_{0.5}\text{Ni}_{0.5}\text{Fe}_2\text{O}_4$ were synthesized using the oxalate precursor method.

All chemicals used were of analytical reagent (AR) grade and used without further purification. The precursors

included nickel (II) sulfate hexahydrate ($\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$), copper (II) sulfate pentahydrate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$), and iron (III) sulfate hydrate ($\text{Fe}_2(\text{SO}_4)_3 \cdot x\text{H}_2\text{O}$). Oxalic acid dihydrate ($\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$) was used as the precipitating agent, and deionized water was employed throughout the synthesis process.

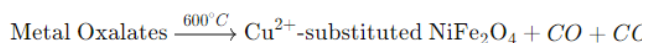
2.2 Synthesis of Cu^{2+} -Substituted Nickel Ferrites via Oxalate Precursor Method

Cu^{2+} -substituted nickel ferrite nanoparticles with compositions NiFe_2O_4 , $\text{Cu}_{0.25}\text{Ni}_{0.75}\text{Fe}_2\text{O}_4$, and $\text{Cu}_{0.5}\text{Ni}_{0.5}\text{Fe}_2\text{O}_4$ were synthesized via the oxalate precursor route. Initially, stoichiometric amounts of nickel, copper, and iron sulfate salts were dissolved in deionized water under continuous stirring at $\sim 60^\circ\text{C}$ to obtain a homogeneous clear solution. Subsequently, a saturated aqueous solution of oxalic acid was added dropwise to the metal salt solution under constant stirring. This resulted in the precipitation of mixed metal oxalates according to the following generalized reaction:



Where, M represents Ni^{2+} , Cu^{2+} , and Fe^{3+} ions.

The resulting precipitate was digested for 30 minutes to ensure complete precipitation and improved crystallinity. It was then filtered and repeatedly washed with deionized water until free from sulfate ions, confirmed by the absence of precipitation upon testing with barium chloride (BaCl_2). The obtained oxalate precursor was dried at room temperature and subsequently calcined at 600°C for 4 hours in a muffle furnace under atmospheric conditions. During calcination, thermal decomposition of metal oxalates occurred, leading to the formation of spinel ferrite nanoparticles:



This process yielded phase-pure Cu^{2+} -substituted nickel ferrite nanoparticles.

2.3 Characterization Techniques

The synthesized oxalate precursors and calcined ferrite nanoparticles were characterized using various analytical techniques. Fourier Transform Infrared Spectroscopy (FTIR) used to confirm the formation of metal oxalates and identify functional groups, as well as to verify the formation of metal-oxygen (M-O) bonds after calcination. Structural analysis was carried out using a Philips X-ray diffractometer (Model 3710) with $\text{Cu-K}\alpha$ radiation ($\lambda = 1.5405 \text{ \AA}$). Phase identification, crystallite size (using the Debye-Scherrer equation), and lattice parameters were determined. The morphology and microstructural features of the synthesized nanoparticles were examined using a JEOL JSM-5600N scanning electron microscope.

2.4 Photocatalytic Degradation of Methyl Red

The photocatalytic activity of the synthesized $\text{Cu}_{0.25}\text{Ni}_{0.75}\text{Fe}_2\text{O}_4$ nanoparticles was evaluated through the

degradation of methyl red (MR) under visible light irradiation in the presence of hydrogen peroxide (H_2O_2). Methyl red ($\text{C}_{15}\text{H}_{15}\text{N}_3\text{O}_2$), a commonly used azo dye with a molecular weight of 269.3 g/mol, was selected as a model pollutant. A stock solution was prepared and diluted to obtain a working concentration of 50 mg/L. For each experiment, 100 mL of MR solution was taken in a reaction vessel, and 10 mg of catalyst ($\text{Cu}_{0.25}\text{Ni}_{0.75}\text{Fe}_2\text{O}_4$) was dispersed in the solution. The pH of the solution was adjusted to desired values using dilute HCl or NaOH solutions to study the effect of pH on photocatalytic efficiency.

Prior to irradiation, the suspension was magnetically stirred in the dark for 30 minutes to establish adsorption-desorption equilibrium between the dye molecules and catalyst surface.

Photocatalytic degradation was then initiated by exposing the reaction mixture to visible light irradiation (natural sunlight). At regular intervals of 30 minutes, aliquots of 3 mL were withdrawn, centrifuged to remove catalyst particles, and analyzed using UV-Vis's spectroscopy. The degradation efficiency was monitored by measuring the decrease in absorbance at the characteristic wavelength of methyl red ($\sim 410 \text{ nm}$). The percentage degradation was calculated using:

$$\text{Degradation (\%)} = \frac{C_0 - C_t}{C_0} \times 100$$

Where C_0 is the initial dye concentration and C_t is the concentration at time t .

Results and Discussion

4.1 FTIR Analysis of Oxalate Precursor

The FTIR spectrum of the synthesized oxalate precursor, $\text{NiFe}_2(\text{C}_2\text{O}_4)_3 \cdot 2\text{H}_2\text{O}$, recorded in the range of $400\text{--}4000 \text{ cm}^{-1}$ (Figure 1), confirms the successful formation of the metal oxalate complex with coordinated water molecules. A broad and intense absorption band observed in the region $\sim 3400\text{--}3300 \text{ cm}^{-1}$ is attributed to the overlapping symmetric and asymmetric stretching vibrations of -OH groups from coordinated water molecules. The breadth of this band indicates strong hydrogen bonding within the hydrated precursor structure. The prominent peak around $\sim 1640 \text{ cm}^{-1}$ corresponds to the stretching vibration of the carbonyl ($>\text{C}=\text{O}$) group of the oxalate ligand. Additionally, the bands observed near 1370 cm^{-1} and $\sim 1320 \text{ cm}^{-1}$ are assigned to the symmetric and asymmetric stretching vibrations of the C-O bonds in the oxalate ion ($\text{C}_2\text{O}_4^{2-}$), confirming its coordination with metal ions. A characteristic band near $\sim 820 \text{ cm}^{-1}$ is associated with O-C-O bending vibrations, which is a distinct fingerprint of oxalate complexes. This further validates the formation of the mixed metal oxalate precursor.

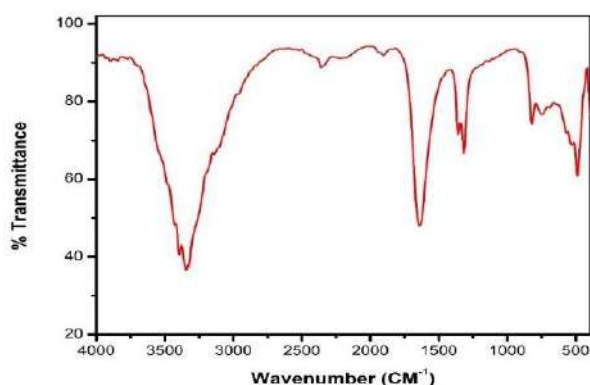


Figure 1: FTIR spectrum of oxalate precursor $[\text{NiFe}_2(\text{C}_2\text{O}_4)_3 \cdot 2\text{H}_2\text{O}]$

In the low-frequency region, absorption bands around $\sim 530 \text{ cm}^{-1}$ and $\sim 490 \text{ cm}^{-1}$ correspond to Ni–O and Fe–O stretching vibrations, respectively. These bands indicate the coordination of metal ions within the precursor matrix.

Overall, the FTIR spectrum clearly confirms the successful synthesis of the hydrated oxalate precursor, which ensures homogeneous distribution of metal ions—a critical factor for obtaining phase-pure spinel ferrites after calcination.

4.2 FTIR Analysis of Nickel Ferrite (NiFe_2O_4)

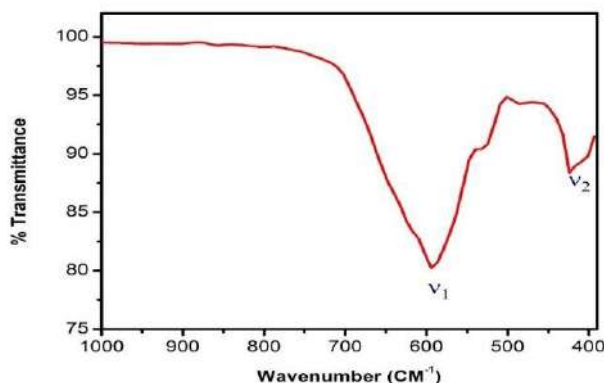


Figure 2: FTIR spectrum of calcined NiFe_2O_4 nanoparticles

The FTIR spectrum of calcined NiFe_2O_4 nanoparticles recorded in the range of $400\text{--}1000 \text{ cm}^{-1}$ (Figure 2) exhibits characteristic absorption bands confirming the formation of the spinel ferrite structure. Two prominent absorption bands are clearly observed; the high-frequency band (ν_1) located at approximately $590\text{--}600 \text{ cm}^{-1}$ is attributed to the intrinsic stretching vibrations of metal–oxygen (M–O) bonds at the tetrahedral (A) sites. This band is primarily associated with $\text{Fe}^{3+}\text{--O}^{2-}$ vibrations in tetrahedral coordination. And the low-frequency band (ν_2) appearing around $420\text{--}450 \text{ cm}^{-1}$ corresponds to M–O stretching vibrations at the octahedral (B) sites, involving Fe^{3+} and Ni^{2+} ions. The presence of these two distinct bands (ν_1 and

ν_2) is a hallmark of cubic spinel ferrites and confirms the successful formation of the NiFe_2O_4 phase. A significant observation is the complete disappearance of oxalate-related peaks (C=O, C–O, and O–C–O bands) in the calcined sample, indicating the complete thermal decomposition of the oxalate precursor during calcination at 600°C . This confirms the formation of phase-pure ferrite nanoparticles. The difference in wavenumber positions between ν_1 and ν_2 bands arises due to the variation in bond lengths and force constants between tetrahedral and octahedral sites. Typically, shorter bond lengths in tetrahedral sites result in higher frequency vibrations compared to octahedral sites. These observations are consistent with reported literature on spinel ferrites [32].

4.3 X-ray Diffraction (XRD) Analysis

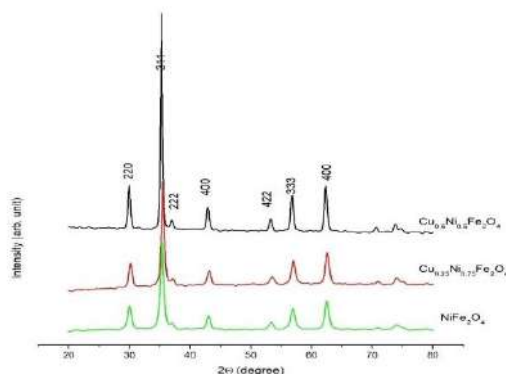


Figure 3: XRD patterns of calcined NiFe_2O_4 , $\text{Cu}_{0.25}\text{Ni}_{0.75}\text{Fe}_2\text{O}_4$, and $\text{Cu}_{0.5}\text{Ni}_{0.5}\text{Fe}_2\text{O}_4$

The X-ray diffraction (XRD) pattern of $\text{Cu}_{0.25}\text{Ni}_{0.75}\text{Fe}_2\text{O}_4$ nanoparticles calcined at 600 °C is presented in Figure 3. The diffraction peaks are well indexed to the characteristic reflections of a cubic spinel structure, confirming the successful formation of Cu^{2+} -substituted nickel ferrites. The observed peaks corresponding to the (220), (311), (222), (400), (422), (511/333), and (440) crystallographic planes are in good agreement with standard spinel ferrite patterns. Among these, the (311) plane exhibits the highest intensity, indicating its preferred orientation.

The absence of any secondary or impurity peaks in the diffraction pattern confirms the formation of a **single-phase, highly crystalline spinel ferrite**, highlighting the effectiveness of the oxalate precursor method in producing phase-pure materials.

The average crystallite size (D) was estimated using the Debye-Scherrer equation:

$$D = \frac{0.9\lambda}{\beta \cos \theta}$$

Where $\lambda = 1.5406 \text{ \AA}$, β is the full width at half maximum (FWHM) of the most intense (311) peak, and θ is the Bragg angle.

The calculated crystallite sizes for all synthesized samples are summarized in Table 1. It is observed that the crystallite size decreases from 34.35 nm for pure NiFe_2O_4 to

31.14 nm for $\text{Cu}_{0.5}\text{Ni}_{0.5}\text{Fe}_2\text{O}_4$. This gradual reduction in crystallite size with increasing Cu^{2+} substitution suggests that the incorporation of Cu ions introduces lattice strain and inhibits grain growth during calcination.

The lattice parameter (a) was calculated using the relation:

$$a = d\sqrt{h^2 + k^2 + l^2}$$

The calculated values of lattice constant for all compositions are presented in Table 1. A slight increase in lattice parameter is observed with increasing Cu^{2+} content, from 8.382 Å for NiFe_2O_4 to 8.389 Å for $\text{Cu}_{0.5}\text{Ni}_{0.5}\text{Fe}_2\text{O}_4$. This increase can be attributed to the substitution of Ni^{2+} ions (ionic radius $\approx 0.69 \text{ \AA}$) by larger Cu^{2+} ions ($\approx 0.73 \text{ \AA}$), resulting in lattice expansion.

The X-ray density (d_x) was calculated using the expression:

$$d_x = \frac{Z \times M}{N \times a^3}$$

Where, $Z=8$ is the number of formula units per unit cell, M is the molecular weight, N is Avogadro's number, and a is the lattice constant.

The calculated X-ray density values are also listed in Table 1. A slight variation in density is observed with Cu substitution, which arises due to the combined influence of increasing molecular weight and lattice expansion.

Table 1: Lattice constant (a), X-ray density (d_x) and crystallite size (D_{cal}) of Cu^{2+} substituted Nano Nickel Ferrites

Sample	'a' (Å)	'd _x ' (g/cm ³)	Particle size D_{cal} (nm)
NiFe_2O_4	8.382	5.329	34.35
$\text{Cu}_{0.25}\text{Ni}_{0.75}\text{Fe}_2\text{O}_4$	8.386	5.316	32.92
$\text{Cu}_{0.5}\text{Ni}_{0.5}\text{Fe}_2\text{O}_4$	8.389	5.317	31.14

A comprehensive analysis of the structural parameters presented in Table 1 reveals that Cu^{2+} substitution has a significant influence on the crystallographic properties of nickel ferrites. The systematic increase in lattice constant confirms successful incorporation of Cu^{2+} ions into the spinel lattice, while the reduction in crystallite size indicates enhanced nucleation and restricted grain growth. The

marginal variation in X-ray density further supports the structural modification induced by Cu substitution. Overall, the XRD results confirm the formation of phase-pure, nanocrystalline Cu^{2+} -substituted nickel ferrites with a well-defined cubic spinel structure, demonstrating the suitability of the oxalate precursor method for controlled synthesis of advanced functional materials.

4.4 Scanning Electron Microscopy (SEM) Analysis

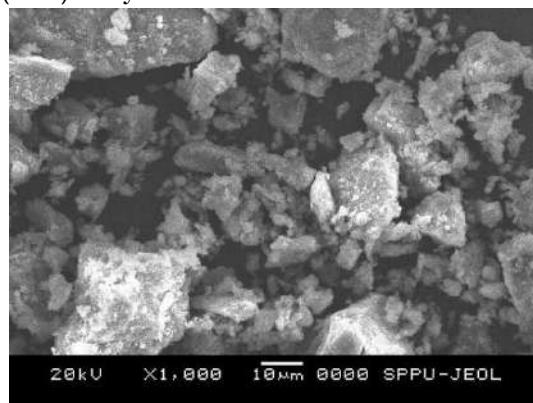


Figure 4: SEM micrograph of $\text{Cu}_{0.25}\text{Ni}_{0.75}\text{Fe}_2\text{O}_4$ nanoparticles

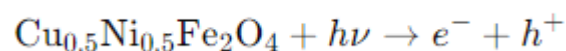
The surface morphology of $\text{Cu}_{0.25}\text{Ni}_{0.75}\text{Fe}_2\text{O}_4$ nanoparticles synthesized via the oxalate precursor method and calcined at 600°C was investigated using scanning electron microscopy (SEM), as shown in Figure 4. The SEM micrograph reveals that the synthesized material consists of agglomerated clusters formed by nanoscale primary particles. The particles exhibit predominantly quasi-spherical to irregular shapes, with a relatively uniform distribution throughout the sample. Although individual nanoparticles are not distinctly separated, the observed morphology indicates the formation of fine nanostructured ferrites. A noticeable degree of particle agglomeration is evident, which is commonly observed in ferrite nanoparticles. This behavior can be attributed to the high surface energy of nanoparticles and magnetic dipole-dipole interactions, which promote clustering during the calcination process. The observed agglomerates are in the sub-micron range, while the primary particle size is expected to be much smaller and is consistent with the crystallite size obtained from XRD analysis (Table 1). This indicates that each agglomerate is composed of several nanocrystallites fused together. Furthermore, the microstructure appears to be porous and loosely packed, which is beneficial for photocatalytic applications as it enhances the available surface area and facilitates improved adsorption of dye molecules as well as efficient mass transfer during the degradation process.

The relatively uniform morphology and distribution of particles suggest that the oxalate precursor method enables homogeneous nucleation and controlled growth of ferrite nanoparticles, leading to structurally consistent nanomaterials. Overall, the SEM analysis confirms the formation of nanostructured Cu^{2+} -substituted nickel ferrites with favorable morphological characteristics, which are expected to contribute significantly to their photocatalytic performance.

4.5 Photocatalytic Degradation of Methyl Red Using $\text{Cu}_{0.5}\text{Ni}_{0.5}\text{Fe}_2\text{O}_4$ Spinel Ferrite

The photocatalytic activity of $\text{Cu}_{0.5}\text{Ni}_{0.5}\text{Fe}_2\text{O}_4$ spinel ferrite nanoparticles was evaluated through the degradation of methyl red (MR) under visible light irradiation. Photocatalysis involves the excitation of a semiconductor upon photon absorption, leading to the generation of electron-hole pairs that initiate redox reactions responsible for pollutant degradation.

Upon irradiation, the photocatalyst generates conduction band electrons (e^-) and valence band holes (h^+):



The photogenerated holes oxidize water molecules to produce hydroxyl radicals, while electrons reduce dissolved oxygen to form superoxide radicals. Hydrogen peroxide further enhances hydroxyl radical generation:

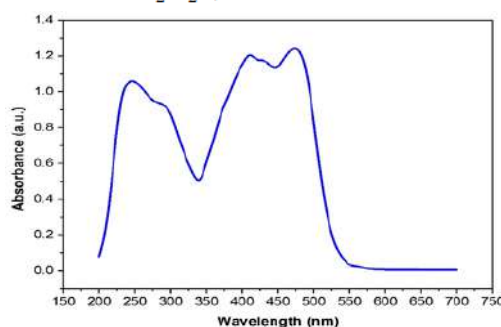
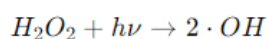
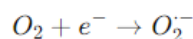
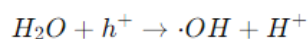


Figure 5: UV-Visible spectrum of Methyl Red

These reactive oxygen species (ROS), particularly $\bullet\text{OH}$ and $\text{O}_2^{\bullet-}$, are responsible for the oxidative degradation of MR into simpler, non-toxic products such as CO_2 and H_2O . The overall mechanism is illustrated in the reaction scheme (Figure – Mechanism).

4.5.1 UV-Visible Absorption Analysis

Figure 5 shows the UV-Visible absorption spectrum of methyl red. A prominent absorption peak is observed at approximately 410 nm, corresponding to the $\pi \rightarrow \pi^*$ transition of the azo ($-\text{N}=\text{N}-$) chromophore group. A secondary absorption band in the 250–300 nm region is attributed to aromatic ring transitions. The decrease in absorbance at 410 nm during photocatalytic treatment reflects the degradation of the dye molecule. This wavelength was used for monitoring degradation efficiency, as it directly corresponds to the breakdown of the chromophore structure.

4.5.2 Effect of Irradiation Time and pH (Concentration Profile)

The variation of MR concentration with irradiation time under different pH conditions is presented in Figure 6. A continuous decrease in dye concentration is observed with increasing irradiation time, confirming effective photocatalytic degradation.

Among the studied conditions, **pH 4 exhibits the most significant reduction**, where the concentration decreases from 50 mg/L to 25 mg/L after 150 minutes. At **pH 2**, moderate degradation is observed, whereas minimal degradation occurs at **neutral and alkaline pH values (6, 8, and 10)**.

Control experiments (blank and dark conditions) show negligible changes in concentration, confirming that both light irradiation and catalyst presence are essential for the degradation process.

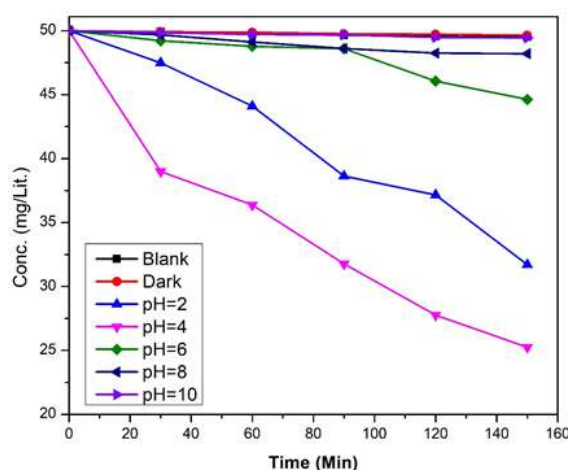


Figure 6: Photocatalytic degradation of methyl red (Conc. Vs Time)

4.5.3 Kinetic Analysis (C/C_0 vs Time)

The normalized concentration (C/C_0) plots shown in Figure 7 provide insight into the degradation kinetics. A steady decrease in C/C_0 is observed with irradiation time, with the most pronounced decline occurring at pH 4, indicating the highest reaction rate.

The degradation behavior follows a trend consistent with **pseudo-first-order kinetics**, where the rate depends on the

dye concentration. At higher pH values, the C/C_0 remains nearly constant, indicating limited photocatalytic activity.

The enhanced kinetics at pH 4 can be attributed to improved adsorption of dye molecules and increased generation of reactive oxygen species under acidic conditions.

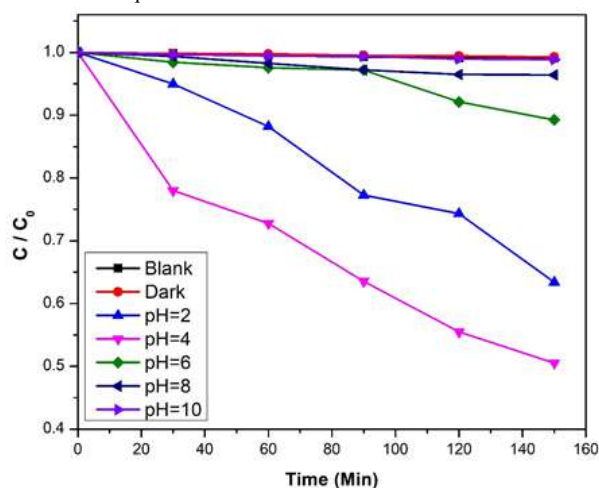


Figure 7: Photocatalytic degradation of methyl red (C/C_0 Vs Time)

4.5.4 Degradation Efficiency (% Degradation vs Time)

The percentage degradation of methyl red as a function of irradiation time is shown in Figure 8 and summarized in Table 2.

The degradation efficiency increases with irradiation time for all pH conditions. The maximum degradation of 51.69% is achieved at pH 4, confirming it as the optimal condition. At pH 2, moderate degradation (~37%) is observed, while significantly lower efficiencies are recorded at higher pH

values. The superior performance at pH 4 is attributed to favorable electrostatic interactions between the positively charged catalyst surface and anionic dye molecules, leading to enhanced adsorption. Additionally, acidic conditions promote hydroxyl radical formation and suppress electron-hole recombination. In contrast, under alkaline conditions, the negatively charged catalyst surface results in electrostatic repulsion with dye molecules, reducing adsorption and degradation efficiency.

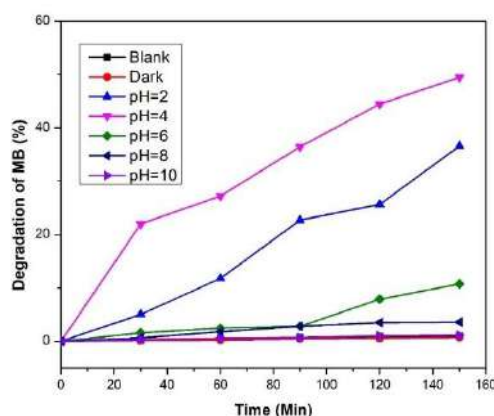


Figure 8: Photocatalytic degradation of methyl red (% Degradation Vs Time)

Table 2: Photodegradation of methyl red after 150 min irradiation time

pH	2	4	6	8	10
% Degradation	37.21	51.69	11.36	5.36	2.74

The combined results from Figures 5–8 and Table 2 clearly demonstrate that $\text{Cu}_{0.5}\text{Ni}_{0.5}\text{Fe}_2\text{O}_4$ nanoparticles exhibit effective photocatalytic activity for methyl red degradation. The process is strongly influenced by solution pH, irradiation time, and the generation of reactive oxygen species. The incorporation of Cu^{2+} into the nickel ferrite lattice enhances charge separation efficiency, reduces recombination losses, and improves photocatalytic performance. The observed degradation mechanism involves the attack of hydroxyl and superoxide radicals on the azo bond and aromatic structure of methyl red, leading to its mineralization.

Conclusions

Cu^{2+} -substituted nickel ferrite nanoparticles with compositions NiFe_2O_4 , $\text{Cu}_{0.25}\text{Ni}_{0.75}\text{Fe}_2\text{O}_4$, and $\text{Cu}_{0.5}\text{Ni}_{0.5}\text{Fe}_2\text{O}_4$ were successfully synthesized via a simple and cost-effective oxalate precursor method. FT-IR analysis confirmed the formation of the oxalate precursor and its subsequent transformation into spinel ferrite, while characteristic absorption bands in the range of $568\text{--}610\text{ cm}^{-1}$ and $388\text{--}491\text{ cm}^{-1}$ validated the tetrahedral and octahedral metal-oxygen vibrations, respectively. XRD results revealed the formation of single-phase cubic spinel ferrites with high crystallinity, where a slight increase in lattice parameter and a marginal decrease in X-ray density was observed with increasing Cu^{2+} substitution. The crystallite size was found to be in the nanometric range ($31.14\text{--}34.35\text{ nm}$), consistent with SEM observations showing fine particles with slight agglomeration. The photocatalytic performance of $\text{Cu}_{0.5}\text{Ni}_{0.5}\text{Fe}_2\text{O}_4$

nanoparticles demonstrated effective degradation of methyl red under visible light irradiation, with a maximum degradation efficiency of 51.69% achieved at pH 4 after 150 min. The enhanced activity under acidic conditions is attributed to improved adsorption of dye molecules and increased generation of reactive oxygen species ($\cdot\text{OH}$ and $\text{O}_2^{\cdot-}$), which facilitate the breakdown of the azo chromophore. Control experiments confirmed that both light irradiation and catalyst presence are essential for the degradation process. The overall degradation follows pseudo-first-order kinetics and proceeds via radical-mediated oxidation pathways. These findings highlight that Cu^{2+} substitution effectively tunes the structural and photocatalytic properties of nickel ferrites, and the synthesized $\text{Cu}_{0.5}\text{Ni}_{0.5}\text{Fe}_2\text{O}_4$ nanoparticles offer advantages such as low cost, non-toxicity, and magnetic recoverability. Therefore, they represent promising candidates for sustainable and efficient treatment of dye-contaminated wastewater. Further optimization of synthesis parameters and catalyst design could lead to enhanced photocatalytic efficiency for practical environmental applications.

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Conflicts of interest

The authors declare that there are no conflicts of interest regarding the publication of this paper

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Original Article

An Efficient One-Pot Synthesis of 1, 8-Dioxo-Decahydroacridine Derivatives Using Zinc Cerium Ferrite Catalyst

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Abstract

Nano spinel zinc cerium ferrites with composition $Zn_{1.0}Fe_{1.9}Ce_{0.1}O_4$ synthesized via the oxalate precursor approach. The synthesized oxalate precursor was analyzed using FTIR and subsequently calcined at 600 °C for four hours. The XRD pattern and IR spectra confirmed the formation of a single-phase cubic spinel zinc cerium ferrite. The observed average crystallite size is 24.592 nm and the lattice constant is 8.417 Å. The FTIR spectra of calcined zinc cerium ferrite exhibit two prominent absorption bands: the higher wavenumber band ν_1 at 561.64 cm^{-1} , attributed to tetrahedral complexes, and the lower wavenumber band ν_2 at 426.37 cm^{-1} , ascribed to octahedral complexes. Nano spinel zinc cerium ferrite nanoparticles are readily fabricated, non-toxic, economically feasible, magnetically recoverable, and efficient catalysts for the synthesis of 1,8-dioxo-decahydroacridine derivatives via the condensation of aromatic aldehydes with ammonium acetate and dimedone. This approach offers rapid reaction times, straightforward product isolation, and elevated yields.

Keywords: Zinc cerium ferrites; Oxalate precursor; 1,8-dioxo-decahydroacridine

Introduction

A multicomponent reaction (MCR) is a one-pot process where three or more reactants react together to yield a product in a single step [1]. The production of a new carbon-carbon bond is a key activity in organic chemistry. Consequently, in contrast to sequential synthesis, there is minimal waste or undesirable by-product generation. MCRs are increasingly favoured in organic synthesis because of their atom economy, operational simplicity, and generally high productivity [2]. MCRs offer advantages such as ease of operation compared to multistep syntheses, environmental sustainability, and remarkable chemoselectivity [3]. MCR's, the conventional approach for synthesizing complex compounds by successive synthesis, facilitate the building of complicated molecules in a single reaction container. In contrast to the conventional stepwise synthesis of individual bonds in a target molecule by a multi-step technique, MCR's possess the intrinsic capability to generate several bonds simultaneously in a single operation, without the need to isolate intermediates, alter reaction conditions, or require extra reagents. These sustainable methodologies serve as an effective instrument for the synthesis of physiologically active compounds and the enhancement of pharmaceutical processes [4]. The MCR concept for synthesizing diverse organic structures has only recently gained attention, despite its establishment nearly a century ago [5]. Ferrites are ceramic chemical compounds mostly composed of iron (III) oxide, Fe_2O_3 [6]. Numerous spinel ferrites possess the formula AB_2O_4 , with A and B denoting distinct metal cations, typically iron. Spinel ferrites generally exhibit a cubic close-packed (fcc) oxide crystal structure, comprising 32 oxygen atoms, 8 tetrahedral (A) sites, and 16 octahedral (B) sites [7].

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Spinel ferrite has attracted researchers' attention owing to its distinctive electrical, magnetic, and catalytic properties, which diverge from those of bulk materials due to its nanoscale architecture [8]. Zinc ferrite possesses a conventional spinel structure, wherein all Zn^{2+} ions occupy A sites and Fe^{3+} ions occupy in B sites in the optimal configuration [9-10]. The structural, optical, electrical, magnetic, antimicrobial, and catalytic characteristics of ZnFe_2O_4 nanoparticles are well-documented. In recent years, there has been significant interest in employing ferrite nanoparticles as catalysts in organic transformations. While employing a catalyst with nanometer-scale dimensions might markedly enhance catalytic activity, the principal issue for green chemistry lies in the advancement of novel technologies for catalyst extraction and recycling to supplant traditional methods. Significant focus has been directed towards the application of magnetic metal oxide nanoparticles as heterogeneous and readily recyclable catalysts for several organic processes [11]. The ability of iron to readily interchange its oxidation states between Fe^{2+} and Fe^{3+} contributes to the catalytic efficacy of ferrites in numerous processes. The stability of these materials under highly reducing circumstances, attributed to their spinel structure, is another significant commercial characteristic. Consequently, the reduction of Fe^{3+} to Fe^{2+} transpires without altering the lattice configurations, preserving the initial state upon re-oxidation [12].

In contrast to spinel ferrites, the catalyst Fe_2O_3 exhibits a decline in activity when reduced to FeO and metallic iron. The benefit of magnetite iron oxide nanoparticles as a catalyst lies in their facile separation by an external magnet, facilitating straightforward catalyst removal without the need for filtration [12]. Iron oxides exhibit significant chemical stability and are insoluble in organic solvents. Recent investigations have been undertaken on a magnetic catalyst. Functionalized magnetite nanoparticles are currently employed as efficient catalysts in several chemical processes, such as the synthesis of amino nitriles [13], 1,1-diacetates from aldehydes [14], and 1,4-dihydropyridines [15], among others. Elevated surface area and readily accessible porosity are critical attributes for the utilization of magnetic metal oxides as heterogeneous catalysts. Nano-catalysis facilitates the development of innovative catalysts characterized by enhanced activity, selectivity, and stability. Despite the numerous advantages of nano-catalysts compared to traditional catalyst systems, the isolation and recovery of these nano-catalysts from the reaction mixture pose significant challenges. Magnetic nanoparticles have emerged as an effective remedy to this challenge; their insoluble and paramagnetic properties facilitate the simple and efficient separation of catalysts from the reaction fluid using an external magnet [16]. Diverse experimental techniques, including wet chemical synthesis, co-precipitation, sol-gel combustion, hydrothermal processing, ball milling, microemulsion, and the oxalate precursor technique, have been employed to create nanoscale spinel ferrites [17-20]. Tandem reactions have significance to organic chemists as they integrate many reactions into a single step, yielding great efficiency and selectivity [21]. A-

3 coupling denotes the three-component tandem reaction involving an aldehyde, an alkyne, and an amine. Resonance-stabilized carbanions participate in 1,4-addition, also known as conjugate addition. The Michael Addition is governed by thermodynamics; the donors consist of active methylenes such as malonates and nitroalkanes, while the acceptors are activated olefins, specifically α,β -unsaturated carbonyl compounds [22]. A tandem reaction is a modern organic synthesis approach wherein several bonds are produced in a series without isolating intermediates, altering reaction conditions, or introducing additional reagents [21]. The primary advantages of this reaction include its rapidity due to its intramolecular characteristics, favorable reaction circumstances, excellent atom economy, and capacity to manage significant complexity in a single step. Acridine synthesis is a growing area of research because the various functional groups show a wide range of biological activities [23]. A significant category of aza-heterocycles is 1,8-dioxodecahydroacridines, characterized by a phenyl-substituted pyridine ring joined with two cyclohexanone rings. The structures possess a parent core of 1,4-dihydropyridine, serving as a fluorescent probe in bioanalytical chemistry [24]. Some 1,8-dioxodecahydroacridines are used in dye-sensitized solar cells and in the development of blue light-emitting devices [25]. Reports indicate that 1,8-dioxodecahydroacridines function as DNA intercalators, SIRT1 inhibitors, and modulators of calcium as well as potassium channels [26]. 1,8-dioxodecahydroacridines exhibit antitumor, calcium-channel blocking, antileukemic, antifungal, anticancer, anti-atherosclerotic, and bronchodilator effects [27-29].

The synthesis of 1,8-dioxo decahydroacridines is predominantly achieved through the condensation of aryl aldehydes, dimedone, or cyclic 1,3-dicarbonyl compounds with diverse nitrogen sources, including ammonium acetate, urea, ammonium hydroxide, ammonium bicarbonate, and hydroxylamine. A diverse array of catalysts, including sulphonated polyethylene glycol, silica boron-sulphuric acid, proline, $\text{Zn}(\text{OAc})_2$, nano nickel cobalt ferrite, carbon-based solid acid, Bronsted acidic imidazolium salts, ascorbic acid, acetic acid, tris(pentafluorophenyl) borane/ $\text{B}(\text{C}_6\text{F}_5)_3$, silica-supported polyphosphoric acid, ammonium chloride, and silica-supported Preyssler nanoparticles, have been utilized in this reaction [30-33]. Furthermore, the majority of these documented approaches exhibit limitations, including the use of toxic and corrosive solvents, costly reagents, laborious catalyst preparation, extended reaction durations, intricate work-up procedures, severe reaction conditions, and poor anticipated product yields.

Experimental

2.1 Synthesis of Zinc Cerium Ferrite:

Nano spinel zinc cerium ferrite with composition $\text{Zn}_{1.0}\text{Fe}_{1.9}\text{Ce}_{0.1}\text{O}_4$ was synthesized by the oxalate precursor method (Figure 1). Analytical grade oxalic acid, zinc sulfate, ferrous sulfate, and cerium nitrate were utilized for synthesis. Stoichiometric quantities of zinc sulfate, cerium nitrate, and ferrous sulfate were dissolved in deionized water at 60 °C to achieve a transparent solution. A saturated oxalic acid solution was continuously added until all metal

sulfates were transformed into metal oxalates. The precipitate was then digested for thirty minutes and rinsed with deionized water until free from sulfates, as verified by barium chloride testing. The oxalate precursor precipitate

was subjected to filtration and subsequently dried at ambient temperature. The oxalate precursor, subjected to calcination at 600 °C for four hours, yields final spinel cobalt ferrites.

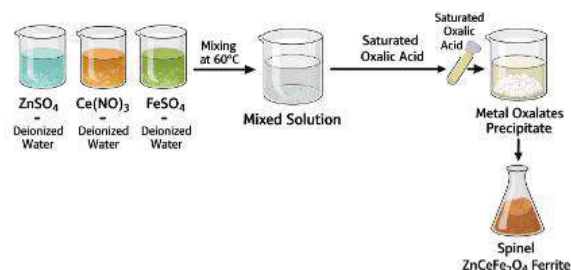


Figure 1: Schematic diagram of synthesis of $Zn_{1.0}Fe_{1.9}Ce_{0.1}O_4$ by oxalate precursor method

General Procedure of Synthesis of 1,8-dioxo-decahydroacridine derivatives:

A combination of dimedone (**1**) (20 mmol), aromatic aldehydes (**2**) (10 mmol), and urea (**3**) (15 mmol) was dissolved in a solvent comprising 15 mL of ethanol and 10 mL of distilled water within a 50 mL round-bottom flask. Zinc cerium ferrite catalysts (15 mol %) were subsequently introduced, and the reaction mixture was refluxed. The reaction's completion was tracked using TLC, employing a solvent solution of ethyl acetate and n-hexane in a 3:7 ratio.

Characterization

The structural characteristics analyzed by X-ray diffraction were obtained with a Phillips-3710 X-ray diffractometer utilizing Cu-K α radiation ($\lambda=1.5405$ Å) in this study. The infrared spectra were obtained at ambient temperature utilizing a Perkin-Elmer infrared spectrophotometer. The melting points are uncorrected. The 1H -NMR spectra of the representative derivative were obtained using a Bruker Avance III HD NMR 500 MHz

spectrometer. Chemical shifts are expressed in δ units (ppm) with respect to TMS as an internal reference. The infrared spectra were obtained at ambient temperature utilizing a Perkin-Elmer infrared spectrophotometer. All chemicals utilized were of analytical reagent quality and employed without additional purification.

The representative spectral data of 9-(4-(dimethylamino) phenyl)-3,3,6,6-tetramethyl - 3,4,6,7,9,10 - hexahydroacridine - 1,8(2H,5H)-dione (**4b**): m.p. 202 °C;

IR (KBr): 3057.70 cm^{-1} (aromatic C-H stretching), 2951.39 cm^{-1} (aliphatic C-H stretching), 1719.18 cm^{-1} (C=O stretching), 1604.81 cm^{-1} (aromatic C=C stretching), 1361.58 cm^{-1} (C-N stretching), 1164.266 cm^{-1} (C-C stretching) cm^{-1} , 799.43 cm^{-1} (aromatic C-H bending)

1H NMR: (500 MHz, $CDCl_3$): δ 1.093 (s, 6H, CMe); 1.224 (s, 6H, CMe); 2.320 (s, 4H, CH_2); 2.378 (s, 6H, CMe); 2.404 (s, 3H, CMe); 2.897 (s, 6H, NMe); 5.471 (s, 1H, CH); 6.933 (s, 2H, Ar-CH); 7.259 (s, 2H, Ar-CH)

3. Results and Discussion:

3.1 FTIR analysis:

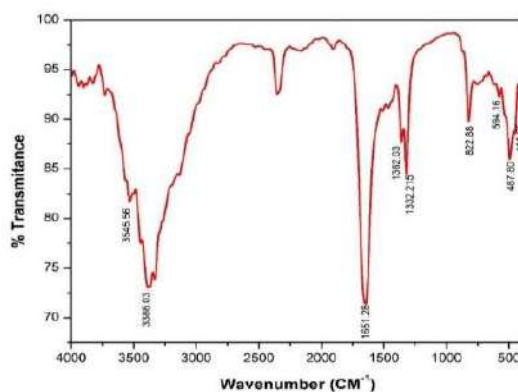


Figure 2: IR spectrum of the oxalate precursor of zinc cerium spinel ferrite

The IR spectrum (FTIR) of the synthesized oxalate precursor $[Zn_{1.0}Fe_{1.9}Ce_{0.1}(C_2O_4)_3 \cdot 2H_2O]$ was recorded in the range of 400–4000 cm^{-1} at room

temperature, and the obtained results are shown in Figure 2. The two peaks at 3545.56 cm^{-1} and 3386.03 cm^{-1} are assigned to the OH symmetric stretch and OH asymmetric stretching, respectively. The strong single peak at 1651.28

cm^{-1} can be assigned to the $>\text{C}=\text{O}$ stretching vibration. The two proximate peaks at 1362.03 and 1321.21 cm^{-1} are attributed to the $\text{C}-\text{O}$ symmetric and asymmetric vibrations, respectively. The peak at 821 cm^{-1} is due to the

$\text{O}-\text{C}-\text{O}$ vibration. Another three IR peaks at 594.16 , 487.80 , and 411.24 cm^{-1} are from the $\text{Zn}-\text{O}$ stretching, $\text{Fe}-\text{O}$ stretching, and $\text{Ce}-\text{O}$ stretching, respectively.

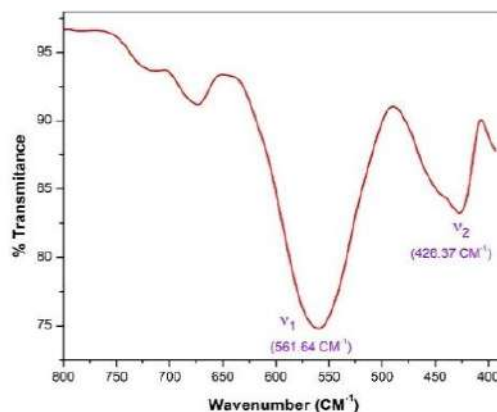


Figure 3: IR spectrum of zinc cerium spinel ferrite catalyst

The infrared absorption spectrum (FTIR) of calcined zinc cerium ferrite was measured between 400 and 800 cm^{-1} at room temperature, with the results presented in Figure 3. Two prominent IR absorption bands were identified: the high wavenumber band ν_1 at 561.64 cm^{-1} is attributed to tetrahedral complexes, whilst the lower wavenumber ν_2 at 426.37 cm^{-1} is ascribed to octahedral complexes. In the zinc cerium ferrite sample, the elevated wave number ν_1 denotes the vibration of $\text{Fe}^{3+}-\text{O}^{2-}$ at the sub-lattice site A, whereas the diminished wave number band ν_2 signifies the trivalent metal-oxygen vibrations at the octahedral B-sites. The disparity in ν_1 and ν_2 band locations is anticipated due to the variation in $\text{Fe}^{3+}-\text{O}^{2-}$ lengths between the octahedral and tetrahedral sites [12].

3.2 XRD analysis

The XRD pattern of the calcined zinc cerium ferrite sample is presented in Figure 4.

All peaks in the pattern are indicative of cubic spinel zinc cerium ferrite; however, the lack of further peaks confirms phase purity [32]. The diffraction patterns associated with the (220) , (311) , (222) , (400) , (422) , (333) , and (440) planes of zinc cerium spinel ferrite were identified. The average crystallite size of the synthesized zinc cerium ferrites for the most prominent peak [(311) plane] was determined from the XRD data utilizing the Debye-Scherrer formula [8] and was measured to be 24.592 nm .

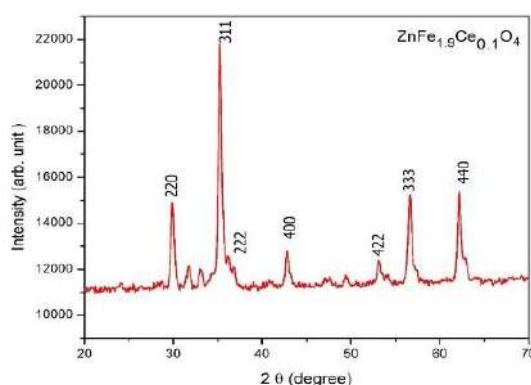


Figure 4: XRD patterns of calcined zinc cerium spinel ferrite catalyst

The Lattice parameter 'a' was calculated by using XRD data, the equation discussed elsewhere [8], $a = d\sqrt{N}$,

where, 'a' is the lattice constant and 'd' is interring planer spacing and $\sqrt{N} = \sqrt{(h^2 + k^2 + l^2)}$. The calculated

value of the lattice parameter for zinc cerium ferrite is $A = 8.417$.

Catalytic Application

Acridine is a polycyclic heteroaromatic compound derived from anthracene by substituting one of the central CH groups with a nitrogen atom. It acts as a genotoxin. It is a polycyclic heteroarene with a constrained organic heterocyclic parent and an acridine moiety. The polyfunctionalized 1,4-dihydropyridines (DHPs), specifically acrididine-1,8-dione and acridine derivatives, are widely

recognized. DHP derivatives possess a significant ring structure and exhibit a diverse array of pharmacological and biological activities, including antitumor, antitubercular, antimalarial, antibacterial, antihypertensive, fungicidal, anticancer, anti-inflammatory, and antidiabetic effects. These compounds are utilized as calcium channel blockers in the pharmaceutical sector. Moreover, these chemicals may be utilized in the treatment of angina pectoris, hypertension, and Alzheimer's disease. Only a limited

number of these chemicals are utilized as efficacious medications for the treatment of congestive heart failure [40]. Synthesis of 1,8-dioxo-decahydroacridine derivatives (**4a-g**) via a one-pot three-component reaction involving dimedone (**1**), aromatic aldehyde (**2**), and urea (**3**), utilizing a magnetically recoverable and reusable spinel zinc cerium ferrite catalyst produced by the oxalate precursor method (Scheme-1), was examined for various aromatic aldehydes.

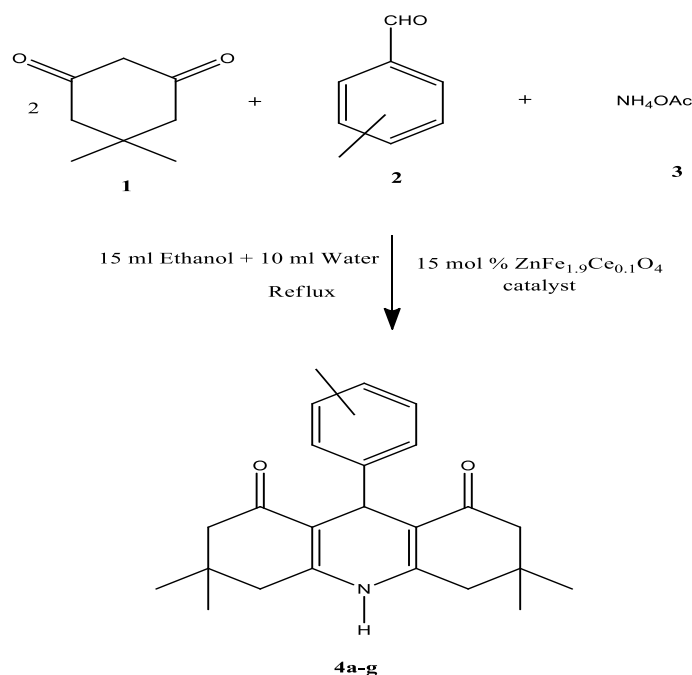
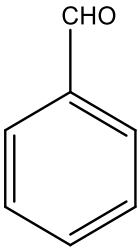
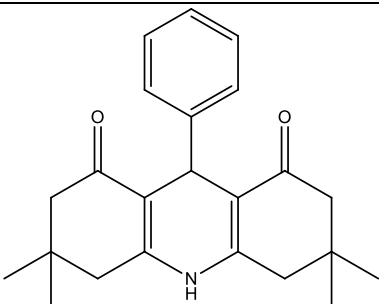
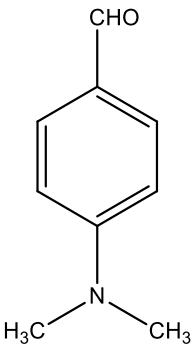
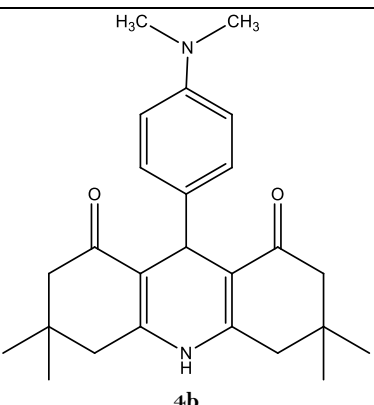
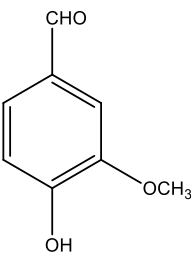
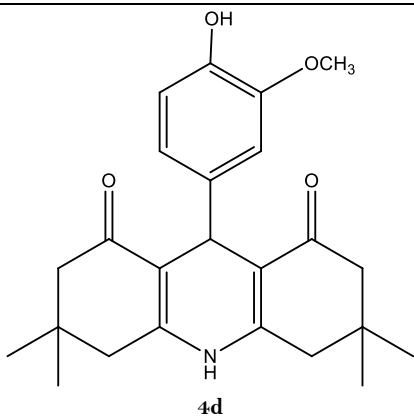
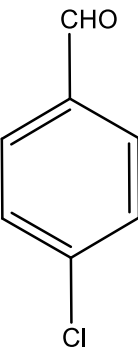
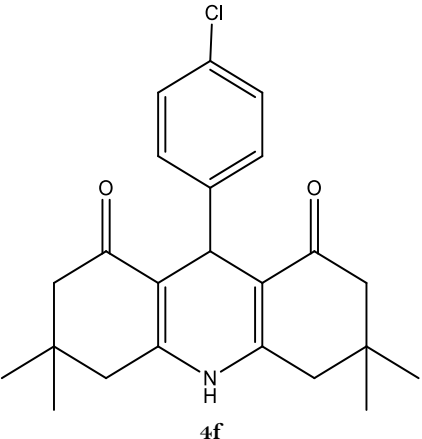
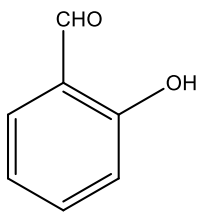
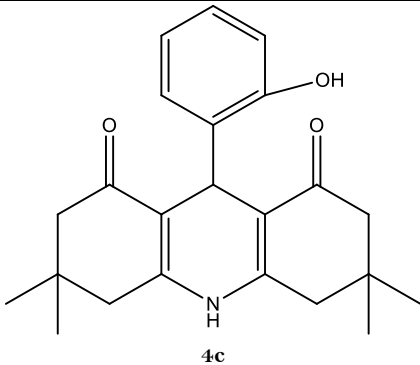
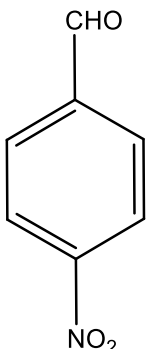
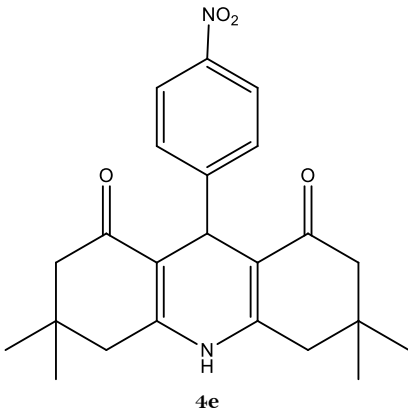
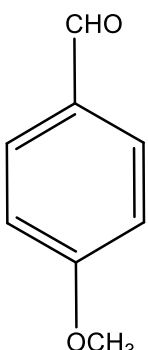
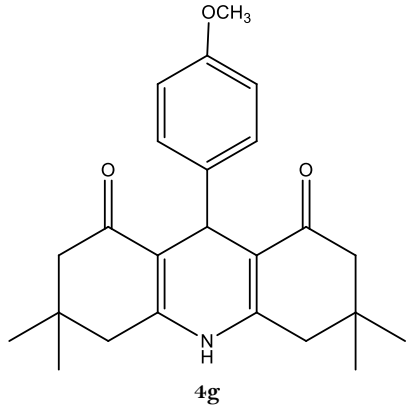


Table 2: Synthesis of 1,8-dioxo-decahydroacridine derivatives using zinc cerium ferrite nanoparticles (**4a-g**).

Entry	Aromatic Aldehyde	Product	Time / min	Yield / %	M.P.
1		 4a	25	97.04 %	180 °C
2		 4b	15	96.63%	202 °C
3		 4d	15	96.36 %	204 °C
4		 4f	15	97.22 %	270 °C

Entry	Aromatic Aldehyde	Product	Time / min	Yield / %	M.P.
5		 4c	20	95.92%	244 °C
6		 4e	25	98.32%	222 °C
7		 4g	15	95.77%	248 °C

Recycle & Reusability of catalyst:

The reusability of catalysts is a significant issue in heterogeneous catalysis. The catalyst's recovery and reusability were examined in this reaction for the model reaction (4a). The catalyst was repeatedly employed five times without any discernible decline in its catalytic efficacy

(Cycle number and yield of 4a: 1- 98.02 %; 2- 97.56 %; 3- 95.60 %; 4- 94.20 %; 5- 94.75 %) as illustrated in Figure 5. These catalysts possess significant magnetic properties, enabling their efficient and nearly total separation using an external magnet, which is a considerable advantage for heterogeneous catalysis

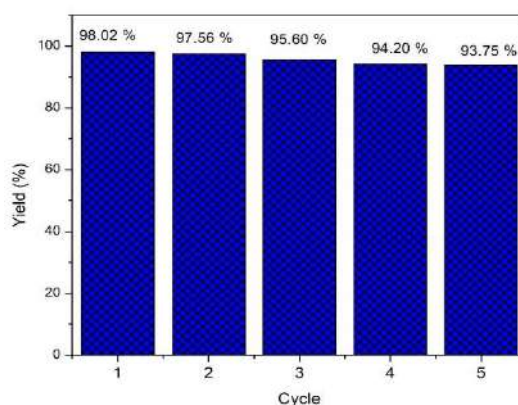


Figure 5: Catalyst reusability

Conclusions

Spinel zinc cerium ferrite nanoparticles were effectively synthesized via the oxalate precursor technique. The XRD pattern and IR spectra confirmed the synthesis of a single-phase cubic spinel zinc cerium ferrite. The mean crystallite size is 24.592 nm, and the lattice parameter is 8.417 Å. Nano spinel zinc cerium ferrite nanoparticles are easily fabricated, non-toxic, cost-effective, magnetically recoverable, and efficient catalysts for the synthesis of 1,8-dioxo-decahydroacridine derivatives through the condensation of aromatic aldehydes with ammonium acetate and dimedone. This approach provides rapid reaction times, straightforward product isolation, and elevated yields.

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Conflicts of interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

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Original Article

Synthesis and characterization of functionalized Quinazolin-2,4-dione derivatives with diverse substituents

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Abstract

To investigate the structural diversity and possible pharmacological significance of quinazoline-2,4-(1H,3H)-dione derivatives, a new series of these compounds were synthesized. 1-ethoxycarbonylmethyl-2,4-dioxo-1,4-dihydro-2H-quinazolin-3-yl)-acetic acid ethyl ester (TM-1), 1,3-diacetylquinazoline-2,4-(1H,3H)-dione (TM-2), 1,3-dibenzoyl-1H-quinazoline-2,4-dione (TM-3), and 1,3-ditosylquinazoline-2,4-(1H,3H)-dione (TM-4). The quinazoline moieties undergo properly substitution and acylation processes to prepare these derivatives. The chemical structure of synthesized compounds was confirmed using FT-IR, ¹H NMR and ¹³C NMR. The functional groups such as acetyl, benzoyl and tosyl scaffolds were introduced through structural changes at N-1 and N-3 locations of quinazoline moieties which are significantly affect biological properties. Literature survey shows quinazoline-based compounds are capable of possessing different pharmacological properties, such as anti-inflammatory, antibacterial and anticancer effects however small compounds have been for biological activities in this area of research. The synthesized derivatives might be having interesting biological potency due to quinazoline core and pharmacologically active substituents. In order to create new quinazoline-based medicinal medicines, our work lays the groundwork for future biological screening and structure-activity relationship (SAR) investigations. The present work plays important role for future biological screening and structure activity relationship examination in order to fabricate novel quinazoline – based medicinal medicines.

Keywords: Quinazoline, Quinazoline derivatives, Quinazolinone

Introduction

Nitrogen-containing heterocyclic compounds are an important class of molecules in current medical and synthetic chemistry due to their vast range of uses and structural diversity. Among these, the quinazoline framework has become a well-known scaffold because of its chemical stability and structural modification adaptability. As a result, various quinazoline derivatives have demonstrated significant biological activities such as antimicrobial [27–30], anticancer [1–23], antibacterial [24–25], and antimalarial [26]. Quinazoline-2,4-dione derivatives in particular have drawn interest due to their easily functionalized reactive sites and clearly defined core structure. Particularly at the N-1 and N-3 positions, where the addition of various functional groups can dramatically change molecular characteristics like polarity, solubility, and reactivity, the quinazoline-2,4-dione nucleus offers a great platform for substitution. These modifications frequently affect how the molecule interacts with biological systems, which makes these derivatives appealing for additional pharmacological research. Substituents such as ester, acyl, aromatic, and sulfonyl groups are known to alter steric orientation and electron distribution, which may have an effect on the compounds' overall behavior.

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In order to investigate their possible uses, recent research has concentrated on creating structurally varied quinazoline derivatives. The synthesis of molecules with desired chemical and biological properties has been made possible by the ability to fine-tune the quinazoline scaffold through systematic replacement. Structural design based on accepted chemical principles can offer important insight into the possible activity of freshly synthesized compounds even in the absence of biological screening. In this context, the present study involves the design and synthesis of a series of quinazoline-2,4-dione derivatives (TM-1 to TM-4) bearing different substituents such as ethoxycarbonyl methyl, acetyl, benzoyl and tosyl groups at key positions of the heterocyclic core. These modifications are expected to influence the electronic and steric environment of the molecules, thereby making them suitable candidates for future biological evaluation. The work reported here primarily focuses on the preparation and structural characterization of these derivatives which contributing to the ongoing efforts in the development of functionalized quinazoline compounds for advanced studies.

Materials and Methods

Chemicals and Reagents

All chemicals and reagents used to perform the present study were of analytical grade and obtained from reputable commercial suppliers. Solvents were filtered and desiccated according to standard protocols prior to utilization when required. The advancement of reactions was observed by thin-layer chromatography (TLC) utilizing silica gel plates, with visualization performed under UV light or via iodine staining.

Instrumentation

Infrared (IR) spectra were obtained utilizing a Fourier-transform infrared spectrophotometer to detect distinctive functional groups. Proton nuclear magnetic resonance (¹H NMR) and carbon-13 nuclear magnetic resonance (¹³C NMR) spectra were acquired utilizing appropriate deuterated solvents, with chemical shifts reported in parts per million (ppm) relative to tetramethylsilane (TMS) as an internal standard. High-resolution mass spectrometry (HRMS) was employed to verify the molecular mass of the produced molecules.

Standard protocol for the production of Quinazoline derivatives

i) Synthesis of Quinazolin-2,4-dione (I):

A mixture of anthranilic acid (50 g, 0.36 mol) and urea (109 g, 1.82 mol) was cooked in a round-bottom flask equipped with a mechanical stirrer at 135°C to 140°C for 3 hours, without solvent, using an air condenser. The molten reaction mixture was introduced into 500 cc of crushed ice while maintaining continuous agitation for 30 minutes. The solid was filtered using a Buchner funnel, rinsed with water (3X100 mL), and subsequently dried. The result was sufficiently pure for use in the subsequent stage. Yield: 74%; melting point: >250°C.

ii) Synthesis of 1-ethoxycarbonylmethyl-2,4-dioxo-1,4-dihydro-2H-quinazolin-3-yl acetic acid ethyl ester (TM-1):

Quinazolin-2,4-dione (1 g) was combined with 1.7 g of K₂CO₃ and 10 ml of dimethylformamide (DMF), stirred for 15 minutes at room temperature, followed by the addition of 12.4 mmol of ethyl bromoacetate. The mixture was stirred at room temperature for 24 hours, then poured into crushed ice. The resulting precipitate was filtered, dried, weighed, and characterized.

iii) Synthesis of 1,3-diacetylquinazoline-2,4(1H,3H)-dione (TM-2):

Quinazolin-2,4-dione (1 g) was combined with 1.7 g of K₂CO₃ and 10 ml of dimethylformamide (DMF), stirred for 15 minutes at room temperature, followed by the addition of 12.4 mmol of acetyl chloride. The mixture was stirred at room temperature for 24 hours, then poured into crushed ice. The resulting precipitate was filtered, dried, weighed, and characterized.

iv) Synthesis of 1,3-dibenzoyl-1H-quinazolin-2,4-dione (TM-3):

Quinazolin-2,4-dione (1 g) was combined with 1.7 g of K₂CO₃ and 10 ml of dimethylformamide (DMF), stirred for 15 minutes at room temperature, followed by the addition of 12.4 mmol of benzoyl chloride. The mixture was stirred at room temperature for 24 hours, then poured into crushed ice. The resulting precipitate was filtered, dried, weighed, and characterized.

v) Synthesis of 1,3-ditosylquinazoline-2,4(1H,3H)-dione (TM-4):

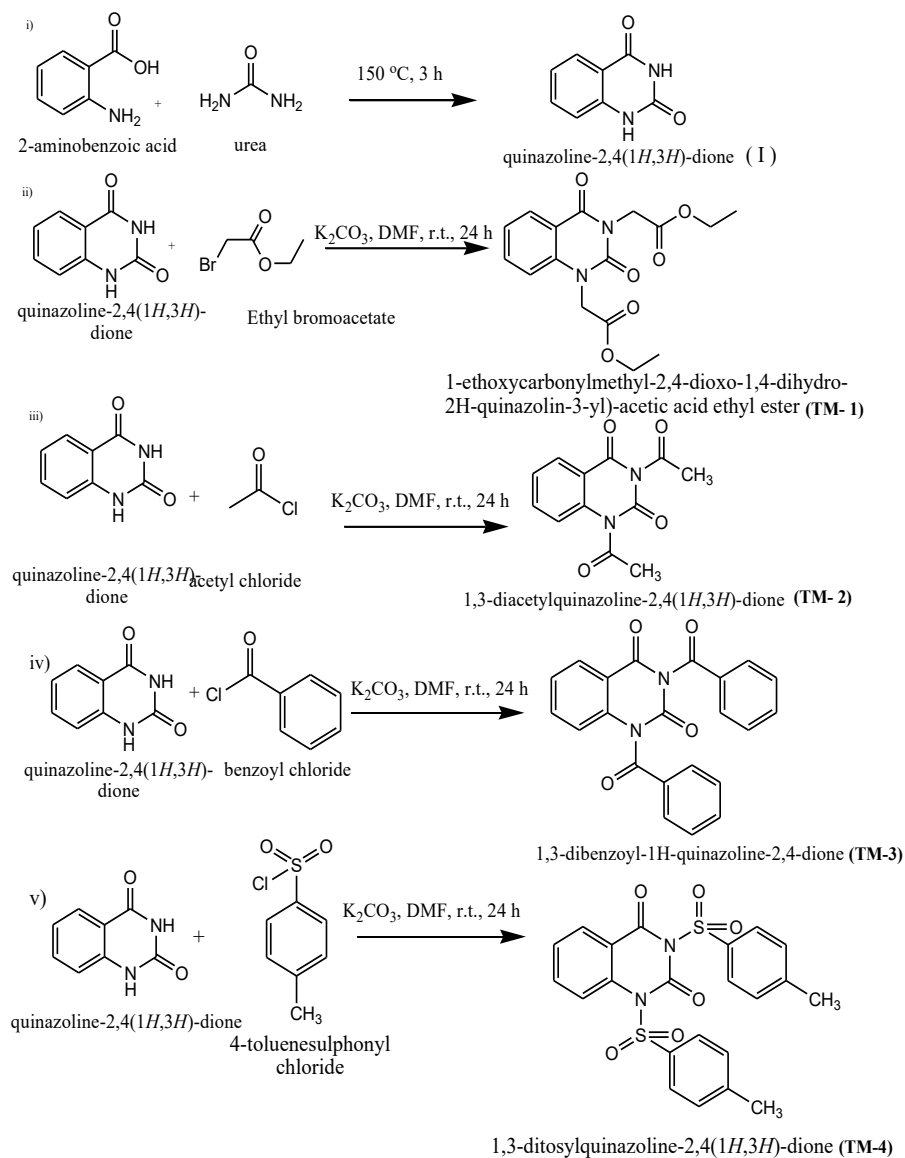
Quinazolin-2,4-dione (1 g) was combined with 1.7 g of K₂CO₃ and 10 ml of dimethylformamide (DMF), stirred for 15 minutes at room temperature, followed by the addition of 12.4 mmol of p-toluenesulfonyl chloride. The mixture was stirred at room temperature for 24 hours, after which it was poured into crushed ice. The resulting precipitate was filtered, dried, weighed, and characterized.

Results and Discussion

Synthesis of Quinazoline Derivatives (TM-1 to TM-4)

A series of substituted quinazoline-2,4-dione derivatives (TM-1 to TM-4) were synthesized effectively utilizing modified literature techniques. The synthesis route entailed functionalization at the N-1 and N-3 positions of the quinazoline core, facilitating the incorporation of several substituents, including ester, acetyl, benzoyl, and tosyl groups. The reactions occurred seamlessly under regulated conditions, yielding the target chemicals in moderate to good quantities. Among the synthesized derivatives, TM-1 was obtained via incorporation of an ethoxycarbonylmethyl moiety, leading to the formation of an ester-functionalized product. TM-2 was synthesized by acetylation, introducing electron-withdrawing acetyl groups at the 1,3-positions. TM-3 and TM-4 were obtained through acylation and sulfonylation reactions, respectively, resulting in bulkier substituents such as benzoyl and tosyl groups.

Scheme I:



Structural Confirmation

The structures of all produced compounds were validated by spectrum methods. The infrared spectra of the compounds displayed distinctive absorption bands associated with carbonyl (C=O) stretching vibrations of the quinazoline-2,4-dione ring structure, generally detected in the anticipated region. Supplementary peaks indicative of ester (TM-1), ketone (TM-2 and TM-3), and sulfonyl (TM-4) functions further corroborated effective substitution. The ¹H NMR spectra exhibited evidence indicative of aromatic protons from the quinazoline ring and the substituent phenyl groups. Aliphatic protons associated with ethyl and methyl groups were distinctly identified in TM-1 and TM-2, respectively. The detection of supplementary aromatic protons in TM-3 and TM-4 validated the inclusion of benzoyl and tosyl groups. The ¹³C NMR spectra confirmed the existence of carbonyl and aromatic carbons at anticipated chemical shift values.

1,3-ditosylquinazoline-2,4(1H,3H)-dione (TM-4)

1-ethoxycarbonylmethyl-2,4-dioxo-1,4-dihydro-2H-quinazolin-3-yl)-acetic acid ethyl ester (TM-1)

FTIR (cm⁻¹): 3254, 2895, 2352, 1720, 1645, 1615, 1565, 1430, 1358, 1275, 1240, 1121, 1024, 968, 858, 742, 704, 604;
¹H NMR (500 MHz, DMSO) δ : 8.05 (d, *J* = 9.1 Hz, 1H), 7.75 (t, *J* = 8.6 Hz, 1H), 7.42 (d, *J* = 8.5 Hz, 1H), 7.33 (t, *J* = 7.5 Hz, 1H), 5.05 (s, 2H), 4.45 (s, 2H), 4.55 (dq, *J* = 14.0, 7.0 Hz, 4H), 1.20 – 1.15 (m, 6H); **¹³C NMR (126 MHz, DMSO) δ :** 168.37 (s), 169.15 (s), 145.60 (s), 139.15 (s), 145.39 (s), 135.60 (s), 122.03 (s), 118.23 (s), 112.72 (s), 56.78 (s), 59.60 (s), 42.31 (s), 44.91 (s), 14.55 (s)

1,3-diacetylquinazoline-2,4(1H,3H)-dione (TM-2)

FTIR (cm⁻¹): 3242, 2356, 1648, 1568, 1452, 1330, 1245, 1115, 1048, 955, 854, 737, 661;
¹H NMR (500 MHz, DMSO) δ : 7.22 (d, *J* = 7.8 Hz, 1H), 7.62 (t, *J* = 8.4 Hz, 1H), 7.14 (t, *J* = 8.5 Hz, 2H), 2.80 (s, 3H), 2.74 (s, 3H); **¹³C NMR (126 MHz, DMSO) δ :** 163.32 (s), 150.79 (s), 141.40 (s), 135.40 (s), 127.40 (s), 122.75 (s), 115.83 (s), 114.79 (s), 36.25 (s), 31.23 (s)

1,3-dibenzoyl-1H-quinazolin-2,4-dione (TM-3)

FTIR (cm⁻¹): 3233, 2363, 1709, 1685, 1576, 1507, 1451, 1358, 1229, 1181, 1145, 834, 757, 709; **¹H NMR (500 MHz, DMSO) δ** : 7.97 – 7.92 (m, 1H), 7.77 – 7.65 (m, 4H), 7.64 – 7.59 (m, 1H), 7.50 – 7.34 (m, 7H), 7.21 – 7.14 (m, 1H); **¹³C NMR (126 MHz, DMSO) δ** : 165.30 (s), 162.87 (s), 161.66 (s), 150.85 (s), 138.15 (s), 135.12 (s), 131.27 (s), 129.04 (s), 128.15 (s), 126.31 (s), 117.98 (s)

Synthesis of 1,3-ditosylquinazoline-2,4(1H,3H)-dione (TM-4) :

FTIR (cm⁻¹): 3252, 3186, 3113, 3049, 2355, 1668, 1587, 1531, 1485, 1456, 1393, 1308, 1252, 1177, 1148, 1069, 1005, 878, 818, 760; **¹H NMR (500 MHz, DMSO) δ** : 8.05 – 7.97 (m, 3H), 7.83 – 7.77 (m, 3H), 7.52 (td, J = 7.8, 4.5 Hz, 1H), 7.34 – 7.30 (m, 4H), 7.24 – 7.20 (m, 1H), 2.31 (s, 6H); **¹³C NMR (126 MHz, DMSO) δ** : 170.22 (s), 144.44 (s), 142.98 (s), 140.96 (s), 138.37 (s), 134.99 (s), 129.61 (s), 127.68 (s), 119.90 (s), 118.60 (s), 21.32 (s).

Comparative Analysis of Substituent Effects

The incorporation of various substituents at the N-1 and N-3 positions markedly affected the physicochemical properties of the produced molecules. TM-1, possessing an ester group, is anticipated to demonstrate comparatively greater polarity than other derivatives. TM-2, possessing acetyl substituents, exhibited a moderate electron-withdrawing nature, potentially affecting reactivity and biological interactions. TM-3 and TM-4, featuring substantial benzoyl and tosyl groups, respectively, exhibited enhanced molecular dimensions and aromaticity. The existence of these groups may augment lipophilicity and promote interactions with hydrophobic areas in biological systems. The sulfonyl group in TM-4 exerts significant electron-withdrawing effects, potentially influencing the compound's chemical properties.

Integrity and Output

All synthesized compounds were acquired in adequate yields and exhibited good purity, as verified by thin-layer chromatography, which revealed single spots under UV inspection. Recrystallization yielded analytically pure chemicals appropriate for subsequent investigations.

Possible Biological Significance

The absence of biological evaluation in this study notwithstanding, the structural characteristics of the synthesized quinazoline derivatives imply possible pharmacological activity. Quinazoline derivatives are recognized for their many biological features, encompassing antibacterial, anticancer, and anti-inflammatory effects. The inclusion of electron-withdrawing groups and aromatic substituents in TM-2, TM-3, and TM-4 may augment binding affinity to biological targets, suggesting their potential for further exploration.

Conclusion

This study effectively designed and synthesized a series of quinazoline-2,4-dione derivatives (TM-1 to TM-4) by modifying existing synthetic techniques. Diverse structural modifications at the N-1 and N-3 locations of the quinazoline core facilitated the integration of multiple

functional groups such as ester, acetyl, benzoyl, and tosyl moieties. The implemented synthesis techniques proved efficient, yielding the target molecules in adequate quantities with high purity. The structures of all synthesized derivatives were validated via standard spectroscopic methods, including IR, ¹HNMR, and ¹³CNMR, confirming the effective synthesis of the desired compounds. Substituent variations were shown to affect the physicochemical features of the compounds, especially regarding polarity, electronic dispersion, and steric attributes. Despite the lack of experimental evaluation of biological activity, the existence of many functional groups and the recognized pharmacological significance of the quinazoline scaffold imply that these derivatives may exhibit considerable biological potential. Consequently, the produced compounds may function as significant intermediates or lead molecules for forthcoming pharmacological research. This study offers a valuable methodology for the synthesis and structural alteration of quinazoline derivatives, enhancing studies in heterocyclic chemistry and pharmacological development.

Abbreviations

DMF: Dimethyl formamide

DMSO: Dimethyl Sulfoxide

FTIR: Fourier Transform Infra-Red Spectroscopy

MTT: (3-(4,5-Dimethylthiazol-2-yl) -2,5-Diphenyltetrazolium Bromide

¹H NMR: Proton Nuclear Magnetic Resonance Spectroscopy

¹³C NMR: Carbon-13 Nuclear Magnetic Resonance Spectroscopy

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Conflicts of interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

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Compliance With Ethical Standards

This article does not contain any study involving human participants or animals performed by any of the authors.

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Original Article

An Efficient and Reusable Heterogeneous Organocatalyst Chitosan for the Transamination of Carboxamides with Amines

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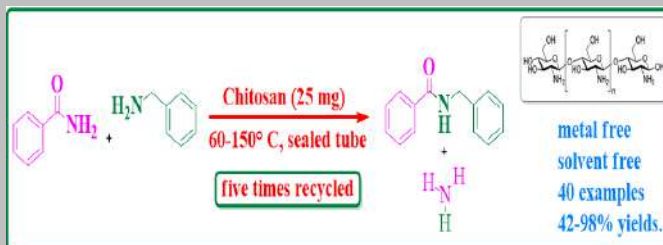
Abstract

This work describes a chitosan-mediated transamidation of carboxamides with amines. A broad set of amide substrates, together with structurally demanding aryl and long-chain alkyl amines, were efficiently converted to the corresponding transamidated products, a class of compounds commonly encountered among bioactive molecules and pharmaceutical agents. Under comparable conditions, treatment of 2-aminothiophenol with formamide delivered the cyclized heterocycle benzo[d]thiazole. Because the chitosan catalyst could be recovered and reused without loss of activity, near-quantitative product yields were maintained across repeated cycles.

Keywords: Chitosan; Heterogeneous organocatalyst; Transamidation; Carboxamides; Amines; Green chemistry; Solvent-free synthesis; Recyclable catalyst; Hydrogen bonding catalysis; N-Formylation; Benzothiazole synthesis; Sustainable catalysis.

Introduction

The amide linkage ranks among the most important functional groups in both biological systems and synthetic chemistry.¹ Its formation underlies the assembly of complex, structurally homogeneous glycoproteins, and amide bonds form the backbone of proteins, often described as the fundamental molecular currency of living organisms. Beyond biology, amides serve as key intermediates in organic synthesis and feed into the production of pharmaceuticals, agrochemicals, polymers, and other functional materials.² A range of strategies has therefore been devised to construct this linkage³, including coupling of activated acid derivatives with amines⁴, amidation of aldehydes⁵, direct synthesis from alcohols and amines⁶, hydroamination of alkynes⁷, and nitrile hydration⁸. What remains particularly sought after, however, are operationally simple, catalytic methods that minimize or eliminate the use of stoichiometric activating reagents. In addition to being complementary, transamidation can also serve as an alternative approach in which the components of two different amide bonds are interchanged directly. This topic has gained considerable attention in recent years among several research teams⁹, including ours.¹⁰ Despite some rare exceptions, most of the current transamidation strategies employ transition-metal-based catalysis, which, however, brings about a problem associated with separating the catalyst from the end product, especially when it comes to pharmaceutical production where metal impurities are undesirable. In addition to posing environmental issues related to generating wasteful byproducts, metal-catalyzed reactions are undesirable due to their non-green nature. Although efficient transamidation protocols are currently available, relatively little effort has been expended on developing strategies involving recoverable and recyclable heterogeneous catalysts.



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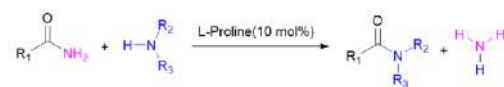
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As part of our ongoing research in green and sustainable methods of synthesis we have developed a new method of carboxamides' transamidation with amines using chitosan

11 as a catalyst. To the best of our knowledge, transamidation under heterogeneously catalyzed and recyclable conditions has not yet been achieved.

Our previous work [Homogeneous organo catalyst]



Present Work [heterogeneous organo catalyst]

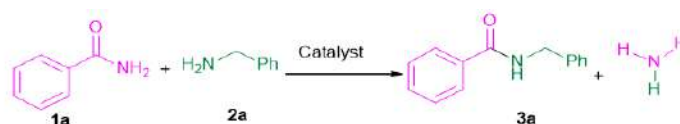


Scheme 1

Results and Discussion

We have earlier reported an organocatalytic transamidation of carboxamides.¹⁰ Even though the catalyst was found effective, it was non-recoverable for re-use. Following up on the above work, along with the earlier reported literature 9f, h that suggested hydrogen-bonding mechanism of transamidation reaction, we tried to look for a heterogenous organocatalyst that would catalyze such a reaction and chitosan appeared to be an interesting candidate. The chitosan molecule has four hydroxyl groups and one amine group per repeat unit, which would allow hydrogen-bonding with amide substrates. The polymer is stable at high temperature and recoverable as well. Benzamide 1a and benzylamine 2a were chosen as the starting substrates, with 25 mg of chitosan in toluene at 120 °C for 36 hours, but no product was observed (Table 1, entry 1). Testing the efficiency of different solvents (DMA, NMP, DMSO, DMF, water, and chlorobenzene) was also unsuccessful (Table 1, entries 2–7). The use of neat reaction conditions at 120 °C resulted in 13% yield of the desired product, N-benzyl benzamide 3a, based on GC-MS analysis (Table 1, entry 8), while increasing the temperature up to

150 °C improved the yield of 3a to 99% (Table 1, entry 9). A decrease in the amount of the catalyst (from 10 to 5 wt%) or in the duration of the reaction time (from 30 to 24 h) led to a considerable decline in yield of 3a (Table 1, entries 10–13). On this basis, the conditions selected for further study were 25 mg chitosan, 150 °C, neat, 36 h. Having determined the optimal reaction conditions, we further studied the substrate scope for this catalytic reaction. As is demonstrated in Table 2, transamidation of benzamide 1 with various benzylamines 2 was readily achieved in excellent yield in the absence of any solvent. It was found that the presence of electron-donating (Me) and electron-withdrawing (Cl, F) groups on the benzylamines had little influence on this reaction (Table 2, entries 2–7). The aniline derivatives also proved to be suitable for this reaction (Table 2, entries 8 and 9). In addition, alkyl-aryl, aliphatic, and cyclic amines also could serve as starting materials, and satisfactory yields of the transamidation products were observed (Table 2, entries 10–15). Most importantly, 2-aminoethanol could selectively participate in this reaction, giving the corresponding product in 73% yield without consumption of the alcohol moiety (Table 2, entry 16).



s.no.	catalyst	temperature (° c)	solvent	conversion ^b
1	chitosan	120	Toluene	nr
2	chitosan	150	DMA	nr
3	chitosan	150	NMP	trace
4	chitosan	150	DMSO	nr
5	chitosan	150	DMF	nr
6	chitosan	120	H ₂ O	nr
7	chitosan	120	Chloro benzene	trace
8	chitosan	120	–	13
9	chitosan	150	–	>99
10	chitin	150	–	77
11	chitosan ^c	150	–	95

Table-1 Optimization of reaction conditions

a Reaction Conditions: Amide (1 mmol), Amine (2 mmol), Catalyst (25 mg), and Solvent (2 mL) in sealed tube, 36 h. b Percent yield calculated from GC-MS analysis. c Reaction time was 30 hours. Next, we expanded the scope to include several other substrates such as benzoic amides (both electron-rich and electron-poor) as well as heteroaromatics, aliphatics, and secondary amides along with an assortment of amines (electrons-neutral and electrons-rich benzylamines as well as aromatic, aliphatic, alkyl-aromatic, and cyclic secondary amines). This resulted in excellent yields of the transamidated products (Table 2, entries 17–32). However, one exception to this rule was 2-aminopyridine which is a heteroaromatic amine giving lower yields due to its electron-withdrawing properties (Table 2, entry 33). Encouraged by the success of this transamidation chemistry under neat, chitosan-catalyzed

conditions (20 wt %), we next asked whether the same approach could deliver *N*-formylated products from a broader set of amines (Table 3). The transformation proved general across a wide range of amine substrates, furnishing the corresponding *N*-formylated anilines under these mild, previously unreported conditions (Table 3, entries 4–8). The propargylic amine substrate gave the *N*-formylated product in quantitative yield (Table 3, entry 9), while, as observed for the transamidation series, *N*-formylation of 2-aminopyridine with formamide again gave a lower yield, consistent with the electron-poor pyridine ring (Table 3, entry 10). Unexpectedly, treating 2-aminothiophenol, 1,2-diaminobenzene, and 2-aminophenol with formamide under the same conditions afforded the cyclized products **4k**, **4l**, and **4m** in 88%, 61%, and 45% isolated yield, respectively.

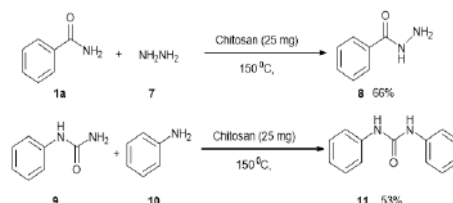
entry	benzamide	amine	product	Yield(b%)
1				92
2				82
3				96
4				89
5				86
6				84
7				75

Table-4. Scope of the transamidation of phthalimide with various amines

a Reaction conditions: amide (1 mmol), amine (2 mmol), catalyst (25 mg) in a sealed tube at 150 °C, 36 h. b Isolated yields.

Applying the same protocol to phthalimide, we examined its transamidation with benzyl, alkyl-aromatic, and aromatic amines (both electron-rich and electron-poor) (Table 4). All

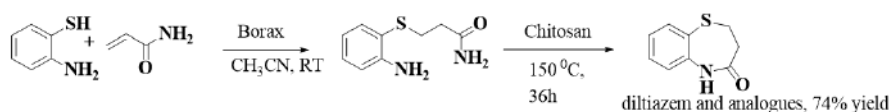
primary amines tested reacted cleanly, giving the corresponding *N*-substituted phthalimides in good-to-excellent yields (75–96%). The transamidation of benzamide and phenylurea with hydrazine and aniline, respectively, also proceeded well, giving the desired products in 66% and 53% yield (Scheme 2).



Finally, an intramolecular variant of this transamidation gave heterocyclic product **12** in 74% yield using 25 mg of

chitosan, providing a practical entry point toward diltiazem and related analogues (Scheme 3).¹²

Scheme 2 Synthesis of 2,3-dihydro-5H-benzo[b]-1,4-thiazepin-4-one via intramolecular transamidation



Scheme 3

Catalyst recyclability was evaluated using the benzamide/benzylamine model reaction under the optimized conditions (Table 1, entry 9). After each run, the catalyst was recovered by simple filtration, washed with water, and oven-dried for 30 min (see Experimental Section). Across five successive cycles, the transamidation

proceeded in essentially quantitative yield with no detectable loss of catalytic activity (Figure 1), underscoring the practicality of chitosan as a recoverable, heterogeneous catalyst for transamidation across a structurally diverse set of carboxamides and amines under solvent-free conditions.

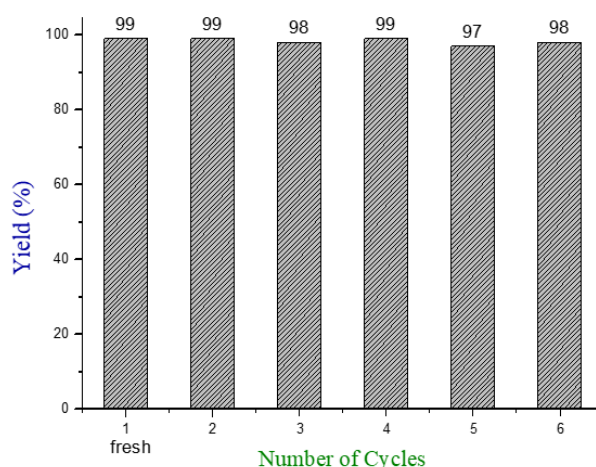
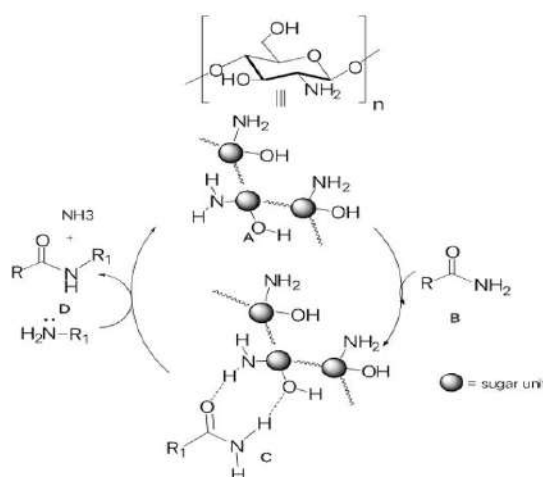


Fig 1. Recyclability study of the chitosan catalyst



Scheme 4 Plausible mechanism

Drawing on precedent in the literature together with the observations reported here, a plausible mechanistic pathway is proposed (Scheme 4). Hydrogen bonding between chitosan **A** and the amide substrate **B** first generates intermediate **C**; the amine partner then reacts with **C** to furnish the transamidated product **D** while regenerating the chitosan catalyst **A**. In summary, we have developed a chitosan-catalyzed transamidation that proceeds

selectively and efficiently. Relative to existing transamidation catalysts, chitosan offers the advantages of low cost, ready availability, and reusability with no measurable decline in catalytic performance, while delivering near-quantitative yields. The method tolerates a broad range of benzylic, aromatic, aliphatic, propargylic, and heteroaromatic amines, affording transamidated products in good-to-excellent yield, and the reaction of aminothiophenol



with formamide under analogous conditions provides direct access to benzothiazoles.

General

Commercial chemicals and reagents were used as received unless stated otherwise. ^1H and ^{13}C NMR spectra were acquired at 500/200 MHz and 125/50 MHz, respectively, using CDCl_3 as solvent. Multiplicities are abbreviated as s (singlet), d (doublet), t (triplet), m (multiplet), and dd (doublet of doublets); coupling constants (J) are reported in Hz, and chemical shifts in ppm relative to TMS. Residual solvent peaks for CDCl_3 were referenced at ^1H δ 7.2 and ^{13}C δ 77.0. Mass spectra were collected by electron-impact (EI) ionization, and reaction progress was followed by TLC. Products were purified by column chromatography on 200–400 mesh silica gel, typically eluting with dichloromethane/ethyl acetate (80:20) unless noted otherwise.

General experimental procedure

Amide (1 mmol), amine (2 mmol), and chitosan (25 mg) were combined and stirred in a sealed tube at the temperature and for the duration specified for each substrate (see Schemes 1–4 and Table 2). Once the mixture had cooled to room temperature, the crude product was isolated by recrystallization or by silica-gel column chromatography (200–400 mesh), eluting with dichloromethane, or dichloromethane/ethyl acetate, or methanol as appropriate. Isolated yields are reported in Tables 1–4 and Scheme 2.

Catalyst recycling experiment

The chitosan catalyst was carried through five consecutive transamidation cycles (36 h each), recovering it after every run. At the end of each cycle, the catalyst was isolated by filtration, rinsed with dichloromethane, oven-dried for 30 min, and reintroduced into the next reaction without further treatment.

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Original Article

Solvent Less Synthesis of Chromeno[2,3-D] Pyrimidines

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Abstract

A simple and one-pot synthesis of new chromeno[2,3-d] pyrimidine synthesized by a three-component condensation reaction of malononitrile, substituted salicylaldehydes and cyclic amines in solventless condition in ultrasound condition. Two cyclohexane-1,3-diones, product Chromeno[2,3-d] pyrimidines. the advantages of this new method are operational simplicity, good yields and easy work-up procedures employed. Chromeno[2,3-d] pyrimidines constitute an important class of fused heterocyclic compounds owing to their significant biological and pharmaceutical activities. In the present study, an efficient, solvent-free, one-pot synthetic protocol was developed for the preparation of novel chromeno[2,3-d] pyrimidine derivatives through a three-component condensation reaction involving substituted salicylaldehydes, malononitrile, and cyclic amines under ultrasonic irradiation. The methodology eliminates the use of hazardous solvents and catalysts, making it an environmentally friendly and sustainable synthetic approach. The reaction proceeds smoothly under mild conditions, affording the desired products in good to excellent yields with simple work-up and high operational efficiency. The ultrasonic-assisted solvent-free conditions significantly reduce reaction time while improving product selectivity and minimizing waste generation. The developed protocol demonstrates the advantages of green chemistry by combining operational simplicity, cost-effectiveness, and eco-friendly reaction conditions. This method offers a practical and efficient strategy for the synthesis of biologically important chromeno[2,3-d] pyrimidine derivatives and may find wide applications in medicinal and synthetic organic chemistry.

Keywords: chromeno[2,3-d] pyrimidine; malononitrile; salicylaldehydes.

Introduction

With the emergence of high throughput screening the demand for new drug molecules has increased manifold. Combinatorial chemistry and parallel automated synthesis has met this demand to a certain extent. However, these methods have certain limitations particularly in providing diverse molecules. The one of the emerging fields in this regard is the multicomponent synthesis. These multicomponent reactions provide considerable diversity particularly providing nitrogen heterocycles. Functionalized nitrogen-heterocycles are attractive targets in medicinal chemistry. The importance of pyrimidine scaffold in nature needs no mention. Pyrimidine compounds are known as antibacterial antiviral and antitumor agents. Fused pyrimidine displays interesting pharmacological profiles. Pyridopyrimidines acts as analgesics and CNS depressants. Benzopyranopyrimidines shows anti-inflammatory, analgesic and in vitro anti-aggregating activities. Fused molecules sharing benzopyran moiety have shown platelet anti-aggregative activity comparable to that of acetylsalicylic acid. As a part of continuing research interest of our group to develop green approaches to biologically relevant molecules we were interested in synthesizing Benzopyrano[2,3-d] pyrimidine. We have developed a solvent free, catalyst free protocol for the synthesis Chromeno[2,3-d] pyrimidines.

Experimental

General Melting points were measured on an Electrothermal 9100 apparatus and are uncorrected. Mass spectra were recorded on a FINNIGAN-MAT 8430 mass spectrometer operating at an ionization potential of 70 eV. IR spectra were recorded on a Shimadzu IR-470 spectrometer. ¹H and ¹³C-NMR spectra were recorded on a BRUKER DRX-300 AVANCE spectrometer at 300.13 and 75.47 MHz, respectively. Elemental analyses were performed using a Heracus CHN-O-Rapid analyzer

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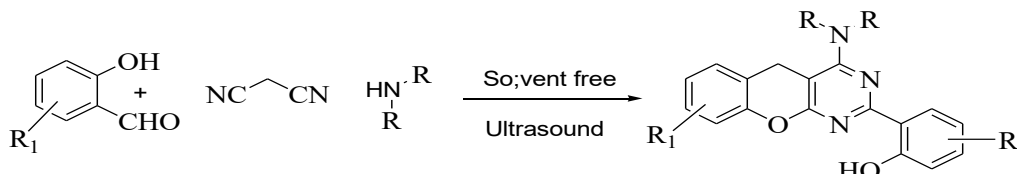
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Reaction Scheme:



During the studies it was observed that instead of exclusive formation of expected product (70%) an additional oxidized product (30%) was also obtained. Efforts to get exclusive oxidized product has so far not succeeded.

Conclusion

In conclusion, a simple, efficient, and environmentally friendly solvent-free protocol has been developed for the synthesis of chromeno[2,3-d] pyrimidine derivatives through a one-pot, three-component reaction involving substituted salicylaldehydes, malononitrile, and cyclic amines under ultrasonic irradiation. The developed methodology offers several advantages, including operational simplicity, shorter reaction time, good product yields, and easy work-up without the use of hazardous organic solvents or catalysts. These features make the protocol consistent with the principles of green chemistry and sustainable organic synthesis.

The synthesized chromeno[2,3-d] pyrimidine derivatives represent an important class of fused heterocyclic compounds with significant pharmaceutical potential due to their reported antibacterial, antiviral, anti-inflammatory, and antitumor activities. Although the formation of a minor oxidized by-product was observed during the reaction, the desired products were obtained in satisfactory yields, demonstrating the effectiveness of the proposed synthetic approach. Further optimization of reaction conditions and biological evaluation of the synthesized compounds may provide valuable insights into their medicinal applications and expand the scope of this environmentally benign synthetic methodology.

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Conflicts of interest

The authors declare that there are no conflicts of interest regarding the publication of this paper

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Original Article

Zinc Oxide (Zno): Synthesis, Structural Characterization, And Their Photocatalytic Application for Methylene Blue (MB) Dye Degradation Under Solar Radiation

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Abstract

Zinc oxide nanoparticles were fabricated by the sol-gel auto-combustion route and examined using XRD, FT-IR, FE-SEM, and EDS techniques. The synthesized ZnO was then tested for the solar-light-assisted removal of MB dye from aqueous solution. The role of solution pH was studied by carrying out degradation experiments under different pH conditions. Among the tested media, pH 8 showed the most favorable response, giving complete removal of MB dye under sunlight. The results indicate that the fabricated ZnO nanoparticles can serve as a promising photodegradation catalyst for treating dye-containing wastewater.

Keywords: ZnO nanoparticle, Photocatalyst, Methylene blue, Solar radiation.

Introduction

Nanotechnology deals with materials whose dimensions are generally within the 1–100 nm range [1]. At this scale, particles often show behavior different from their bulk forms due to their smaller size and large surface-to-volume ratio. These changes can influence mechanical strength, thermal behavior, surface activity, and chemical reactivity [2]. Among nanomaterials, metal oxide nanoparticles are widely studied because of their stability, large active surface area, and usefulness in optoelectronic devices, sensors, catalysts, and electronic applications [3]. The characteristics property of nanomaterials such as chemical stability, thermal resistance, magnetic response, and biocompatibility made it important for biological studies. Several nanomaterials have therefore been used for anticancer, antioxidant, antibacterial, and antifungal applications [4]. There are many metal oxide nanoparticles are widely applied as photocatalyst for waste water treatments such as MoO₃ [5], CeO₂ [6], ZnO [7], TiO₂ [8] WO₃ and metal sulfides like ZnS [9, 10]. ZnO has been commonly studied for photo degradation applications because of its tunable optoelectronic properties, chemical stability and high electron mobility, [11]. In addition to that ZnO has remarkable ability to form H₂O₂ molecules, which is crucial for photocatalytic reactions [12]. Due to the large band gap energy 3.30–3.32 eV, ZnO shows characteristics semiconducting properties [13]. Because of their high catalytic effectiveness in photocatalytic degradation processes, zinc oxide nanoparticles have gained a lot of attention. Sol-gel derived metal oxide nanoparticles have strong antibacterial properties and a greater capacity to break down organic contaminants. Furthermore, ZnO nanoparticles have demonstrated great efficacy in the photocatalytic elimination of colored dyes and chemical pollutants [14].

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Numerous techniques, including co-precipitation [15], chemical oxalate approach [16], sol-gel method [17], hydrothermal [18], micro emulsion [19], and sonochemical [20, 21], are used to create zinc oxide nanoparticles. Each of the aforementioned techniques has advantages of its own, but the sol-gel method has several noteworthy benefits, such as ease of use, high production efficiency, and the capacity to fabricate high purity materials, homogeneity, and uniformity. The leather, pharmaceutical, textile, and paper industries frequently utilize Methylene Blue as a cationic dye. Methylene blue dye from these businesses is released into bodies of water, causing major harm to the environment and human health. Excessive levels of methylene blue dye in wastewater disrupt aquatic ecosystems, decrease light penetration, and have harmful impacts on living things. The potential of ZnO for useful environmental remediation applications was recently demonstrated by the successful degradation of MB dye under solar illumination utilizing microwave-assisted manufactured ZnO structures [22]. Similarly, for wastewater treatment, generally adsorption and coagulation techniques are used but these conventional methods not successful fails to for removal of toxic dye compounds from wastewater completely, and it may generate secondary pollutants. Semiconductor photocatalysis offers a practical route for treating dye-contaminated wastewater, as it can degrade pollutants under light irradiation with limited secondary waste. Among the available photocatalysts, ZnO is widely preferred because it is inexpensive, chemically stable, photosensitive, and have capacity for production of reactive species required for dye oxidation. [23]. Under sunlight, ZnO generates electron-hole pairs while absorbing photon energy exceeds its band gap. The photogenerated charges react with water, hydroxide ions, and dissolved oxygen to create $\bullet\text{OH}$ and $\text{O}_2\bullet^-$ radicals. These active species attack methylene blue molecules and promote their conversion into CO_2 , H_2O , and other degraded products. [24] Solar radiation induced dye removal is highly important because sun light is an abundant and inexpensive, renewable source of energy, making it sustainable and economically feasible for applications in huge wastewater treatment. Recently it is observed that ZnO nanostructured materials exhibit excellent photocatalytic potency for degradation of dye under solar radiation due to its greater surface area and improved charge separation properties. Atta et al observed increased photo degradation efficiency of zinc oxide nanoparticle thin films toward methylene blue degradation due to improved crystallinity and surface morphology [25]. Nowadays, modified and metal-doped ZnO photocatalysts have also shown improved visible-light absorption and reduced electron-hole recombination, leading to higher degradation efficiency [26]. Solar

radiation enhanced photocatalytic degradation is not only removes color from wastewater but also reduce toxicity and chemical oxygen demand, which improves water quality. In order to treat industrial dye effluents and protect aquatic habitats from dangerous pollutants, nanostructured zinc oxide (ZnO) assisted photocatalysis using solar radiation has emerged as an appealing, environmentally friendly, and effective technique. In the present investigation, zinc oxide nanoparticles were synthesized through a sol-gel auto-combustion process, characterized, and evaluated as a photocatalyst for MB dye removal under sunlight. The effect of solution pH on the reaction was examined by performing degradation tests at pH (pH = 2, 4, 6, 8, and 10). The obtained results were used to examine the most favorable pH condition for ZnO-assisted dye degradation.

Materials And Methods

Analytical-reagent-grade zinc nitrate hexahydrate [$\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$], citric acid monohydrate [$\text{C}_6\text{H}_8\text{O}_7 \cdot \text{H}_2\text{O}$], and ammonia solution (NH_3) were used as received. The phase and crystallographic phase of the prepared ZnO powder were examined by Powder X-ray diffraction, Philips-3710 diffractometer and Cu K α radiation ($\lambda = 1.5405 \text{ \AA}$). FT-IR study carried out using a PerkinElmer infrared spectrophotometer. The elemental composition was determined by EDAX analysis using an INCA Oxford system with SEM attached. Surface characterization was analyzed with a JEOL JSM-5600N scanning electron microscope. The absorption measurements were examined with the help of single-beam UV-visible spectrophotometer from Biotechniques India.

A stoichiometric amount of Zinc nitrate ($\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, and Citric acid ($\text{C}_6\text{H}_8\text{O}_7 \cdot \text{H}_2\text{O}$) are taken in a separate beaker 100 ml and 25 ml deionized water added each beaker and stirred for complete dissolution of compounds. After dissolving completely these two solutions are mixed in 500 ml beaker. The solution was continuously stirred and slowly dropwise conc. ammonia solution is added upto solution reach at pH = 7. After that solution stirred and heated constantly at 80°C temperature. On continuous heating sol is formed on continuous heating sol is transformed into gel. After further continuous heating gel auto-combustion takes place to form porous carbonaceous powder and cured using clean and dry mortar. The resulting powder was sintered at 500 °C for 3hr in muffle furnace in air atmosphere to form pure Zinc Oxide.

Results And Discussions

After successful synthesis of by sol-gel autocombustion method, their surface characterization such as geometry, presence of bonding, Morphology, presence of metals, etc were analyzed by using following techniques.

X-ray Diffraction Analysis

The XRD analysis is used to identify the structure of synthesized powdered zinc oxide by sol-gel autocombustion method. The figure 1 shows a typical XRD pattern of ZnO. The XRD diffraction peaks obtained can be assigned to the characteristic planes of hexagonal ZnO, including (100), (002), (101), (102), (110), (103), (200), (112), and

(201). These well-defined reflections indicated the crystalline hexagonal wurtzite structure of ZnO [23, 25]. These XRD peaks are matched with the literature indicates the formation and hexagonal structure of ZnO synthesized by sol-gel autocombustion method. No any secondary phase peaks observed confirms the formation zinc oxide (ZnO).

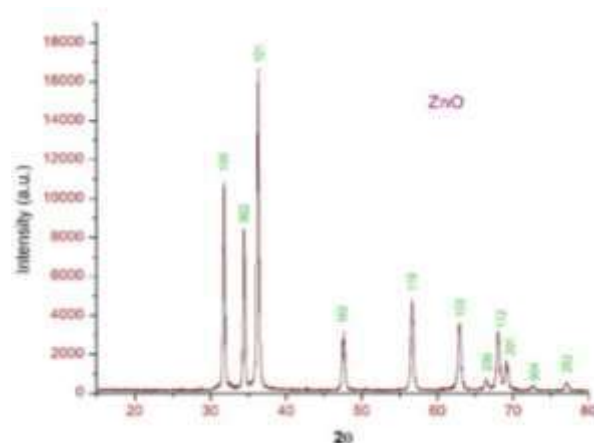


Figure 1: XRD Pattern zinc oxide (ZnO) calcinated at 500°C.

FT-IR Spectra: -

FT-IR spectroscopy was employed to study the bonding features of the synthesized ZnO nanoparticles. The spectrum shown in Figure 2 contains absorption bands near 3746, 3610, 3524, 2348, 1178, 548, and 419 cm^{-1} . The low-wavenumber bands at about 548 and 419 cm^{-1} are assigned to Zn–O vibrations, confirming the

formation of ZnO [23,25]. The band around 1178 cm^{-1} may arise from residual C–O-related vibrations associated with the precursor or combustion process. The peaks observed at 3746, 3610, and 3524 cm^{-1} are characteristics of O–H stretching vibrations of adsorbed water molecules [22]. The band near 2348 cm^{-1} ascribed to adsorbed atmospheric CO_2 .

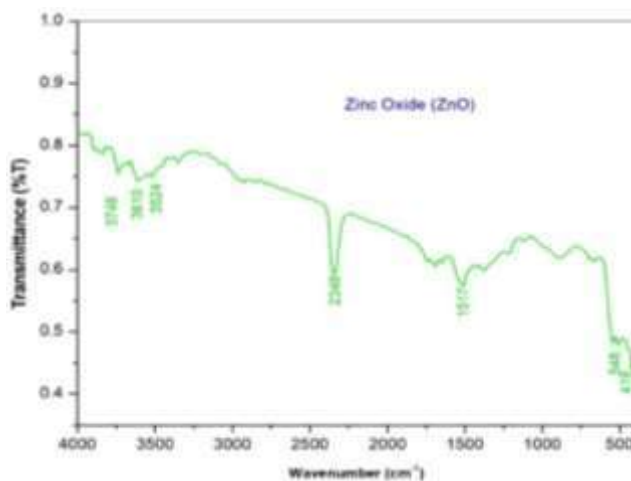


Figure 2: FT-IR Spectra of pure ZnO calcinated at 500 °C.

Scanning Electron Microscopy (SEM) and EDAX analysis

The SEM technique is used to study morphology of zinc oxide as illustrated in the following figure 3. Figure 3 illustrates that the SEM images zinc

oxide, prepared via the sol-gel technique reveal that the pure ZnO nanoparticles have a mostly spherical morphology with a little presence of rod-like structures, accompanied by minimal agglomeration. The average particle size of ZnO ranges from 20 nm to 44 nm. The

composition (elemental) and presence of metal was examined using EDAX techniques illustrated in figure 3.

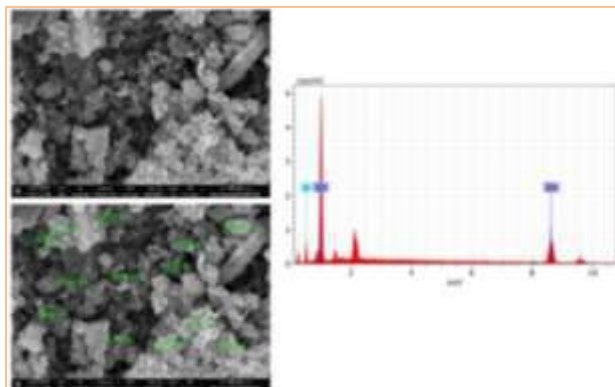


Figure 3: SEM image and EDS spectra of ZnO calcinated at 500°C.

Table 1: Atomic and weight percentage of Zn, O in zinc oxide (ZnO) calcinated at 500°C.

Element	Weight Percentage (%)	Atomic Percentage (%)
Zn	93.75	78.60
O	6.25	21.40
Total	100	100

The FE-SEM picture and EDS chemical mapping of ZnO are presented in Figure 4. Figure 4 illustrates a densely packed morphology of closely spherical to irregular nanograins with minimal agglomeration. The homogeneous surface distribution verifies the successful production of nanoscale ZnO. The

porous, coarse texture often provides a substantial surface area, advantageous for catalytic applications. The elemental mapping reveals a uniform distribution of Zn (green) and O (blue) without any impurity phases, confirming the purity and consistent composition of ZnO nanoparticles.

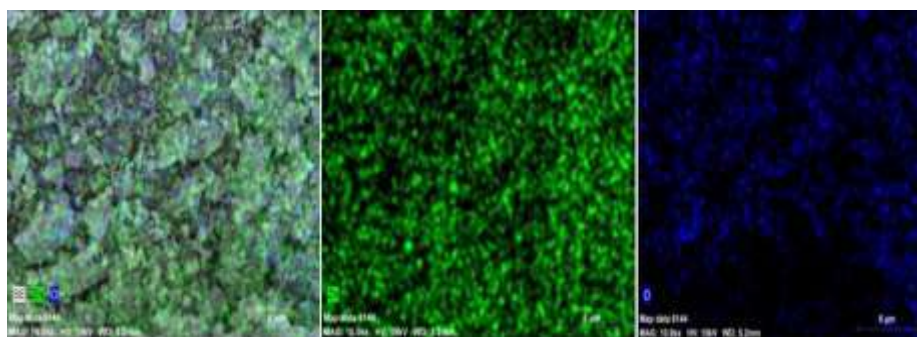


Figure 4 FE-SEM and EDS chemical mappings of ZnO calcinated at 500°C.

Experimental procedure for photo degradation of methylene blue:

This study employed methylene blue (MB) as a model dye for degradation via a ZnO photocatalyst. MB is a well-known cationic dye frequently employed in the textile sector. Methylene blue, or 3,7-bis(dimethylamino)-phenothiazine-5-in chloride (tetramethyl thionine), with 319.85 g/mol. molecular weight. The correlation between catalytic activity and pH was analyzed by assessing the breakdown of MB at five distinct pH levels. The degrading process was achieved under visible light (<400 nm) utilizing natural sunlight. In every experiment, 10 mg

of ZnO photocatalyst was mixed with 100 mL of MB solution of 5 mg/L, concentration. The solution pH was modified utilizing dilute H_2SO_4 or NaOH. To attain adsorption/desorption equilibrium, the mixture was agitated in the absence of light for 30 minutes. The photocatalytic degradation was thereafter commenced by exposing the suspension to sunshine. The experiment setup is represented in figure 5. Every 30 minutes, 3 mL samples were extracted, centrifuged, and examined via UV-Vis spectroscopy. The deterioration was observed by measuring the absorption peak at 660 nm.



Figure 5: Experimental setup for photocatalytic degradation of MB under sunlight

The photocatalytic oxidation of MB to H_2O and CO_2 is a crucial method for eliminating the dye from industrial wastewater. The process occurs in the presence of a photocatalyst, a semiconductor substance activated by

photon absorption, which can expedite a reaction without being depleted.

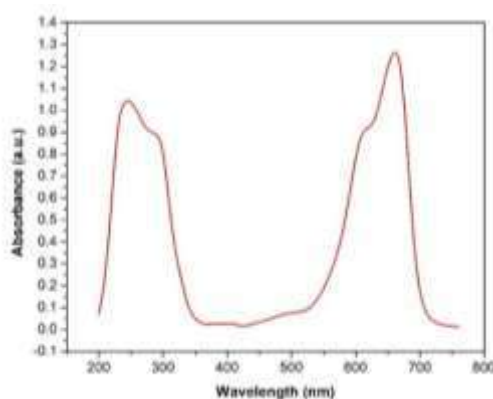
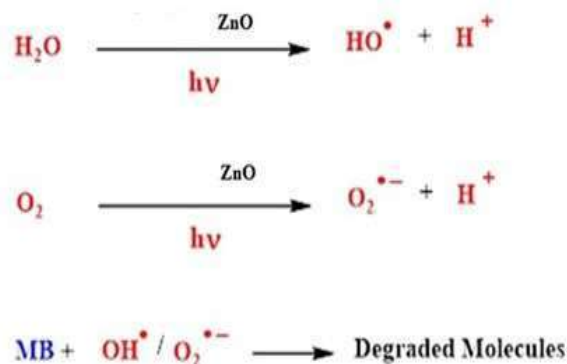


Figure 6: UV – visible spectrum of methylene blue

In photocatalytic process, photogenerated holes in the valence band oxidize surface-adsorbed H_2O or OH^- species to produce hydroxyl radicals ($\bullet OH$). Electrons in the conduction band, at the same time are captured by dissolved oxygen, leading to the formation of superoxide

radical anions ($O_2^{\bullet -}$). These reactive species participate in successive oxidation–reduction reactions and are mainly responsible for the breakdown of MB dye molecules. The possible reaction steps are as follows:



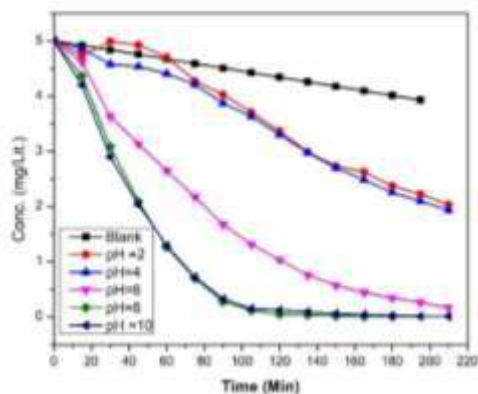


Figure 7: Photocatalytic breakdown of MB dye (Conc.Vs Time)

The photocatalytic breakdown of MB is conducted utilizing ZnO nanoparticles at different pH levels. To ascertain the UV-spectroscopic absorbance of MB, tests were conducted on the photocatalyst,

specifically ZnO nanoparticles (Figure 6). Methylene blue exhibits pronounced absorption bands at 291 nm and 660 nm, with the latter diminishing progressively as the irradiation duration is extended (Figure 6.)

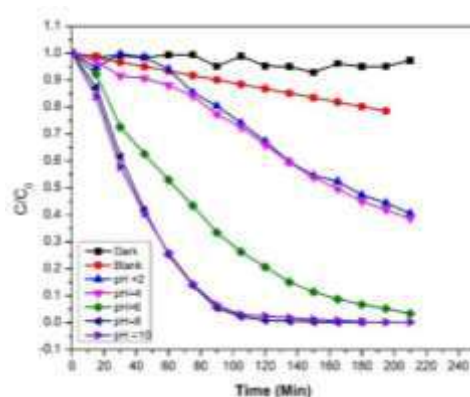


Figure 8: Photocatalytic degradation of methylene blue (C/C_0 Vs Time)

A photocatalyst can initiate the destruction or elimination of MB upon exposure to visible light. The decolonization of MB may be achieved through oxidative degradation or two-electron reduction. After 210 minutes, the quantity of methylene blue dye significantly

diminished. The degradation versus time graph indicates that pH levels of 8 and 10 exhibit superior degradation efficiency relative to pH levels of 2, 4, and 6 (Figures 7, 8, and 9).

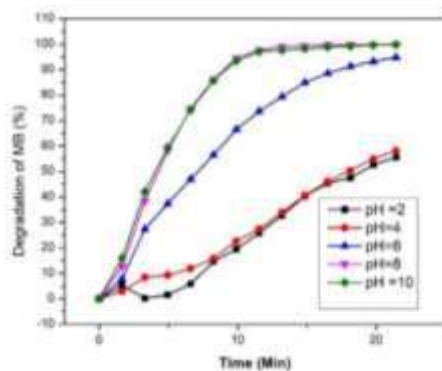


Figure 9: Photocatalytic degradation of methylene blue (% Degradation Vs Time)

The photocatalytic degradation potency of MB dye was determined by change in optical density (absorbance) using the following relation:

$$\text{Degradation \%} = \frac{C_0 - C_t}{C_0} \times 100$$

$$\text{Degradation \%} = \frac{A_0 - A_t}{A_0} \times 100$$

Where, C_0 is initial dye concentration and C_t is dye concentration after irradiation time t . A_0 = initial absorbance of MB solution before irradiation, A_t = absorbance of MB dye on irradiation time t . Both the forms are commonly used for photocatalytic removal of dyes.

Effect of Irradiation Time:

The exposure time and equilibrium adsorption between MB dye and the photocatalyst are important aspect are important in the photo catalytic degradation process. The methylene blue dye decomposition is

proportional exposure time. Thus, thereby increasing irradiation time or exposure decomposition of dye molecules also increases. The distinctive absorption peak of MB at 660 nm reduces slowly with time, by colour change from blue to colorless. The decrease in absorption is related to the breakdown of the MB chromosphere. Firstly, the photocatalytic decomposition of MB dye consistently increases with exposure time though, the breakdown rate ultimately stabilizes approaching to attainment of equilibrium. Figure 8 and 9 represents the impact of exposure time on MB dye degradation with the absorbance at 660 nm reflects reduction on time. Figure 10 illustrates the reduction in MB concentration, further substantiating the hypothesis that prolonged irradiation time augments degradation. Control experiments conducted without the photocatalyst (blank) and in the absence of light demonstrated that the MB concentration remained constant, underscoring the essential roles of both light and the ZnO catalyst for successful degradation.



Figure 10: M B degradation with irradiation time using ZnO photocatalyst.

The pH Effect on Photodegradation of Methylene Blue:

The pH significantly affects the characteristics of dyes and the processes of photocatalytic reactions, such as hydroxyl radical production, direct oxidation by photogenerated holes (h^+), and reduction through the conduction band. The surface charge of the photocatalyst fluctuates with pH, influencing its interaction with dyes. Methylene blue (MB) is a cationic dye, and its adsorption onto the photocatalyst is augmented when the catalyst surface possesses a large negative charge.

Figures 8 and 9 illustrate how pH of solution affects the photodegradation of MB. Under basic conditions, the photocatalyst attains a negative charge, resulting in enhanced adsorption of the positively charged MB molecules via electrostatic attraction. Conversely, under acidic circumstances, H^+ ions predominate and

compete with MB for adsorption sites on the catalyst surface, hence diminishing the dye's adsorption. This reduced adsorption restricts the interaction between hydroxyl radicals ($-OH$) and MB, hence diminishing the degradation efficiency.

At elevated pH levels, competition between $-OH$ and MB molecules is negligible it is because of repulsion of hydroxyl ions by the negatively charged catalyst surface, resulting in their increased presence in the solution. Consequently, MB adsorption is most effective in alkaline media, where the negative surface charge of the ZnO photocatalyst amplifies the electrostatic attraction between the catalyst nanoparticles and MB molecules. ZnO as a photocatalyst enhances the rate of MB dye photodegradation with increasing solution pH, shown in Table 2 and Figure 10.

Table 2: Photodegradation of MB dye after 210 min irradiation time.

pH	2	4	6	8	10
% Degradation	59.42	61.39	96.69	100	99.87



At pH = 8 of solution, with ZnO photocatalyst higher photodegradation of methylene blue dye (100 %) achieved.

Conclusion

A zinc oxide nanoparticle was easily fabricated using simple sol-gel auto-combustion route. From X-ray diffraction confirms the prepared ZnO exist in hexagonal crystalline phase without any impurity. The FT-IR spectra gave characteristics absorption peaks which indicates the formation of Zone nanoparticles. The SEM technique used to examine surface morphology of ZnO which is primarily spherical particles with minor rod-like geometry, along with small aggregation was noticed. From EDAX analytical techniques the presence of zinc (Zn) and Oxygen (O) element and its composition is verified. The photocatalytic decomposition of methylene blue dye reveals that of MB indicated that, under dark conditions with zinc oxide and in the absence of a photocatalyst (control) the MB dye concentration persisted unaffected. In the presence of ZnO photocatalyst under sun light, the rate of photo degradation of MB dye increased and this increase of photocatalytic degradation of MB strictly dependent upon the pH of solution. At pH = 8, 100% degradation efficiency of MB dye was achieved. Finally in short zinc oxide synthesized using simple sol-gel auto-combustion method are easy, cheap and safe to fabricate ZnO nanoparticle and the synthesized zinc oxide shows excellent photocatalytic breakdown of methylene blue dye implies that ZnO is a sustainable material for wastewater treatment.

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Nil.

Conflicts of interest

The authors declare that there are no conflicts of interest regarding the publication of this paper

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Original Article

Effect of La^{3+} Doped Ni-Co-Zn Nanoferrite on Optical and Electrical Properties

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Abstract

The influence of La^{3+} doping on the optical and electrical characteristics of Ni-Co-Zn nanoferrites was examined. A series of nanoferrite samples with the chemical formula $\text{Ni}_{0.4}\text{Co}_{0.4}\text{Zn}_{0.2}\text{La}_x\text{Fe}_{2-x}\text{O}_4$ ($x = 0.025, 0.050, 0.075, 0.100, \text{ and } 0.125$) was synthesized by the sol-gel process, ensuring optimal compositional uniformity and nanoscale particle development. The optical characteristics of the synthesized samples were analyzed by UV-Vis and FTIR spectroscopy. FTIR spectra shows a distinct absorption bands around 600 cm^{-1} , which indicates metal-oxygen vibrations in the spinel ferrite structure, and confirms the successful synthesis of ferrite. UV-Vis spectroscopy was done to know the optical absorption properties and the influence of La^{3+} substitution on the optical features. The two-probe approach was useful to investigate the electrical properties and assess the impact of lanthanum doping on electrical conductivity. The findings demonstrated that La^{3+} incorporation markedly influenced the optical and electrical characteristics of the produced nanoferrites.

Keywords: Ni-Co-Zn nanoferrite, UV, FTIR, dc resistivity, Sol gel method.

Introduction

Recently, researchers are interested in the field spinel ferrites due to its important unique dielectric, optical, magnetic and electrical properties. Spinel ferrite materials are frequently used for many applications due to their cubic spinel structure, good chemical stability, high electrical resistivity, and low eddy current losses. The spinel ferrites materials are used for many applications such as a sensor material, in microwave devices, in transformers, magnetic recording devices and high-frequency electronic devices. Nowadays, spinel ferrites materials and its optical, electrical and magnetic properties can be modified by doping with rare earths and transition metal ions into spinel ferrite lattice. The sol-gel auto-combustion method is simple and advantageous method because it gives very good stoichiometric ratio, homogeneous sample with purity, required low temperature, and fabricate material with equal particle-size distribution. This technique has produced lanthanum-substituted nickel ferrite nanoparticles which have been thoroughly investigated. The structural and electrical properties of the ferrites are improved as decrease in particle size with increasing lanthanum concentration [1]. X-ray diffraction (XRD), Fourier Transform Infrared (FTIR) spectroscopy, and scanning electron microscopy (SEM) have been used to study analytical structure of Ni-Co-Zn spinel ferrite nanoparticles prepared by the sol-gel auto-combustion method. It also provides important details about phase identification, lattice characteristics, and cation distribution inside the crystal lattice, the XRD spectra confirmed the formation of a single-phase cubic spinel structure. While SEM images show spherical structure of nanoparticles with aggregation due to their nanoscale dimensions, FTIR measurements confirmed the distinctive metal-oxygen vibrations linked to the spinel ferrite structure [2].

Due to their huge significance in electronic and communication equipment, ferrites' electrical characteristics have been deeply studied. The four-probe method has been used to analyze the conductivity properties of cobalt ferrite nanoparticles doped with lithium. The result shows that conductivity drops when lithium concentration drops, this means dopant ions plays an important role in charge transport processes. These ferrites are used as for microwave and high-frequency applications due to their advanced electrical characteristics [3].

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Similarly, this sol-gel method also used to prepare single-phase $\text{Ni}_x\text{Co}_{1-x}\text{Nd}_y\text{Fe}_{2-y}\text{O}_4$ nanoferrites having a cubic spinel structure. By the references on electrical research, resistivity decreases with increasing temperature but dc resistivity increases with increasing neodymium concentration. This behavior shows these ferrites' potential application in semiconducting and electrical devices and validates their semiconducting nature [4]. We also studied the dc resistivity and ac conductivity of chemically produced $\text{Cu}_x\text{Co}_{1-x}\text{Fe}_2\text{O}_4$ nanoferrites. The data shows that the ferrite system's electrical conduction behavior and charge transport mechanism were affected by the addition of copper ions [5]. In other study, $\text{Ni}_{1-x}\text{Cd}_x\text{Fe}_2\text{O}_4$ ferrites prepared by using the sol-gel auto-combustion method showed a decrease in dc resistivity as the temperature increases. Most of ferrite materials have semiconducting properties, as demonstrated by the reported temperature dependency of resistivity [6]. Numerous studies have also been conducted on Ni-Zn ferrites produced using the traditional solid-state reaction technique. The two-probe method of electrical tests showed that dc resistivity falls with increasing temperature. Additionally, it was noted that the Curie temperature drops as the zinc content rises, indicating the impact of cation substitution on the ferrite system's electrical and magnetic characteristics [7]. According to several studies the electrical conduction mechanism of ferrites is vary with temperature. The semiconducting nature of ferrites is confirmed by the dc resistivity generally decreases with increasing temperature because of increased electron hopping between Fe^{2+} and Fe^{3+} ions at the octahedral sites of the spinel lattice [8,9]. Now a days researchers pay attention on $\text{NiFe}_{2-x}\text{Gd}_x\text{O}_4$ ferrites synthesized by the sol-gel combustion method because it increases dc resistivity beyond $x = 0.1$, but magnetic properties decrease with increasing Gd^{3+} concentration. These results show the effect of rare-earth ion substitution significantly on ferrite nanoparticles' electrical and magnetic properties [10].

The study of review of the literature shows that the structural, magnetic, optical, and electrical properties of spinel ferrites are significantly affected by rare-earth and transition-metal substitutions. Thus, synthesizing and characterizing lanthanum-doped Ni-Co-Zn ferrite nanoparticles is crucial to comprehending their possible uses in optoelectronic, magnetic, and electrical devices.

Material And Method

$\text{Ni}_{0.4}\text{Co}_{0.4}\text{Zn}_{0.2}\text{La}_x\text{Fe}_{2-x}\text{O}_4$ nanoparticles ($x=0.025, 0.050, 0.075$, and 0.100) were prepared by the sol-gel auto combustion method. AR grade ferric nitrate ($\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$), nickel nitrate ($\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$), zinc nitrate ($\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$), cobalt nitrate ($\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$), citric acid ($\text{C}_6\text{H}_8\text{O}_7$), and ammonium hydroxide solution (NH_4OH). Distilled water was mixed with the stoichiometric ratio of all nitrates and citric acid. maintain the pH at 7, the ammonium hydroxide dropwise added while stirring. Sol was obtained; this solution was constantly agitated at 100°C for three to four hours. Auto-combustion done when sol converts in a thick brown gel. The fine powder of $\text{Ni}_{0.4}\text{Co}_{0.4}\text{Zn}_{0.2}\text{La}_x\text{Fe}_{2-x}\text{O}_4$ nanoferrite is finally obtained. This powder was calcinated at 600°C for four hours.

Result And Discussion

1. Optical Properties

(i) UV-Visible Spectroscopy:

In order to investigate optical behavior of synthesized $\text{Ni}_{0.4}\text{Co}_{0.4}\text{Zn}_{0.2}\text{La}_x\text{Fe}_{2-x}\text{O}_4$ nanoferrites ($x = 0.025, 0.050, 0.075$, and 0.100) by UV-Visible absorption spectra was recorded in the $200\text{--}800\text{ nm}$ range of wavelength. At lower wavelength in ultraviolet region UV spectra gave strong absorption. If the wavelength shifted towards visible region, then absorption intensity decreased for all composition of samples. This result indicates the synthesized nano ferrites, $\text{Ni}_{0.4}\text{Co}_{0.4}\text{Zn}_{0.2}\text{La}_x\text{Fe}_{2-x}\text{O}_4$ nanoferrites ($x = 0.025, 0.050, 0.075$, and 0) have excellent optical absorption at lower wavelengths. The optical behavior of ferrite materials is greatly influenced chemical composition, particle size, fabrication method, and distribution of cation within the spinel ferrite lattice. The electronic transitions between metal ions are altered when La^{3+} ions are incorporated into the Ni-Co-Zn ferrite structure, leading to changes in the absorbance properties. The band structure and optical sensitivity of the material are affected by La substitution, according to data analysis the observed variations in the absorption spectra occurs due to increasing lanthanum concentration. There is drop in absorption at higher wavelengths because of reduced photon energy and a lesser likelihood of electronic transitions. These results confirms that Ni-Co-Zn- $\text{La}_x\text{Fe}_{2-x}\text{O}_4$ nanoferrites' optical characteristics are composition-dependent and may be modified by lanthanum doping for possible optoelectronic uses. The band gap energy was calculated and it varies from 1.5060 eV to 1.5560 eV . This type of nanoferrite shows that the semiconductor behavior.

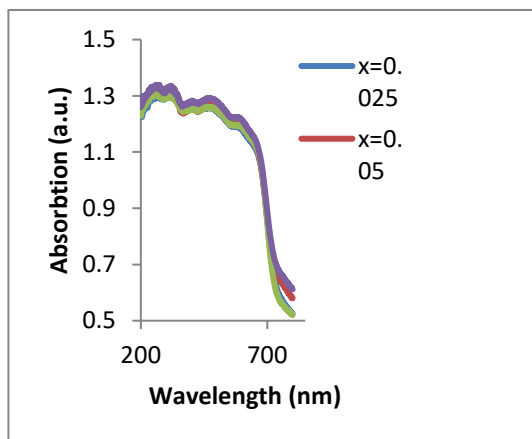


Fig.1: UV-Visible Spectrum of $\text{Ni}_{0.4}\text{Co}_{0.4}\text{Zn}_{0.2}\text{La}_x\text{Fe}_{2-x}\text{O}_4$ nanoferrite

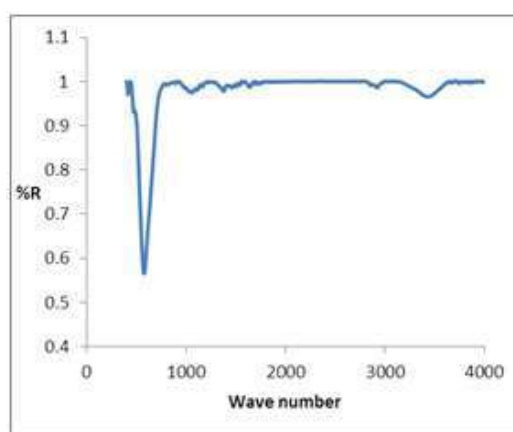


Fig.2: FTIR of $\text{Ni}_{0.4}\text{Co}_{0.4}\text{Zn}_{0.2}\text{La}_x\text{Fe}_{2-x}\text{O}_4$ nanoferrite at $x = 0.05$

(ii) Fourier Transform Infrared (FTIR) Spectroscopy:

The FTIR spectrum of the synthesized $\text{Ni}_{0.4}\text{Co}_{0.4}\text{Zn}_{0.2}\text{La}_x\text{Fe}_{2-x}\text{O}_4$ nanoferrite with $x = 0.05$ was recorded to analyse the spinel ferrite structure, which is given in Fig. 2. The spectrum shows a strong absorption band at $\sim 600 \text{ cm}^{-1}$, which is typical of spinel ferrites. The stretching vibrations of metal-oxygen (M-O) bonds at the spinel lattice's tetrahedral sites are responsible for this absorption band. The occurrence of these distinctive bands shows that the synthesized ferrite materials have the spinel crystal structure and confirms the ferrite phases successfully developed.

Electrical Properties

The dc electrical resistivity (ρ) of $\text{Ni}_{0.4}\text{Co}_{0.4}\text{Zn}_{0.2}\text{La}_x\text{Fe}_{2-x}\text{O}_4$ nanoferrites with $x = 0.025, 0.050, 0.075$, and 0.100 was determined using the two-probe method. This method is commonly used to investigate the electrical transport properties of ferrite materials and to understand how dopant concentration influences charge conduction. The change in dc resistivity with applied voltage at a constant temperature of 200°C is depicted in Figure 3. At lower voltages, it was discovered that all of the nano ferrite samples had comparatively high dc resistivity, which gradually decreased as the applied voltage increased. The resistance lowering with voltage indicates an increase in

electrical conductivity. The increased mobility or movement of charge carriers and improved electron hopping between Fe^{2+} and Fe^{3+} ions in the octahedral locations of the spinel lattice might be responsible for this behavior. Low resistivity may occur from charge carriers or electrons gaining enough energy to contribute more successfully to the conduction process as the applied voltage increases. Figure 4 shows the connection between dc resistivity and lanthanum concentration at 200°C . The result shows that as the La^{3+} content increases, dc resistivity systematically decreases. This decrease in resistivity suggests that the substitution of lanthanum has improved the electrical characteristics of the nano ferrite samples. Charge carriers can flow more easily when La^{3+} ions are incorporated into the ferrite lattice because they alter the cation distribution and charge transport mechanism. Higher lanthanum concentrations thereby encourage electrical conduction and lessen the material's total resistivity. The semiconducting characteristic of the produced $\text{Ni}_{0.4}\text{Co}_{0.4}\text{Zn}_{0.2}\text{La}_x\text{Fe}_{2-x}\text{O}_4$ nano ferrites is confirmed by the observed reduction in resistivity with increasing voltage and lanthanum content. These results imply that lanthanum doping significantly influences the material's electrical characteristics, making it a viable option for high-frequency and electronic device applications.

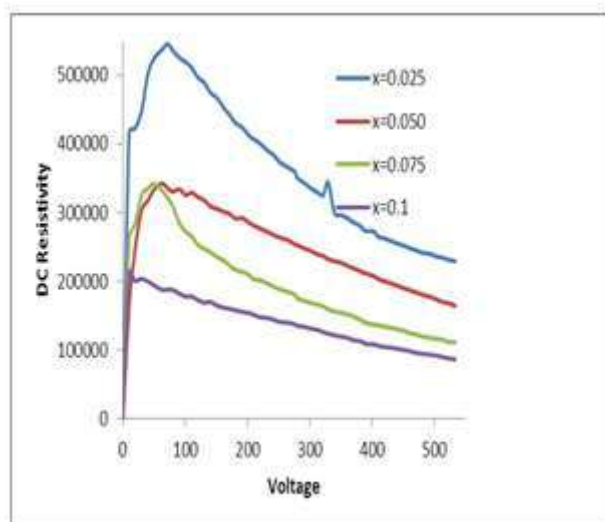


Fig.3: Plot of DC Resistivity with Voltage of $\text{Ni}_{0.4}\text{Co}_{0.4}\text{Zn}_{0.2}\text{La}_x\text{Fe}_{2-x}\text{O}_4$ nanoferrite at 2000°C

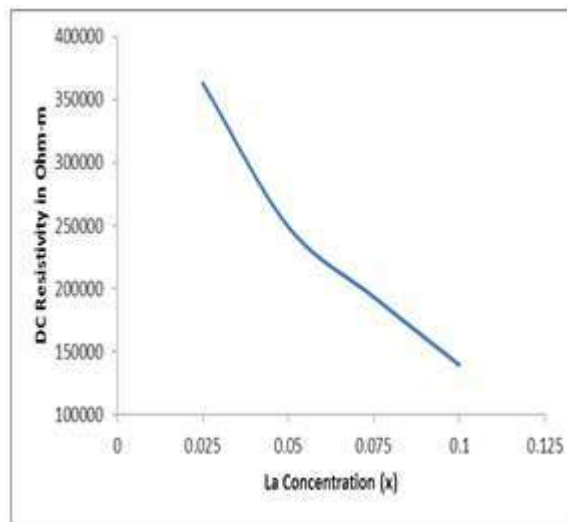


Fig.4: Plot of DC Resistivity with La Concentration of $\text{Ni}_{0.4}\text{Co}_{0.4}\text{Zn}_{0.2}\text{La}_x\text{Fe}_{2-x}\text{O}_4$ nanoferrite at 2000°C

Conclusion

Sol-gel auto combustion method was successfully used to synthesis of $\text{Ni}_{0.4}\text{Co}_{0.4}\text{Zn}_{0.2}\text{La}_x\text{Fe}_{2-x}\text{O}_4$ where $x=0.025, 0.050, 0.075$, and 0.100 nanoferrite. According to the UV-visible spectrum, as absorption decreases the wavelengths increases. And the range of the band gap energy is 1.5060 eV to 1.5560 eV . The nanoferrites shows semiconductor behavior. At 600 cm^{-1} , a significant absorption bond is observed, providing the spinel structure's distinctive bond. As voltage increases, $\text{Ni}_{0.4}\text{Co}_{0.4}\text{Zn}_{0.2}\text{La}_x\text{Fe}_{2-x}\text{O}_4$ nanoferrite's resistivity falls. The dc resistivity of $\text{Ni}_{0.4}\text{Co}_{0.4}\text{Zn}_{0.2}\text{La}_x\text{Fe}_{2-x}\text{O}_4$ nanoferrite reduces with increasing Lanthanum content.

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Original Article

Non-Alcoholic Fatty Liver Disease: Pathophysiology, Emerging Drug Delivery Strategies, Nanotechnology, and the Growing Role of Artificial Intelligence in Diagnosis and Management

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Abstract

Non-alcoholic fatty liver disease, widely known as NAFLD, has quietly become one of the most widespread chronic diseases of our time. Roughly one in four adults around the world is believed to carry some form of this condition [1], a figure that keeps climbing alongside the global rise in obesity, sedentary lifestyles, and metabolic disorders like type 2 diabetes. What makes NAFLD particularly challenging to manage is that it does not sit still — it moves along a disease continuum, starting from harmless fat build-up in liver cells and potentially advancing, in a significant subset of patients, to a far more dangerous inflammatory state called non-alcoholic steatohepatitis or NASH [2]. From there, the road can lead to cirrhosis and even liver cancer. For many years, clinicians had little to offer beyond lifestyle advice, but the 2024 approval of resmetirom changed that picture considerably [3]. This review takes a broad and critical look at NAFLD — examining the biology of disease progression in depth, surveying the pharmacological landscape including newer agents, and exploring two rapidly evolving frontiers: nanoparticle-based drug delivery systems that can carry medicines directly to diseased liver cells with precision [4,5], and artificial intelligence tools that are beginning to transform how we diagnose, stage, and treat this condition [6]. The authors also address the practical hurdles that stand between these promising innovations and real-world clinical use.

Keywords: Non-alcoholic fatty liver disease; NASH; hepatic steatosis; drug delivery; nanotechnology; lipid nanoparticles; artificial intelligence; machine learning; liver-targeted therapy; metabolic syndrome.

Introduction

Among the many organs that quietly sustain human life, the liver carries a particularly heavy burden. It filters blood, metabolizes nutrients, synthesizes proteins, and regulates fat storage — tasks that place it at the intersection of nearly every major metabolic pathway in the body. Non-alcoholic fatty liver disease is the clinical expression of metabolic disruption in the liver, and it has grown into one of the most significant public health concerns of the twenty-first century. Prevalence estimates suggest that approximately one quarter of the global adult population lives with some form of NAFLD, with particularly high rates observed in South and East Asia, the Middle East, and parts of Latin America [1].

To understand NAFLD is to understand a spectrum rather than a single disease. At one end lies simple fatty liver — an accumulation of lipid droplets within hepatocytes that may remain medically silent for years. At the other end lies cirrhosis and hepatocellular carcinoma, conditions that carry grim prognoses and limited treatment options [2]. The pivotal middle ground is NASH, a state in which fat accumulation is accompanied by active liver cell injury, inflammation, and scarring. Identifying patients who are on track to progress through this continuum — and intervening before irreversible damage occurs — remains one of the central challenges in hepatology today [3]. Until recently, the absence of approved pharmacological therapies for NAFLD forced clinicians into a frustrating position: recommending lifestyle changes that most patients struggle to maintain, while watchfully waiting for disease to progress. The regulatory approval of resmetirom in 2024 offered the first real pharmacological option for patients with at-risk NASH [4]. Yet this single approval does not fully address the heterogeneity of disease presentations or the range of pathogenic mechanisms involved.

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Two fields — nanotechnology and artificial intelligence — are increasingly recognized as holding the keys to a more comprehensive solution [4,6]. This review synthesizes what is currently known across all these dimensions and tries to articulate where the field is heading.

Pathophysiology and Molecular Mechanisms of NAFLD

1. Moving Beyond the Two-Hit Model

Early attempts to explain how NAFLD progresses drew on a simple framework: a first hit causes fat to accumulate in the liver, and a second hit — typically oxidative injury — tips that fat-laden liver into active inflammation and damage. However, as research deepened, the model began to show its limitations [2]. Today, most researchers favor a multi-hit framework that recognizes NAFLD as the product of numerous simultaneous and interacting biological insults: excess fat delivery to the liver, systemic insulin resistance, dysregulation of gut bacteria, genetic predisposition, endoplasmic reticulum stress, and impaired fat clearance, all converging on the same organ at the same time [2,8]. These factors do not take turns; they act together, and their interactions are what drive the variable and often unpredictable clinical course of the disease.

2. How Fat Accumulates and Why It Becomes Harmful

The liver is not supposed to be a fat storage organ. When fat arrives faster than the liver can process it, it accumulates within hepatocytes as lipid droplets — a state called steatosis. This excess fat enters the liver from three main directions: fat released by adipose tissue during obesity-related insulin resistance, dietary fats absorbed from the gut, and fat manufactured by the liver itself through *de novo* lipogenesis [2]. The real danger is not the lipid droplets themselves but the toxic molecular species generated from excess fat — things like ceramides, diacylglycerol, and lysophosphatidylcholine. These molecules switch on stress pathways inside hepatocytes, trigger inflammation, damage mitochondria, and ultimately drive cell death. It is this lipotoxicity, rather than steatosis per se, that appears to initiate the progression from simple fatty liver to NASH [1].

3. The Role of Insulin Resistance

Insulin resistance sits at the heart of NAFLD in a way that is both cause and consequence — a feedback loop that makes the disease self-perpetuating. When tissues stop responding normally to insulin, adipose cells release fatty acids at an uncontrolled rate, flooding the liver with raw material for fat synthesis. The liver simultaneously loses the ability to suppress glucose production while retaining — or even enhancing — its sensitivity to insulin's fat-building signals [2]. As fat accumulates in the liver, it further degrades insulin signaling in surrounding tissues, worsening the systemic resistance that started the problem in the first place. Genetic factors add another layer of complexity; variants in genes such as PNPLA3, TM6SF2, and GCKR are strongly associated with increased NAFLD risk and partly explain why some individuals develop severe disease despite having metabolic profiles that look unremarkable [2].

4. Oxidative Stress and the Inflammatory Response

When a fatty liver is forced to burn large quantities of fat for energy, the mitochondria responsible for that process produce reactive oxygen species as a byproduct. In steatotic hepatocytes, the sheer volume of fat oxidation generates oxidants faster than they can be neutralized, and

the resulting oxidative stress begins to damage cellular structures — lipids in membranes, proteins in metabolic pathways, and DNA itself [2]. These damaged molecules act as distress signals that activate Kupffer cells, which respond by secreting inflammatory mediators including tumor necrosis factor- α and the interleukins. This inflammatory response, when sustained over years, activates hepatic stellate cells that begin producing scar tissue — collagen and fibrous proteins that progressively replace functional liver architecture. This fibrosis is the final common pathway through which NAFLD destroys liver function, and fibrosis stage is the strongest predictor of liver-related morbidity and mortality [1].

5. The Gut-Liver Connection

One of the more surprising discoveries in NAFLD research over the past decade has been the importance of the gut microbiome in shaping liver disease. The gut and liver are anatomically linked by the portal vein, which carries blood from the intestinal tract directly to the liver. In many patients with NAFLD, the intestinal lining becomes compromised, allowing bacterial products — especially lipopolysaccharide — to leak into the portal circulation and activate innate immune receptors in the liver [2]. Beyond this direct inflammatory effect, the microbiome also shapes the liver's metabolic environment by influencing how bile acids are processed, how dietary choline is metabolized, and even by producing small amounts of ethanol through fermentation. Dysbiosis — an imbalance in gut microbial community composition — appears to make all of these effects worse, and there is growing interest in whether restoring microbial balance might offer a therapeutic entry point for NAFLD [2].

Current and Emerging Pharmacological Treatment Strategies

1. The Case for Lifestyle Change — and Its Limits

Before any medication enters the picture, the most fundamental treatment for NAFLD remains the same as it has always been: lose weight, eat better, and move more. Studies have shown that losing between five and ten percent of body weight produces measurable improvements in liver fat, inflammation scores, and even early fibrosis. Lose more than ten percent, and a meaningful fraction of patients achieve something approaching remission — NASH resolves, fibrosis regresses [1]. Mediterranean-style diets, rich in olive oil, vegetables, legumes, and whole grains, have shown particular benefit because of their antioxidant content and their ability to reduce the dietary fat and sugar load that drives hepatic lipid synthesis. The problem is that sustained lifestyle change is one of the hardest things a person can be asked to do, and the gap between what lifestyle modification can achieve and what patients can actually sustain is precisely the space that pharmacological therapy is being asked to fill [2].

2. A New Era of Drug Treatment

The approval of resmetirom — marketed as Rezdiffra — by the US Food and Drug Administration in 2024 was genuinely significant for this field. Resmetirom works by selectively activating a thyroid hormone receptor beta subtype in liver cells, controlling fatty acid metabolism without triggering the heart and bone effects that made older thyromimetic drugs dangerous. Clinical trials showed that patients with NASH and moderate-to-severe fibrosis who took resmetirom for a year were significantly more

likely to show NASH resolution and fibrosis improvement than those who received placebo [3] — the first time any drug had met both of these endpoints simultaneously in a Phase III trial. Beyond resmetirom, a pipeline of agents is advancing through clinical development: obeticholic acid acts on farnesoid X receptors to regulate bile acid and lipid metabolism; lanifibranor activates pan-PPARs to collectively reduce liver fat, inflammation, and fibrosis; and semaglutide, a GLP-1 agonist already established in diabetes and obesity, has shown liver-specific benefits that have prompted dedicated NASH trials [4]. The mechanistic diversity of these candidates suggests that combination regimens targeting multiple pathways simultaneously may eventually offer the durable remissions that monotherapy is unlikely to achieve.

3. Why Getting Drugs to the Liver Is Harder Than It Sounds

Even when an effective drug molecule exists, delivering it precisely to the diseased cells in the liver is a formidable pharmaceutical challenge. Drugs taken orally pass through the intestinal wall and enter the portal circulation before reaching the liver, where a portion is metabolized before reaching systemic circulation — the well-known first-pass effect. Drugs given intravenously face a different problem: Kupffer cells are highly efficient at clearing particles from the blood, taking up a significant fraction of an administered dose before it can reach its intended target [5]. Drug distribution to the specific hepatic cell populations most relevant to disease pathogenesis — hepatocytes, stellate cells, and Kupffer cells — remains non-selective in most conventional formulations, resulting in off-target exposure and dose-limiting toxicity. These biological barriers have increasingly motivated the application of nanotechnology as a means of achieving precise, cell-type-specific hepatic drug delivery [6].

Nanotechnology and Nanoparticle-Based Drug Delivery in NAFLD

1. Why the Liver Is Actually Well-Suited to Nanoparticle Delivery

There is something almost poetic about the fact that the same features of liver anatomy that complicate conventional drug delivery actually make it a particularly suitable organ for nanoparticle-based approaches. The blood vessels lining the liver's sinusoids are perforated by small gaps called fenestrations, with typical diameters in the range of 100 to 200 nanometers [7]. Nanoparticles engineered within this size range can access hepatocytes directly through these fenestrations, while larger particles remain confined to the vessel lumen. By coating nanoparticle surfaces with polyethylene glycol, engineers can shield particles from Kupffer cell recognition and extend their circulation time. By attaching targeting ligands — small molecules that bind to receptors expressed specifically on hepatocytes, such as the asialoglycoprotein receptor — they can direct nanoparticles to specific cell types within the liver with a degree of selectivity that no conventional formulation can match [8].

2. Lipid Nanoparticles — From Vaccines to the NAFLD Liver

Lipid nanoparticles attracted enormous public attention during the COVID-19 pandemic as the delivery vehicles for mRNA vaccines, but their potential in liver disease has been recognized by researchers for considerably

longer. These particles are composed of lipid components that naturally merge with cellular membranes, facilitating the release of their cargo inside cells — a property that makes them particularly effective for delivering nucleic acid therapeutics like small interfering RNA and messenger RNA [9]. In the context of NAFLD, lipid nanoparticle systems have been used to deliver mRNA encoding hepatocyte growth factor, a protein that supports liver regeneration, achieving measurable reductions in steatosis in animal models. Separate work has explored lipid nanocarriers incorporating vitamin D3, targeting the gut-liver axis by reinforcing intestinal barrier integrity and thereby reducing the bacterial product leakage that drives hepatic inflammation [5]. The clinical maturity of lipid nanoparticle technology — already proven safe at scale in humans — gives these applications a translational pathway that more novel nanocarrier platforms have yet to establish.

3. Polymeric Nanoparticles for Sustained and Selective Delivery

Biodegradable polymeric nanoparticles, most commonly built from poly (lactic-co-glycolic acid), offer a different set of advantages. PLGA breaks down gradually inside the body into lactic acid and glycolic acid — both normal metabolic byproducts — releasing its drug payload over days to weeks in a controlled fashion [5]. Researchers have exploited the preferential uptake of PLGA nanoparticles by Kupffer cells to deliver anti-inflammatory drugs directly to the cells most responsible for sustaining hepatic inflammation. In one well-characterized approach, PLGA particles loaded with a spleen tyrosine kinase inhibitor were taken up selectively by Kupffer cells, dampening their inflammatory activity and producing significant improvement in NASH histology in rodent models [4]. Another line of research has used rapamycin-loaded PLGA nanoparticles to inhibit the mTOR pathway in hepatocytes, reducing both lipid accumulation and inflammatory signaling simultaneously. The capacity to independently tune particle size, surface charge, and drug release kinetics affords polymeric systems a degree of formulation flexibility that is particularly advantageous given the cellular and mechanistic heterogeneity of NAFLD [1].

4. Smart Nanoparticles That Respond to Disease Signals

Perhaps the most intellectually exciting development in this field is the emergence of stimuli-responsive nanoparticles — drug carriers that remain inert in healthy tissue but release their payload in response to specific pathological signals present at the disease site. The NASH liver is characterized by elevated levels of reactive oxygen species, generated as a byproduct of the excessive fat oxidation described earlier [2]. Engineers have designed nanoparticles with structural bonds that are specifically cleaved by ROS, so that drug release occurs preferentially in the oxidative microenvironment of the diseased liver and not in healthy surrounding tissue [4]. Butyric acid-releasing particles of this type have demonstrated measurable therapeutic effects in NASH animal models, triggering fat metabolism pathways and reducing lipid accumulation at precisely the locations where ROS levels are highest. Dual-purpose nanoconstructs that combine a therapeutic agent with a diagnostic imaging agent — for example, iron oxide nanoparticles that can be visualized by MRI while simultaneously delivering an anti-fibrotic drug — represent



yet another frontier, pointing toward a future in which treatment and monitoring are conducted by the same platform [5].

Artificial Intelligence and Machine Learning in NAFLD Diagnosis and Management

1. Teaching Computers to Read Liver Biopsies

A liver biopsy remains the most definitive way to assess what is happening in NAFLD. A small sample of liver tissue, examined under a microscope, can reveal the degree of fat accumulation, the extent of inflammatory damage, the severity of hepatocyte injury, and how much scar tissue has formed [6]. The problem is that translating what is seen under the microscope into a standardized score involves a degree of judgment that varies between pathologists, between institutions, and even within the same pathologist on different days. Artificial intelligence, in the form of deep learning algorithms trained on thousands of digitized biopsy images, is beginning to offer something better: computerized image analysis that is fully reproducible and capable of extracting quantitative measurements that capture pathological severity more precisely than any ordinal score [6]. Studies have shown that well-trained neural networks can match or exceed expert hepatopathologists in detecting individual NASH features, and the prospect of using AI-derived quantitative endpoints in clinical trials — as more sensitive measures of drug efficacy — is already being seriously discussed by regulatory agencies.

2. Finding NAFLD Without a Needle

Liver biopsy, for all its diagnostic power, is an invasive procedure that carries real risks and is impractical for population-level screening or frequent longitudinal monitoring. Machine learning is changing this picture by enabling the simultaneous analysis of dozens or hundreds of variables — laboratory values, imaging-derived measurements, clinical parameters, genetic markers, gut microbiome profiles, and metabolomics data — to construct composite diagnostic models that outperform any individual marker [6]. These models do not require the researcher to know in advance which variables matter most; they discover the most informative combinations directly from the data. Clustering algorithms applied to multi-omics datasets are also revealing that NAFLD may in fact encompass several biologically distinct subtypes with different dominant mechanisms, different rates of progression, and different responses to specific therapeutic targets [7]. Identifying which subtype a particular patient has could eventually guide precision treatment selection in a way that current generic staging systems cannot.

3. Using AI to Find Better Drugs Faster

Drug development for NAFLD has historically been a slow and expensive process, with a high proportion of agents that show impressive results in animal models ultimately failing to demonstrate efficacy in human trials. Part of this failure can be attributed to the challenge of selecting the right patients for trials [7]. AI tools trained on clinical and molecular data from completed trials are being used to identify patient characteristics — genetic profiles, biomarker patterns, histological features — that are most predictive of response to specific therapeutic mechanisms, enabling enriched trial designs that improve the probability of detecting a real treatment effect [6]. On the earlier end of the drug development pipeline, generative AI models are

proposing novel molecular structures with optimized properties — high binding affinity for NAFLD targets, favorable metabolic stability, and acceptable toxicity — reducing the time between target identification and candidate selection. The aggregate effect of these AI applications could substantially compress the timeline from scientific discovery to approved treatment [6].

4. Personalizing Care Through Predictive Modeling

One of the most clinically consequential applications of AI in NAFLD is the prediction of individual disease trajectories. As any hepatologist will attest, NAFLD is unpredictable at the individual level: some patients with advanced fibrosis remain clinically stable for years, while others with apparently mild disease progress rapidly to cirrhosis [7]. Machine learning prognostic models that incorporate longitudinal data — sequential measurements of liver stiffness, biomarker trends, treatment adherence, and lifestyle variables over time — are showing superior ability to identify patients at highest risk of rapid progression compared to conventional clinical scoring tools [6]. Such models allow clinical resources to be directed toward those who will benefit most from early aggressive intervention. The long-term vision is a precision medicine framework in which AI-derived patient profiles direct individualized therapeutic selection, dosing, and follow-up schedules — an aspiration that the current convergence of clinical informatics, multi-omics science, and machine learning capability is beginning to make achievable [7].

Challenges and Regulatory Considerations

1. The Long Road from Mouse Models to Human Patients

The history of hepatology is littered with promising treatments that worked beautifully in mice and failed in humans, and NAFLD nanomedicine is not immune to this risk. Mouse livers differ from human livers in ways that are subtle but consequential — the density of Kupffer cells, the architecture of sinusoidal fenestrations, and the metabolic response to high-fat feeding all differ in ways that can make rodent efficacy data misleading [8]. Beyond these biological differences, the path from a laboratory batch of nanoparticles to a pharmaceutical-grade product manufactured consistently at scale involves formidable technical and regulatory challenges: ensuring batch-to-batch uniformity, achieving long-term colloidal stability, and generating the comprehensive toxicology and pharmacokinetic data packages that regulatory agencies require before human trials can begin [8]. These challenges require resources and expertise that demand sustained industry partnership to advance.

2. Making AI Trustworthy Enough for Clinical Use

An AI tool that performs brilliantly on a test dataset drawn from the same clinical center that generated the training data may perform considerably less well when applied to patients from a different hospital, a different country, or a different demographic group [6]. Addressing this generalizability problem requires prospective external validation studies conducted in populations that differ meaningfully from the training cohort — something that demands coordinated effort across multiple institutions and significant investment in data sharing infrastructure. Beyond performance generalizability, clinicians need to be able to understand why an AI model has made a particular prediction or recommendation. This explainability

requirement is both a scientific challenge and an ethical one: a physician cannot responsibly delegate clinical decisions to a system whose reasoning is opaque, no matter how impressive its aggregate performance metrics may appear [7].

3. Who Benefits — and Who Gets Left Behind

There is a troubling irony embedded in the promise of AI-powered and nanotechnology-enhanced NAFLD care. The populations most burdened by this disease — lower-income communities, ethnic minorities with high genetic susceptibility, populations in low- and middle-income countries — are precisely the populations least likely to be represented in the research datasets used to train AI models [6]. AI diagnostic tools trained predominantly on data from well-resourced academic medical centers may perform less accurately in the populations with the greatest disease burden, creating a technological equity gap that mirrors and amplifies existing healthcare disparities. Addressing this challenge requires deliberate effort at every stage of the research and development process, and advocating for pricing and access models that do not restrict advanced therapies to the wealthy [8].

Future Perspectives

The trajectory of NAFLD research and treatment over the next decade is likely to be shaped by the deepening integration of precision diagnostics, targeted therapeutics, and intelligent computational frameworks [7]. On the nanotechnology front, the development of hepatic stellate cell-targeted nanocarriers — exploiting surface ligands with affinity for vitamin A receptors or platelet-derived growth factor receptor beta, which are upregulated on activated stellate cells — holds particular promise for anti-fibrotic applications, potentially enabling delivery of RNA-based therapeutics that silence pro-fibrotic gene expression [1]. Exosome-mimetic and cell-membrane-coated nanoparticles, which leverage endogenous cellular recognition mechanisms to evade immune clearance and achieve biologically guided targeting, represent an emerging frontier that may overcome immunogenicity barriers limiting conventional synthetic nanocarriers [4].

On the AI front, the convergence of large language models, federated learning architectures — which enable model training across distributed clinical datasets without centralizing sensitive patient data — and multi-omics integration platforms is expected to substantially enhance the biological depth and clinical utility of computational NAFLD tools [6]. Digital twin technologies, in which patient-specific computational models calibrated on longitudinal clinical and omics data simulate individual disease trajectories and therapeutic responses, offer a particularly ambitious vision of personalized NAFLD management [7]. The coming decade is also likely to witness the emergence of companion diagnostic AI tools co-developed alongside targeted nanomedicines, enabling patient stratification based on molecular features most predictive of response — a paradigm shift from one-size-fits-all to molecularly guided NAFLD therapy [6].

Conclusion

Non-alcoholic fatty liver disease is, in many ways, a disease that reflects the world we have built — one of nutritional excess, physical inactivity, chronic metabolic stress, and widening health inequalities. The molecular

science of NAFLD has matured considerably, and we now have a reasonably clear picture of why fat accumulates in the liver, how that fat becomes injurious, and what drives the inflammatory and fibrotic cascades that turn a benign condition into a potentially fatal one [2]. What has lagged behind is our ability to translate that mechanistic knowledge into effective clinical interventions — and it is here that the developments reviewed in this article become most significant. Nanoparticle-based delivery systems offer a way to put the right drug in the right place at the right time, bypassing the biological barriers that have made conventional hepatic pharmacotherapy so frustratingly imprecise [4,5]. Artificial intelligence offers a way to see patterns in complex, multi-dimensional clinical data that would be invisible to human analysis, enabling earlier and more accurate diagnosis, smarter trial design, and eventually individualized therapeutic decisions [6]. The foundations are in place, and the direction of travel is clear: toward a future in which NAFLD is not merely managed but genuinely understood and effectively treated, one patient at a time.

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The authors declare that there are no conflicts of interest regarding the publication of this paper

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Original Article

Cyanuric chloride as highly efficient catalyst for esterification reaction.

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Abstract

Using cyanuric chloride, an effective and solvent-free esterification reaction between alcohols and acetic acid has been developed. High yields, fast reaction times, and room temperature conditions are benefits of this method. The development of sustainable and efficient methodologies for esterification reactions remains an important objective in organic synthesis. In the present study, cyanuric chloride has been employed as an effective Lewis acid catalyst for the solvent-free esterification of a wide range of alcohols with acetic acid under ambient conditions. The optimized reaction protocol utilizes 10 mol% of cyanuric chloride, affording the corresponding acetate esters in excellent yields within 25–35 minutes. The method eliminates the need for hazardous organic solvents and harsh reaction conditions, making it environmentally benign and economically attractive. Various substituted aromatic and aliphatic alcohols were successfully converted into their respective esters with high selectivity and without the formation of significant by-products. The catalyst demonstrated superior performance compared with several conventional Lewis acids under identical reaction conditions. The protocol offers operational simplicity, short reaction times, high product yields, and broad substrate compatibility, making it a practical and green alternative for ester synthesis in both laboratory and industrial applications.

Keywords: Cyanuric chloride; Esterification; Green chemistry; Solvent-free synthesis; Acetic acid; Lewis acid catalyst; Acetate esters; Organic synthesis; Sustainable catalysis; Room-temperature reaction.

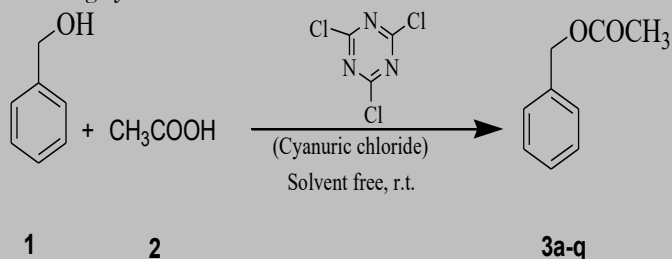
Introduction

In organic chemistry, esterification of alcohols with carboxylic acids is a basic and frequently used functional reaction.¹ Transesterification or the solication of acylating/formylating agents is how the reaction proceeds.² Since the only byproduct of the esterification reaction is water, the use of acetic acid as an acetylating agent is crucial from an environmental viewpoint. When compared to the use of anhydrides or acid chlorides, this approach is primarily useful for industrial requests.³ Alcohols and acid have undergone an esterification reaction using a multiplicity of catalytic devices.^{6–17} However, some of these include the requirement for a large amount of catalyst and challenging reaction conditions. In this reaction, ionic liquid can also be utilised as a solvent and catalyst.^{3b} Ionic liquids are too expensive and unsuitable from an industrial perspective, despite being one of the environmentally friendly esterification reaction methods.⁴ Esters are used to make flavors, pharmaceuticals, fragrances, food preservers, solvents, cosmetics, and plasticizers.⁵

The majority of these catalysts (metal triflates) are either extremely costly or water-sensitive, and all of these procedures made use of organic solvents.^{18–24} Organic solvents are flammable and expensive, an issue to the environment. Rajabi et al. studied into the supported iron oxide nanoparticles' acetylation.²⁷ Heraviet and associates. described the esterification process using Fe(ClO₄)₃·6H₂O.²⁵ Here, we investigated green Fischer esterification using cyanuric chloride as a catalyst as part of our ongoing research into developing new techniques.

Present method

Here, we need to share our research on the room-temperature, solvent-free esterification of various alcohols with acetic acid using cyanuric chloride.



Scheme 1: Acetic acid esterification of alcohols.

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Reaction conditions: Alcohol (1 mmol), acetic acid (3 mmol), and cyanuric chloride (10 mol%) were mixed for 30 to 40 minutes at room temperature and solvent free.

Result and discussion:

Cyanuric chloride-based reagents have gained importance in recent years as adaptable reaction media for a variety of organic transformations, including epoxide hydrolysis and regio- and stereoselective alcoholysis²⁶. In contrast, cyanuric chloride is among the most accessible, making it one of the most affordable and eco-friendly choices. Our goal is to provide a simple and efficient way to acetylate alcohols using acetic acid and a catalytic amount of cyanuric chloride Scheme: 1. In a model reaction involving

benzyl alcohol 1a (1 mmol) and acetic acid 2 (3 mmol) under solvent-free conditions to produce benzyl acetate 3a, various Lewis acids were assessed (Table 1). TLC analysis failed to detect the presence of the product when the reaction took place in the absence of a catalyst (entry 1 in Table 1). using common Lewis acids, like BF_3 , at catalytic concentrations of 5 mol%. With yields of 10%, 16%, 17%, 19%, 25%, 20%, and 21%, respectively, Et_2O , SnCl_2 , TiCl_4 , BiCl_3 , InF_3 , $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$, and ZnBr_2 produced the desired product (Table 1, entries 2-8). It was found that cyanuric chloride was the most efficient way to boost the reaction (Table 1, entry 10).

Table 1 shows how the catalyst affects the reaction between 1a and 2 to produce benzyl acetate 3a (Scheme 1)a.

Entry	Catalyst	Catalyst mol %	Time h/min	percent(%) of yield
1 ^b	None	--	45	0
2	$\text{BF}_3 \cdot \text{Et}_2\text{O}$	5	46	10
3	SnCl_2	5	45	16
4	TiCl_4	5	50	17
5	BiCl_3	5	21	19
6	InF_3	5	22	25
7	$\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$	5	18	20
8	ZnBr_2	5	25	21
9	Cyanuric Chloride	5	65	70
10.	Cyanuric Chloride	10	25	91

aUnless otherwise noted, the following reaction conditions were employed: room temperature, solvent-free, benzyl alcohol 1a (1 mmol), acetic acid 2 (3 mmol), and cyanuric chloride (5 mol percent). Condition of bReaction: 50°C.

cAt room temperature, a 10 mole percent catalyst is employed.

The chosen catalyst, cyanuric chloride, was then optimized using a traditional method to determine the highest yield with the shortest reaction time. Catalyst loadings of 0, 1, 2, 5, 10, 15, and 20 cyanuric chloride were employed. Scheme 1 illustrates that, of all these different concentrations, 10 mol percent produced the most efficient value with a maximum yield of 91 percent in 25–35 minutes of reaction time. The yield value rises gradually, but it is known to fall at catalyst concentrations greater than 15

mol%.

After optimizing catalyst loading, we acylated benzyl alcohol 1a (1 mmol) using 10 mol percent cyanuric chloride and acetic acid 2 (3 mmol) in a variety of solvents, including DMSO, DMF, acetonitrile, toluene, THF, methanol, and dioxane. The results showed that the reaction time was extended (40–60 min) in all solvents and that a reduction was seen at room temperature under unimpaired conditions (Table 1, entry 10).

Table 2: Optimization of reaction conditions

Entry	Solvent	Time (min)	Yield (%)
1	DMSO	45	65
2	DMF	45	60
3	CH_3CN	60	50
4	Toluene	60	45
5	THF	40	55
6	CH_3OH	40	60
7	Dioxane	60	45

A variety of structural alcohols were treated with acetic acid. (Table 2, entries 1-7) were exposed to acetic

acid to generate high yields of the corresponding acetates. At room temperature, the reactions proceeded



without the need for solvents. Table 3's results clearly illustrate the protocol's applicability and breadth in relation to different types of alcohols. The reactions went very well and there were no byproducts. By reacting the

substrates with chloro, methyl, methoxy, and nitro groups, the tolerance of different functional groups under the current reaction conditions has been investigated.

Table 3: Synthesis of esters using alcohols and acetic acid.

Entry	Benzyl alcohols	Product	Product	Time h/min	Yield (%) ^{a,b}
1.			3a	/30	91
2.			3b	/35	89
3.			3c	/25	90
4.			3d	/22	91
5.			3e	/23	88
6.			3f	/30	86
7.			3g	/27	87
8.			3h	/35	80
9.			3i	/37	81
10.			3j	/27	87
11.			3k	/22	89
12.			3l	/25	85
13.			3m	/30	80
14.	1-hexenol		3n	/16	88
15.	1-octanol		3o	/19	87
16.	1-decanol		3p	/17	86
17.	1,8-octane diol		3q	/26	80

^ayield in isolation.

Section for experimentation:

General Procedure:

A mixture of alcohol (1 mmol), acetic acid (3 mmol), and cyanuric chloride at 10 mol percent was stirred at room temperature without solvent for the duration indicated in the table. After the reaction was finished, as determined by TLC, the reaction mixture was extracted using diethyl ether (2x15 ml). After being repeatedly rinsed with saturated NaHCO₃ solution and dried under low pressure, the organic layer was purified using column chromatography with pet-ether:ethyl acetate (9:1) as the eluent.

Conclusion:

The esterification of alcohols using acetic acid has been successfully developed as a highly efficient and practical method. The advantages of this method include the

^b Products are identified by HNMR and IR.

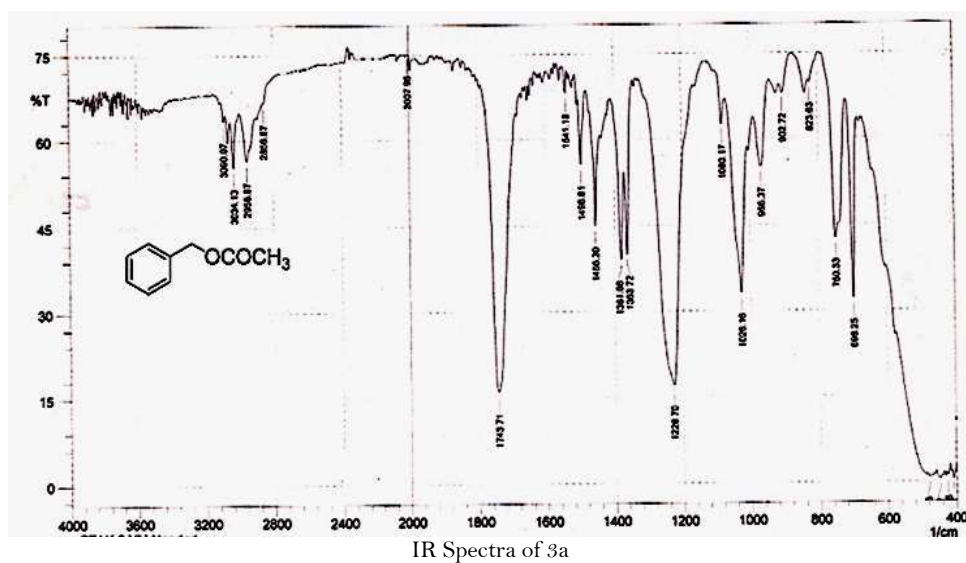
utilization of a minimal quantity of catalyst and reaction occurring at room temperature.

Compound spectral data:

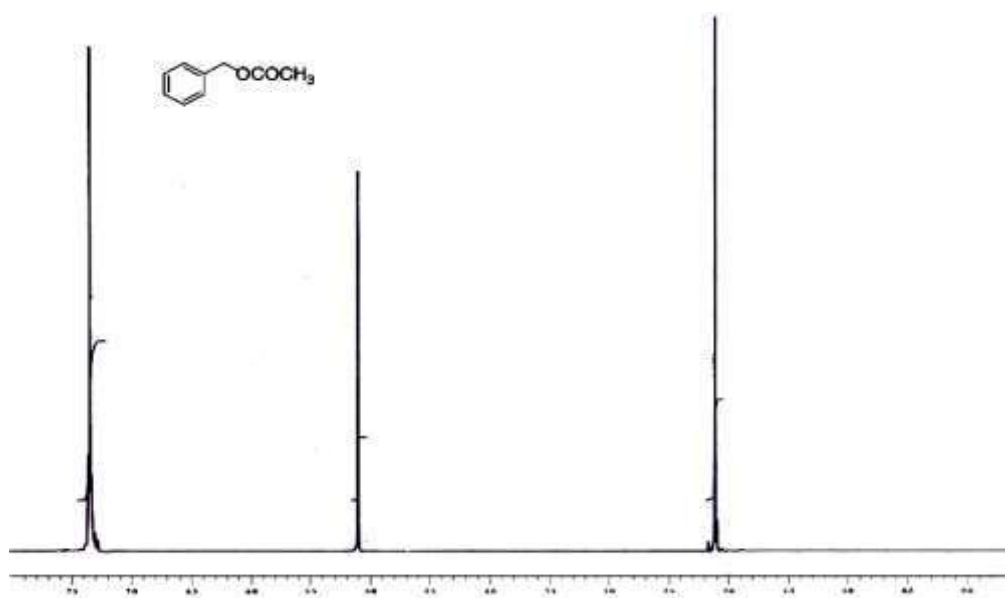
Benzyl acetate (3a) is a transparent liquid. The IR (KBr, cm⁻¹) 3061, 1745, 1456, and 1228. ¹H NMR (400 MHz, CDCl₃) exhibits δ = 2.2 (s, 3H), 5.3 (s, 2H), and 7.3 (s, 5H). MS m/z 150.2

2 4-Nitrobenzyl acetate (3b) is a white crystalline substance The IR (KBr, cm⁻¹) values for 3374, 3262, 1732, 1593, 1610, 1445, and 1229 ¹H NMR (400 MHz, CDCl₃): δ = 2.1 (s, 3H), 3.80 (s, 3H), 5.21 (s, 2H), 6.91 (d, 2H, J = 8.6 Hz), 7.30-7.32(d, J = 8.6 Hz, 2H); MS: m/z = 195 (M⁺).

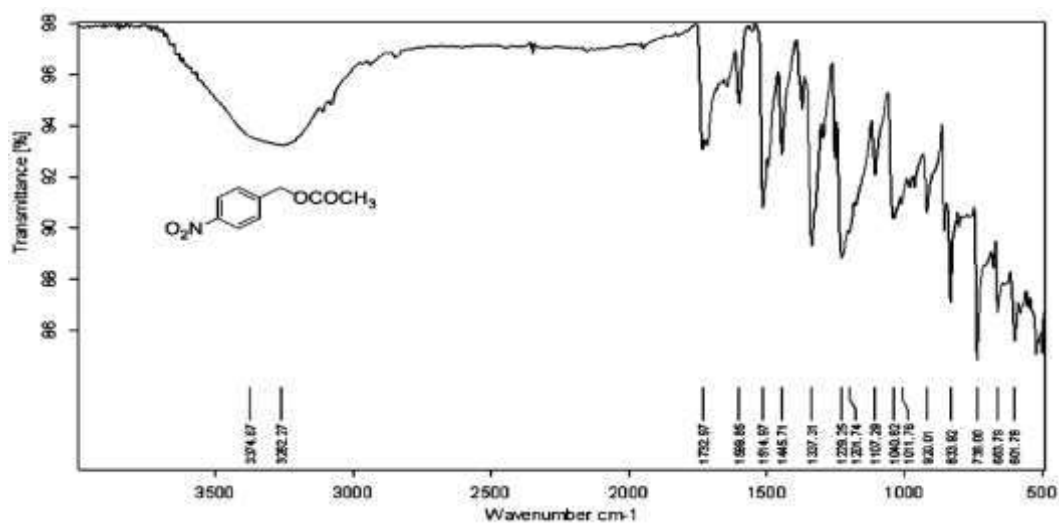
4 4-Methoxybenzyl acetate (3d), White crystalline substance; m.p: 122 °C; IR(KBr, cm⁻¹): 3022, 2921, 1711, 1611, 1508, 1212 ¹H NMR (400 MHz, CDCl₃): δ = 2.3 (s, 3H), 5.09 (s, 2H), 7.18-7.3(d, J = 8.6 Hz, 2H); MS: m/z = 180.18 (M⁺).



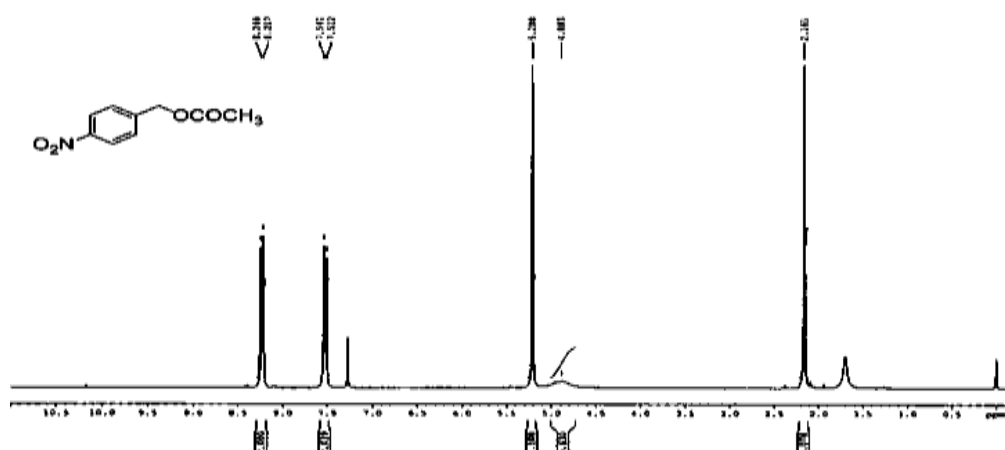
IR Spectra of 3a



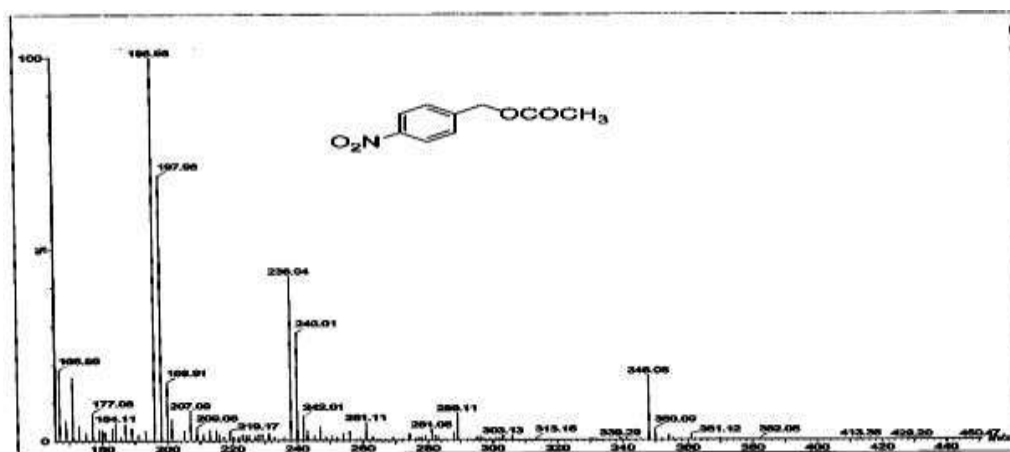
¹H NMR Spectra of 3a



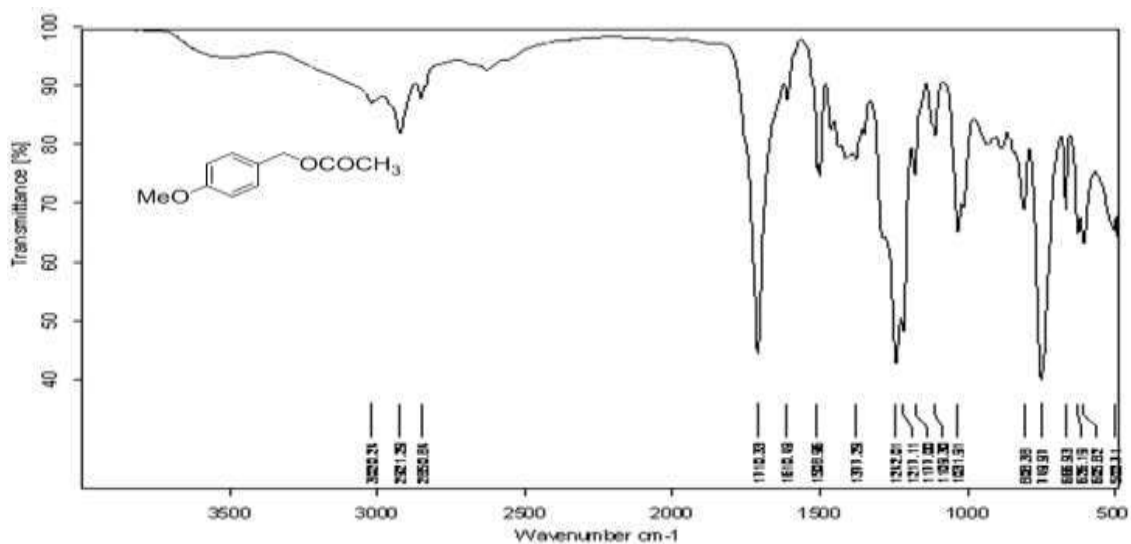
IR Spectra of 3b



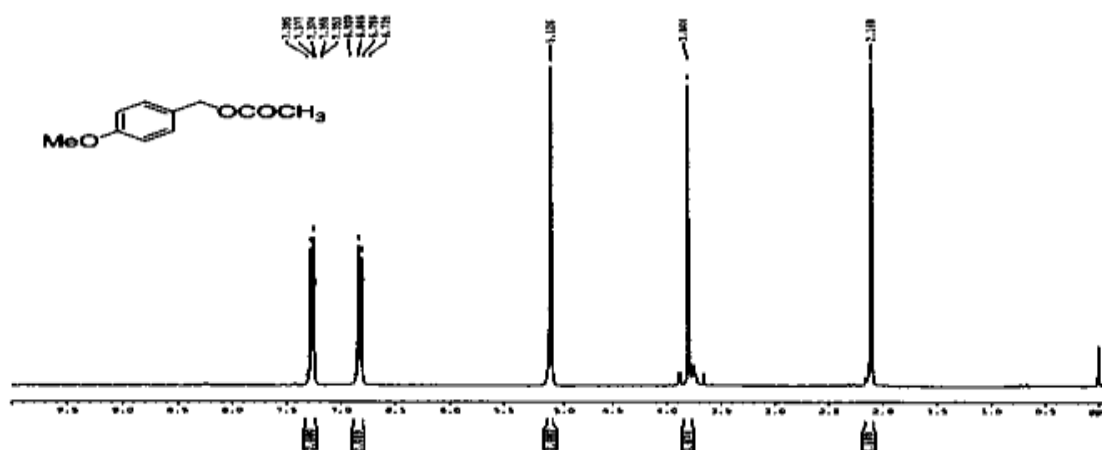
¹H NMR Spectra of 3b



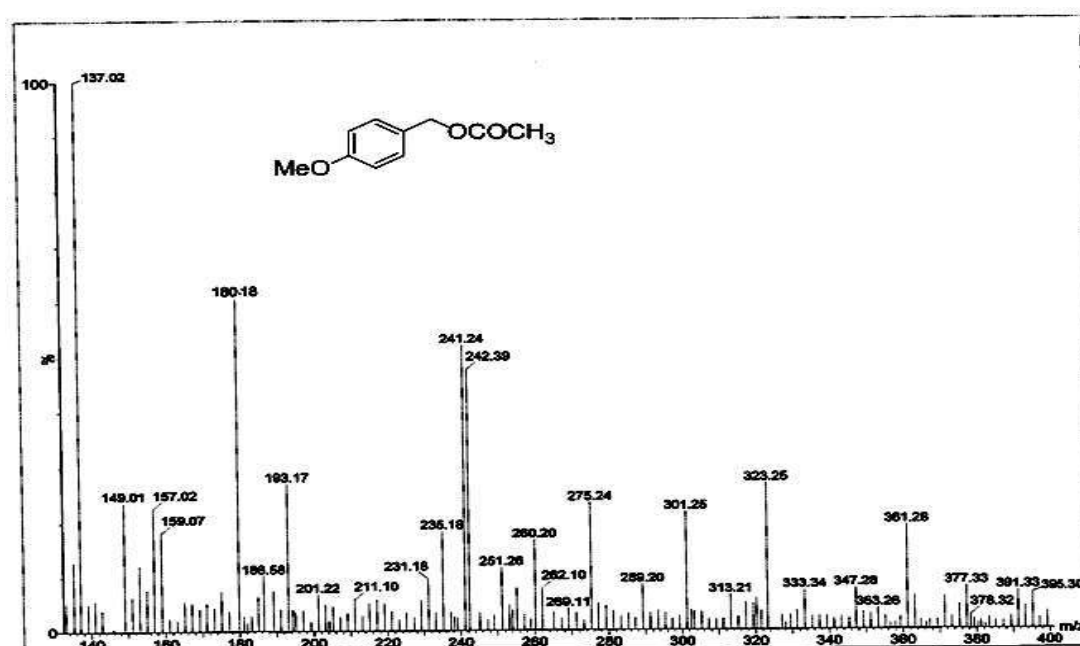
Mass Spectra of 3b



IR Spectra of 3d



¹H NMR Spectra of 3d



Mass Spectra of 3d

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Conflicts of interest

The authors declare that there are no conflicts of interest regarding the publication of this paper

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Original Article

Analysis of the Flower colour mutant in Coriander with respect to Phytonutrients

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Abstract

An established method for improving crop plants genetic makeup by introducing heritable variants is called mutation breeding. By creating new genetic variability within already-existing crop varieties, induced mutagenesis improves desired agronomic and nutritional characteristics. The commercially significant leafy herb *Coriandrum sativum* L. (coriander), a member of the Apiaceae family, is widely grown and consumed throughout India. In Maharashtra, it is referred to as *Kothembiri*. In order to produce morphological variations in coriander, the current work used induced mutagenesis using gamma rays and ethyl methane sulphonate (EMS). The physical traits of the resulting mutant populations were assessed, with a focus on flower color mutation, and their phytonutrient composition was compared. When compared to the untreated control, the induced flower color mutants showed a notable variety in phytonutrient content, demonstrating the efficacy of EMS and gamma irradiation in producing beneficial genetic variability. The data illustrate the efficacy of mutant breeding as a significant approach for enhancing coriander's nutritional and phenotypic characteristics.

Keywords: Mutation breeding, *Coriandrum sativum* L., Ethyl Methane Sulphonate (EMS), Gamma rays, Flower colour mutants, Phytonutrients.

Introduction

A member of the Apiaceae family, *Coriandrum sativum* Linn. is a valuable medicinal and commercial plant that is grown all over the world for its leaves and seeds. The plant, commonly referred to as coriander, is widely used as a flavoring and spice in cooking. It is natively called as *Kothembiri* in Maharashtra, although it is often referred to as *Dhania* throughout India. The Greek word *Koris* (bedbug), which refers to the distinctive strong smell released by the plant's aerial parts, especially the young fruits, is where the term "coriander" originates. Due to its strong nutritional, commercial, and therapeutic importance, coriander holds a prominent position in Indian agriculture. In both traditional and contemporary cooking methods, the fresh leaves and dried seeds are frequently used. In addition to its culinary significance, *C. sativum* has long been acknowledged as a significant medicinal plant in traditional medicine, especially Ayurveda, where its therapeutic qualities are utilized to treat a variety of illnesses Salve and More (2019). Much study has been focused on genetically improving coriander because of its importance in agriculture and commerce. A successful method for creating new genetic variability and enabling the creation of better crop varieties is mutation breeding. By generating favorable mutants that have been directly released as novel cultivars or used in breeding programs, induced mutagenesis using physical and chemical mutagens has greatly improved crops Gottschalk and Wolf (1983). The current study uses varied concentrations of ethyl methanesulphoate (EMS) and gamma ray dosages to examine the induction of floral color mutation in *C. sativum*. The objective is to assess these treatments' mutagenesis efficacy and describe the resultant blossom color variations for possible use in coriander improvement initiatives.

Material And Methods

Tamil Nadu Agricultural University in Coimbatore, Tamil Nadu, India, provided the *Coriandrum sativum* L. cv. CS-287 seeds. To produce genetic variations, seeds were treated with a physical mutagen, gamma rays and chemical mutagen, Ethyl Methane Sulphonate (EMS). The seed in the control group were not treated. To raise the M1 generation, seeds from each treatment and the control were sown under field condition following the mutagenic treatment using a Complete Randomized Block Design (CRBD) with three replications.

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Flower colour mutants were detected and chosen for extra analysis after successive generations were produced using traditional breeding techniques. The selected flower colour mutations are subjected to biochemical investigation in the M3 and M4 generations. According to Hesham H. A. Rassem et al. (2016), the hydrodistillation method was used to assess the essential oil content of fruits and seeds. The anthrone method Hedge and Hofreiter (1962) was used to estimate the total carbohydrate content, while the Lowery et al. (1951) method was used to quantify the total protein content in leaves, fruits, and seeds. In order to compare the mutant lines and the control, all biochemical parameters were quantitatively measured, and the results were documented and examined.

Result And Discussion

Compared to the control genotype, the flower-color mutant showed a noticeably increased carbohydrate content. Similarly, in both fruits and leaves, the mutants protein concentration were higher than those of the control. Furthermore, the overall essential oil content of the mutant lines was higher, indicating that the accumulation of primary and secondary metabolites was positively impacted by mutagenesis.

These findings consistent with previous research on induced mutagenesis in *Coriandrum sativum* L. Salve and More (2019) discovered that while dwarf mutants had lower biochemical parameters, tall mutants had higher carbohydrate content, higher protein level in leaves and fruits and increased essential oil yield. Salve and Bhosale (2024) found that late-flowering coriander mutants had higher quantities of protein in leaves and fruits, carbohydrates and essential oil than to the control. Similarly, dark green leaves and luxuriant mutants of *C. sativum* were reported to have more biochemical contents by Salve and More (2018). According to all these results, induced mutations may boost metabolic activity, which may enhance the production and accumulation of biochemical components important to the economy.

Similar reactions have been seen in other aromatic and medicinal species. Iwo et al. (2013) showed that Ginger mutants produced by gamma irradiation had higher oleoresin content and rhizome yield, indicating increases in

both biochemical composition and productivity. In a similar vein, gamma-irradiated *Centella asiatica* acquired substantially higher total flavonoid content than untreated controls, according to Moghaddam et al. (2011). The authors also noted that when plants progressed through different stages of growth, biochemical contents gradually increased. Latif et al. (2011) showed in another study that a low dose of gamma radiation improved plant growth, boosted the production of phytohormones, stimulated the biosynthesis of essential oils, and raised the concentrations of amino acids in coriander, underscoring the advantages of an optimal irradiation dose on plant metabolism.

A member of the Apiaceae family, *Coriandrum sativum* L. is a commercially significant fragrant and medicinal herb that is extensively grown for its culinary and medicinal uses. Coriander essential oil has a wide range of biological activities, such as antibacterial, anthelmintic, insecticidal, allelopathic, antioxidant, antidiabetic, anticonvulsant, antidepressant, and hepatoprotective qualities, according to Al-Khayri et al. (2023). The genetic enhancement of coriander by mutant breeding has been the subject of much research due to its significant economic and therapeutic value. In order to create genetic variety and enable the selection of superior genotypes with desired agronomic and biochemical traits, chemical mutagens like ethyl methansulphonate (EMS) and physical mutagens like gamma irradiation have been widely used. The efficiency of induced mutagenesis as a crop development tool was demonstrated by Kashtwari et al. (2018), who revealed that in vitro mutagenesis employing EMS successfully produced significant genetic variability and desirable features in saffron.

One of the best physical mutagens for creating advantageous genetic variety in coriander is gamma irradiation. Gamma rays improve the biochemical and functional quality of the crop by influencing the biosynthesis and accumulation of phytochemicals, secondary metabolites, chlorophyll, and antioxidant substances in addition to causing heritable mutations. According to Lin et al. (2022), gamma irradiation increased the concentration of these bioactive components in coriander, indicating that it may be a useful technique for creating new cultivars with better nutritional, therapeutic, and commercial value.

Table No.: 1 – Biochemical composition protein, carbohydrates and essential oil of Flower Colour Mutants compared to the control.

Sr. No.	Morphological Mutants	Percentage Protein Leaf (± SE)	Percentage Protein in fruits and seeds (± SE)	Percentage Carbohydrates (± SE)	Percentage Essential oil (± SE)
1	Control	2.50 (± 0.02)	2.15 (± 0.02)	5.01 (± 0.01)	0.30 (± 0.01)
2	Tested Mutant	2.61 (± 0.03)	2.33 (± 0.01)	5.26 (± 0.02)	0.61 (± 0.02)

(± SE) = standard error

Conclusion

Research has shown that *Coriandrum sativum* has a great deal of potential for genetic enhancement through mutation breeding. It has been demonstrated that the use of induced mutagenesis improves important agronomic and biochemical characteristics, such as higher protein,

carbohydrate, and essential oil yields. Additionally, relatively slight changes were found in the phytonutrient composition of the M3 and M4 mutant generations after mutagenic treatment, suggesting the relative stability of these beneficial substances. These results highlight the efficacy of mutation breeding as a genetic modification



technique for the creation of superior coriander genotypes with improved nutritional and therapeutic qualities.

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Conflicts of interest

The authors declare that there are no conflicts of interest regarding the publication of this paper

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Original Article

Effect of Gamma Radiation and EMS on Pollen Sterility and Survival of plants in *Vigna radiata* (L). Wilczek

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Abstract

Vigna radiata (L.) Wilczek (Mungbean or green gram) is a supreme pulse crop of the family Fabaceae, valued for its short growth duration, high economic return, and role in sustainable cropping systems. The present study aimed to assess the impact of physical and chemical mutagens on pollen sterility and plant survival in the M₁ generation of Mungbean, cultivar Phule Chetak (PM-707-5). Seeds were treated with gamma radiation at doses of 100, 150, 200, 250, 300, 350, 400 and 450 Gy and Ethyl Methane Sulphonate (EMS) at concentrations of 10, 20, 30 and 40 mM. The treated seeds were grown under field conditions, and observations were recorded on pollen sterility and plant survival. Both mutagenic treatments markedly affected pollen viability and plant survival. Pollen sterility increased progressively with higher doses of gamma radiation and greater concentrations of EMS indicating a dose-dependent response. The highest pollen sterility in EMS-treated plants was observed at 40 mM, while the lowest was recorded at 10 mM. Similarly, maximum pollen sterility in gamma-irradiated plants was observed at 400 and 450 Gy, whereas the minimum was recorded at 100 Gy. A progressive decline in plant survival was also observed with increasing mutagen intensity. The findings of study indicate that increasing doses of gamma radiation and EMS significantly enhanced the pollen sterility and adversely affect plant survival in *Vigna radiata*. These findings provide valuable information for selecting appropriate mutagen doses in Mungbean mutation breeding programmes.

Keywords: *Vigna radiata* (L.) Wilczek, Mungbean, gamma radiation, mutation breeding, Ethyl Methane Sulphonate (EMS), pollen sterility, plant survival.

Introduction

Mungbean (*Vigna radiata* (L.) Wilczek) occupies an important position among pulse crops due to its high dietary and nutritional value (Patel *et al.*, 2015). It is widely cultivated in Asia and has gradually expanded to several countries outside Asia because of its multiple uses and economic importance. Mungbean is recognized globally as an important pulse crop contributing to food and nutritional security.

Agricultural development is essential for meeting the food and nutritional needs of increasing global population. At present, food availability and food security are major challenges worldwide, as population growth continues to exert pressure on agricultural production systems. In India, a large proportion of the population follows a vegetarian diet and depends on cereals and pulses as primary sources of energy and protein. Pulses are highly nutritious, economical, and rich in protein, making them an essential component of the daily diet. They are often regarded as the lifeline of the vegetarian population in India. In addition to their nutritional importance, pulses improve soil fertility through biological nitrogen fixation and contribute significantly to sustainable agriculture.

Pulses constitute a major component of Indian diets, particularly among low-income rural populations. However, achieving food self-sufficiency remains a challenge for these communities. Consequently, extensive efforts have been undertaken to enhance pulse productivity and yield. Mungbean is a predominantly self-pollinated crop and is commonly cultivated in crop rotation systems. It belongs to the family Fabaceae, the second most agriculturally important plant family and a major source of vegetable protein for human consumption. Among pulse crops, Mungbean commands a high market price in India. Owing to its superior nutritional quality, the global demand for Mungbean has increased considerably in recent years. India ranks first in both production and consumption of Mungbean and cultivates the crop over approximately 3.8 million hectares. Several international collaborations, including the International Mungbean Improvement Network (IMIN), the Indian Institute of Pulses Research (IIPR), and the Australian Centre for International Agricultural Research (ACIAR), have played significant roles in Mungbean breeding programmes aimed at strengthening future nutritional security.

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Mutation breeding is an effective approach for developing novel and desirable traits in crop plants. It has been widely utilized to create genetic variability within a relatively short period, thereby facilitating crop improvement programmes. According to Shah et al. (2008), gamma radiation induces genetic alterations, disrupts gene linkages and generates favorable variations that can be exploited for crop improvement. Induced mutations have been successfully employed to increase genetic diversity and enhance crop productivity. Advanced breeding methods, including mutation breeding, are valuable tools for improving crop genotypes and increasing agricultural production (Wright, 1935). The effectiveness of induced mutagenesis largely depends on the selection of suitable mutagens and treatment conditions (Solanki et al., 1994; Mahabatra et al., 1983; Anwar et al., 2013; Ali et al., 2014a,b,c; and Jahangir et al., 2013). Physical as well as chemical mutagens are widely used to induce mutations in crop plants for genetic improvement. The determination of LD₅₀ values is important for assessing the sensitivity of plants to mutagenic treatments and for selecting effective mutagen doses.

Genetic variability forms the foundation of plant breeding and crop improvement. Plant breeders continuously seek innovative approaches to enhance crop productivity, adaptability, and sustainability. The issues arising from rapid population growth, climate change and declining natural resources have intensified the need for efficient breeding strategies capable of generating improved crop varieties. In this context, mutation breeding offers a valuable tool for creating useful genetic variation and accelerating the development of superior Mungbean cultivars.

Materials And Methods

Plant Material

The study was carried out using the Mungbean (*Vigna radiata* (L.) Wilczek) variety Phule Chetak (PM-707-5), developed at the Oilseed Research Station, Mahatma Phule Krishi Vidyapeeth (MPKV), Jalgaon, Maharashtra, India. The variety was developed by pedigree selection method using cross between SML-668 and Naval. Healthy, well-filled, uniform-sized, and dry seeds were obtained from the Oilseed Research Station, MPKV, Jalgaon, and used for the study.

Salient Features of Mungbean Variety Phule Chetak (PM-707-5)

Sr. No.	Character	Feature
1	Plant height (cm)	40–55
2	Plant habit	Erect
3	Leaf type	Trifoliate
4	Stipule	Broad
5	Stem colour	Green
6	Seeds per pod	11–13
7	Seed colour	Green
8	Seed coat colour	Green
9	Seed size	Small to medium bold
10	Seed lustre	Shiny
11	Pod position	Terminal
12	Hilum	Flat

Mutagenic Treatments

The physical and chemical mutagens were used in the study. The gamma radiation is physical mutagens and Ethyl Methan Sulphonate (EMS) as chemical mutagens. Gamma irradiation treatments were administered using a Cobalt-60 (⁶⁰Co) gamma source installed in the Gamma Chamber at the Department of Chemistry, Savitribai Phule Pune University, Pune, Maharashtra, India. The chemical mutagen EMS (CH₃SO₂OC₂H₅; molecular weight: 124.16 g mol⁻¹) was procured from Rathod Enterprises Chemicals. 500 seeds were pre-soaked in distilled water for 6 h before treatment. The pre-soaked seeds were then subjected to EMS treatment by immersing them in mutagenic solutions at a volume ten times greater than the seed volume to ensure uniform absorption. The treatment was carried out for 4 h at room temperature. Seeds without any mutagenic treatment were maintained as the control for comparison.

Raising of M₁ Generation

The treated and control seeds were sown immediately in the field for raising the M₁ generation. The experiment was

conducted using a Randomized Block Design (RBD) with three replications. Each treatment consisted of three rows, maintaining a spacing of 30 cm between rows. Standard agronomic practices were maintained and the plots were monitored regularly until maturity.

Assessment of Pollen Sterility

For the assessment of pollen fertility and sterility, 25 plants were randomly selected from each treatment. Fresh flower buds were collected from selected plants at the flowering stage. For slide preparation, floral parts such as the corolla, wings, and keel petals were carefully removed using a scalpel. Pollen grains were collected by dusting mature anthers onto clean glass slides and stained with 1% acetocarmine solution.

Pollen grains showing complete staining were considered fertile whereas partial stained or unstained pollen grains were recorded as sterile. The stained pollen grains were observed under a microscope and counted to determine pollen fertility and sterility percentages.

Pollen fertility percentage was calculated using the following formula:

$$\text{Pollen Fertility(\%)} = \frac{\text{Number of well-stained pollen grains}}{\text{Total number of stained and unstained pollen grains}} \times 100$$

Pollen sterility percentage was calculated using the following formula:

$$\text{Pollen Sterility(\%)} = \frac{\text{Number of unstained pollen grains}}{\text{Total number of stained and unstained pollen grains}} \times 100$$



Assessment of Plant Survival

Plant survival was recorded at crop maturity and the survival percentage was calculated from the number of plants surviving in each treatment. The effect of mutagenic

treatments on plant survival was evaluated by comparing treated populations with the untreated control. A gradual reduction in plant survival was observed with increasing doses of gamma radiation and higher concentration of EMS.

Observation Table:

Table 1. Pollen Sterility induced by EMS and Gamma Radiation

Sr. No	Mutagen	Dose/ Concentration	Fertile Pollens (%)	Sterile Pollens (%)	Survival of plants at maturity (%)	Survival of plants at maturity (%)
1	Control	-	97.56%	2.44%	298	99.33%
2	EMS	10mM	95.71%	4.29%	291	97.00%
		20mM	94.53%	5.47%	289	96.33%
		30mM	92.61%	7.39%	283	94.33%
		40mM	89.27%	10.73%	280	93.33%
3	Gamma Radiation	100Gy	96.02%	3.98%	293	97.66%
		150Gy	95.15%	4.85%	290	96.66%
		200Gy	93.71%	6.29%	287	95.66%
		250Gy	92.58%	7.42%	282	94.00%
		300Gy	89.05%	10.95%	279	93.00%
		350Gy	85.35%	14.65%	271	90.33%
		400Gy	83.77%	16.23%	269	89.66%
		450Gy	81.51%	18.49%	261	87.00%

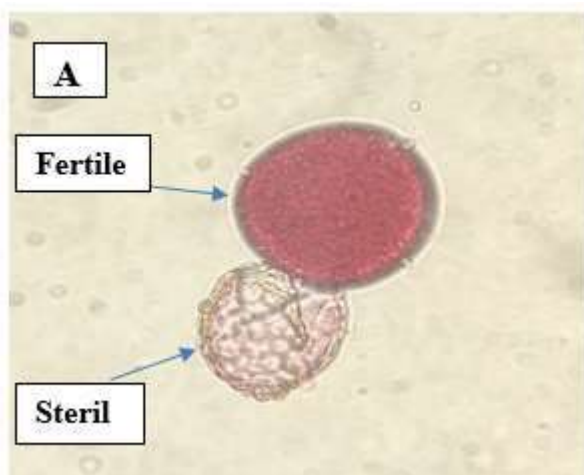
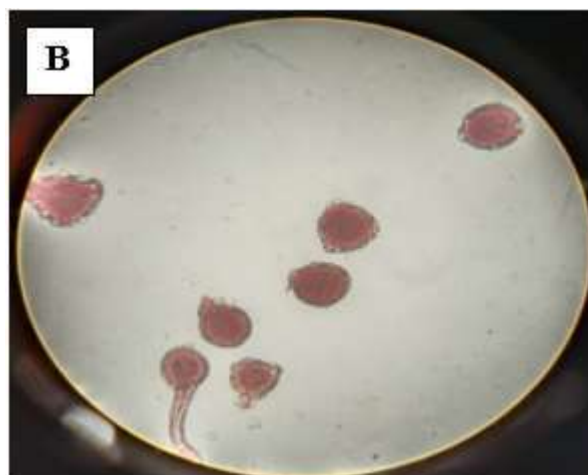
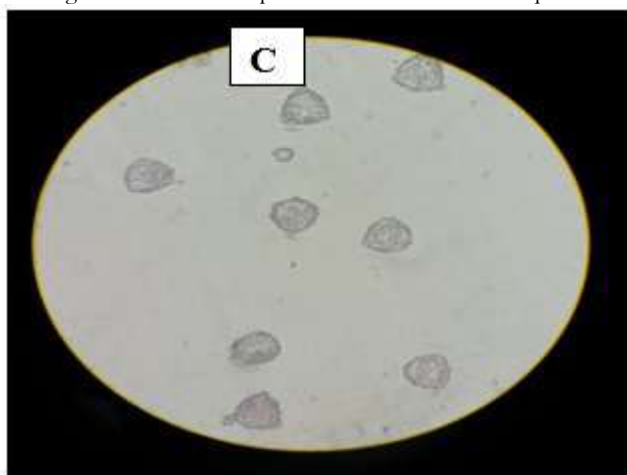


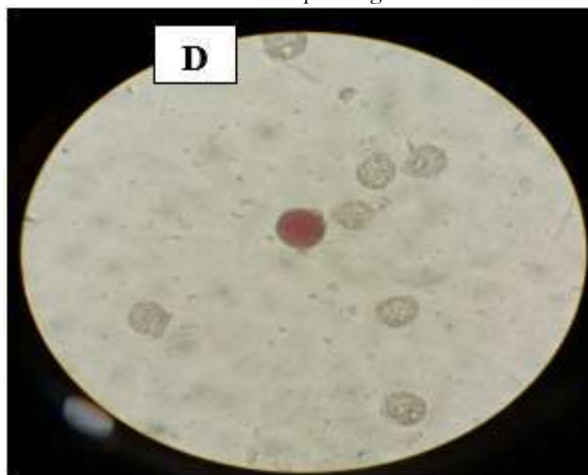
Fig A. Fertile stained pollen and sterile unstained pollen



B. Well stained and viable pollen grains of the control



C. Completely sterile pollen grains



D. Only one pollen fertile and others are sterile.

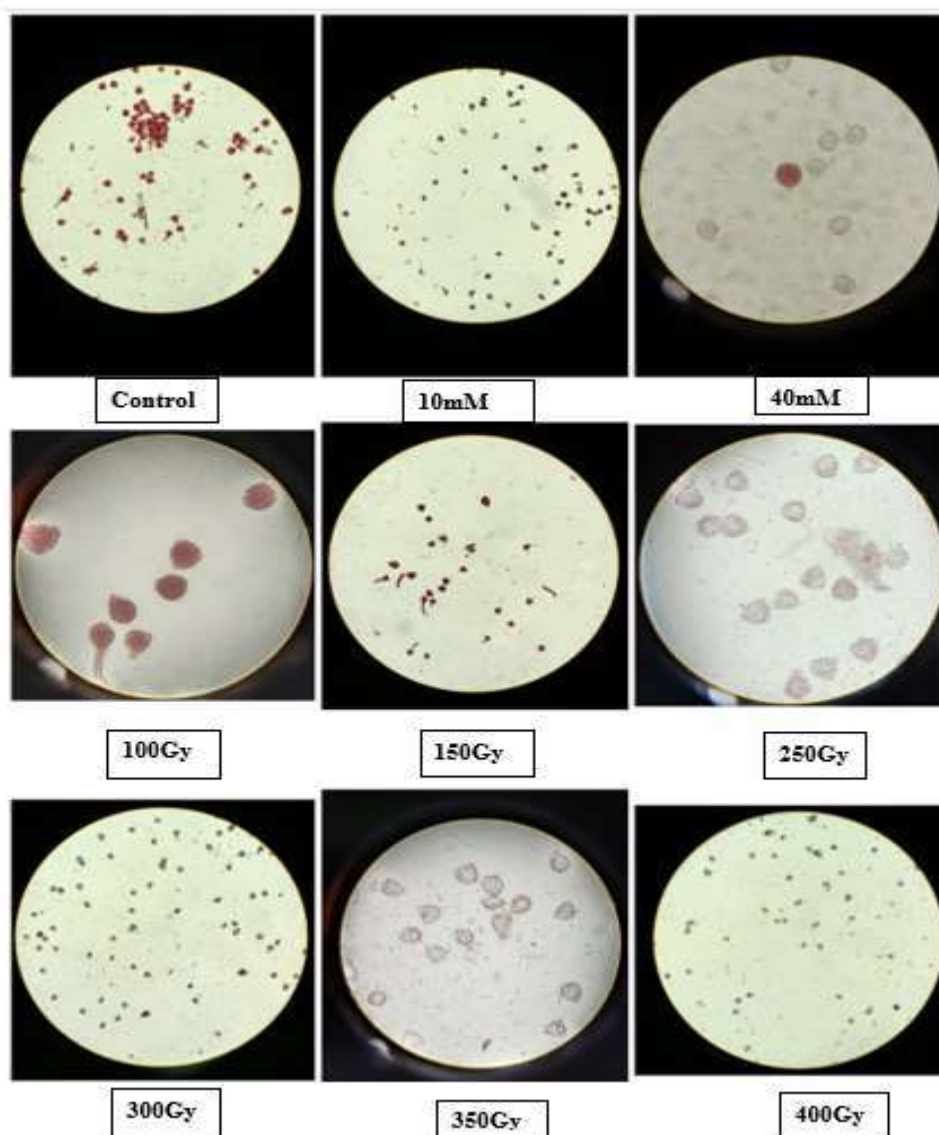


Fig. Different Treatment wise photographs of pollen sterility.

Results And Discussion

In the present investigation, biological damage in the form of pollen sterility was induced and recorded in the M_1 generation of *Vigna radiate* (L.) Wilczek var. Phule Chetak (PM-707-5) following treatments with the chemical mutagen Ethyl Methane Sulphonate (EMS) and physical mutagen gamma radiation. In both mutagenic treatments, pollen sterility increased progressively with increasing doses or concentrations of the mutagens, indicating a dose-dependent response. Among the EMS treatments, the maximum sterility of pollen (10.73%) was recorded at 40 mM, whereas the minimum pollen sterility (4.29%) was recorded at 10 mM. Conversely, the highest pollen fertility (95.71%) was observed at 10 mM, while the lowest pollen fertility (89.27%) was recorded at 40 mM. Similarly, in gamma radiation treatments, the maximum pollen sterility (18.49%) was observed at 450 Gy and the minimum pollen sterility (3.98%) at 100 Gy. The highest pollen fertility (96.02%) was recorded at 100 Gy, whereas the lowest pollen

fertility (81.51%) was observed at 450 Gy. These results clearly demonstrate that increasing doses of both EMS and gamma radiation adversely affected pollen viability and enhanced pollen sterility.

Several researchers have reported similar findings. Monica, S. and Seetharaman, N. (2015) reported increased pollen sterility following mutagenic treatments in garden bean (*Lablab purpureus* (L.) Sweet var. typicus cv. CO (Gb)). Taziun, T. *et al.* (2017) observed comparable results in lentil (*Lens culinaris* Medik.) cultivars Pant L-406 and DPL-62. Similar observations were also reported by Wani, M.R. *et al.* in lentil, Ganesh, B.K. (2011) in horse gram [*Macrotyloma uniflorum* (Lam.) Verdcourt], and Sangle, S.M. *et al.* in pigeonpea. Pollen sterility is considered an important biological parameter for assessing the effectiveness and efficiency of mutagenic treatments. S. Ignacimuthu and C.R. Babu (1989) also reported increased the pollen sterility with increasing mutagen dose or concentration in various *Vigna* species including *Vigna radiata*, *Vigna mungo* and *Vigna sublobata*. The dose-dependent increase in pollen sterility



may be attributed to meiotic abnormalities and chromosomal aberrations induced by mutagenic treatments, as reported by Kashid and Kulthe (2011). Chemically induced pollen sterility has also been investigated in several crop species by Gaul H. and Aastveit K. (1966), Kothekar V.S. (1978), Deshpande N.M. (1980), and Hakande T.P. (1992).

Plant survival at maturity was also affected by mutagenic treatments. In the control population, plant survival was recorded as 99.33%. In EMS-treated populations, plant survival ranged from 97.00% at 10 mM to 93.33% at 40 mM. Similarly, in gamma radiation-treated populations, plant survival ranged from 97.66% at 100 Gy

to 87.00% at 450 Gy. The reduction in plant survival with increasing mutagen dose or concentration may be due to physiological disturbances, genetic damage, and reduced cellular viability caused by mutagenic treatments.

The present findings indicate that both EMS and gamma radiation effectively induced biological changes in Mungbean. However, gamma radiation produced a comparatively greater increase in pollen sterility and a more pronounced reduction in plant survival than EMS. Therefore, pollen sterility and plant survival may be considered reliable indicators for evaluating the biological effects of mutagens and for determining suitable mutagen doses in Mungbean mutation breeding programmes.

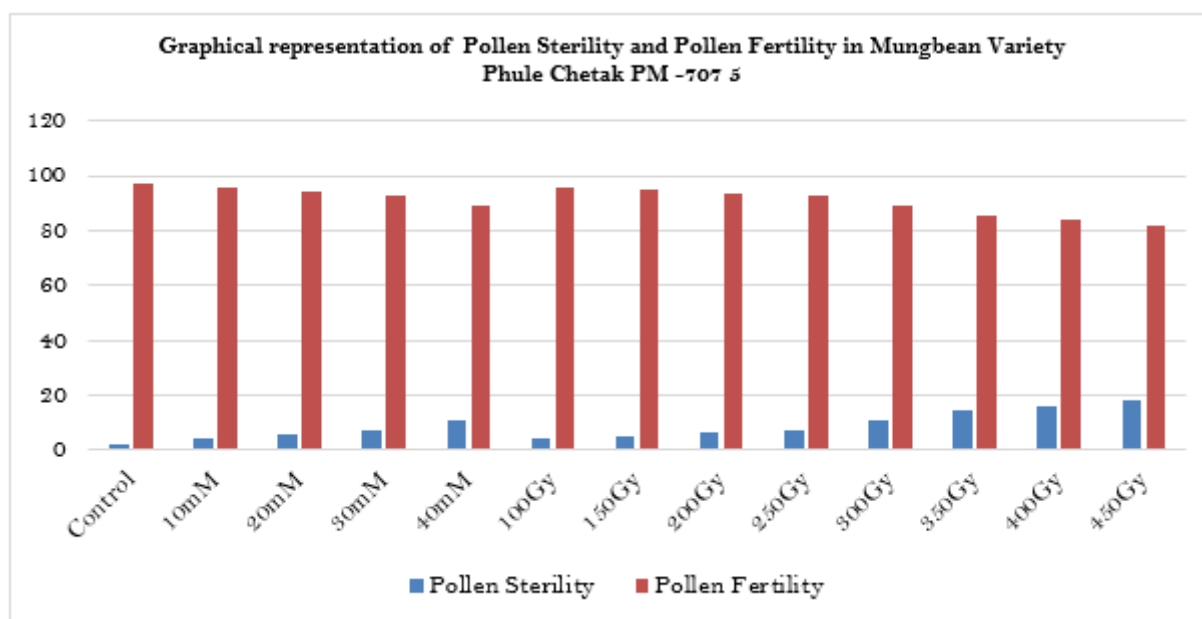


Fig. Graphical representation of Pollen Sterility and Pollen Fertility in Mungbean Variety Phule Chetak PM-707 5

Conclusion

The present study demonstrated that both physical and chemical mutagenic treatments, *viz.* gamma radiation and Ethyl ethane Sulphonate (EMS), significantly increased pollen sterility in *Vigna radiata* (L.) Wilczek var. Phule Chetak (PM-707-5). Pollen sterility increased with higher mutagen doses and concentrations, indicating a dose-dependent response. Simultaneously, plant survival at maturity decreased progressively with increasing mutagen intensity, suggesting an inhibitory effect of both mutagens on plant growth and development.

Among the EMS treatments, the lower concentration (10 mM) was found to be more effective and less detrimental than the higher concentrations (20, 30 and 40 mM). Similarly, reduced doses of gamma radiations (100, 150 and 200 Gy) were comparatively more suitable than the higher doses (400 and 450 Gy), as they induced lower pollen sterility and higher plant survival. Similar findings in Mungbean have been reported by Das, T.R. and Baisakh, B. (2011); Das, T.R. and Baisakh, B. (2020); Das, T.R. and Misra, R.C. (2005) and Das, T.R. and Prusti, A.M. (2020) in Mungbean.

The results indicate that both gamma radiation and Ethyl Methane Sulphonate (EMS) are efficient mutagens for stimulating genetic variability in Mungbean and can be utilized in mutation breeding programmes for crop improvement. However, all induced mutations are not

necessarily beneficial. The successful application of mutation breeding depends on the selection of an optimal mutagen concentration, the identification of desirable mutant traits, and the use of suitable breeding strategies. Therefore, careful screening and selection of mutants are essential for developing improved Mungbean varieties with enhanced yield and other suitable agronomic characteristics.

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The authors declare that there are no conflicts of interest regarding the publication of this paper

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Original Article

Barnyard Millets *Echinocloa esculanta* L. (Phule Barti-1) "Recipe: Important Milestone in Indian Fasting Rituals"

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Abstract

Millets have been an essential part of human diet for centuries. Minor millets are widely recognized as functional food due to their exceptional richness in nutritional components, bioactive compounds, antioxidant, antidiabetic, and anticancer activity. Barnyard millets are known as minor grain. It belongs to Poaceae family. Present research work explores the value-added nutritional food products of Barnyard millets viz instant foods, sun dried foods, deep fried foods, intrusion foods, fermented foods, cooking foods etc. However, a diverse range of barnyard millet-based products had been developed, including instant foods, sun-dried foods, deep-fried foods, intrusion foods, fermented foods, cooking foods etc. Nutritionally, all these products revealed that barnyard millets products are rich in protein, fibre, carbohydrates, vitamins, minerals, phenolic compounds, etc. Millets promotes health and reduces risk of several diseases. The prospects of millet-based products involve in increasing awareness of health benefits of the human beings.

Keywords: Barnyard Millet, Value added Food, Nutritional Parameters

Introduction

Millets are an excellent source of macro and micronutrients, mineral profile and essential amino acid, composition are superior to other major cereals such as wheat and rice (Mal et al., 2010; Singh et al., 2012). Regular intake of millets has been reduced risk of many degenerative diseases due to richness in phytonutrients and biologically active components such as dietary fibre, phenolic acids, flavonoids and phytosterols, It also has other health benefits including anti-oxidative, anti-ulcerative, hypoglycaemic and anti-inflammatory properties in addition to cholesterol-lowering potential (Shobana & Malleshi, 2007; Schoenlechner et al., 2013; Sharma & Gujral, 2019a). Millet carbohydrates have lower starch digestibility modulating blood glucose level due to slower absorption (Chethan & Malleshi, 2007). Despite their long-standing presence, their full potential is still unexplored. Their perceived lack of palatability and limited awareness about their nutritional benefits contribute to them under appreciation (Majid and Priyadarshini, 2020). For centuries in India, millets were served as a staple food but now it is marginalised in specific regions (Harish et al., 2024). Attributing to their phytochemical profile and contribution to human wellness, they are being explored for applications in the food industry as potential functional ingredients for gluten-free and low-glycemic products (Sharma & Gujral, 2019). Throughout history, there has been a recognition of the abundant nutritional value and extensive potential inherent in these small seeds. It is already known that millets are a pack of minerals and vitamins, and they are viewed as important for the health and wellness of people (Obadina et al., 2017). Millets are recognised as a functional food due to their promotion of health benefits and reduction of disease risk attributed to the presence of several bioactive compounds (Banerjee and Ray 2019). Millets are well-known crops for individuals who are gluten intolerant have a weak immune system or suffering from celiac disease (Annor et al., 2017; Kavitha et al., 2024; Bhasin et al., 2024).

Nutritional Profiling of Barnyard Millet-Based Products (Dhokla)

Millets are highly beneficial as they are rich sources of protein, fibre, carbohydrate and many bioactive components.

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Milletts are now produced in diverse forms worldwide including alcoholic and nonalcoholic beverages, breads, muffins, cakes, snacks, cookies, biscuits, chips, bars, pasta and noodles (Alavi et al., 2019; Murugkar, 2020; Birania et al., 2020; Rashwan et al., 2021; Yousaf et al., 2021). Researchers are actively investigating ways to make these superfoods appealing to consumers by considering both health benefits and taste (Yadav & Up sana, 2024). Contemporary research endeavors have placed notable emphasis on millet-based products, delving into their nutritional, functional, and bioactive components. Considering the growing demand for diverse food options, there is an increasing acceptance of healthier alternate.

Collectively, millets are exerting a substantial influence in the food industry, offering consumers nutritious and palatable alternatives. Baked products the demand for ready-to-eat baked products has experienced substantial growth, gaining acceptance across diverse age groups globally. In response to the rising prevalence of gluten intolerance, there has been a shift towards using millet-based flours instead of their gluten containing counterparts.

Food categories can be prepared from barnyard millets

1. Instant foods- appe, upma, shira, soup
2. Sun-dried foods- machine made papad, steamed papad, sandga, vermicelli, dried chakli
3. Deep-fried foods – pakoda, shev, chakli, gulabjamun
4. Intrusion foods- Murmura, poha, etc.
5. Fermented foods- Ambil, idli, dosa, sour rice, curd rice
6. Cooking foods- Bhakri, rice, khimat, paratha, thalipith
7. Roasted food- laddoos

Material and Method

Research material- Barnyard Millet seeds (Phule Bharti-1) procured from Research Station, Zonal Agriculture Research Centre Shendapark, Kolhapur.

Material Used- Floor of Barnyard Millet (Phule Barti-1)

1. Barnyard Millet Laddo

Ingredient- Barnyard millet flour – 1 cup, Jaggery powder – $\frac{3}{4}$ cup, Ghee – $\frac{1}{4}$ cup, Roasted peanuts / cashews – 2 tbsp, Cardamom powder – $\frac{1}{2}$ tsp.

Method

1. Dry roast barnyard millet flour on low flame till aromatic.
2. Add ghee and mix well.
3. Switch off flame, add jaggery and cardamom.
4. Mix in roasted nuts.
5. When warm, shape into small ladoos.

2. Barnyard millet idli

Ingredients- Barnyard millet (raw) – 1 cup, Urad dal – $\frac{1}{2}$ cup, Fenugreek (methi) seeds – $\frac{1}{2}$ tsp, Salt – to taste

Method

1. Wash and soak barnyard millet, urad dal, and methi seeds for 5–6 hours.
2. Grind urad dal fluffy; grind millet slightly coarse.
3. Mix both batters, add salt, and ferment overnight (8–10 hrs).
4. Pour batter into greased idli plates.
5. Steam for 10–12 minutes.

3. Barnyard millet Meduwada

Ingredients- Barnyard millet (raw) – 1 cup, Urad dal – $\frac{1}{2}$ cup, Onion (optional) – 1 small, finely chopped Green chilli

– 1–2, finely chopped, Ginger – 1 tsp, grated, Cumin seeds – 1 tsp, Curry leaves – few, chopped, Black pepper – $\frac{1}{2}$ tsp, Salt – to taste, Oil – for frying

Method

1. Wash and soak barnyard millet & urad dal for 5–6 hours.
2. Grind urad dal very smooth and fluffy; grind millet slightly coarse.
3. Mix both batters well, add salt, spices, onion, and curry leaves.
4. Heat oil. Take batter, make a hole in the centre.
5. Fry on medium flame till golden and crisp.

4. Barnyard Millet Appe

Ingredients- Barnyard millet (raw) – 1 cup, Urad dal – $\frac{1}{4}$ cup, Curd – $\frac{1}{4}$ cup, Onion – 1 small, finely chopped, Green chilli – 1, finely chopped, Ginger – 1 tsp, grated, Cumin seeds – 1 tsp, Curry leaves – few, chopped, Salt – to taste, Oil – for greasing appe pan

Method

1. Wash and soak barnyard millet & urad dal for 4–5 hours.
2. Grind to a slightly coarse batter, add curd and salt.
3. Rest for 30–60 minutes (optional light fermentation).
4. Mix in onion, chilli, ginger, cumin & curry leaves.
5. Heat appe pan, grease lightly, pour batter.
6. Cook covered, flip, and cook till golden.

5. Barnyard millet Bhakari

Ingredients- Barnyard millet flour – 1 cup, Warm water – as needed, Salt – to taste

Method

1. Take flour and salt, add warm water little by little.
2. Knead into a soft, crack-free dough.
3. Make a ball, flatten gently using fingers or between cloth.
4. Roast on hot tawa flip and cook both sides.
5. Roast directly on flame till it puffs (optional).

6. Barnyard Millet Soup

Ingredients- Barnyard millet – 2 tbsp, Water – 2 cups, Onion – 1 small (chopped), Garlic – 2 cloves, Ginger – $\frac{1}{2}$ tsp, Mixed vegetables – $\frac{1}{2}$ cup, Cumin seeds – $\frac{1}{2}$ tsp, Black pepper – $\frac{1}{4}$ tsp, Turmeric – a pinch, Salt – to taste, Oil – 1 tsp

Method:

1. Soak millet 15 min. Heat oil, add cumin, onion, garlic, ginger.
2. Add veggies, salt, turmeric.
3. Add millet + water, cook till soft (2 whistles). Add pepper, mash/blend & serve hot.

7. Barnyard Millet Gulab jamun

Ingredients- Barnyard millet flour – 1 cup, Milk powder – $\frac{1}{2}$ cup, Baking soda – a pinch, Ghee – 1 tsp, Milk – to knead, Oil/Ghee – for frying, **Sugar Syrup**, Sugar – 1 cup, Water – 1 cup, Cardamom – 2 (crushed)

Method

1. Mix millet flour, milk powder, soda, ghee. Knead soft dough with milk.
2. Make small balls. Fry on low flame till golden.
3. Boil sugar, water & cardamom to make syrup.
4. Soak hot jamuns in warm syrup for 1–2 hours.



A broad spectrum of baked items derived from millets have been developed, including Dosa, Bhakari, Ladoo, Soup, Shev,

as detailed in Table 1.

Food Products Prepared from Barnyard Millet

Result and Discussions

Result



Barnyard Millet Seed



Research Plot



Research Plot



Mature Panicle



Panicle Structure



Barnyard Millet Dhokla



Barnyard millet Idli



Barnyard Millet Meduwada



Barnyard Millet Appe



Barnyard Millet Bhakari



Barnyard Millet Ladddu



Barnyard Millet Pakoda



Barnyard millet dises



Barnyard millet dishes



Barnyard millet Chakli

Sr. No.	Parameter	Observed Value	Unit	Reference Method
1	Energy	211	Kcal	Nutritive Value of Indian foods (by calculation); 1971
2	Carbohydrates	23.15	G	Manual of Methods of Analysis of foods (As per FSSAI) 5 th edition (Clause 1.10.5): 2021
3	Fat	11	G	IS: 13354
4	Protein	4.85	G	Manual of methods of Analysis of foods (As per FSSAI) 5 th edition (clause 3.2.6): 2021
5	Fibre	4.7	g	Manual of Method of Analysis of Foods (As per FSSAI) 5 th edition (clause 2.4.34) 2021

Discussions

Barnyard millet has immense importance in Indian spiritual context as a healthy rice particularly during fasting known as "Vrat ke Chawal" (fasting rice). In Indian tradition the devotees of Lord Vitthala do fast (upwas) fortnightly is commonly known as Ekadashi. It is also prominently consumed during nine days fasting that is Shardiya Navratra (Sharad season) and Chaitra Navratra in (Vasant season) to seek blessings from Goddess Durga. In rural India, it is customary practice to prepare sun-dried food products during summer season by using different grains. In present study various range of healthy products prepared out of this variety of barnyard millet (Phule Barti-1), list number 1

Recently youngster facing lifestyle diseases due to instant fast food, work pattern, and lack off knowledge about inclusion of millets in the diet. To overcome health issues in youngest and to keep lifestyle diseases under control, barnyard millet will work miraculously. Barnyard millet used as a direct substitute for rice or in khichadi and pulao. It is natural unpolished and dried in direct sunlight to keep flavor and texture, often with a more than 12-month shelf life. It has held high nutrition packed with iron, calcium, protein, and magnesium. It enhanced to keep health parameters under control by providing low calories, anti-inflammatory, antioxidants, and dietary fiber. Barnyard millet is alternatives for baking and cooking snacks of wheat and rice for healthy non-fried, and gluten free. Successful improvement of these attributes would be a crucial key to expand the spectrum of applications of millet grains. Future trends should focus on the millet consumption in the developed countries that could help its industrial revolution. These nutritional parameters obtained from Bioresource Laboratory, Hadapsar Pune.

Conclusion

In conclusion, Barnyard millets stand for a valuable and underutilised resource in the realm of nutrition and food production. Despite their rich nutritional profile and historical significance, barnyard millets not included in modern diets. Millets offer a diverse range of products, including each with unique nutritional attributes. Their inclusion in various food categories enhances the nutritional profile of the products and it also contributes to addressing global challenges such as food security and health consciousness. The exploration and promotion of barnyard millet-based products should be fully harnessing their potential in promoting human health and sustainable food systems.

Barnyard millet has high nutrition, high carbohydrate, energy content, and gluten-free minor grain. Millet has dietary and nutritional balance in foodstuffs for millions of people in Africa and Asia. In the market, raw seeds (in the form of rice) and flour is available. It is very essential to move further, to explore for healthy dietary processed food items like instant foods, sun dried foods, deep fried foods, intrusion foods, fermented foods, cooking foods etc.

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Original Article

Review On Bats: Bio-Indicators and Ecosystem Engineers, Synthesizing Roles, Threats and Conservation Priorities for Chiropteran Diversity in India

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Abstract

Bats or *Carpe noctem*, act as vital bio-indicators amid rapid habitat alteration and climate shifts. This ecosystem engineers play important economic and ecological roles such as pollinators, seed dispersers, pest suppressors, bio-indicators, guano suppliers, predators, bush meat etc. Due to their creepy appearance and lack of information and misconceptions, these have remained neglected. Drawing from national surveys Chiroptera in India are estimated to be included in 9 families, 47 genera and 135 species. Yet, anthropogenic threats—habitat loss, hunting, deforestation, pesticides and wind turbines, jeopardize 25 globally critically endangered species. This paper analyzes data to propose targeted conservation framework. Recommendations emphasize legislation, awareness, and monitoring for sustainable management.

Keywords: Chiroptera, bioindicators, anthropogenic threats, diversity, conservation strategies.

Introduction

The Bats, unique for flight and echolocation, evolved during Eocene-era and constitute 20% of mammals worldwide. India with 137 species reflect hotspots like Western Ghats, but research scarcity persists amid urbanization. Zoonotic associations (rabies, Nipah) amplify scrutiny, yet ecological/economic values—pollinators, guano are overlooked. Ten times more Research work on bats is carried out internationally than in India on diversity, echolocation calls, reproduction, the diseases concerned with bats etc. [1,2,3]. Regional documentation of Indian bats shows 7 bat species in IISc campus [4], 25 species in western ghats [5], 28 species in Himachal Pradesh [6], 13 species in Delhi and 135 species in all over India [7,8,9]. However, owing to their nocturnal feeding habits, dark roosting or residing places, misconceptions in laymen's mind, lack in scientific knowledge, and social taboos; there is no reliable information on their ecology and conservation status in most places of India. Also, India's bat populations, face intensifying anthropogenic pressures that undermine their ecological services. This paper analyzes data from a key review document to propose targeted conservation frameworks.

Bio-indicator and ecosystem engineer role:

Bats are good biodiversity indicators of the ecosystem. The predatory behavior of bats, the sensitivity towards wide array of stresses, their occurrence world-wide except for polar regions, their roosting habit together in a single cave; the nocturnal habit of feeding and other activities, the strong flight in the air, the usage of echolocation and sonar radiation in detection of prey and their adaptability to man-made structures due to habitat destruction make the chiropterans good bio-indicators. Also, insectivorous bats occupy high trophic levels, they are sensitive to accumulations of pesticides and other toxins, and changes in their abundance may reflect changes in populations of arthropod prey species [1, 10]. Bats, the ecosystem engineers also provide several ecosystem services, and hence reflect the status of the plant populations on which they feed and pollinate as well as the productivity of insect communities [1].

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Bats of two families - Pteropodidae and Phyllostomidae pollinate 528+ species angiosperms worldwide [11, 12,13]. Chiropterans devour a palate of different sized invertebrates and vertebrates like insects, fish, arthropod orders, fishes, frogs, and other small mammals etc. [14,15] Previous studies on Microchiropteran bats show direct and indirect effects on the herbivorous animals and producers through both density mediated (consumption) and trait-mediated (behavioral) interactions and for nature balance [16]. Studies also indicated the predatory and insect population role of insectivorous bats like *Myotis austroriparius*, *M. septentrionalis*, *Nycticeius humeralis* and *Eptesicus fuscus* which consumed more than 25% of their body mass, and eliminated 99 % of potential insect pests [17,18].

Synthesizing role

Synthesizing role suggests guano mining by humans from bat roosting caves for utilization as agricultural crops fertilizers [19]. Studies on bats of Africa, Asia and Pacific islands suggest that bush meat of bats *Eidolon helvum*, *Pteropus*, *Tadarida*, *Cynopterus*, *Megaderma* etc. is considered highly proteinaceous, nutritious and cheaper [20]. Although, the bat bush meat generates income and serves as a luxury food, there are certain drawbacks associated with it in terms of spread of Zoonotic diseases like Ebola and Corona over hunting of the specific fruit bats [21]. Besides the above, the anticoagulant compound- Salivary plasminogen activator (DSPA -alpha-1 /desmotepase) present in Vampire Bat's saliva has been found effective in stroke treatment [22]. Also, chiropteran echolocation and the sonar radiation are used to produce BATMAVs (bat-like motorized aerial vehicles), bio medical ultrasounds, sensors for automatic systems and also in wireless communication [23].

Comprehensive Threat Landscape:

The major anthropogenic threats faced directly by the bats are the in the form of Habitat fragmentation and destruction, deforestation, mining, encroachment, direct hunting, intensive farming, use of pesticides in farming and wind turbines. The non- anthropogenic threats faced by the bats are cat attacks, White Nose Syndrome (WNS) disease, predation by spiders [24,25,26,27,28] etc.

Conservation Framework and Policy Implications:

Conservation Recommendations suggested are implementation of legislative laws for chiropteran conservation like Banning of bat hunting and Protecting bat roosting sites or trees; Reducing pesticide usage; Debunking the myths surrounding bats; NBSAP integration and achieving bioindicator monitoring and acoustic surveys.

Conclusions

Urgent policy implementation and sustaining India's ecosystems can elevate bats from neglected to prioritized

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Original Article

Promoting Agave americana leaf fibers for manufacturing ropes and cords

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Abstract

Like ornamental plant agave Americana is cultivated widely due to its adaptability to climatic conditions. It is naturalized in many regions, including Southern California, the West Indies, South America, the Mediterranean Basin, Africa, the Canary Islands, India, China, Thailand and Australia. The plant is significant source of natural fibers. Agave americana Fibers are prized for their, smoothness, durability, flexibility and resistance to wear and also used in an industrial application. These fibers have been used for manufacturing the, ropes, twines, cords, mats and other utility products which are used in the agricultural and construction fields. Sustainable and eco-friendly materials make fibers as renewable resource. The present work attracts the attention of Agave americana fibers for their uses in the production of high-quality ropes and cords.

Key words: Agave americana, agricultural, Synthetic fibers, natural fibers

Introduction

Agave americana (century plant, maguey or American aloe) is a species of flowering plant in the family Asparagaceae, native to Mexico and the United States. For extraordinary adaptation to unfavorable climatic conditions this species is known. This species is endemic to Mexico and areas of the southern United States, Texas. This species is planted an ornamental plant due to its spectacular look and low maintenance. Agave americana is monocarpic, which means it flowers only once on its lifetime. The plant produces a high blooming stalk and it dies after completion of seed development. This plant occurs around railway tracks, roadsides, riverbanks and wastelands and it occurs in arid and semi-arid areas, naturally where some other crops can be survived (Hulley et al., 2015). The uses of natural fibers as sustainable alternatives to synthetic materials have increased in recent decades. The synthetic fibers are prepared from non-renewable sources and cause health issues and environmental and during the manufacturing and disposal procedures (Pappu et al., 2016). On the other hand, natural fibers have garnered much interest of academics and industries for their eco-friendly, biodegradable, low density, renewability and local availability. Additionally, a lot of natural fibers have mechanical qualities equivalent to those of synthetic fibers and are utilized in a variety of industrial applications (Teklu, 2021). Thus, Agave americana fibre has been recognized as a potential natural resource for the development of sustainable composite materials and rope making applications.

Material and Method

Agave Americana plant

Kingdom: Plantae
Clade: Angiosperms
Order: Asparagales
Family: Asparagaceae
Subfamily: Agavoideae
Genus: *Agave*
Species: *A. americana*

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Agave Americana plant



Agave Americana leaf fibers

Plant characteristics

Agave americana has long life and has adaptation to grow in adverse weather conditions. Plant grow and reach of about 150 kg. In reproduction, it shows hermaphrodite blooms which have male and female both reproductive parts. Pollinating agents are moths, butterflies, and bats. For species' growth sandy and loamy soils are best. It flourishes in a different type of soil, including acidic, neutral, and alkaline. Agave americana grows in desert and semi-arid areas and show drought resistance (Kolte and Daberao, 2012).

Agave americana Fiber Extraction

Extraction by Mechanism

Extracted of fiber from Agave americana leaves is done by hand in past time, it was a labor-intensive and time-consuming work. In mechanical decortications method, blunt knives are fitted with revolving wheels and used to crush and beat the leaves. The fibrous bundles remain back from non-fibrous materials. Washing process is done for elimination of any remaining plant matter, the collected

fibers are dried in the sun (Kolte and Daberao, 2012; Hulley, 2015).

Extraction of Chemicals

Acids, alkalis, or enzymes are used for chemical extraction techniques. Acid treatments increase fiber crystallinity. Alkali treatments increase surface area, dissolve cementing elements between fibers and lowering the degree of polymerization. Enzymatic extraction enhances fiber swelling, decreases polymerization, and improves softness and flexibility. Fibers produced by enzymatic treatment are not difficult to process and more malleable (Kolte and Daberao, 2012; Hulley, 2015).

Agave americana fibers are used in the production of strong ropes, cords, twines.

Characteristics of Agave americana Leaf Fibers

Agave americana leaf fibers can be used for making ropes and cords because they have smooth, flexible, and strong tensile qualities. Fibers show hydrophilic nature and they absorb water in amounts greater than their weight. They burn like other cellulosic fibers and after ignition they create an odor similar to burning paper (Hulley et al., 2015).

Chemical Composition of Agave American fibers

Sr.No.	Composition	%
1.	Cellulose	68.42
2.	Hemi cellulose	15.67
3.	Lignin	4.85
4.	Moisture	7.69

(Mylsamy and Rajendran, 2010)

Application of fibers

Additionally, non-woven, bioplastic, geotextiles, and fiber-bound molded furniture are made from agave americana fibers. (Hulley and Kadole, 2014). Mat, rope, broom, cable, bags, clothes, shoes, and other household items can be made from the fibers. (Zwane and others, 2011)

In the past, ropes were made from cotton, dogbane, sisal, jute, hemp, flax abaca (Manila hemp), coir (coconut husks), leather, and hair. Fibers were used to make twines and ropes for agricultural uses. Agave Americana fibers are biodegradables. The ropes and other materials are recycled into paper. Man-made synthetic fibers include polyester,



nylon, and acrylic they are hazardous and are not environmentally friendly. While burning, it melts and spread a chemical odor. (Aarthi and Saryana, 2022). It shrinks and forms beads that adhere to the skin as it melts. The findings of a survey of 15 people on Agave americana fibers by expert craftsmen who manufacture various sorts of strong, durable ropes, cords, and nets. Sangola Taluka, Dist-Solapur (MS), was the site of the survey.

Some observations got according to 15 people about Agave americana fibers are as follows,

1. Agave americana plants are easily available but harvesting and fiber extraction is time consuming process. It needs man power.
2. They are biodegradable
3. They eco-friendly
4. They can Store at home or under shade.
5. They provide small business.

Final thoughts and suggestions:

Agave Americana fibers are environmentally friendly and biodegradable. Agave Americana plant cultivation will give farmers and laborers access to small businesses. The manufactured ropes may be utilized for tying and handling livestock as well as agricultural uses. It will serve as a substitute for synthetic fiber. Natural fibers like lingo cellulose and proteins are completely sustainable, renewable, and biodegradable, according to Saranya and Aarthi's research from 2022. They are extensively utilized in the clothing industry and interior design and have a number of advantages over synthetic fibers. Unfortunately, their application is relatively limited due to their flammability and lack of resistance to biodegradation; however, non-durable finishers can be used to protect natural fibers from flammability. Agave americana fibers can be utilized in technological applications such reinforced composite materials, paper manufacturing, non-woven fabrics, and geotextiles, according to research done by Hulley et al. (2015). According to Msahali et al.'s 2005 paper on the characteristics of agave Americana leaf fibers, the fibers can be utilized in technological applications such geotextiles and reinforced materials. Additionally, it was stated that fibers are traditionally extracted in Tunisia and used to make twines and ropes for use in agriculture and the maritime industry. According to Zawane et al. (2011), the majority of women in Swaziland, particularly those living in rural areas, have found that the Agave americana, also known as the century plant, is a plant of life. According to Sreeremya's (2018) research, agave americana fibers can be utilized as reinforced materials. According to Mylsamy's (2011) research, Agave americana fiber are used for producing lightweight composite materials. The fibers have biodegradability. According to Patil and Yadav (2023), natural fibers have different benefits like readily availability, and its ecologically importance. Agave americana cultivation and its applications will provide massive jobs in agricultural and construction sectors without environmental pollution because the fibers of Agave americana are ecofriendly and biodegradable.

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Conflicts of interest

The authors declare that there are no conflicts of interest regarding the publication of this paper

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