

Synthesis and Characterisation of Fe₃O₄ Nanoparticles for Photo-Fenton Degradation of Methylene Blue Dye

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ABSTRACT

Developing efficient and recyclable photocatalytic materials for dye degradation is therefore of significant importance. In the present work, magnetite (Fe₃O₄) nanoparticles were synthesised via chemical co-precipitation using FeCl₃·6H₂O and FeCl₂·4H₂O as precursor salts. The XRD patterns confirmed the formation of crystalline Fe₃O₄ with a characteristic spinel structure. Optical analysis revealed a pronounced absorption in the ultraviolet region centred near 320 nm, corresponding to an estimated band-gap energy of 3.80 eV. SEM micrographs demonstrated the formation of irregularly distributed mesoporous particles, which provide a high surface area favourable for catalytic applications. The photo-Fenton catalytic performance of the synthesized Fe₃O₄ nanoparticles was evaluated by employing methylene blue (MB) as the target contaminant. Furthermore, optimisation of operational parameters showed that a catalyst loading of 60 mg per 100 mL solution, combined with the addition of 20 µL of 30% H₂O₂, resulted in nearly complete degradation (99%) of methylene blue within 30 min, with a rate constant of 0.0269 min⁻¹. These findings demonstrate that the synthesised Fe₃O₄ nanoparticles possess significant potential as efficient, magnetically recoverable photocatalysts for wastewater purification and the treatment of dye-contaminated water.

Keywords: Fe₃O₄; UV light, photocatalyst; dye degradation.

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1. INTRODUCTION

Iron-based nanomaterials have attracted considerable attention in environmental remediation owing to their exceptional catalytic activity and effectiveness in removing a wide range of organic and inorganic contaminants from aqueous systems [1,2]. Their favourable characteristics, including strong redox behaviour, low toxicity, cost-effectiveness, facile

recovery, and significant absorption within the visible-light spectrum, make them promising candidates for the treatment of emerging water pollutants [3]. Furthermore, several iron-containing nanostructures exhibit intrinsic magnetic properties, enabling efficient separation from treated solutions when an external magnetic field is applied [4]. This feature not only facilitates catalyst recovery and

reuse but also minimises the release of secondary nanoparticle pollutants into the environment, thereby enhancing the sustainability of wastewater treatment technologies [5].

Among the various magnetic iron oxides, magnetite (Fe₃O₄) has developed as a widely investigated material due to its unique combination of magnetic, chemical, and catalytic properties [6]. Fe₃O₄ nanoparticles have been employed in numerous applications, including environmental remediation, biomedical engineering, sensing technologies, and heterogeneous catalysis. In advanced oxidation processes, particularly Fenton and photo-Fenton reactions, iron-based catalysts play a crucial role in activating hydrogen peroxide to generate highly reactive oxygen species that degrade persistent organic contaminants. Under acidic conditions, the catalytic cycling between Fe²⁺ and Fe³⁺ significantly enhances the decomposition of hydrogen peroxide, thereby generating hydroxyl radicals with strong oxidative potential^{7,8}. The incorporation of light irradiation in photo-Fenton systems further accelerates these reactions by promoting iron regeneration and increasing radical production, thereby improving pollutant degradation efficiency and mineralisation rates. Over the past decades, photo-Fenton technology has demonstrated remarkable effectiveness in eliminating various recalcitrant organic compounds, converting them into environmentally benign end products such as carbon dioxide and water [6,9]. Consequently, this process has been widely explored as an efficient wastewater treatment strategy and as a complementary approach to conventional remediation methods. The physicochemical properties of Fe₃O₄ nanoparticles, including particle size, morphology, crystallinity, magnetic behaviour, and catalytic performance, are strongly influenced by the synthesis methodology. Therefore, selecting an appropriate preparation route is essential for tailoring material characteristics to specific applications [7,10]. Various synthetic methods were developed for the production of magnetite

nanoparticles, including sol-gel processing, microemulsion methods, co-precipitation, and hydrothermal or solvothermal approaches. Each technique offers distinct advantages and limitations related to particle uniformity, reaction time, temperature requirements, scalability, and cost.

In the present study, Fe₃O₄ nanoparticles were synthesised via co-precipitation. The catalytic performance of the synthesised magnetically recoverable nanocatalysts was evaluated by UV-light-assisted photo-Fenton degradation of methyl blue, a model emerging pollutant, at an initial concentration of 20 ppm. Particular emphasis was placed on understanding the relationship between synthesis conditions and catalyst characteristics, including particle size, magnetic response, and photocatalytic efficiency, to optimise their applicability in wastewater treatment processes.

2. COPRECIPITATION-ASSISTED SYNTHESIS OF Fe₃O₄ NANOPARTICLES

Fe₃O₄ nanoparticles were prepared through the use of Ferric Chloride Hexahydrate and Ferrous Chloride Tetrahydrate (Ammonium Hydroxide purity, Rankem lab). In this preparation, we used 5.4 g of Ferric Chloride Hexahydrate and 1.99 g of Ferrous Chloride Tetrahydrate dissolved in 50 ml of water in a beaker. The mixture was heated up to 80 °C while being stirred using a magnetic stirrer. The pH value was adjusted to 10 using NH₄OH (28%) solution until the formation of precipitates occurred. The precipitates were then heated to 60 °C to obtain Fe₃O₄ nanoparticles.

3. RESULTS AND DISCUSSION

Phase analysis of Fe₃O₄ was performed using XRD, and the results are shown in Figure 1. Purity of the Fe₃O₄ has diffraction patterns in agreement with the orthorhombic phase (JCPDS No 75-1609). The crystallite size of Fe₃O₄ was 104.36 nm. The specific diffraction peaks for Fe₃O₄ were identified as corresponding to 2θ values of 30, 35, 45, 54, and 62, namely (220), (311), (400), (422), (511), and (440).

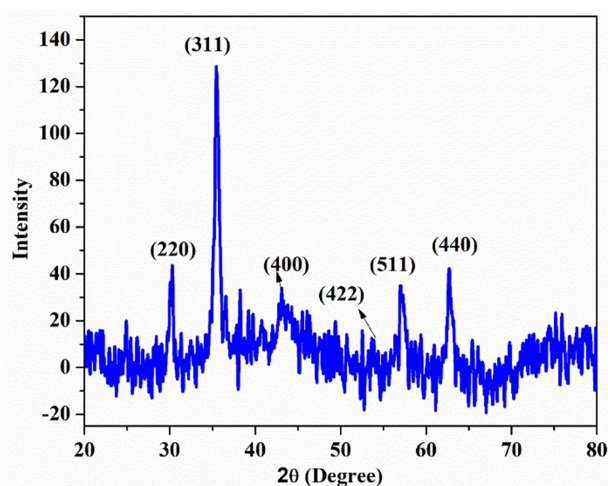


Figure 1: XRD pattern of Fe_3O_4 nanoparticles

The pristine Fe_3O_4 microstructure and size have been studied through FESEM. The micrograph of Fe_3O_4 provides insight into the growth of irregular nanoparticles measuring 20-25 nm (Figure 2a-b). UV-Visible Diffused Reflection Spectroscopy has been performed to ascertain

the bandgap of Fe_3O_4 . Figure 3a shows that Fe_3O_4 pristine exhibits strong absorption in the UV range. Tauc Plot $(\alpha h\nu)^2$ vs $h\nu$ (eV) has been shown in Figure 3b. The band gap energy of Fe_3O_4 has been estimated as 3.80 eV (Figure 3b).

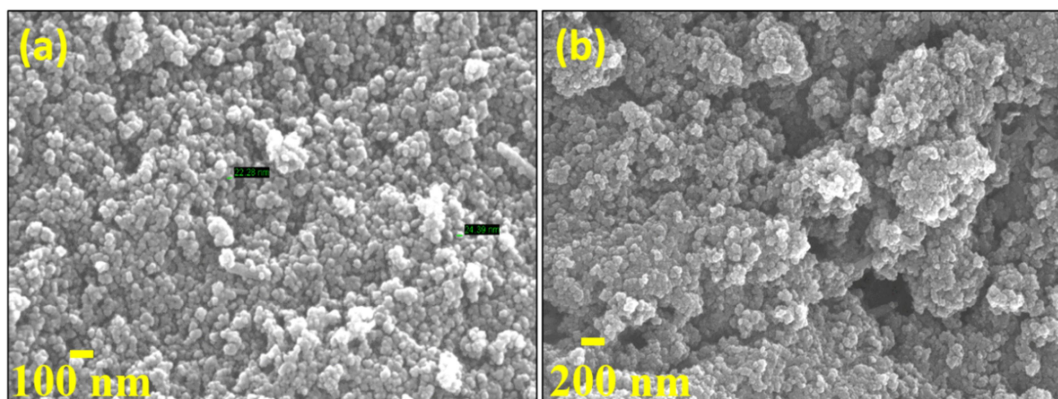


Figure 2: (a-b) FESEM Image of Fe_3O_4

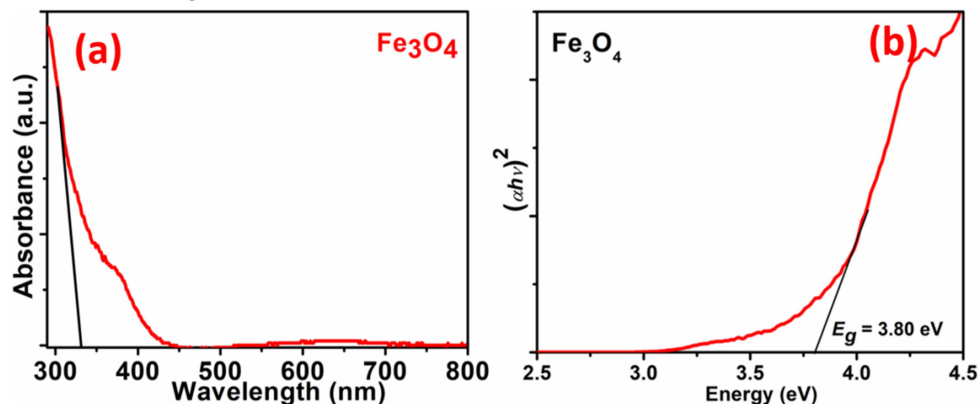


Figure 3: (a) Diffuse reflectance UV-Vis spectra of Fe_3O_4 nanoparticles and (b) optical band gap determination from the Tauc plot.

The chemical functionalities present in the synthesised Fe₃O₄ nanoparticles were investigated using Fourier Transform Infrared (FTIR) spectroscopy. As illustrated in Figure 4, broad absorption bands observed at 3435 and 1632 cm⁻¹ correspond to the stretching and bending vibrations of hydroxyl (-OH) groups associated with adsorbed water molecules and surface hydroxyl species. A prominent absorption band in the 580–590 cm⁻¹ region is

characteristic of Fe–O lattice vibrations, confirming the formation of magnetite nanoparticles. Additionally, a band detected near 1300 cm⁻¹ can be attributed to carboxylate-related functional groups and C–O–C stretching vibrations [11]. The presence of these oxygen-containing groups may contribute to nanoparticle stabilisation and influence the nucleation and growth processes during Fe₃O₄ formation

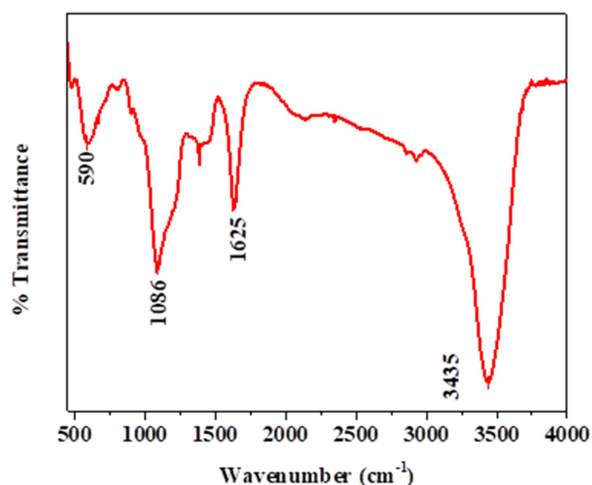


Figure 4: FTIR pattern of Fe₃O₄ nanoparticles.

3.1 Photo-Fenton catalytic degradation

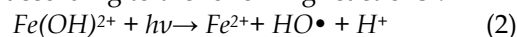
The synthesised Fe₃O₄ nanoparticles were assessed for their photocatalytic activity by degrading methylene blue (MB) under photo-Fenton reaction conditions. As illustrated in Figure 5a, the introduction of a small quantity of hydrogen peroxide (20 µL of 30% v/v H₂O₂) into the Fe₃O₄/UV system significantly enhanced dye removal compared with photocatalysis alone. The synergistic interaction among the catalyst, hydrogen peroxide, and ultraviolet irradiation accelerated the generation of reactive oxygen species, thereby promoting rapid MB degradation. This combined oxidation pathway is characteristic of the photo-Fenton process. A pseudo-first-order kinetic model was employed to investigate the degradation behaviour of the pollutant.

$$\ln(C_0/C_t) = kt \quad (1)$$

A linear relationship between $\ln(C_0/C_t)$ and reaction time confirmed the applicability of the kinetic model (Figure 5b). Under optimised

operating conditions, the apparent rate constant reached 0.022 min⁻¹, demonstrating the high catalytic efficiency of the Fe₃O₄-based photo-Fenton system. Control experiments revealed that direct photolysis of MB under UV irradiation, in the absence of both catalyst and oxidant, resulted in negligible degradation. Likewise, experiments conducted using only H₂O₂ or only Fe₃O₄ showed considerably lower removal efficiencies than the complete photo-Fenton system. These findings confirm that the simultaneous presence of Fe₃O₄ nanoparticles, hydrogen peroxide, and UV light is essential for achieving efficient dye mineralisation. The contribution of light irradiation was further assessed by comparing degradation under illuminated and dark conditions. In the absence of UV light, the Fe₃O₄/H₂O₂ system behaved as a conventional Fenton process, achieving only about 60% degradation after 30 min. The slower degradation rate observed in the dark indicates that UV irradiation is critical for accelerating hydroxyl radical production and catalyst

regeneration. In contrast, the reaction mixture containing only H_2O_2 , without a catalyst or irradiation, exhibited minimal dye removal, indicating that MB was not susceptible to direct oxidation under these conditions. The enhanced activity under UV irradiation can be attributed to additional radical-generation pathways. Light promotes the photoreduction of ferric species to ferrous ions while simultaneously inducing hydrogen peroxide photolysis, leading to increased formation of hydroxyl radicals according to the following reactions¹:



The influence of operational parameters on dye degradation was systematically investigated. Among the tested conditions, acidic media exhibited superior catalytic activity, with optimum performance obtained at pH 3 (Figure 6a). Although degradation occurred at near-neutral pH values, the reaction rate was significantly lower because iron cycling and hydroxyl radical generation are more efficient under acidic conditions. The coexistence of Fe^{2+} and Fe^{3+} species at low pH facilitates continuous radical production and sustains the oxidation process. Catalyst loading also strongly affected the degradation efficiency (Figure 6b). Increasing the Fe_3O_4 concentration from 0.4 to 0.6 mg mL^{-1} enhanced the reaction rate due to the availability of a greater number of active sites. However, a further increase to 0.8 mg mL^{-1} resulted in a decline in the degradation rate. This reduction may be associated with particle agglomeration, light-scattering effects, and decreased utilisation of active catalytic sites at excessive catalyst concentrations [12]. The initial MB concentration also influenced photocatalytic performance (Figure 6c). Faster degradation kinetics were observed in 20 ppm MB solutions

compared with 40 ppm MB solutions. At higher dye concentrations, the increased number of pollutant molecules competes for reactive species, reducing light penetration through the solution and ultimately slowing the oxidation process. Among all investigated variables, solution pH and catalyst dosage exerted the greatest influence on degradation efficiency, whereas hydrogen peroxide was indispensable for initiating and sustaining the photo-Fenton reaction. Maintaining the pH within the narrow range of 2.8–3.2 was particularly important for preserving catalytic activity throughout the reaction. The oxidation mechanism involves continuous redox cycling between Fe^{2+} and Fe^{3+} species present on the Fe_3O_4 surface. These reactions generate highly reactive hydroxyl radicals, responsible for dye degradation⁹:



Hydroxyl radicals have an extremely high oxidation potential and can oxidise dye molecules through a series of reactions, leading to their decomposition [13]. However, at elevated pH values, iron ions tend to form insoluble hydroxide precipitates, thereby decreasing the availability of catalytically active species and adversely affecting process efficiency. To validate the catalytic role of Fe_3O_4 nanoparticles, comparative experiments were performed with and without the catalyst, and with and without hydrogen peroxide. The significantly lower degradation observed in these control systems confirms that the remarkable dye removal efficiency results from the synergistic interaction between Fe_3O_4 nanoparticles, hydrogen peroxide, and ultraviolet irradiation within the photo-Fenton process.

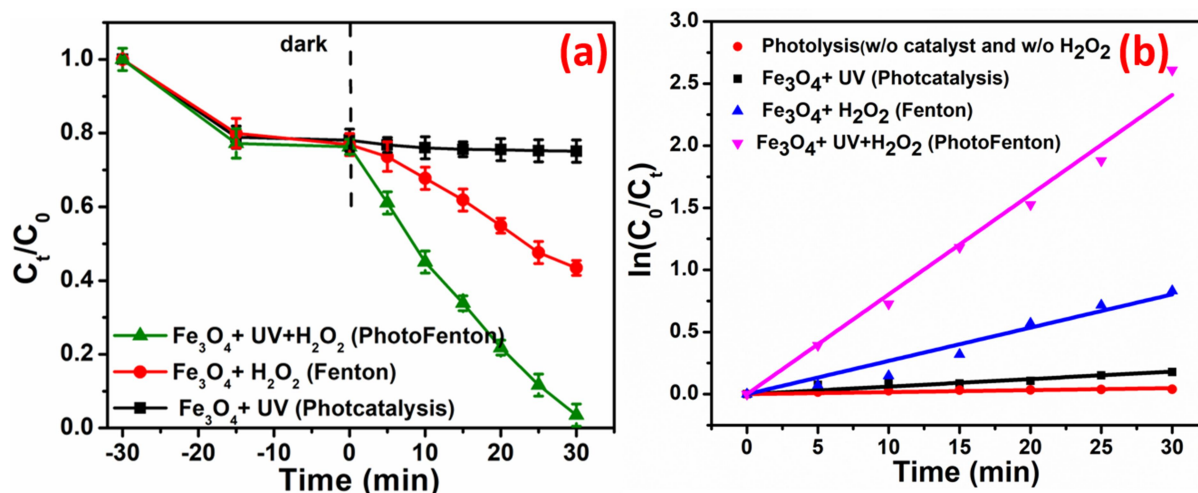


Figure 5: (a) MB degradation by Fe₃O₄ nanoparticles, (b) Kinetics study.

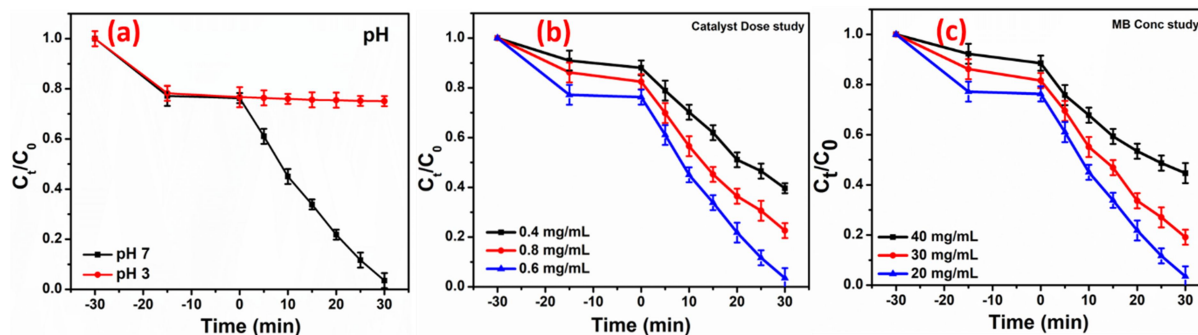


Figure 6: Effect of pH 3 and 7, Fe₃O₄ dose and RhB concentration on photo Fenton degradation of MB by Fe₃O₄ nanoparticles.

4. CONCLUSION

Magnetite (Fe₃O₄) nanoparticles were successfully synthesised via a simple, cost-effective co-precipitation route. The characterisation results confirmed the formation of crystalline Fe₃O₄ nanoparticles with morphological and optical properties suitable for catalytic applications. The photocatalytic performance of the synthesised material was investigated through the UV-assisted photo-Fenton degradation of methylene blue (MB). Experimental optimisation demonstrated that the degradation efficiency was strongly influenced by operational parameters, with optimal conditions at pH 3, a catalyst loading of 0.06 mg mL⁻¹, and an initial dye concentration of 20 mg L⁻¹. Under these conditions, the photo-Fenton process exhibited excellent catalytic activity, yielding a pseudo-first-order rate constant of 0.026 min⁻¹ and rapid dye removal. The enhanced degradation performance was attributed to the efficient generation of reactive

oxygen species resulting from the synergistic interaction among Fe₃O₄ nanoparticles, hydrogen peroxide, and UV irradiation. Overall, the results highlight the potential of Fe₃O₄ nanoparticles as efficient, reusable, and environmentally benign catalysts for advanced oxidation processes. Their high catalytic efficiency and facile recovery make them promising candidates for treating dye-contaminated wastewater and other emerging organic pollutants in aquatic environments.

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