

Article

Evaluating the Photocatalytic Activity of Green Synthesized Iron Oxide Nanoparticles

Devendra Khadka ^{1,2}, Prayas Gautam ¹, Rabin Dahal ³, Moses D. Ashie ³ , Hari Paudyal ¹ , Kedar Nath Ghimire ¹, Bishweshwar Pant ^{4,*} , Bhoj Raj Poudel ^{5,*} , Bishnu Prasad Bastakoti ³  and Megh Raj Pokhrel ¹

¹ Central Department of Chemistry, Tribhuvan University, Kathmandu 44600, Nepal; meghrajpokhrel2000@gmail.com (M.R.P.)

² Department of Chemistry, Tribhuvan Multiple Campus, Tribhuvan University, Tansen 32500, Nepal

³ Department of Chemistry, College of Science and Technology, North Carolina Agricultural and Technical State University, Greensboro, NC 27411, USA

⁴ Carbon Composite Energy Nanomaterials Research Center, Woosuk University, Wanju 55338, Republic of Korea

⁵ Department of Chemistry, Tri-Chandra Multiple Campus, Tribhuvan University, Kathmandu 44600, Nepal

* Correspondence: bisup@woosuk.ac.kr (B.P.); bhoj.poudel@trc.tu.edu.np (B.R.P.)

Abstract: Water pollution from dyes is a major environmental challenge, demanding advanced materials for efficient degradation. In this study, we synthesized iron oxide nanoparticles (IONPs) using an aqueous extract of *Senegalia catechu* leaves and evaluated their photocatalytic activity in methylene blue (MB) dye degradation under sunlight irradiation. The IONPs were characterized by UV-visible spectroscopy (UV-vis), Fourier Transform Infrared Spectroscopy (FT-IR), X-ray Diffraction (XRD), Scanning Electron Microscopy (SEM), and Energy Dispersive X-ray Spectroscopy (EDS). XRD pattern showed a highly crystalline structure with an average crystallite size of 34.7 nm, while SEM images revealed predominantly spherical particles with uneven surface texture. Photocatalytic efficiency exceeded 80% MB dye degradation after 120 min of sunlight exposure. Optimization of catalyst dose, pH, dye concentration, and other parameters is essential for maximizing degradation efficiency. The IONPs demonstrated reusability over four degradation cycles, retaining effective photocatalytic performance. This study underscores the potential of green-synthesized IONPs as eco-friendly photocatalysts for wastewater treatment and environmental remediation.

Keywords: environmental remediation; green synthesis; *Senegalia catechu*; methylene blue degradation; photocatalyst



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1. Introduction

Rapid industrialization and urbanization over the past few decades have significantly increased the discharge of synthetic dyes into water bodies, resulting in severe environmental and health hazards [1]. Among these pollutants, methylene blue (MB) dye is an extensively used cationic dye in the textile, paper, and plastic industries. It leads to its abundant presence in industrial effluents [2]. It is a toxic, carcinogenic, and nonbiodegradable chemical [3]. This dye seriously threatens public health and the environment if it enters water sources due to improper disposal [4]. Therefore, treating these effluents to remove the dye before discharging them into water bodies is essential. Traditional wastewater treatment methods often fail to remove dyes like methylene blue (MB) completely and may generate harmful by-products, underscoring the need for more efficient and sustainable approaches. Photocatalysis is a promising and eco-friendly method for degrading pollutants, including dyes such as methylene blue. It involves the use of light to activate catalysts, which then degrade pollutants into harmless products [5,6].

Iron oxide occurs in three distinct crystalline structures: FeO, Fe₂O₃, and Fe₃O₄. Within these nanocrystalline phases, Fe₂O₃ is found in four polymorphs: alpha, beta, gamma, and epsilon Fe₂O₃ [7]. Magnetic forms of iron oxide nanoparticles, such as maghemite (γ-Fe₂O₃) and magnetite (Fe₃O₄), have biocompatibility, strong superparamagnetic properties, and low toxicity [8]. Iron oxide, mainly hematite (α-Fe₂O₃) nanoparticles, have emerged as efficient photocatalysts due to their abundance, stability, non-toxicity, low cost, and suitable bandgap (1.56–2.1 eV) for sunlight absorption [9–11]. However, conventional synthesis techniques are often associated with the use of harmful chemicals, high energy demands, and highly expensive equipment, raising environmental and economic concerns. In contrast, the biosynthesis of IONPs using plant extracts offers an eco-friendly and sustainable alternative. This green approach leverages natural reducing and stabilizing agents in plant extracts, minimizing the need for harmful chemicals and energy-intensive processes [12]. Various studies have shown that biosynthesized hematite nanoparticles are effective in photocatalytic applications, indicating their potential for large-scale environmental remediation by producing unique morphologies and properties that enhance photocatalytic activity [13].

This study introduces the novel use of *Senegalia catechu* leaf extract as an efficient reducing and capping agent for the synthesis of IONPs. Chemical analysis of *Senegalia catechu* identifies abundant phenolics, tannins, and flavonoids, such as catechin, epicatechin, epigallocatechin, taxifolin, and procyanidin [14]. These compounds can act as both reducing agents, facilitating nanoparticle formation, and capping agents, promoting its stability [15]. This innovative biosynthetic approach allows for a low-temperature synthesis process, eliminating the need for expensive and toxic surfactants and reducing high energy demands. This method not only improves the economic feasibility of the synthesis but also minimizes its environmental impact, making it a sustainable alternative to conventional methods. Moreover, the study explores the efficacy of the biosynthesized IONPs as photocatalysts for the degradation of MB dye under sunlight irradiation. The photocatalytic performance of the IONPs is systematically evaluated, demonstrating significant degradation efficiency. Additionally, the study assesses the reusability of the catalyst, examining its stability and effectiveness over multiple cycles of degradation.

2. Results and Discussion

2.1. UV–Visible Spectroscopic Analysis

The addition of aqueous leaf extract to a solution of FeSO₄·7H₂O caused an immediate color change from light yellow to black, indicating the formation of IONPs [16]. UV–visible spectroscopic analysis confirmed this by revealing the characteristic absorption peak of IONPs, indicating their successful synthesis. The dark color originated from the vibration induced by surface plasmon excitation in the IONPs [17]. The presence of an absorption band at 580 nm, as shown in Figure 1, confirmed the formation of IONPs. The formation of IONPs occurs through a process that involves the complexation of iron salt followed by the encapsulation of iron by phenolic compounds [17,18]. The absorption band observed in the UV range (around 300–350 nm) is attributed to ligand-to-metal charge transfer from O^{2−} to Fe³⁺ [19], while the distinct peak observed around 580 nm in the visible region corresponds to electronic transitions within the Fe³⁺ ions, likely involving 3d orbitals. These findings align with those reported by Da'na et al. and Kaur et al. [16,20].

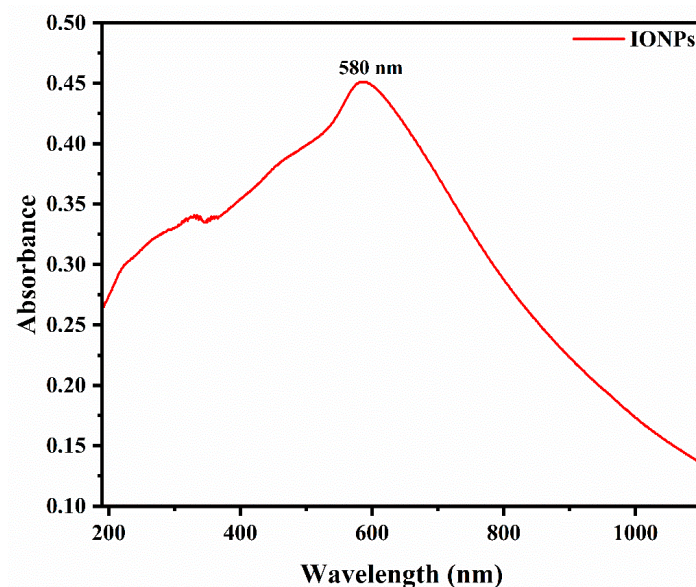


Figure 1. UV-visible spectra of synthesized IONPs.

2.2. FTIR Analysis

FTIR analysis was conducted to identify the functional groups in the phytochemicals present in leaf extract responsible for metal ion reduction. This analysis also elucidated the key chemical bonds, particularly the Fe–O bonds, which significantly contribute to improving photocatalytic performance by facilitating light absorption and charge transfer. The detailed FTIR spectra provided insights into the surface chemistry and bonding interactions, directly linking these features to the nanoparticles' photocatalytic efficiency. The FTIR spectra were obtained from the 4000 to 400 cm^{-1} range. Figure 2 presents the FTIR spectra, showing distinct absorption bands at wave numbers 527, 1057, 1402, 1584, and 3224 cm^{-1} for the leaf extract and at 436, 525, 1119, and 1644 cm^{-1} for the IONPs. This indicates that the various functional groups are involved in the interaction between the leaf extract and the iron precursor during the formation of IONPs. On the spectra of IONPs, two sharp peaks 436 cm^{-1} and 525 cm^{-1} resemble Fe–O stretching reported by Lassoued et al. [21] and also following the data reported by Hussain et al. [22]. The sharp peaks at 525 cm^{-1} and 436 cm^{-1} correspond to Fe–O stretching and O–Fe–O bending vibrations, respectively, in $\alpha\text{-Fe}_2\text{O}_3$ (hematite) nanoparticles [23]. Similar FTIR spectra were reported by Lohrasbi et al., indicating a peak at 516 cm^{-1} attributed to Fe–O stretching that enhances light absorption and electron transfer, generating superoxide radicals that effectively degrade organic pollutants during photocatalytic reactions under sunlight irradiation [24]. FTIR analysis revealed a broad peak within the 3200–3400 cm^{-1} range, which is characteristic of O–H stretching vibrations in alcohol or phenol [23,25]. Peaks in the 1600–1400 cm^{-1} region are characteristic of aromatic C=C stretching vibrations, while the peak observed in the 1100–1150 cm^{-1} region can be attributed to C–C bond stretching vibrations [26]. The increased contact between the nanoparticles and the MB dye enhances the degradation by bringing the pollutants in closer proximity to the reactive sites on the nanoparticle surface. The sharp peaks 436 and 525 cm^{-1} signify the successful formation of iron oxide nanoparticles. Additionally, other peaks might suggest the phytochemicals present on the exterior of nanoparticles. Changes in the peak positions within the 400–4000 cm^{-1} range can indicate the adsorption of functional groups onto the iron oxide surface. Weak or no peaks at higher wave numbers are likely due to the removal of organic functional groups and the formation of more pure and crystalline iron oxide structures at a higher calcination temperature of 800 $^{\circ}\text{C}$.

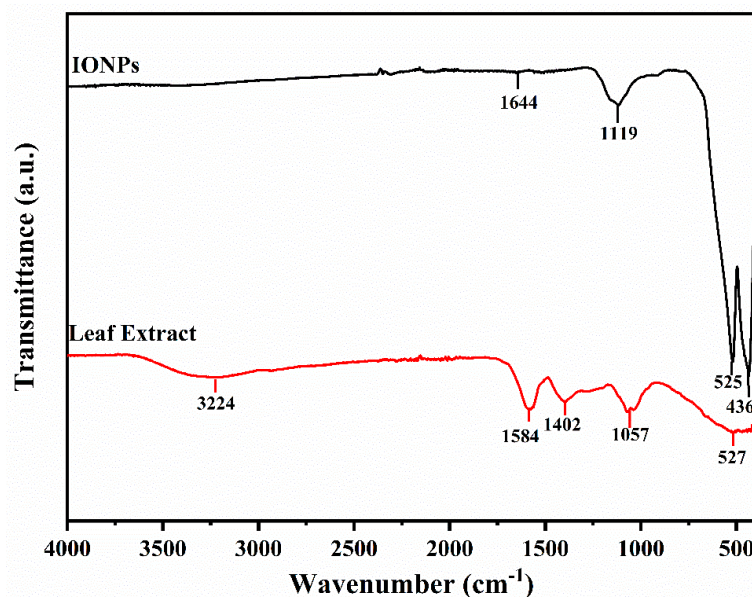


Figure 2. FTIR spectra of *Senegalia catechu* leaf extract and IONPs.

2.3. XRD Study

An X-ray diffraction (XRD) technique was employed to assess the crystallinity of the synthesized IONPs with the resulting pattern shown in Figure 3. The XRD pattern displays sharp and intense peaks, indicating a highly crystalline structure for the IONPs. The distinctive diffraction peaks were detected at angles of 2θ of 24.02° , 33.06° , 35.54° , 40.78° , 49.40° , 54.0° , 57.56° , 62.38° and 63.98° . These diffraction peaks were matched to (012), (104), (110), (113), (024), (116), (122), (214), (300) of crystal planes respectively. Additionally, these values align with the previously reported results [27–29]. The observed diffraction patterns closely match those documented in JCPDS card no. 00-024-0072, suggesting the rhombohedral structure of pure hematite ($\alpha\text{-Fe}_2\text{O}_3$). This is further supported by the low magnetization value of 0.5 emu/g for ($\alpha\text{-Fe}_2\text{O}_3$) calcined at 600°C , indicating the complete phase transition from $\gamma\text{-Fe}_2\text{O}_3$ to $\alpha\text{-Fe}_2\text{O}_3$ [30]. Using X-ray diffraction analysis and the Debye–Scherrer equation, the synthesized IONPs were found to have an average crystallite size of 34.07 nm. Typically, the mechanical and physical characteristics of these attributes could boost their photocatalytic activity [31].

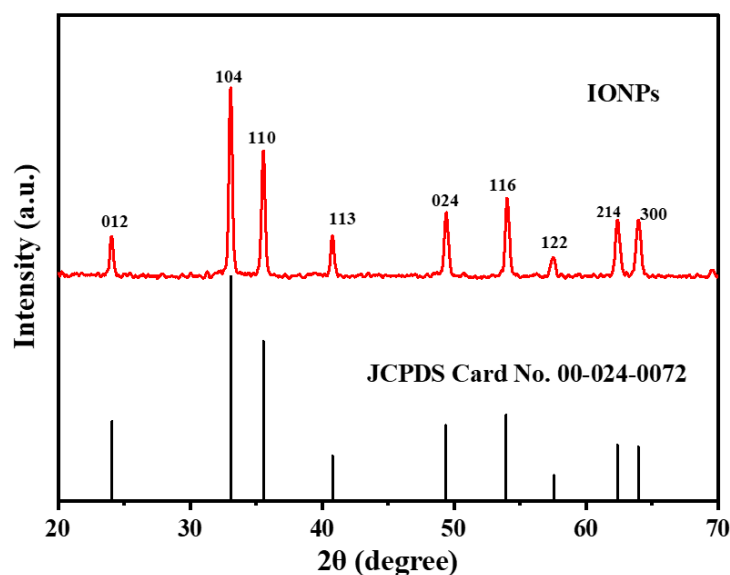


Figure 3. XRD pattern of synthesized IONPs.

2.4. Morphological and Elemental Analysis

The morphology of the synthesized IONPs was examined using SEM. Figure 4 displays SEM images of the synthesized IONPs captured at various magnifications. The images reveal significant morphological details and insights into the aggregation behavior of the particles. At lower magnifications, as seen in images Figure 4a,b, the nanoparticles exhibit a relatively loose, irregular distribution with noticeable aggregation. At moderate magnification, as seen in image Figure 4c, the agglomerates become more pronounced, indicating that the particles tend to cluster rather than remain isolated. Higher magnification images in Figure 4d–f provide a closer view of these aggregates, revealing that the individual nanoparticles are mostly spherical with an uneven surface texture. Lassoued et al. have also documented comparable results [21]. It is evident that hematite nanoparticles predominantly exist in the form of granules containing both small as well as large spherical particles demonstrating a high degree of crystallization. Phytochemicals in the extract, reaction conditions, and precursor concentration significantly influence the morphology of nanoparticles. These results align with the findings of previous studies by Lassoued et al. and Lohrasbi et al., affirming the elemental composition of synthesized IONPs [21,24].

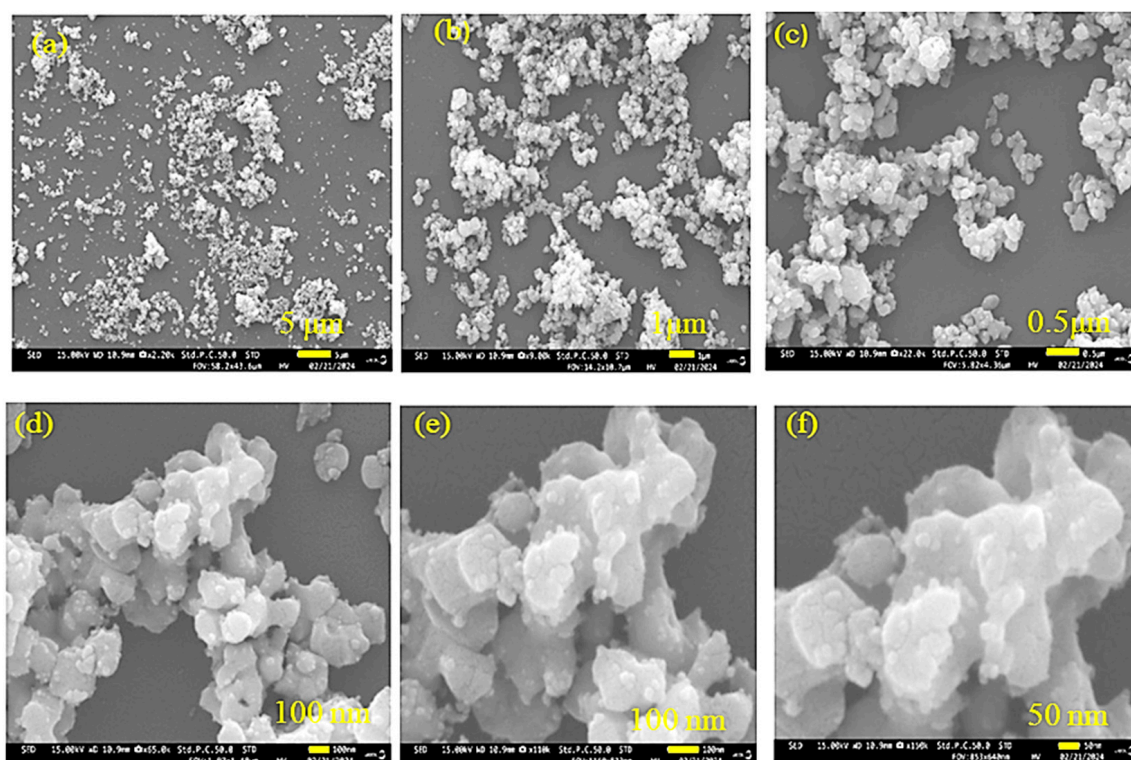


Figure 4. SEM images of IONPs with scale bars of (a) 5 μm , (b) 1 μm , (c) 0.5 μm , (d) 100 nm, (e) 100 nm, and (f) 50 nm.

The quantitative analysis reveals that the nanoparticles primarily consist of iron and oxygen, with weight percentages of 66.07% and 27.95%, respectively, and atomic percentages of 35.95% for iron and 48.90% for oxygen. This composition confirms that iron oxide is the main constituent. The carbon peak significantly influences the nanoparticle morphology [31]. The EDS spectrum of the IONPs (Figure 5) provides a detailed analysis of its elemental composition. The spectrum displays prominent peaks corresponding to oxygen (O) and iron (Fe) and a smaller peak for carbon (C). A prominent K- α peak at 6.4 keV and 0.72 keV, indicating the presence of iron (Fe) atoms within the nanoparticles, and K- α lines at $\approx$ 0.6 keV, characteristic of oxygen (O) atoms. The carbon peak could be attributed to residual organic matter or contamination during sample preparation. The high intensity of the oxygen and iron peaks indicates the successful synthesis of iron oxide nanoparticles.

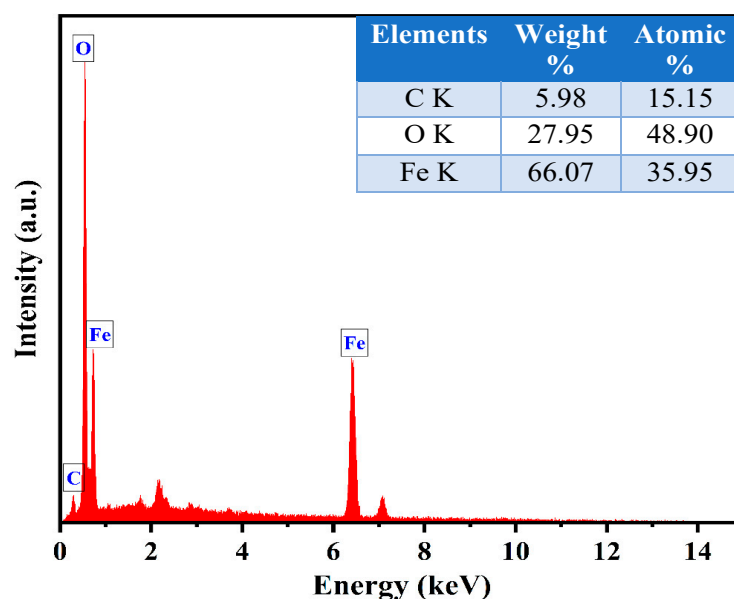
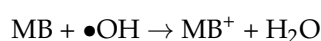
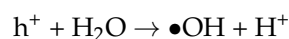
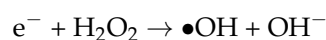
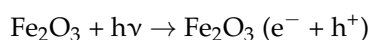


Figure 5. EDS spectrum of synthesized IONPs.

2.5. Photocatalytic Degradation of MB

The effectiveness of the synthesized IONPs in methylene blue dye degradation was assessed. Figure 6 illustrates time-dependent changes in the UV–visible spectrum of MB dye when exposed to sunlight irradiation in the presence of IONPs acting as a photocatalyst. Photocatalytic degradation of MB reaches approximately 85% after 7 h of light exposure without H_2O_2 . However, in the presence of H_2O_2 , this degradation occurs in about 2 h. This demonstrates that H_2O_2 significantly accelerates the photocatalytic degradation of MB. The addition of H_2O_2 in conjunction with a catalyst enhances the formation of highly reactive $\bullet\text{OH}$ radicals, leading to more efficient dye degradation [32]. It involves several steps. Upon irradiation with sunlight, IONPs (Fe_2O_3) generate electron–hole pairs. The photogenerated electrons reduce H_2O_2 , producing hydroxyl radicals ($\bullet\text{OH}$), while the holes oxidize water or hydroxide ions, also forming $\bullet\text{OH}$. These hydroxyl radicals are highly reactive and interact with the MB molecules, resulting in their breakdown into smaller organic fragments and eventually complete mineralization into CO_2 , H_2O , and inorganic ions [33]. Additionally, H_2O_2 helps to minimize electron–hole recombination by scavenging electrons, ensuring continuous production of reactive species [34]. This synergistic action of IONPs and H_2O_2 demonstrates a highly effective approach for the photocatalytic degradation of MB. Despite observing high degradation efficiency in our experiments, we did not carry out radical scavenger experiments to directly identify the specific reactive oxygen species (ROS) contributing to the degradation process. The photocatalytic degradation mechanism proposed in this study is based on literature where ROS generation has been shown to facilitate photocatalytic activity. Further investigation through radical scavenger experiments would be necessary to conclusively determine the contributions of different ROS species to the observed photocatalytic degradation. To achieve the highest removal efficiency, the influence of various reaction conditions on the photocatalytic process was investigated.



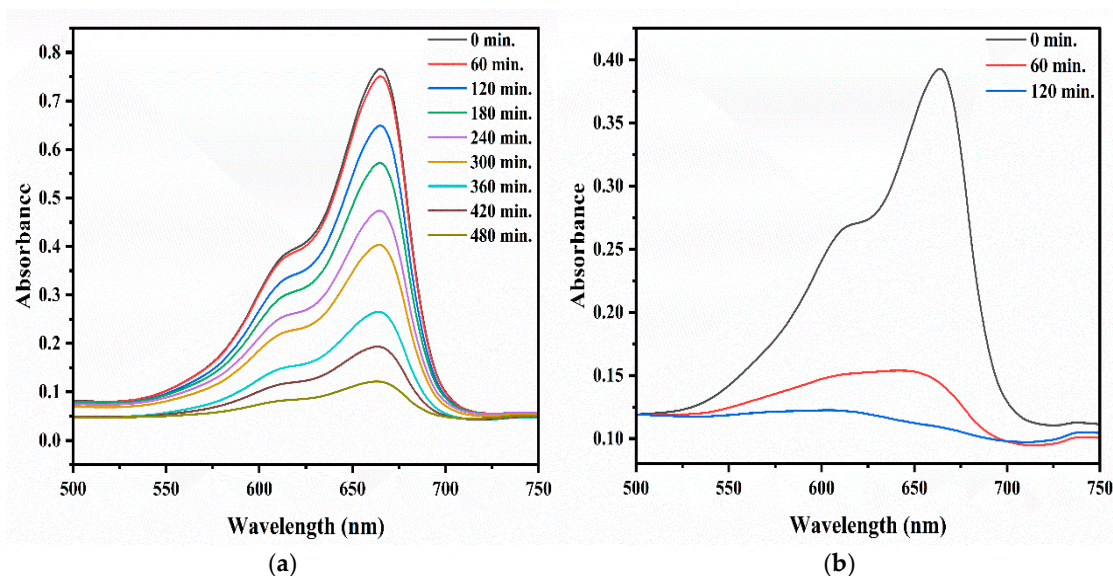
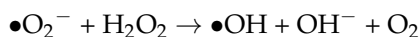
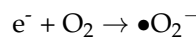
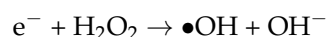
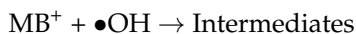


Figure 6. UV–visible spectra of photocatalytic degradation of MB (a) without H_2O_2 (b) with H_2O_2 .

2.5.1. Effect of Concentration of Dye

Experiments were conducted using MB concentrations of 2, 4, 6, and 8 parts per million (ppm) to investigate the effect of IONPs. The results, depicted in Figure 7a, show that photocatalytic degradation decreases gradually with increasing dye concentration. The degradation rate is highest for the 2 ppm dye solution, with reduced rates observed for the 4, 6, and 8 ppm solutions. This observed trend is due to the increased adsorption of dye molecules on the outer surface of the IONPs, which hinders light penetration to the dye molecules on the surface of the catalyst. Consequently, this inhibits the action of the particles and reduces the availability of reactive $\text{OH}^\bullet$ and $\text{O}_2^{\bullet-}$ free radicals that interact with the dye molecules, thus hindering photodegradation [35,36].

2.5.2. Effect of pH

The degradation efficiency of dye is highly sensitive to the hydrogen ion concentration (pH) of the solution. The degradation process is pH-dependent, as it affects the surface ionization of the [37]. Figure 7d shows the highest degradation at pH 12. The increased degradation with rising pH may be attributed to the positively charged methylene blue, which tends to dissociate negatively charged ions [38]. Furthermore, higher pH levels increase the concentration of OH^- ions on the material surface, boosting the number of OH^- radicals as active sites and thereby enhancing MB degradation [39]. Qasim et al. reported higher degradation rates at elevated pH levels [40]. However, at pH 12, the degradation percentage with and without the IONPs was almost similar. This indicates that the increased degradation at pH 12 was not specifically due to the catalytic activity of the IONPs but rather due to the high alkalinity of the solution itself. Similar results (99.84% degradation efficiency at pH 11 under sunlight without photocatalyst) were obtained by Göktaş [41]. Consequently, to accurately assess the photocatalytic activity of the IONPs, further experiments were carried out at the original pH of the dye solution rather than at

pH 12. This approach ensures that the observed degradation is primarily attributable to the catalytic action of the IONPs.

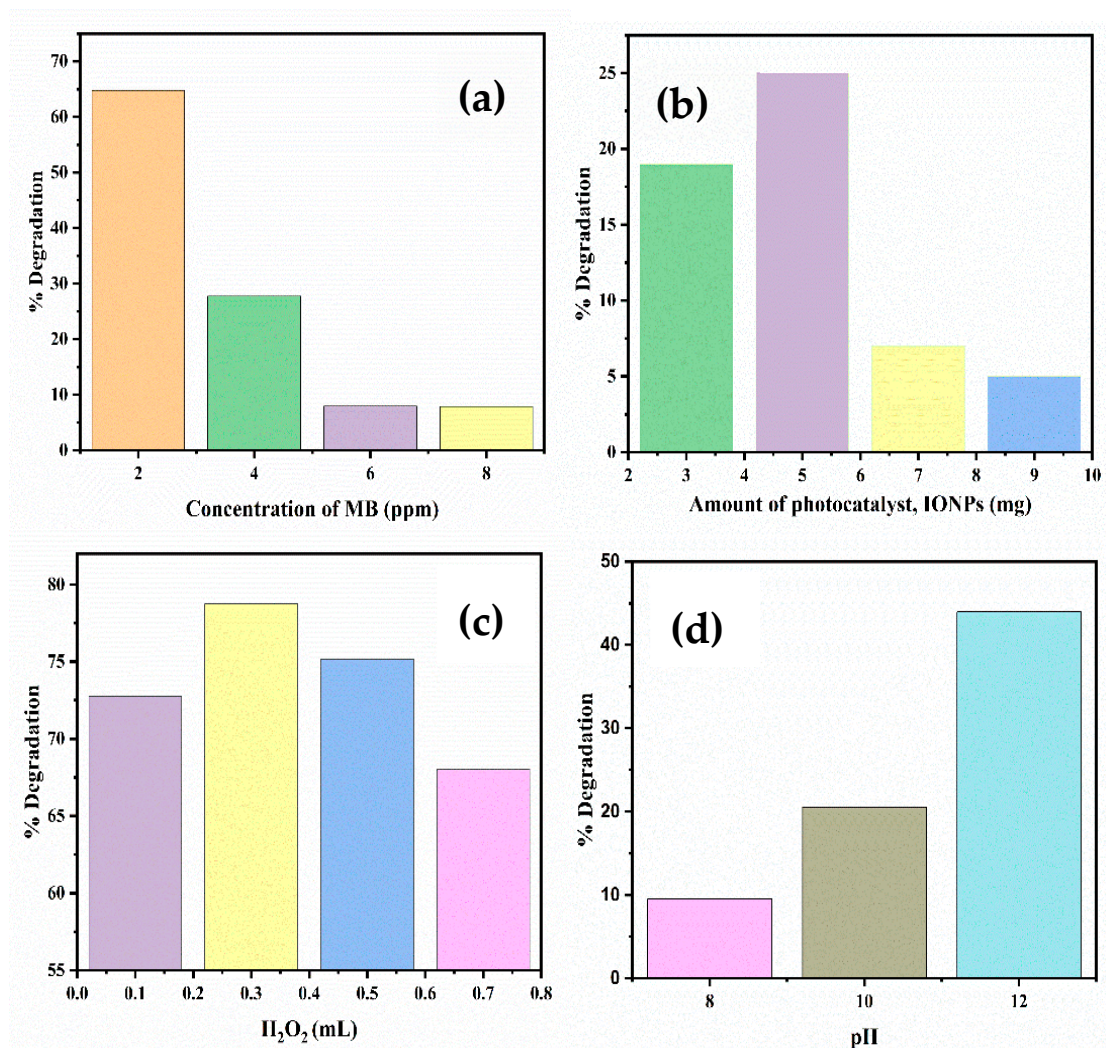


Figure 7. Percentage degradation of MB dye with a variation on (a) concentration of MB, (b) amount of photocatalyst (IONPs), (c) concentration of H_2O_2 , (d) pH.

2.5.3. Effect of Catalyst Dose

Figure 7b demonstrates the effect of varying amounts of IONPs on color elimination under sunlight. Increasing the quantity of particles accelerates the photodegradation process, with the highest degradation efficiency achieved at 5 mg of synthesized IONPs. Beyond this point, the rate decreases at 7 mg and 9 mg. This indicates that the quantity of catalyst used significantly influences the degradation rate, possibly due to increased suspension opacity from the additional IONPs. A key factor may be the increased amount of photocatalyst, which reduces light penetration [32]. Moreover, exceeding the optimal concentration of photocatalyst may lead to nanoparticle coagulation, which reduces the surface area and photoreception, thus decreasing the rate of photocatalytic degradation [36,42,43].

2.5.4. Effect of H_2O_2

In the study of photocatalytic degradation, as depicted in Figure 7c, catalytic activity increases with higher concentrations of H_2O_2 . However, beyond a certain limit, this activity begins to decline. Specifically, using 0.1 mL of H_2O_2 resulted in approximately 75% degradation of MB. The highest degradation of 80% was observed with 0.3 mL of H_2O_2 , followed by a decrease to 78% and 68% with 0.6 mL and 0.9 mL, respectively. The

decrease in photocatalytic activity at higher H₂O₂ dosages may be attributed to hydrogen peroxide's ability to scavenge free radicals responsible for MB degradation [44]. At elevated concentrations, H₂O₂ might also form by-products that interfere with the photocatalytic process or cause deactivation of the photocatalyst, thereby reducing the overall efficiency of the degradation process [45].

2.5.5. Comparative Analysis of Photocatalytic Activity

The photocatalytic degradation of MB by IONPs synthesized using *Senegalia catechu* leaf extract demonstrated above 80% degradation after 120 min under sunlight irradiation. These results align well with previous studies using the plant-mediated synthesis of IONPs for dye degradation as shown in Table 1. For instance, IONPs synthesized using *Wedelia urticifolia* DC. leaf extract achieved 98% degradation of MB under visible light exposure but required a significantly longer reaction time of 360 min [46]. In contrast, *Psidium guajava* and *Moringa oleifera* leaf extracts resulted in only ~20% degradation of methylene blue within 60 min under visible light [47], indicating much lower efficiency compared to our findings. Furthermore, *Mentha pulegium* leaf extract-mediated IONPs demonstrated 78.68% degradation of methylene blue under UV light within 120 min [33], which is closely aligned with the results of our study, though a different light source was employed. This suggests that the sunlight-driven degradation efficiency of IONPs synthesized from *Senegalia catechu* is highly competitive, achieving similar results to those under UV light. Comparative studies using other dyes, such as crystal violet and remazol yellow, also demonstrate varying levels of efficiency. For instance, *Ruellia tuberosa* leaf extract-mediated IONPs degraded 80% of crystal violet under visible light in 150 min [48], while *Carica papaya* leaf extract-derived IONPs achieved 77% degradation of remazol yellow within 360 min under sunlight [49] and *Withania coagulans* mediated IONPs achieved 70% degradation of safranin under 180 min of visible light exposure [40]. These results further reinforce the versatility and effectiveness of plant-based synthesis of IONPs, with our study showing significant degradation performance within a relatively short reaction time. This comparison illustrates that the IONPs synthesized using *Senegalia catechu* leaf extract not only exhibit high photocatalytic efficiency under natural sunlight but also perform favorably in comparison to similar plant-mediated IONPs reported in the literature. The differences in degradation efficiency can be attributed to factors including nanoparticle size, surface area, morphology, and the specific interactions between the nanoparticles and dye molecules.

Table 1. Comparison of photocatalytic activity of green synthesized IONPs with previous studies.

Plant Used	Pollutants	Light Source	Degradation Time (min.)	Degradation Efficiency (%)	References
<i>Wedelia urticifolia</i>	MB	Visible light	360	98	[46]
<i>Psidium guajava</i> and <i>Moringa oleifera</i>	MB	Visible light	60	20	[47]
<i>Mentha pulegium</i>	MB	UV light	120	78.68	[33]
<i>Ruellia tuberosa</i>	Crystal violet	Visible light	150	80	[48]
<i>Carica papaya</i>	Remazol yellow	sunlight	360	77	[49]
<i>Withania coagulans</i>	Safranin	Visible light	180	70	[40]
<i>Senegalia catechu</i>	MB	sunlight	120	80	Present study

2.5.6. Regeneration and Reusability of Iron Oxide Nanoparticles

The regeneration and reusability of IONPs were evaluated through four consecutive cycles of photocatalytic degradation of MB dye. Reusability is a critical factor in determining the long-term efficiency and cost-effectiveness of the photocatalyst. To assess this, we conducted four consecutive cycles under sunlight irradiation over four different days. After each cycle, the IONPs were recovered using centrifugation, rinsed with distilled water, and then dried for reuse in the subsequent cycle. Figure 8 displays the UV–vis spectra related to photocatalytic degradation of MB recorded after each cycle, highlighting the characteristic

absorption peak of MB at 664 nm before degradation and the changes observed in the spectra after each subsequent cycle. The observed shifts in absorbance intensity across the cycles substantiate the efficacy of IONPs in facilitating the degradation process.

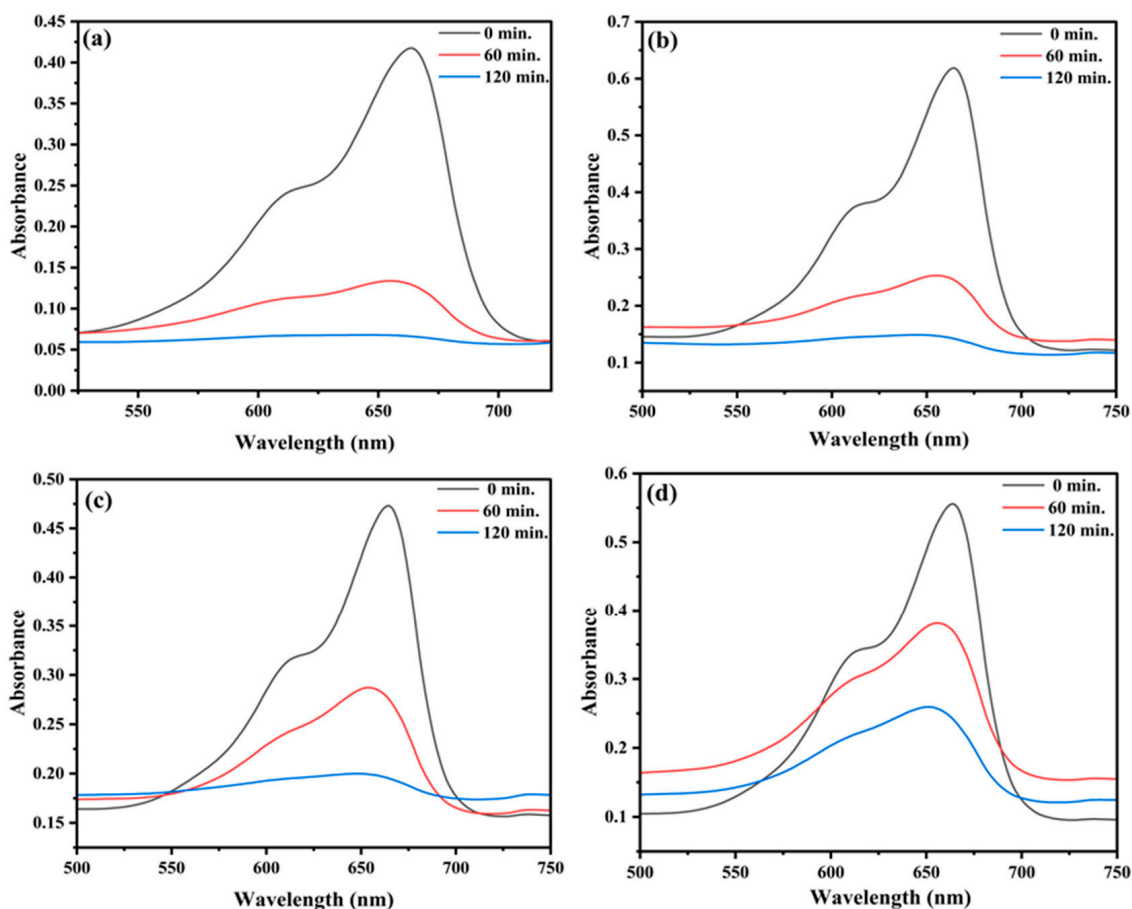


Figure 8. UV-vis spectra of methylene blue degradation across (a) first, (b) second, (c) third, and (d) fourth cycles.

Figure 9 presents a quantitative evaluation of the photocatalytic activity, showing degradation efficiencies achieved in each cycle. Under 120 min of sunlight irradiation per cycle, the degradation rates were 84%, 74%, 58%, and 56% for the first, second, third, and fourth cycles, respectively. These results indicate a noticeable decline in photocatalytic activity with each successive cycle, suggesting several underlying factors affecting the stability and service life of the IONPs. The initial high degradation rate of 84% in the first cycle demonstrates the effectiveness of the IONPs in catalyzing the breakdown of methylene blue dye under sunlight irradiation. However, the observed decrease in efficiency after the second cycle highlights the importance of evaluating the long-term stability of photocatalysts. The reduced degradation efficiency may be attributed to several factors, including catalyst loss during recovery, surface fouling from residual dye, and by-products blocking active sites [50]. Additionally, variability in sunlight irradiation over different days and the accumulation of intermediate by-products likely contributed to the decline [51]. Despite these challenges, the IONPs retained over 55% of their initial efficiency after four cycles, demonstrating reasonable stability for multiple uses in pollutant degradation. Mbachu et al. reported a similar trend in their study of iron oxide NPs [52]. Future research should focus on optimizing regeneration methods, exploring composite materials and surface modifications, and adjusting reaction conditions to enhance degradation efficiency and long-term performance.

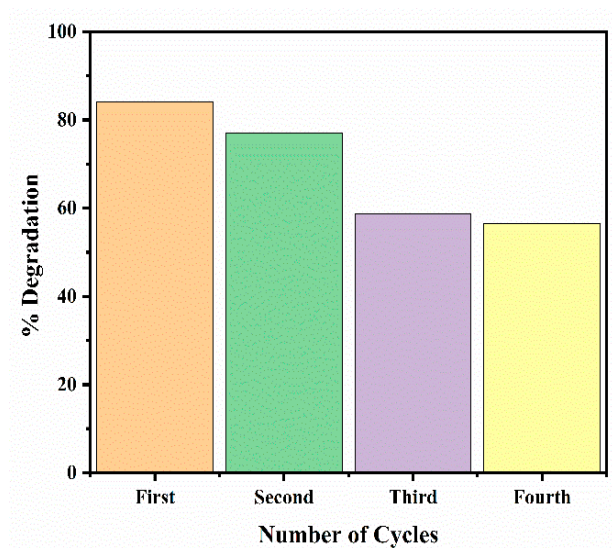


Figure 9. Percentage degradation of MB dye over four cycles using IONPs.

3. Materials and Methods

3.1. Chemicals Used

Analytical-grade chemicals were employed throughout this research. The chemicals used included iron (II) sulfate heptahydrate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$, 98.5%) and hydrogen peroxide (30% H_2O_2) from Merck Life Science Pvt. Ltd., Bengaluru, India, sodium hydroxide (NaOH, 97%) and methylene blue ($\text{C}_{16}\text{H}_{18}\text{ClN}_3\text{S}$) from Thermo Fisher Scientific India Pvt. Ltd., Mumbai, India, and absolute ethanol (99.9%) from Changshu Honsheng Fine Chemical Co., Ltd., Suzhou, China. All required solutions were prepared using distilled water without any additional treatments.

3.2. Preparation of Plant Extract

The fresh leaves of *Senegalia catechu* were collected from Tansen-14 Argali, Nepal, GPS coordinates (latitude: 27.924993°, longitude: 83.469533°). They were authenticated from the National Herbarium and Plant Laboratory (NHPL), Lalitpur, Nepal, with voucher code DK001 (KATH). The freshly collected leaves were rinsed with clean water to remove the dirt present, followed by distilled water, and subsequently air-dried in shade for 15 days. The dried leaves were finely powdered using a mechanical grinder (Baltra, New Delhi, India). Eight-gram leaf powder was mixed with 400 mL distilled water and subjected to magnetic stirring at 60 °C for 2 h. After cooling, the aqueous extract was centrifuged at 8500 rpm for 10 min to remove any solid particles. The supernatant was then subjected to filtration using Whatman (Maidstone, UK) No. 1 filter paper to produce a clarified extract, which was preserved at 4 °C for future utilization.

3.3. Synthesis of Iron Oxide Nanoparticles

The synthesis of IONPs involved the dropwise addition of 50 mL of a freshly prepared *Senegalia catechu* leaf extract to a 50 mL solution of 0.1 M $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ in a beaker. Continuous stirring of the mixture was carried out on a magnetic stirrer (Stuart Scientific Co., Ltd, Redhill, Surrey, UK) at room temperature. The pH of the mixture was maintained by adding 0.1 M NaOH dropwise. The solution turning black provided a visual clue for the formation of iron oxide nanoparticles. Centrifugation (15 min, 8500 rpm) isolated the precipitate, which was subsequently washed three times with ethanol and water to eliminate impurities. The residue was transferred to a crucible and calcined at 800 °C for 4 h in a muffle furnace. Thus, formed iron oxide nanoparticles were stored in vials wrapped with aluminum foil for subsequent analysis.

3.4. Characterization of IONPs

A comprehensive analysis of the synthesized IONPs was performed using several characterization methods. The optical properties were examined with a double-beam UV–visible (UV–vis) spectrophotometer (SPECORD 200 plus, Analytik, Hallertau, Germany). A UV–vis spectrum of a dilute suspension of the sample in distilled water was acquired by scanning the wavelength range from 200 to 800 nm. FT-IR analysis was performed using an (IRTraacer-100, SHIMADZU, Duisburg, Germany) in the range of 4000–400 cm^{-1} . The FT-IR spectra were obtained to analyze the functional groups present. The morphology and surface characteristics of the samples were studied using scanning electron microscopy (SEM) (FESEM, JEOL, JSM-IT 800, Oxford, UK). SEM images were acquired at different magnifications to examine the nanoparticles' size, shape, and distribution. The sample's elemental composition was assessed using energy-dispersive X-ray spectroscopy (EDS) with an octane-plus detector (Jeol Ltd., Tokyo, Japan). Parameters included an accelerating voltage of 15 kV, a takeoff angle of 70.67 degrees, a count rate of 2911 counts per second (CPS), a dead time (DT) of 2.6%, a live time of 48.731 s, an amplifier time of 3.84 microseconds, and an energy resolution of 127.8725 eV. Quantitative analysis was performed using the eZAF Smart Quant method, accounting for atomic number (Z), absorption (A), and fluorescence (F) effects, providing weight and atomic percentages for the detected elements. X-ray Diffraction Spectroscopy (XRD) analysis was employed to investigate the crystallinity and phase composition of the synthesized nanoparticles using an XRD (Rigaku Miniflex 600, Cedar Park, TX, USA) with $\text{K}\alpha$ radiation (wavelength, $\lambda = 0.209013$ nm). X-ray diffraction (XRD) analysis was performed on the sample using a scan range of 20° to 70° (2θ) with a step size of 0.020° and a scan rate of 2° per minute. The XRD patterns were examined to determine the crystal phase present in the materials. The average crystallite size was estimated using the Scherrer equation.

$$D = \frac{k \lambda}{\beta \cos \theta} \quad (1)$$

where

D = crystallite size (nm);

K = Scherrer constant (0.9);

λ = Wavelength of X-ray source (0.209013 nm);

β = Full width at half maxima, FWHM (radians);

θ = peak position (radians).

3.5. Photocatalytic Activity of Iron Oxide Nanoparticles

The photocatalytic activity of synthesized IONPs was evaluated by monitoring the degradation of MB dye under irradiation of sunlight. In a typical procedure, a 100 mL beaker containing 50 mL of 4 ppm MB solution was prepared. Then, 5 mg of IONP photocatalyst was introduced, followed by the addition of 0.3 mL of 3% hydrogen peroxide. Before exposure to the sunlight, a magnetic stirrer was employed to stir the mixture for 30 min and then left unstirred for another 30 min for absorption–desorption to maintain equilibrium in the dark. Subsequently, the solution was exposed to sunlight without stirring. At regular intervals of time, a 3 mL portion of the solution was withdrawn, and absorbance was taken at 664 nm using a UV–visible spectrophotometer to monitor the progress of the reaction. The efficiency of degradation was evaluated using the following equation:

$$\text{Percentage (\%) degradation} = \frac{(A_0 - A_t)}{A_0} \quad (2)$$

where A_0 is the initial absorption and A_t is the absorption after a specific time interval t of MB dye solution, respectively.

3.6. Reusability Study

This study evaluated the reusability and recovery of IONPs in the degradation of MB dye, where 20 mg of IONPs was introduced in a beaker with 200 mL of 4 ppm MB solution along with 0.3 mL of 3% hydrogen peroxide. Four separate experiments were conducted following this procedure. The photocatalyst recovered after each experiment by centrifuging at 8500 rpm for 20 min. It was subsequently washed three times with distilled water to eliminate any residual impurities. The purified photocatalyst was then dried at 80 °C for 60 min.

4. Conclusions

This study successfully demonstrated the green synthesis of IONPs using an aqueous extract of *Senegalia catechu* leaves. The synthesis was confirmed by various characterization techniques, including UV–vis, FTIR, XRD, SEM, and EDS analyses. The findings indicate that the IONPs exhibit a distinct absorption peak at 580 nm, corresponding to electronic transitions within Fe^{3+} ions, and display characteristic Fe–O stretching peaks, confirming the successful formation of IONPs. XRD, SEM, and EDS analyses confirm that the IONPs have an average crystallite size of 34.07 nm, exhibit good crystallinity, and possess a spherical morphology, with high weight percentages of iron and oxygen, indicating the purity of the synthesized IONPs. The photocatalytic activity evaluation revealed that the green-synthesized IONPs were effective in degrading MB dye under sunlight irradiation, achieving a degradation percentage of 80%. Optimizing catalyst dosage, dye concentration, pH, and hydrogen peroxide is crucial for efficient photocatalytic degradation of MB. Studies on the reusability of synthesized IONPs revealed their ability to be regenerated and reused for multiple cycles. The degradation efficiency remained above 80% in the first cycle and even reached 55% in the fourth cycle. This suggests that IONPs can be a promising reusable nanomaterial for degradation processes. This eco-friendly approach to IONP synthesis offers a promising alternative to conventional methods. To enhance the long-term performance and practical significance of such material, it is essential to investigate its efficacy in degrading a broader spectrum of pollutants, encompassing phenols, antibiotics, and other wastewater contaminants. Furthermore, examining the photodegradation mechanism of green-synthesized IONPs can provide valuable insights for designing and developing more efficient photocatalysts for environmental remediation applications in the future.

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