



Effectiveness of Zinc Oxide/Iron Oxide Nanocomposites in Treatment of Colored Wastewater

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Abstract

Background & Aims: Industrial wastewater containing persistent dyes is a serious environmental concern, threatening aquatic ecosystems and public health. Although numerous treatment technologies have been developed for the treatment of colored wastewater, many remain ineffective in degrading complex dye molecules. Usage of ZnO/Fe₂O₃ nanocomposites presents a promising approach by enhancement of ultraviolet (UV) absorption and photocatalytic efficiency. This study aimed to evaluate the efficiency of zinc oxide/iron oxide (ZnO/Fe₂O₃) nanocomposites in the removal of methyl orange dye from wastewater with the goal of enhancement of ZnO's photocatalytic properties through modification with Fe₂O₃.

Materials and Methods: In this experimental study, the ZnO/Fe₂O₃ nanocomposite was synthesized using a chemical co-precipitation method. A cylindrical UV reactor with a 1-liter outer volume and an internal UV lamp was used to evaluate photocatalytic efficiency. Effects of photocatalyst dosage, initial dye concentration, pH, and contact time on dye removal were investigated, with experiments conducted using a 500 mL working volume and 30 samples. The experiments were carried out at room temperature, and the initial pH was adjusted by using H_2SO_4 . Descriptive analysis of the data and regression were conducted in Microsoft Excel software.

Results: Under optimal conditions with a pH of 9, 0.25 mg/L nanocomposite dose, 2 mg/L dye concentration, and 90minute contact time, the removal efficiency of methyl orange dye reached 89%. In comparison, UV treatment alone removed only 6% of the dye, and ZnO/Fe₂O₃ without UV achieved 56% removal.

Conclusion: The ZnO/Fe₂O₃ nanocomposites significantly enhance photocatalytic efficiency, owing to the synergistic effect of ZnO and Fe₂O₃ and improved UV absorption. These findings highlight the potential of ZnO/Fe₂O₃ nanocomposites as a cost-effective and eco-friendly solution for industrial wastewater treatment.

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1. Introduction

The textile industries are among the largest global water consumers. As a result, this industry has become a leading source of water pollution. The colored wastewater produced by textile industries, if discharged without prior treatment, can cause aesthetic and health problems and significant environmental damage [1-3]. Due to their synthetic origin and complex molecular structures, they are primarily persistent and non-biodegradable [3]. It is estimated that around 280,000 tons of wastewater containing industrial dyes are released into the environment annually [4].

Dye concentrations as low as 1 mg/L are visible in water, and their accumulation in nature diminishes the aesthetic quality of the environment. The colored layer on the surface of the water prevents sunlight penetration, reducing photosynthesis and dissolved oxygen levels, which degrades water quality and harms aquatic life. Major pollutants in textile wastewater include dyes, detergents, and heavy metals, with their concentrations varying depending on the processes and operations performed in the factories [5]. Dye concentrations in wastewater vary by industry; accordingly, that of the leather dyeing industry ranges from 1,000 to 5,000 mg/L, while that of the textile industry is within the range of 10-200 mg/L [6,7].

This wastewater is characterized by high color concentration, high chemical oxygen demand, low biodegradability, and significant variability. The large fluctuations in pH (ranging from 2 to 12) also pose a problem, primarily due to the wide variety of dyes used [8].

Anionic dyes, such as methyl orange (MO), a common azo dye in the textile industry, are highly toxic when ingested or inhaled [9]. They are resistant to light and washing, which makes them difficult to degrade. These dyes reduce light penetration in water, posing a severe threat to aquatic life. Moreover, their persistence and absorption by plants can lead

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to genetic mutations in future generations $[\underline{10}]$.

Therefore, treatment of colored wastewater before discharge is essential, and removal of these pollutants is critically important. Various methods, such as ion exchange, Fenton processes, electrochemical treatment, membrane filtration, ozonation, activated carbon treatment, UV/H_2O_2 and UV/TiO_2 photooxidation, chemical coagulation, and reverse osmosis, have been used to remove dyes from wastewater with varying degrees of success.

Textile wastewater, containing various dyes, resists biodegradation due to the high molecular weight, synthetic nature, and complex aromatic structures of the dyes [11,12]. Advanced oxidation processes (AOPs), which include chemical, photochemical, and photocatalytic processes, have been developed to address this challenge. Today, oxidation processes that use ultraviolet (UV) radiation are classified as AOPs. These processes generate hydroxyl radicals (OH•), highly reactive and non-selective oxidants, which degrade toxic, non-biodegradable, and persistent organic pollutants. Given the high cost of complete organic degradation, AOPs are often used for partial degradation, oxidation into neutral and less harmful products, and disinfection [13,14].

Recent research has focused extensively on the removal of dyes from industrial wastewater using photocatalysts. Photocatalysts are semiconductors that oxidize organic compounds when exposed to high-intensity light. When the energy of a photon is equal to or greater than the bandgap energy, an electron is excited from the valence band to the conduction band, creating a hole in the valence band. These excited electrons and holes can directly or indirectly generate OH•, converting organic materials into inorganic substances [<u>15</u>]. In Heterogeneous photocatalytic processes, UV lamps or sunlight and solid catalytic agents, such as zinc oxide (ZnO) were used [16]. The ZnO, a semiconductor with a bandgap energy of 3.3 electron volts, is widely used as a catalyst due to its high UV absorption capability, high surface-to-volume ratio, and long operational life. It is also relatively inexpensive, and its ability to absorb a wide range of electromagnetic waves and perform photocatalysis under UVA radiation makes it highly effective [17,18].

Modification of semiconductors, such as doping with

transition metals or rare earth elements, has been shown to improve photocatalytic properties. To enhance costefficiency and optimize the use of nanocatalysts, they are often synthesized as composites, with chemically modified surfaces. Designing nanocomposites composed of metals and semiconductors, such as ZnO/Fe, can improve photocatalytic efficiency [19]. Depositing a noble metal onto semiconductor nanoparticles further maximizes this efficiency [20].

This study aimed to evaluate the efficiency of the $ZnO/Fe_2O_3 + UV$ process in the degradation of MO dye (an azo dye).

2. Materials and Methods

In this experimental study, the effects of several independent variables (nanoparticle dose, initial dye concentration, contact time, and pH) were evaluated on the UV + ZnO/Fe_2O_3 process. The evaluation was first conducted under UV radiation through a series of preliminary experiments. Based on the pretest experiments results, the optimal range for each variable was determined and the experiments were repeated three times to optimize each factor. Contact time, the primary variable, was minimized as much as possible.

The optimization process began with the nanoparticle dose, followed by pH and initial dye concentration. Zinc Nitrate Hexahydrate, with 98% purity, and all other chemicals, in analytical grade, were purchased from Merck (Germany). The data descriptive analysis and regression were conducted in Microsoft Excel software.

Synthesis of the nanophotocatalyst

In this work, the synthesized ZnO/Fe_2O_3 nanocomposite was used as a catalyst. This nanocomposite was synthesized using the simultaneous chemical precipitation method (the steps are illustrated in Figure 1). Details of this method can be found in references [21, 22]. Briefly, a solution of zinc nitrate hexahydrate and a solution of sodium hydroxide were prepared. Both solutions were stirred at room temperature to ensure homogeneity. Afterward, the sodium hydroxide solution was slowly added to the zinc nitrate solution under rapid stirring.



Figure 1. ZnO synthesis steps: a) addition of sodium hydroxide solution to zinc nitrate solution, b and c) transfer of the suspension to a centrifuge, d) deposition of Zn (OH)2 deposited in the tube after centrifugation, e) transfer of Zn (OH)2 to a thermal furnace, and f) preparation of ZnO powder

In the following process, the resultant suspension was transferred into test tubes and placed in a centrifuge. The desired precipitate settled at the bottom of the tubes, while the remaining reaction products formed a clear solution above it. The resultant $Zn(OH)_2$ precipitate was heated in a furnace, yielding ZnO powder (Figure 1).

In the next step, the ZnO/Fe₂O₃ nanocomposite was synthesized using the photochemical precipitation method. In this method, the previously prepared ZnO was mixed with a 6 mM solution of FeCl₃·6H₂O and then exposed to UV radiation for 2 h. After this step, the suspension was allowed to settle for half an hour, and the precipitate was collected and heated at 400 °C for 2 h before cooling. This powder was sampled for chemical characterization and used in experiments as a ZnO/Fe₂O₃ nanophotocatalyst. The general properties of the synthesized nanoparticles were analyzed and controlled using X-ray diffraction (XRD) and energy dispersive X-ray (EDX) techniques.

Model pollutant dye

In this study, MO dye with a molecular weight of 327.22 and the formula $C_{14}H_{14}N_3NaO_3S$, manufactured by Merck, Germany, was used as the model pollutant. The concentration of this dye in aqueous solution was measured using a spectrophotometric method, with absorbance readings taken at a wavelength of 464 nm [23]. To prepare the initial standard solutions and test samples for the evaluated process, a stock solution of MO dye was prepared at a concentration of 2 g/L using deionized water. The stock solution and deionized water were then used to prepare the test samples.

Reactor and experiments

The reactor used in this study, shown schematically in <u>Figure</u> <u>2</u>, consists of two parts. A cylindrical UV reactor, featuring

an outer volume of 1 L and an inner compartment housing the UV lamp, was utilized to evaluate photocatalytic efficiency. Design of the reactor allows for uniform exposure to UV light, optimizing photocatalytic reactions. Variables, such as photocatalyst dosage, initial dye concentration, pH, and contact time, were systematically investigated for their impact on dye removal, with all experiments conducted using a working volume of 500 mL.

Based on preliminary tests and the results of similar research, four concentrations of MO (1, 1.6, 2, 4 mg/L) were prepared from the stock solution for these experiments. The pH was adjusted to acidic, neutral, and alkaline conditions (pH=4, 7, 9), the nanocomposite dosage was varied at three levels (0.125, 0.25, and 0.5 g/L), and the contact times were 0, 0.5, 1, 1.5, and 2 h for the treatment experiments. These experiments were conducted in three separate processes $(UV, ZnO/Fe_2O_3, and UV + ZnO/Fe_2O_3)$, each repeated three times. To determine the contribution of the adsorption process, compared to dye oxidation, an experiment was first conducted in the absence of any light (UV or natural light) under dark conditions, similar to those of the illuminated experiments. The amount of dye removed in this case was attributed to adsorption and subtracted from the total removal efficiency.

3. Results

ZnO/Fe_2O_3 synthesis

Figure 3 (a) shows the XRD pattern of the synthesized ZnO/Fe_2O_3 nanoparticle sample, along with the standard patterns of ZnO and Fe₂O₃. As observed, the XRD peaks in this figure largely correspond to a combination of the ZnO and iron oxide (hematite) patterns.

Figure 3 (b) illustrates the EDX spectrum of the synthesized nanocomposite. As observed in this figure, the elements Zn, O, and Fe constitute 71.83%, 24%, and 4.14% of the synthesized composite, respectively.



Figure 2. A schematic diagram of the reactor used in the study and a decolorizing process using the nanophotocatalyst method in this



Figure 3. The X-ray diffraction pattern of the synthesized ZnO/Fe₂O₃ sample, compared to the standard ZnO patterns, and the energy dispersive X-ray spectrum for the ZnO/Fe₂O₃ sample

ZnO/Fe_2O_3 and UV performance, separately and combined

The separate and combined effects of ZnO/Fe₂O₃ and UV were examined to determine their synergistic impact. The dye degradation was assessed at different contact times using a dye concentration of 1.6 mg/L, a nanocomposite dose of 0.25 mg/L, and a UV lamp. The results, shown in Figure 4, indicate that UV alone is not effective in dye removal. However, the removal rate with ZnO/Fe₂O₃ nanocomposite alone initially increased during the first 30 min, followed by a decrease, likely due to the initial adsorption of the dye onto the nanocomposite and subsequent desorption. The combination of ZnO/Fe₂O₃ and UV demonstrated a significant increase in the efficiency of MO removal, reaching 79% under 30 min contact time. In contrast, UV alone removed only 6% of the dye, while ZnO/Fe₂O₃ without UV achieved a 56% removal rate.

Effects of pH and dye concentration

The pH of the solution can affect the photocatalyst surface charge, the ionization degree of various pollutants, the exposure of functional groups on active sites of the photocatalyst, and the molecular structure of the dye. Therefore, solution pH is a crucial parameter throughout the dye adsorption process. Removal efficiency of MO dye at three pH levels (4, 7, and 9), with an initial dye concentration of 2 mg/L and a photocatalyst dose of 0.25 mg/L, is presented in <u>Figure 5</u> (a). As depicted, the highest dye removal efficiency was observed at pH 9.

To determine the impact of dye dose on process efficiency at the optimal concentration and pH (0.25 mg/L nanocomposite dose and pH 9), the range of MO dye doses from 1 to 4 mg/L was investigated. The results are shown in Figure 5 (b). According to the data, the highest dye removal efficiency was achieved at a concentration of 2 mg/L of MO. At higher concentrations (4 mg/L), dye removal significantly decreased, which may be due to a reduction in available sites for hydroxyl radical generation.

ZnO/Fe₂O₃ nanocomposite dose effects

In this study, considering economic and environmental criteria as well as preliminary tests, the optimal dose range of the nanophotocatalyst was determined at 0.125-0.5 mg/L, with a sample volume of 500 mL, pH of 9, and dye concentration of 1.6 mg/L. The results regarding the effect of ZnO/Fe₂O₃ nanocomposite dose are shown in Figure 6. As illustrated, the highest dye removal efficiency was observed at a nanophotocatalyst concentration of 0.25 mg/L. The optimal dose of ZnO/Fe₂O₃ nanocomposite was found to be 0.25 mg/L, balancing cost and performance. Higher doses did not significantly enhance dye removal and could lead to unnecessary costs.



Figure 4. Effect of the synergistic interaction between ZnO/Fe₂O₃ and ultraviolet on dye removal efficiency at various contact times (nanocomposite dose: 0.25 mg/L; initial dye concentration: 1.6 mg/L for all tests)



Figure 5. Effect of pH (a) and methyl orange dye concentration (b) on dye removal efficiency (nanoparticle dose in all experiments was 0.25 mg/L, and the initial pH for the experiments was 9)



Figure 6. Effect of ZnO/Fe₂O₃ dose on dye removal efficiency at different contact times (pH was 9 in all experiments)



Figure 7. Pseudo first-order reaction kinetics fitting for photocatalytic reaction

Photocatalytic reaction kinetics

After the determination of the optimal conditions, experiments were conducted to ascertain the reaction kinetics. The obtained data are presented in Figure 7, which illustrates the ratio of the remaining dye concentration to the initial dye concentration (C/C_0) over time, considering the optimal conditions. The regression analysis indicates that the reaction follows a pseudo-first-order kinetics.

This study also emphasized that ZnO/Fe_2O_3 nanocomposites are not only cost-effective and efficient but also preferable over other physical-chemical treatment methods due to their lack of secondary pollution. The AOPs using nanocomposites are recognized as one of the most efficient methods for the removal of colored pollutants in industrial wastewater due to the generation of OH_{\bullet} .

Findings of the present research align with those of a study conducted by Hodges (2018) [13], which demonstrated improved UV absorption using ZnO-based nanocomposites. However, the use of Fe_2O_3 in the present study further enhanced dye degradation efficiency, showing that the ZnO/Fe₂O₃ nanocomposite is more effective in the removal of persistent dyes.

Optimization of operational parameters, such as pH, nanocomposite concentration, and contact time, led to a

high removal efficiency of 89% under optimal conditions (pH=9, concentration: 0.25 mg/L, contact time: 90 min). This performance markedly surpassed the use of UV or ZnO alone and offered much higher effectiveness. Similar studies have confirmed that ZnO nanoparticles, particularly when combined with other metals, like Fe₂O₃, have high capabilities for the degradation of organic pollutants due to increased UV light absorption and hydroxyl radical production. Given their high efficiency, these nanocomposites could play a significant role in the reduction of the environmental impact of colored wastewater and the enhancement of advanced treatment methods.

4. Discussion

The findings of this study highlighted the significant potential of ZnO/iron oxide (ZnO/Fe₂O₃) nanocomposites as effective photocatalysts for the treatment of dyecontaminated wastewater, particularly for the removal of MO dye as a persistent dye contaminant. The results demonstrated that the synergistic effects between ZnO and Fe₂O₃ lead to enhanced photocatalytic efficiency.

Similar research, such as a study performed by Hodges (2018), has also shown that ZnO-based composites, with improved UV absorption, can effectively degrade resistant pollutants [13]. These findings not only demonstrate the superior efficiency of ZnO/Fe₂O₃ nanocomposites but also highlight their advantage in avoiding secondary pollution, making them an excellent choice for the treatment of resistant dyes in wastewater.

Photocatalytic efficiency and synergistic effects

High performance of the ZnO/Fe₂O₃ nanocomposite can be attributed to several factors. It is known that ZnO has excellent UV absorption and photocatalytic properties, but its efficiency can be limited by rapid electron-hole recombination during photocatalytic reactions. By combining ZnO with Fe₂O₃, this recombination can be suppressed, leading to a higher yield of reactive species, like OH•, which play a crucial role in the degradation of organic pollutants, such as MO.

Under UV light, the ZnO/Fe₂O₃ nanocomposite showed a maximum dye removal efficiency of 89% under optimal conditions (pH=9, 0.25 mg/L nanocomposite dose, 2 mg/L dye concentration), compared to only 6% dye removal efficiency with UV alone. This significant enhancement demonstrates the synergistic interaction between the two oxides in boosting photocatalytic activity.

Role of pH in dye removal

The pH plays a critical role in influencing the photocatalytic degradation of dyes. Results of this study showed that alkaline conditions (pH=9) yielded the highest removal efficiency for MO. At alkaline pH, the

surface charge of ZnO becomes negative, which improves the adsorption of anionic dyes, like MO. Moreover, Alkaline conditions were optimal for hydroxyl radical production.

Similar findings were reported in a study performed by Dehghani Fard, where higher pH levels led to faster and more extensive hydroxyl radical generation, thereby enhancing photocatalytic efficiency [24]. In acidic conditions, ZnO can lose its photocatalytic properties due to dissolution in water and interaction with H⁺ ions. This further supports the need to optimize pH when applying ZnO-based nanocomposites in wastewater treatment processes.

This study confirms that at pH 9, optimal conditions for photocatalysis are achieved, aligning well with other research demonstrating improved performance in alkaline environments. It should be noted that pH plays a vital role in the removal of dye, as it affects the surface charge of nanoparticles and their interaction with pollutants in the solution. At low pH (acidic conditions), ZnO nanoparticles typically have a positive charge, which can hinder the effective adsorption of anions and negatively charged dyes, leading to reduced removal efficiency.

As pH increases towards basic conditions, the surface charge of nanoparticles becomes negative, enhancing the adsorption of anionic pollutants, such as azo dyes [22]. At pH values above 9, optimal conditions are achieved for OH• generation through nanocomposite activation, which increases the rate of oxidation reactions and more effectively decomposes pollutants. As shown in Figure 5, dye removal efficiency rose with the increase in pH, as the highest removal was observed at alkaline pH. The changes in solution pH affect the positive or negative charge of ZnO nanoparticles, impacting their degradation performance.

In alkaline pH, the behavior of ZnO nanoparticles differs from acidic conditions [19, 23]. When ZnO catalysts are exposed to photons with energy equal to or greater than the energy of the valence band, electron-hole pairs are generated. In neutral and alkaline pH conditions, the released electrons react with oxygen atoms (as electron acceptors), converting O_2 into O_2^- [25]. The resulting electron holes can either separate an electron from organic molecules to form R⁺ or react with H₂O or OH⁻ to produce OH•, leading to the degradation of organic matter.

In acidic pH, ZnO nanoparticles dissolve in water due to oxygen loss (in reaction with H^+ ions), ultimately losing their photocatalytic properties [26]. The study carried out by Dehghani Fard et al. showed that in the alkaline pH conditions, the removal efficiency of aniline via photocatalytic processes with ZnO nanoparticles increased due to faster and more extensive formation of OH• [24]. Studies performed by Kuo and Liao (2006) have shown that the removal efficiency of 4-chlorophenol during photocatalytic processes with kaolinite catalysts increased with pH from 3 to 11, due to higher free radical production and optimized surface conditions of nanoparticles, leading to faster pollutant degradation [27].

Effect of nanocomposite dose and dye concentration

This study also explored the effects of various doses of ZnO/Fe_2O_3 and the initial concentration of MO dye. A nanocomposite dose of 0.25 mg/L was found to be optimal, offering a balance between cost-effectiveness and photocatalytic efficiency. An increase in the dose beyond this level did not result in significantly improved dye removal, indicating that there is a saturation point beyond which additional catalyst does not lead to proportional performance gains. Other research, such as a study conducted by Liu et al. (2015), has also recommended similar dosages of nanocomposites, emphasizing their cost-effectiveness in large-scale applications [22]. The findings support that higher doses do not necessarily improve performance and might increase operational costs without proportionate benefits.

Similarly, the initial dye concentration affected removal efficiency, with the concentration of 2 mg/L achieving the best results. At higher concentrations (e.g., 4 mg/L), the removal efficiency dropped due to the reduced availability of active sites on the photocatalyst surface for hydroxyl radical production. These findings suggest that optimization of the dose of nanocomposites and the pollutant load is crucial for ensuring a cost-effective treatment.

Based on the above-mentioned results, a pH of 9 is considered optimal, with a nanocomposite concentration of 0.25 mg/L and a dye concentration of 2 mg/L as the optimal conditions for this process. Synergistic effect of ZnO and Fe₂O₃ under UV light resulted in significantly improved dye degradation, with a maximum removal of 89% at optimal conditions. These results demonstrated the importance of hydroxyl radical generation in the process.

Photocatalytic reaction kinetics

The degradation of MO dye in the presence of ZnO/Fe_2O_3 nanocomposites followed pseudo-first-order reaction kinetics, as indicated by the linear relationship between the logarithm of the concentration ratio (C/C_0) and time (<u>Figure 7</u>). This is a typical behavior observed in photocatalytic reactions involving semiconductor materials. The pseudo-first-order model suggests that the reaction rate is directly proportional to the concentration of the dye, but the availability of active sites on the photocatalyst surface plays a limiting role as the reaction progresses.

Initially, the ZnO/Fe_2O_3 nanocomposite offers numerous active sites for the generation of OH•, which are responsible for the degradation of the dye molecules. However, as the reaction proceeds, these active sites become saturated with adsorbed dye molecules and intermediates, leading to a decrease in the reaction rate. This decline in available active sites results in a slower dye degradation over time, consistent with pseudo-first-order kinetics.

As active sites on the nanocomposite surface become saturated, the reaction rate decreases over time. This finding aligns with those of the studies performed by **Kuo and Liao (2006)**, which also demonstrated pseudo-first-order kinetics in photocatalytic processes using similar materials [27]. The reaction kinetics observed in the present study confirmed that the ZnO/Fe₂O₃ nanocomposite behaves predictably, making it a reliable photocatalyst for industrial wastewater treatment.

5. Conclusion

This study demonstrated that ZnO/Fe_2O_3 nanocomposites are highly effective in the removal of resistant azo dyes from wastewater. The synergistic interaction between ZnO and Fe_2O_3 enhanced UV absorption and photocatalytic activity, leading to significant pollutant degradation. These findings suggest that ZnO/Fe₂O₃ could be used as a cost-effective and environmentally friendly photocatalyst for industrial wastewater treatment.

Given its high efficiency and eco-friendly nature, ZnO/Fe₂O₃ nanocomposites hold great promise for largescale industrial wastewater treatment. Future studies should explore the scalability and practical applications of this nanocomposite in real-world wastewater systems.

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Authors' Contribution

Conceptualization: Narges Hakimi Data curation: Bahareh Fathi Formal analysis: Reza Nemati Investigation: Bahareh Fathi Visualization: Narges Hakimi Writing of the original draft: Narges Hakimi, Reza Nemati Writing of the review and editing: Reza Nemati

Competing Interests

The authors declare that they have no competing interests.

Ethical Approval

This study was conducted in accordance with ethical guidelines, and approval was obtained from the Ethics Committee of Saveh University of Medical Sciences (Approval Code: IR.SAVEHUMS.REC.1398.021).

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