

Original Article



Synthesis, Structural Characterization, and Sunlight-Induced Photocatalytic Performance of Fe₂O₃@CuO Core–Shell Nanocatalyst for Methylene Blue Degradation in the Presence of H₂O₂

Farinam Faryabi,^{1#} Esmail Mohammadian,^{2#} Galavizh Mandomi,³ Seyyed Mohammad Hassan Hashemi,⁴ Masoud Memar,⁵ Amir Shadboorestan,⁶ Mahmoud Omid,^{7,8*} Omid Safa^{9*}

¹Student Research Committee, Hormozgan University of Medical Sciences, Bandar Abbas, Iran

²Cellular and Molecular Research Center, Research Institute for Health Development, Kurdistan University of Medical Sciences, Sanandaj, Iran

³Environmental Health Research Center, Research Institute for Health Development, Kurdistan University of Medical Sciences, Sanandaj, Iran

⁴Department of Pharmaceutics, Faculty of Pharmacy, Hormozgan University of Medical Sciences, Bandar Abbas, Iran

⁵Food Health Research Center, Hormozgan University of Medical Sciences, Bandar Abbas, Iran

⁶Department of Toxicology, Faculty of Medical Sciences, Tarbiat Modares University, Tehran, Iran

⁷Molecular Medicine Research Center, Hormozgan Health Institute, Hormozgan University of Medical Sciences, Bandar Abbas, Iran

⁸Department of Pharmacology and Toxicology, Faculty of Pharmacy, Hormozgan University of Medical Sciences, Bandar Abbas, Iran

⁹Department of Clinical Pharmacy, Faculty of Pharmacy, Hormozgan University of Medical Sciences, Bandar Abbas, Iran

Article history:

Received: October 24, 2025

Revised: December 19, 2025

Accepted: January 6, 2026

ePublished: June 29, 2026

***Corresponding authors:**

Mahmoud Omid,

Email: toxicology@hums.ac.ir;

Omid Safa,

Email: osafa@hums.ac.ir

Authors Mohammadian and Faryabi contributed equally.

Abstract

Introduction: The discharge of colored industrial wastewater—especially organic thiazine dyes, which are toxic, mutagenic, and carcinogenic—poses serious environmental and health problems. Methylene blue, a common dye, is toxic and non-biodegradable, and thus requires effective removal methods.

Methods: This study designed and evaluated a photocatalytic degradation system based on Fe₂O₃@CuO nanoparticles for methylene blue removal. The nanoparticles were synthesized via chemical precipitation and characterized using FT-IR, XRD, FESEM, TEM, EDS, and DLS. Their photocatalytic performance was evaluated under sunlight irradiation in a batch system, investigating the effects of catalyst dosage, H₂O₂ concentration, and solution pH. Dye concentration was measured using a UV-Vis spectrophotometer.

Results: Characterization confirmed the formation of nanoparticles with irregular geometric shapes and uniform distribution. The highest degradation efficiency (99.6%) was achieved within 45 minutes at a pH of 9, a catalyst dosage of 150 mg/L, and 6% H₂O₂. The lowest destruction efficiency (15.3%) occurred in the absence of hydrogen peroxide. Efficiency increased with higher H₂O₂ and catalyst doses due to increased radical generation. The nanocatalyst showed excellent reproducibility, retaining 97.1% of its initial efficiency after five cycles with no structural changes.

Conclusion: Fe₂O₃@CuO nanoparticles demonstrated high efficiency in the photocatalytic degradation of methylene blue under sunlight. The use of non-toxic, readily available materials, excellent reusability, and a free light source makes this process an economically viable and practical method for treating dye-containing effluents.

Keywords: Fe₂O₃@CuO, Core-shell, Colored pollutants, Nanoparticles, Photocatalysis

Please cite this article as follows: Faryabi F, Mohammadian E, Mandomi G, Hashemi SMH, Memar M, Shadboorestan A, et al. Synthesis, structural characterization, and sunlight-induced photocatalytic performance of Fe₂O₃@CuO core–shell nanocatalyst for methylene blue degradation in the presence of H₂O₂. J Adv Environ Health Res 2026;14(2): 132-141. doi:10.34172/jaehr.1473

Introduction

The production of industrial wastewater has been increasingly recognized in recent decades as one of the

major sources of environmental pollution.¹⁻³ Among these, the textile industry, due to its high water consumption and the generation of large volumes of colored and toxic



© 2026 The Author(s); Published by Kurdistan University of Medical Sciences. This is an open-access article distributed under the terms of the Creative Commons Attribution License (<https://creativecommons.org/licenses/by/4.0>), which permits unrestricted use, distribution, and reproduction in any medium, provided the original work is properly cited.

wastewater, is considered one of the most polluting industries in the world.^{4,5} Discharging such wastewater into water resources not only causes undesirable visual changes but can also lead to severe toxic effects on the health of humans, animals, and plants.⁶⁻⁹ Therefore, treating colored wastewater, especially in the textile sector, is an environmental and public health necessity.

Among the various dyes found in textile wastewater, thiazine-based organic dyes such as methylene blue pose particularly serious risks to humans and aquatic organisms because of their toxic, mutagenic, and carcinogenic nature.^{10,11} Given the highly harmful effects of these compounds, their removal from water and wastewater is of great importance.¹² Studies have shown that among different treatment methods, advanced oxidation processes and adsorption have demonstrated higher efficiency in removing these compounds.¹³⁻¹⁷

This high efficiency is mainly because of the strong oxidation ability of hydroxyl radicals, which can convert organic pollutants into harmless inorganic compounds such as CO₂ and H₂O.^{18,19} In advanced oxidation processes, hydroxyl radicals are produced through several steps, including UV irradiation of semiconductors, formation of electron-hole pairs, adsorption of OH and O₂ on the semiconductor surface, and generation of reactive radicals ($\cdot\text{OH}$) and O₂ $\cdot^-$), which ultimately lead to effective dye degradation.^{2,18,20}

Previous studies show that using sunlight or artificial irradiation in the presence of metal-oxide-based photocatalysts is one of the most effective approaches for degrading organic dyes.²¹ Among these photocatalysts, copper-based compounds have been introduced as efficient options for removing industrial dyes because of properties such as their p-type semiconducting nature with a relatively narrow band gap (≈ 1.2 eV),²² good stability, abundance, and low cost.^{4,7,20} In addition, core-shell nanoparticles offer much better photocatalytic performance than regular nanomaterials; this is because their heterogeneous structure improves surface reactions, enhances charge transfer and separation at the interface, allows tuning of the band structure, increases the effective surface area, and boosts light-resistance against corrosion.^{6-11,23} These features make core-shell nanostructures highly effective in water treatment applications and especially in the photocatalytic degradation of persistent dyes.

Because core-shell nanostructures and metal-oxide photocatalysts such as copper have demonstrated strong photodegradation performance, investigating other effective metal-based compounds is warranted. Iron-containing nanocomposites, in particular, have been shown to possess significant photocatalytic properties. Their wide range of reported biological effects adds to the appeal of further research on these nanoparticles.²⁴⁻²⁷ Goudjil et al evaluated the efficiency of green-synthesized hematite nanoparticles in removing the industrial dye methylene blue. The results showed that these nanoparticles were able to degrade about 78% of methylene blue within 120 minutes.²⁸ Furthermore, Turkoglu et al evaluated the

performance of ZnO nanoparticles in the photocatalytic degradation of methylene blue and were able to achieve complete efficiency, meaning 100% dye removal, within 270 minutes.²⁹

Considering the importance of environmental pollution, the toxicity of organic thiazine dyes, the role of hydroxyl radicals in dye degradation, and the extraordinary photocatalytic efficiency of core-shell nanoparticles, this study designed and evaluated a photocatalytic degradation system based on Fe₂O₃@CuO nanoparticles for removing methylene blue from aqueous solutions. While numerous studies have utilized single-oxide semiconductors (e.g., pure Fe₂O₃ or CuO) for photocatalytic dye degradation, their practical efficiency remains limited due to rapid electron-hole recombination and suboptimal light absorption and charge transfer properties. In contrast, heterojunction or core-shell architectures provide a promising route to overcome these intrinsic drawbacks by promoting more effective separation of photogenerated charge carriers, enhancing light harvesting, and increasing the number of active sites on the catalyst surface.³⁰ Specifically, core-shell composites can stabilize the semiconductor core, reduce photocorrosion and agglomeration, and maintain long-term activity, offering superior durability and reusability compared to bare oxides.³¹ Nevertheless, relatively few studies have investigated the photocatalytic degradation of industrial dyes such as methylene blue using a well-characterized Fe₂O₃@CuO core-shell nanocatalyst under solar irradiation. Therefore, developing and comprehensively evaluating such a nanocomposite fills an important research gap and has the potential to advance cost-effective and sustainable wastewater treatment technologies. The main innovation of this study was the use of Fe₂O₃@CuO core-shell nanoparticles as an efficient and cost-effective photocatalyst for removing methylene blue under sunlight irradiation—a structure that remains largely unexplored for dye removal. The combination of hematite and copper oxide nanoparticles creates a stable heterojunction that enhances charge transfer, reduces electron-hole recombination, and increases the generation of reactive radicals, all of which significantly improve photocatalytic performance. The synthesis of this nanocomposite with a uniform structure, nanoscale size, high stability, and recyclability—along with a comprehensive evaluation of key influencing parameters (catalyst dose, pH, and H₂O₂ concentration)—represents another innovation that distinguishes this study from previous research. Using sunlight as the energy source further guides this work toward developing a green, low-cost, and practical technology for the treatment of real wastewater.

Materials and Methods

Materials and Instruments

All chemical reagents were obtained commercially from Fluka and Merck, and they were used exactly as supplied, requiring no additional purification. FT-IR spectra were recorded using a PerkinElmer C-92264 spectrophotometer

(KBr pellets, 4000–450 cm^{-1}) A Shimadzu dual-beam spectrophotometer was used to record the UV/Vis diffuse reflectance spectra (DRS) in the 200–800 nm range. X-ray measurements were obtained with Cu-K α radiation ($\lambda = 1.54056 \text{ \AA}$) on a Bruker D8 ADVANCE. The EDS analysis and SEM images were acquired with the use of the TESCAN-XMU MIRA3 apparatus. The Philips EM 208S transmission electron microscope (TEM) was used to capture the TEM pictures. A Horiba SZ-100 piece of equipment was used to measure the particle sizes and zeta potential. It should be noted that all materials used in this study were sourced from Merck without any additional purification. Also, all the experiments were repeated three times, and, in all cases, the RSD below 10% was obtained.

Synthesis of the $\text{Fe}_2\text{O}_3/\text{CuO}$ Nanoparticle

Mahmood Abid and colleagues previously introduced a procedure for synthesizing Fe_2O_3 nanoparticles.³² In 100 mL of deionized water, 3 mM of the synthesized Fe_2O_3 nanoparticles were dispersed for 20 minutes (ultrasonic frequency of 20 kHz). The suspension above was diluted with 50 mL of $\text{Cu}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ solution at a concentration of 0.65 mM. After stirring for 12 hours at room temperature, the mixture was collected. After filtering and drying the mixture at 100°C , it was calcined for six hours at 300°C in an electric furnace under static air.

Batch Experiments

$\text{Fe}_2\text{O}_3/\text{CuO}$ nanoparticles were investigated for their photocatalytic performance for dye removal using the method described by M.U. Anu Prathap et al.^{33,34} Nanoparticles (0, 5, 10, and 15 mg) were mixed with dye solutions containing 10 mg/L for each experiment. The experiments were carried out at room temperature over a pH range of 2.0 to 12.0 to more closely examine the dependence of the adsorption process on pH. The pH was adjusted using 0.1 M NaOH and 0.1 M HCl solutions. The mixture was then added 0, 2, 4, and 6 mL of hydrogen peroxide (30 w/w%) and stirred under dark conditions for 30 minutes. The reaction mixture was then exposed to sunlight at 20°C . The dye concentration was measured using a UV-Vis spectrophotometer at a wavelength of 664 nm before and after each experiment. The dye removal efficiency was calculated using Equation (1):

$$\% \text{ Degradation} = \frac{(C_0 - C_t)}{C_0} \times 100 \quad (1)$$

where, C_0 and C_t are the initial concentration and the equilibrium dye concentration, respectively. Also, all experiments were conducted under the optimized conditions obtained in this study. Each test was performed in triplicate, and the standard error remained below 5%, indicating high repeatability and reliability of the measurements.

Results and Discussion

Characterization of the Photocatalyst

The FT-IR spectrum of $\text{Fe}_2\text{O}_3/\text{CuO}$ nanoparticles is shown

in Figure 1. The FT-IR spectrum shows a broad band in the 3200–3550 cm^{-1} range, which is due to the O–H stretching vibration of water molecules.³⁵ The bands at 1640 and 1340 cm^{-1} arise from the bending vibration of water molecules.³⁶ The OH stretching vibration of the lattice water molecules is responsible for the band at 1120 cm^{-1} .³⁷ Vibrationally, the Cu–O and Fe–O bands extend at 495 and 524 cm^{-1} , respectively.^{36,38}

Figure S1 displays the XRD pattern related to the $\text{Fe}_2\text{O}_3/\text{CuO}$ nanoparticles. It is noteworthy that at $2\theta = 24.28, 33.44, 35.86, 72.66,$ and 75.96° , the characteristic peaks of Fe_2O_3 appeared. Peaks related to the Cu–O phase appeared at $2\theta = 35.78, 38.89, 48.83, 58.37,$ and $61.69, 66.36, 68.11, 72.59, 75.33^\circ$. Scherrer's equation, used to predict the crystallite size of photocatalysts, gave a size of 36 nm.

Field emission scanning electron microscopy (FE-SEM) was used to examine the photocatalyst's morphology. FE-SEM images of $\text{Fe}_2\text{O}_3/\text{CuO}$ nanoparticles are shown in Figure 2a. The FE-SEM micrograph revealed that these photocatalysts showed that they have an irregular geometric shape. The photocatalyst's particle sizes estimated from FE-SEM are in good agreement with the XRD findings. The TEM images of the $\text{Fe}_2\text{O}_3/\text{CuO}$ nanoparticles are shown in Figure 2b, and they confirm the irregular geometric shape of the nanocomposite.

To determine the photocatalyst's purity, EDS analysis was performed. The photocatalyst's elemental composition derived from EDS is shown in Figures 2c and 3d. The photocatalyst contains no impurities, based on the results. Moreover, the gathered information verifies that the desired elements (O, Cu, and Fe) are present. Additionally, elemental mapping images demonstrate the homogeneous distribution of the components.

Dynamic Light Scattering (DLS) and Zeta Potential

At room temperature, the zeta potential and particle size distribution were determined using a DLS instrument. For sonication, 1 mg of the material was used in 2 mL of deionized water for 20 min at room temperature.³⁹ After adding 400 L of H_2O_2 to the prepared solution, it was left outside in the sun for about 5 min. Following the formation of $\cdot\text{OH}$ and OH^- on the catalyst surface, as well as the e-/h+ couples in the nanoparticles, the zeta potential and particle size distribution were evaluated once more. The

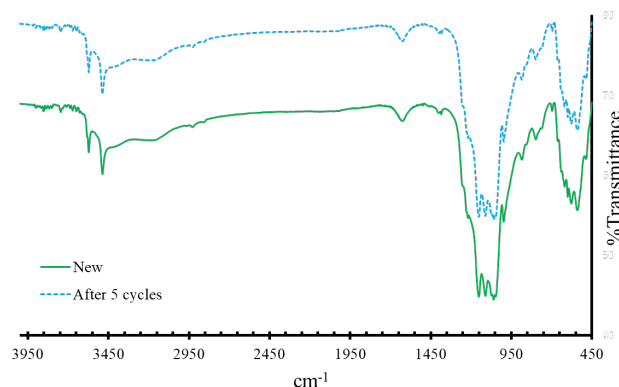


Figure 1. FT-IR Spectrum of $\text{Fe}_2\text{O}_3/\text{CuO}$ Nanoparticles

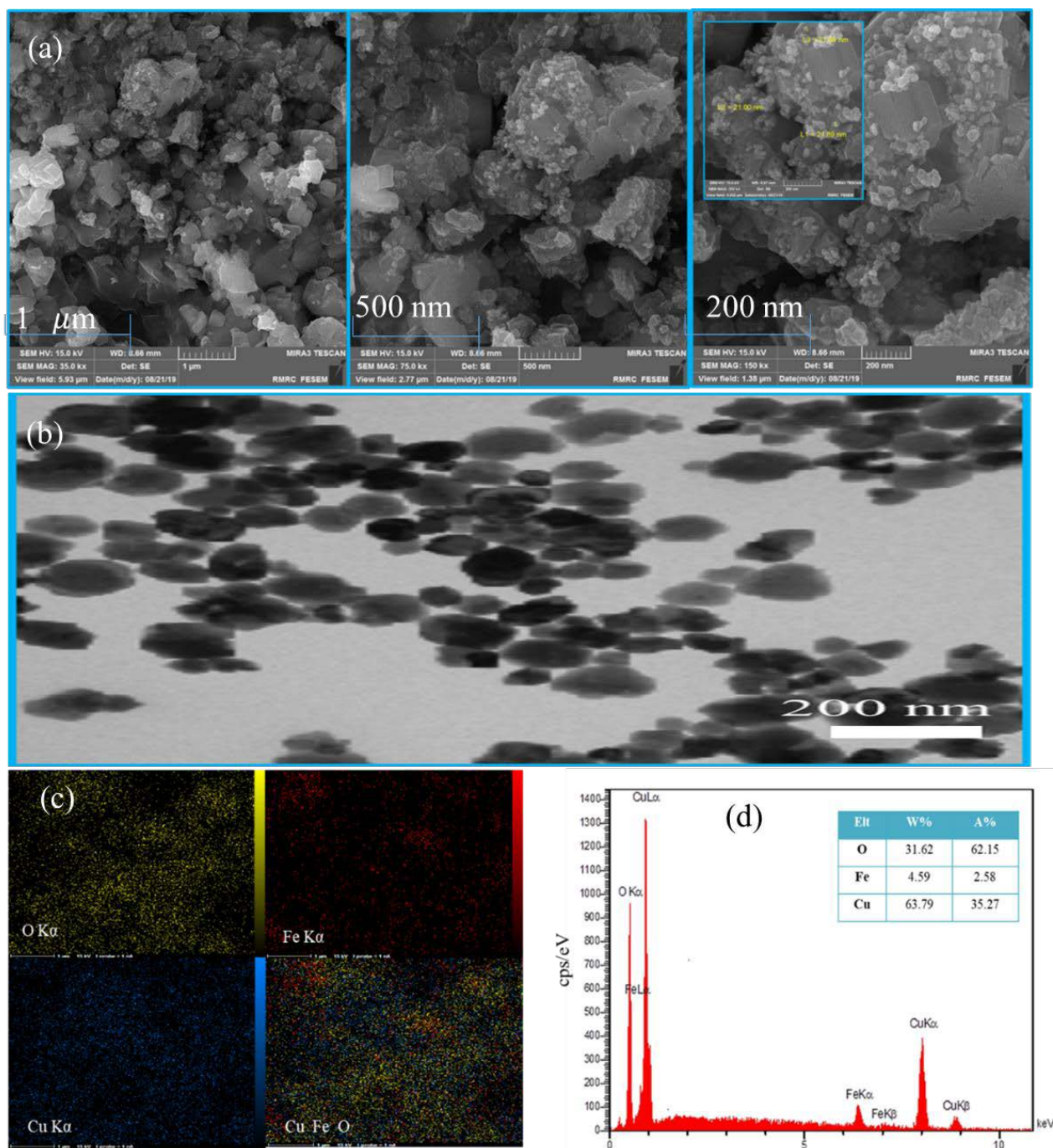


Figure 2. (a) FE-SEM Images of Fe₂O₃@CuO Nanoparticles. (b) TEM Images of Fe₂O₃@CuO Nanoparticles. (c) EDS Elemental Maps of O-Kα, Fe-Kα, and Cu-Kα, along with the Combined Cu-Fe-O Map, Confirm the Uniform Spatial Distribution of the Constituent Elements. (d) EDS Spectrum and Quantitative Analysis (W% and A%) Showing the Dominant Presence of Cu, Followed by O and a Smaller Amount of Fe

particle size distribution of the Fe₂O₃@CuO nanoparticles is shown in Figure S2a and b. The DLS method determines particle size based on the hydrodynamic diameter of colloidal particles that scatter light at a constant rate. In this case, the thickness of the adsorbed solvation shell affected the size of the metallic core; hence, DLS recorded a larger size than the XRD and FE-SEM techniques. Figure S2c and d displays the zeta potential of the samples scattered in water and with H₂O₂. The zeta potential, which reflects electrostatic repulsion forces and can prevent nanoparticle aggregation, often indicates the surface charge of nanoparticles.⁴⁰ Low electrostatic repulsion (less than 17.9 mV in magnitude) is indicated by the zeta

potential values of the as-produced nanoparticles. As seen in Figure S2c and d, the zeta potential becomes negative upon addition of H₂O₂ and exposure to sunlight. This implies that the nanoparticles' surfaces include OH[•], and OH⁻. In the Fe₂O₃@CuO photocatalytic system, the primary photochemical process begins with the absorption of visible/near-visible photons by the semiconductors, leading to the generation of electron-hole pairs (e⁻/h⁺). The effective transfer of these charge carriers from the core (Fe₂O₃) to the shell (CuO) or vice versa, depending on the type of electronic interface (p-n, Z-scheme, or S-scheme, as reported in different studies), increases charge separation and reduces the recombination rate.⁴¹⁻⁴³ This

creates favorable conditions for the production of more reactive oxygen species (ROS). Specifically, the presence of CuO in the heterojunction improves charge transfer at the contact interfaces and reduces the effective band gap of the composite, which enhances visible-light absorption and charge separation efficiency. This interfacial charge transfer mechanism between Fe₂O₃ and CuO (the effect of the interfacial junction and charge transfer) has been identified in previous experimental and analytical studies as the main factor responsible for increasing photocatalytic activity.^{41,44} After the partial separation of charges, the pathways for generating active radicals can be summarized as follows: the excited electrons (e⁻) can reduce dissolved molecular oxygen to superoxide anion (O₂^{•-}), and the holes (h⁺) can oxidize hydroxide ions (OH⁻) or water molecules to produce hydroxyl radicals (OH[•]). In addition, adding H₂O₂ to the system plays multiple roles: H₂O₂ can act as an electron acceptor (reducing e⁻/h⁺ recombination) and can be converted directly, or through reactions with electrons/superoxides, into OH[•] and HO₂, which increases the concentration of strong oxidizing species and accelerates pollutant degradation. The dominant reactive species (OH[•] versus O₂^{•-} or h⁺) depends on the system conditions (pH, presence of H₂O₂, surface charge of the catalyst, and band structure), but in many reports, the hydroxyl radical has been identified as the main oxidizing agent in the degradation of organic dyes.⁴⁵⁻⁴⁷

Aspects of Optics

The optical properties of the Fe₂O₃@CuO nanocomposite, including band gap and light-harvesting characteristics, were examined using UV-Vis reflectance spectroscopy (Figure 3a). Due to the charge transfer from the valence band (VB) to the conduction band (CB), activation by UV and visible light of the nanocomposite was observed in the obtained results. Tauc's equation (Equations 2 and 3) was used to calculate the band gap energy (E_g).^{40,48,49}

$$(\alpha h\nu)^n = B (h\nu - E_g) \quad (2)$$

$$\alpha = \frac{2.303 A}{t} \quad (3)$$

where, α , $h\nu$, n , B , A , and t are the absorption coefficient, energy of photons, constant, proportionality constant, absorbance, and thickness of the sample, respectively. After doing the calculations, the values of E_g for the Fe₂O₃@CuO nanoparticles are 2.18 eV (Fe₂O₃) and 1.45 eV (CuO) (Figure 3b).⁵⁰

Multiple studies have shown that integrating CuO, a p type semiconductor with a relatively narrow band gap (≈ 1.4 eV), into heterostructures with wide- or medium bandgap oxides significantly enhances visible light absorption and promotes charge separation, thereby overcoming limitations of bare oxides.⁵¹⁻⁵³ For example, in a ZnO-CuO composite the coupling shifts the absorption edge into the visible region and facilitates electron-hole separation via a p-n junction, which markedly boosts photocatalytic degradation of dyes under visible light.⁵⁴

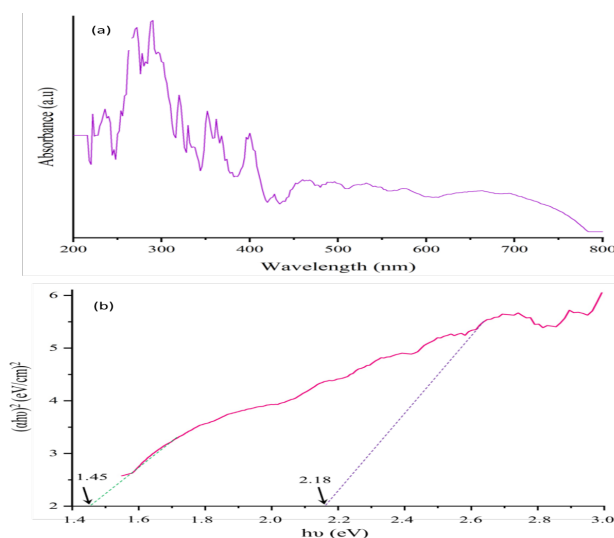


Figure 3. (a) Absorption Spectra of Fe₂O₃@CuO. (b) Tauc Plot of Fe₂O₃@CuO

Similarly, for an Fe₂O₃/CuO heterojunction photoanode, formation of the junction induces an internal electric field that improves movement of photoexcited charge carriers, significantly reduces electron-hole recombination, and enhances photogenerated charge utilization. Consequently, the effective optical band gap of the composite becomes smaller than that of the pure wide-gap oxide, allowing activation under lower-energy (visible) photons and improving photocatalytic degradation efficiency. Given that pure Fe₂O₃ often suffers from rapid recombination of photogenerated carriers despite absorption in the visible region, the formation of a Fe₂O₃@CuO core-shell or heterojunction structure can address this drawback by lowering the effective band gap, boosting visible light absorption, enhancing charge separation, and thereby increasing photocatalytic performance. This mechanistic advantage underpins the rationale for employing the Fe₂O₃@CuO system for visible-light-driven dye degradation (Figure S3). The energy band alignment and charge transfer mechanism of the α -Fe₂O₃/CuO heterogeneous bond are shown in Figure S3 based on the results of a study conducted by Kyesmen et al.⁵¹

Dye Removal Experiments

Effect of H₂O₂

Fe₂O₃@CuO was used to efficiently degrade MB, and the amount of H₂O₂ was optimized to achieve this under sunshine. Figure 4a illustrates that in the absence of H₂O₂, less than 15.3% of the methylene blue was destroyed in 45 min. However, the degradation efficiency rose to 98.6% when the H₂O₂ concentration reached 6%. Research indicates that the introduction of hydrogen peroxide into the system results in the generation of OOH[•] and OH[•] radicals on the photocatalyst's surface, driven by the holes and electrons produced within the catalyst. The increased radical content accelerates the degradation rate.⁵⁵ The main mechanism of this enhancement is the improved separation of electron-hole pairs and the increased production of reactive oxygen radicals. H₂O₂ acts as an electron acceptor; it captures the free electrons in the catalyst and prevents

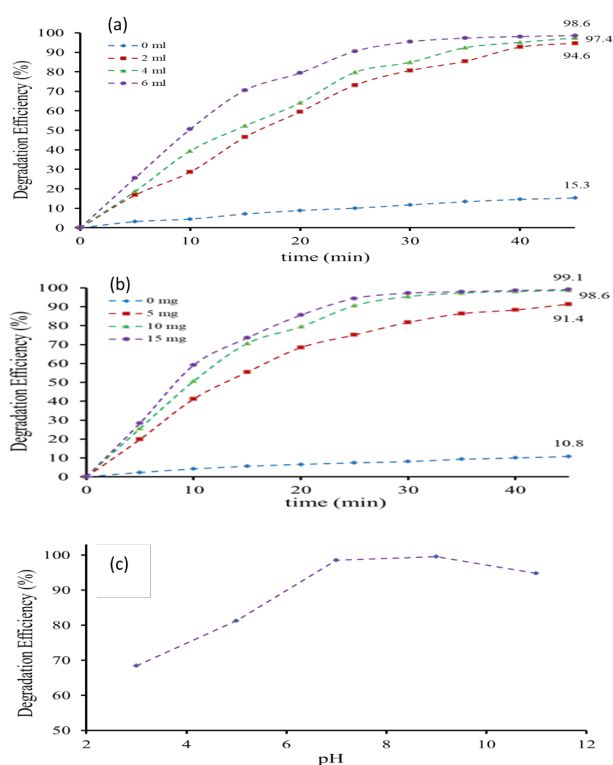


Figure 4. (a) Effect of Different H₂O₂ Dosages on the Photocatalytic Degradation of MB by Fe₂O₃@CuO at Various Time Intervals (MB Concentration: 10 mg/L; Catalyst Dosage: 10 mg; pH: 7; Sunlight Irradiation). (b) Effect of Different Fe₂O₃@CuO Dosages on the Sunlight-Driven Degradation of MB (MB Concentration: 10 mg/L; pH: 7; H₂O₂ Volume: 6 mL; Sunlight Irradiation). (c) Effect of Solution pH on the Degradation of MB by Fe₂O₃@CuO after 45 min (MB Concentration: 10 mg/L; Catalyst Dosage: 15 mg; H₂O₂ Volume: 6 mL; Sunlight Irradiation)

their recombination with the holes, while itself being converted into OH[•] and other radical species, which are highly oxidative. The results of the study by Saha et al confirmed the findings of the present study.⁵⁶ However, the literature shows that the relationship between H₂O₂ concentration and degradation efficiency is not simply linear. In some studies, excessive H₂O₂ leads to a decrease in efficiency.^{57–59} This is because at high concentrations, H₂O₂ can act as an OH[•] radical scavenger, eliminating the active radicals, or it can increase the production of less reactive radical species such as HO₂[•].⁵⁹

Effect of Catalyst Dosage

The amount of nanocatalyst is one of the most crucial factors in degradation reactions. The optimal quantity must be determined to avoid using excess catalyst. An MB concentration of 10 mg/L and different doses, including 0, 50, 100, and 150 mg/L of the Fe₂O₃@CuO nanocatalyst and 6% of hydrogen peroxide, were used to study the effect of the nanocatalyst concentrations on photocatalytic degradation. Figure 4b illustrates how the breakdown of the MB solution is affected by varying concentrations of Fe₂O₃@CuO nanocatalyst. As the figures demonstrate, there is a significant increase in reaction speed with increasing photocatalyst dosage. This is because there are more active sites on the photocatalyst's surface, which increases the quantity of OOH[•] and OH[•] radicals.⁶⁰ Zyoud et al studied the efficiency of the CuS@TiO₂ nanocatalyst in the removal

and complete degradation of the pesticide imidacloprid from water under simulated sunlight. One key aspect of this research was the effect of catalyst concentration on removal efficiency. Experiments were conducted with different amounts of CuS@TiO₂ ranging from 0.05 to 0.25 g in 100 mL of a 40 ppm imidacloprid solution. The results showed that increasing the catalyst dose up to 0.20 g led to a continuous rise in degradation efficiency from 50 to 93%, which was mainly due to the increased number of active sites for pollutant adsorption and the generation of reactive oxygen species. These findings are consistent with the results of the present study.⁶¹

Effect of pH

The pH of the solution is an important parameter in surface adsorption. It can also greatly influence the surface charge of the adsorbent as well as the structure and degree of ionization of the chemical compound.⁶² To further investigate the dependence of adsorption and degradation on pH, experiments were carried out at room temperature in the pH range of 2.0 to 12.0. The pH was adjusted with 0.1 M NaOH and 0.1 M HCl solutions.⁶³ MB degradation efficiency increased with rising pH up to a certain point (Figure 4c). After 45 minutes at pH 9, 99.6% photocatalytic activity was observed. Degradation percentages at pH 3, 5, 7, and 11 were 68.4, 81.3, 98.6, and 94.8%, respectively. Higher pH levels enhance the interaction between the photocatalyst surface and the cationic dye because the surface becomes predominantly covered with negatively charged OH[−], resulting in significant adsorption.⁶⁴ At pH 11, the degradation of MB decreased slightly. Degradation may be inhibited at very high pH (pH > 11) due to competition between hydroxyl ions and dye species for active sites.⁶⁵ At lower pH values, MB degradation is reduced due to the presence of H⁺ on the photocatalyst's surface and the decreased generation of reactive species including O₂^{•−}, OH[•], and OH[−].⁶⁶ Since MB is a cationic dye, it has a stronger tendency to adsorb onto negatively charged surfaces in alkaline environments. This leads to higher initial adsorption of the dye and, consequently, enhances the generation and interaction of reactive oxygen species with MB molecules. Such behavior has been repeatedly confirmed in previous experimental studies and comprehensive reviews. For example, the results reported by Khan et al fully align with this pattern and show a significant increase in MB degradation efficiency at alkaline pH levels.⁶⁷ In the study by Zhou et al, modifying fly ash with magnesium resulted in an adsorbent with a negative charge under alkaline pH conditions, which increased the MB removal efficiency to more than 95%. Similar to the results of the present study, the removal efficiency decreased significantly in acidic pH due to the positive surface charge of the adsorbent and electrostatic repulsion, while the highest efficiency was observed in the pH range of 5–11. This similarity shows that the dominant mechanism in both studies is electrostatic adsorption between the negatively charged surface and the cationic MB dye.⁶⁸ In the study by Kalaycıoğlu et al, the CeO₂/GO/PAM composite

hydrogel also showed the highest degradation efficiency ($\approx 90\%$) under alkaline conditions ($\approx \text{pH } 12$), which was attributed to the increased negative surface charge in such environments. This trend is consistent with the findings of the present study; however, in the current work, increasing the pH above 11 leads to a decrease in efficiency. This difference is likely related to the sensitivity of the present photocatalyst to competition between OH^- ions and the dye for active sites, whereas the network structure of the hydrogel in the study of Kalaycıoğlu et al does not show this limitation.⁶⁹

Reproducibility of Photocatalyst Properties

Using Equation (1), Figure 5 shows the nanocatalyst's degradation efficiencies across five cycles. After five cycles for MB degradation, the $\text{Fe}_2\text{O}_3/\text{CuO}$ nanocatalyst's performance declined by 1.5%. After five recovery processes, the FTIR spectra (Figure 1) and XRD (Figure S1) were obtained to confirm the stability of the photocatalyst structure. Comparing the spectra taken before and after the five photocatalytic cycles revealed no significant change in the nanocatalyst's structure. Consequently, the $\text{Fe}_2\text{O}_3/\text{CuO}$ photocatalyst exhibits good reusability for the degradation of the studied dye. This level of efficiency retention compares favorably with some reports; for example, Maleki et al reported that a zinc–lanthanum oxide (Zn–La oxide) nanocatalyst used for the photocatalytic degradation of methylene blue under UV irradiation showed about a 5.4% decrease in efficiency, with the removal rate dropping from 98.8 to 93.4%.⁷⁰ The small decline observed here indicates the structural stability and good reusability of this photocatalyst, making it suitable for large-scale water treatment applications.

One limitation of the present study is that control experiments (photolysis: MB + light only; adsorption in the dark; MB + H_2O_2 + light without catalyst; MB + catalyst in the dark; catalyst + H_2O_2 in the dark) were not performed in the present manuscript. This was due to three main reasons: (1) the primary scope of the current work was to develop and characterize the $\text{Fe}_2\text{O}_3/\text{CuO}$ heterojunction and report its overall photocatalytic performance rather than to fully deconvolute mechanistic contributions; (2) resource and time constraints during preparation of the initial submission; and (3) existing

peer-reviewed literature showing that under comparable experimental conditions, direct photolysis or simple adsorption often contribute minimally to MB removal, while well-designed $\text{Fe}_2\text{O}_3/\text{CuO}$ heterojunctions show superior photocatalytic activity compared with single phases. Representative supporting references are cited.⁷¹ Studies of Fe-containing photo-Fenton and Fe_2O_3 -based heterostructures demonstrate enhanced performance of heterojunctions and the important role of catalyst-mediated ROS generation.⁷² Regarding H_2O_2 , UV/ H_2O_2 systems can degrade MB without a catalyst but typically require UV irradiation and optimized H_2O_2 doses; under visible-light conditions and with the low H_2O_2 amounts used in many heterogeneous photocatalysis studies, the standalone H_2O_2 effect is limited.⁷³

Conclusions

In this study, $\text{Fe}_2\text{O}_3/\text{CuO}$ core–shell nanoparticles were successfully synthesized and applied as an efficient photocatalyst for the degradation of methylene blue under sunlight irradiation. Structural characterization using FT-IR, XRD, FE-SEM, TEM, EDS, and DLS confirmed the successful formation of the nanocomposite, its appropriate crystallinity, homogeneous elemental distribution, and suitable optical properties with band gap energies of 2.18 eV (Fe_2O_3) and 1.45 eV (CuO). Photocatalytic experiments demonstrated that the presence of H_2O_2 significantly enhanced dye degradation through the generation of highly reactive hydroxyl and superoxide radicals. Optimization studies revealed that increasing the catalyst dosage and adjusting the solution pH to alkaline conditions (pH 9) considerably improved the degradation efficiency. Under optimal conditions, the $\text{Fe}_2\text{O}_3/\text{CuO}$ nanocatalyst achieved a removal efficiency of over 99% within 45 min. Furthermore, recyclability tests confirmed the remarkable stability and reusability of the nanocatalyst, with only a 1.5% reduction in efficiency after five cycles and no detectable structural changes in the FT-IR and XRD patterns. Overall, the results indicate that $\text{Fe}_2\text{O}_3/\text{CuO}$ nanoparticles exhibit excellent photocatalytic performance, stability, and reusability, making them promising and cost-effective materials for the removal of organic dyes from wastewater under sunlight irradiation. This work highlights their potential for practical applications in sustainable wastewater treatment systems. However, this study focused only on the removal of methylene blue under controlled laboratory conditions, and the performance of $\text{Fe}_2\text{O}_3/\text{CuO}$ nanoparticles against other pollutants or in real wastewater was not examined. For future research, it is recommended that the efficiency of the nanocatalyst be evaluated on a larger, more realistic scale using industrial wastewater samples to determine its potential for practical application. In addition, assessing the long-term stability of the nanoparticles in the environment, examining possible environmental impacts, and developing modified structures such as those produced by doping, surface functionalization, or combination with other semiconductors could help improve the photocatalytic performance and expand the environmental applications

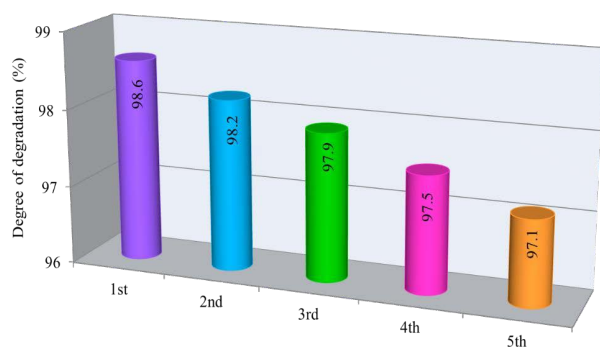


Figure 5. Reusability of the Nanocatalyst for MB Degradation in the Presence of H_2O_2 (MB Concentration: 10 mg/L; Catalyst Dosage: 15 mg; H_2O_2 Volume: 6 mL; pH 9; Under Sunlight Irradiation)

of this nanomaterial.

Acknowledgments

The authors sincerely thank and appreciate Hormozgan University of Medical Sciences, Bandar Abbas, Iran, for its financial support of this study.

Authors' Contribution

Conceptualization: Omid Safa, Mahmoud Omid
Data Curation: Omid Safa
Formal Analysis: Esmaeil Mohammadian, Amir Shadboorestan
Funding Acquisition: Mahmoud Omid
Investigation: Farinam Faryabi
Methodology: Esmaeil Mohammadian, Masoud Memar
Project Administration: Esmaeil Mohammadian
Resources: Farinam Faryabi
Software: Farinam Faryabi
Supervision: Omid Safa, Mahmoud Omid
Validation: Seyyed Mohammad Hassan Hashemi, Masoud Memar
Visualization: Esmaeil Mohammadian
Writing–Original Draft: Farinam Faryabi, Galavizh Mandomi
Writing–Review & Editing: Seyyed Mohammad Hassan Hashemi, Amir Shadboorestan

Competing Interests

The authors declare that there are no conflicts of interest.

Ethical Approval

This study was approved by the Ethics Committee of Hormozgan University of Medical Sciences, Hormozgan, Iran (Ethics Approval Code: IR.HUMS.REC.1401.039).

Funding

This work was supported by the Research Grants from the Hormozgan University of Medical Sciences, Bandar Abbas, Iran (Grant number: IR.HUMS.REC.1401.039).

Supplementary Files

Supplementary file contains Figures S1–S3.

References

- Noorimotlagh Z, Darvishi Cheshmeh Soltani R, Khataee AR, Shahriyar S, Nourmoradi H. Adsorption of a textile dye in aqueous phase using mesoporous activated carbon prepared from Iranian milk vetch. *J Taiwan Inst Chem Eng* 2014;45(4):1783–91. doi:10.1016/j.jtice.2014.02.017
- Darvishi Cheshmeh Soltani R, Jorfi S, Ramezani H, Purfadakari S. Ultrasonically induced ZnO-biosilica nanocomposite for degradation of a textile dye in aqueous phase. *Ultrason Sonochem* 2016;28:69–78. doi:10.1016/j.ultsonch.2015.07.002
- Darvishi Cheshmeh Soltani R, Khataee AR, Godini H, Safari M, Ghanadzadeh MJ, Rajaei MS. Response surface methodological evaluation of the adsorption of textile dye onto biosilica/alginate nanobiocomposite: thermodynamic, kinetic, and isotherm studies. *Desalin Water Treat* 2015;56(5):1389–402. doi:10.1080/19443994.2014.950344
- Ghaedi M, Ghaedi AM, Hossainpour M, Ansari A, Habibi MH, Asghari AR. Least square-support vector (LS-SVM) method for modeling of methylene blue dye adsorption using copper oxide loaded on activated carbon: kinetic and isotherm study. *J Ind Eng Chem* 2014;20(4):1641–9. doi:10.1016/j.jiec.2013.08.011
- Kuriechen SK, Murugesan S, Raj SP, Maruthamuthu P. Visible light assisted photocatalytic mineralization of Reactive Red 180 using colloidal TiO₂ and oxone. *Chem Eng J* 2011;174(2):530–8. doi:10.1016/j.cej.2011.09.024
- Shirvani Ardekani P, Karimi H, Ghaedi M, Asfaram A, Purkait MK. Ultrasonic assisted removal of methylene blue on ultrasonically synthesized zinc hydroxide nanoparticles on activated carbon prepared from wood of cherry tree: experimental design methodology and artificial neural network. *J Mol Liq* 2017;229:114–24. doi:10.1016/j.molliq.2016.12.028
- Bordbar M, Sharifi-Zarchi Z, Khodadadi B. Green synthesis of copper oxide nanoparticles/clinoptilolite using *Rheum palmatum* L. root extract: high catalytic activity for reduction of 4-nitro phenol, rhodamine B, and methylene blue. *J Solgel Sci Technol* 2017;81(3):724–33. doi:10.1007/s10971-016-4239-1
- Ghaedi M, Tavallali H, Sharifi M, Nasiri Kokhdan S, Asghari A. Preparation of low cost activated carbon from *Myrtus communis* and pomegranate and their efficient application for removal of Congo red from aqueous solution. *Spectrochim Acta A Mol Biomol Spectrosc* 2012;86:107–14. doi:10.1016/j.saa.2011.10.012
- Mittal A, Mittal J, Malviya A, Gupta VK. Adsorptive removal of hazardous anionic dye “Congo red” from wastewater using waste materials and recovery by desorption. *J Colloid Interface Sci* 2009;340(1):16–26. doi:10.1016/j.jcis.2009.08.019
- Khalili E, Hassanzadeh-Tabrizi SA. ZnO–CdO nanocomposite: microemulsion synthesis and dye removal ability. *J Solgel Sci Technol* 2017;81(2):475–82. doi:10.1007/s10971-016-4211-0
- Mahmoud GA, Abdel-Aal SE, Badway NA, Elbayaa AA, Ahmed DF. A novel hydrogel based on agricultural waste for removal of hazardous dyes from aqueous solution and reuse process in a secondary adsorption. *Polym Bull* 2017;74(2):337–58. doi:10.1007/s00289-016-1717-0
- Zou W, Bai H, Gao S, Li K. Characterization of modified sawdust, kinetic and equilibrium study about methylene blue adsorption in batch mode. *Korean J Chem Eng* 2013;30(1):111–22. doi:10.1007/s11814-012-0096-y
- Hossein Beyki M, Bayat M, Shemirani F. Fabrication of core-shell structured magnetic nanocellulose base polymeric ionic liquid for effective biosorption of Congo red dye. *Bioresour Technol* 2016;218:326–34. doi:10.1016/j.biortech.2016.06.069
- Chen H, Zhao J, Wu J, Dai G. Isotherm, thermodynamic, kinetics and adsorption mechanism studies of methyl orange by surfactant modified silkworm exuviae. *J Hazard Mater* 2011;192(1):246–54. doi:10.1016/j.jhazmat.2011.05.014
- Murcia JJ, Hidalgo MC, Navío JA, Araña J, Doña-Rodríguez JM. Correlation study between photo-degradation and surface adsorption properties of phenol and methyl orange on TiO₂ Vs platinum-supported TiO₂. *Appl Catal B Environ* 2014;150:151:107–15. doi:10.1016/j.apcatb.2013.12.010
- Qiao WJ, Wang ZZ, Zhai SR, Xiao ZY, Zhang F, An QD. Oxygen-containing/amino groups bifunctionalized SBA-15 toward efficient removal of methylene blue: kinetics, isotherm and mechanism analysis. *J Solgel Sci Technol* 2015;76(2):320–31. doi:10.1007/s10971-015-3779-0
- Robati D, Mirza B, Rajabi M, Moradi O, Tyagi I, Agarwal S, et al. Removal of hazardous dyes-BR 12 and methyl orange using graphene oxide as an adsorbent from aqueous phase. *Chem Eng J* 2016;284:687–97. doi:10.1016/j.cej.2015.08.131
- Batista AP, Carvalho HW, Luz GH, Martins PF, Gonçalves M, Oliveira LC. Preparation of CuO/SiO₂ and photocatalytic activity by degradation of methylene blue. *Environ Chem Lett* 2010;8(1):63–7. doi:10.1007/s10311-008-0192-8
- Ndolomingo MJ, Meijboom R. Kinetic analysis of catalytic oxidation of methylene blue over γ-Al₂O₃ supported copper nanoparticles. *Appl Catal A Gen* 2015;506:33–43. doi:10.1016/j.apcata.2015.08.036
- Mosleh S, Rahimi MR, Ghaedi M, Dashtian K, Hajati S. Sonochemical-assisted synthesis of CuO/Cu₂O/Cu nanoparticles as efficient photocatalyst for simultaneous degradation of pollutant dyes in rotating packed bed reactor: LED illumination and central composite design optimization. *Ultrason Sonochem* 2018;40(Pt A):601–10. doi:10.1016/j.ultsonch.2017.08.007
- Hunge YM, Mahadik MA, Moholkar AV, Bhosale CH.

- Photoelectrocatalytic degradation of oxalic acid using WO₃ and stratified WO₃/TiO₂ photocatalysts under sunlight illumination. *Ultrason Sonochem* 2017;35(Pt A):233-42. doi:10.1016/j.ultsonch.2016.09.024
22. Sasikala R, Karthikeyan K, Easwaramoorthy D, Bilal IM, Rani SK. Photocatalytic degradation of trypan blue and methyl orange azo dyes by cerium loaded CuO nanoparticles. *Environ Nanotechnol Monit Manag* 2016;6:45-53. doi:10.1016/j.enmm.2016.07.001
 23. Navakoteswara Rao V, Lakshmana Reddy N, Preethi V, Karthik M, Yu YT, Yang JM, et al. A critical review on core/shell-based nanostructured photocatalysts for improved hydrogen generation. *Int J Hydrogen Energy* 2023;48(31):11754-74. doi:10.1016/j.ijhydene.2023.01.059
 24. Mohamadian E, Shayanfar A, Khoubnasabjafari M, Jouyban-Gharamaleki V, Ghaffary S, Jouyban A. Analysis of deferiprone in exhaled breath condensate using silver nanoparticle-enhanced terbium fluorescence. *Anal Methods* 2017;9(38):5640-5. doi:10.1039/c7ay01715d
 25. Mohamed AT, Abdel Hameed R, El-Moslami SH, Fareid M, Othman M, Loutfy SA, et al. Facile synthesis of Fe₂O₃, Fe₂O₃@CuO and WO₃ nanoparticles: characterization, structure determination and evaluation of their biological activity. *Sci Rep* 2024;14(1):6081. doi:10.1038/s41598-024-55319-8
 26. Mohammadian E, Rahimpour E, Alizadeh-Sani M, Foroumadi A, Jouyban A. An overview on terbium sensitized based-optical sensors/nanosensors for determination of pharmaceuticals. *Appl Spectrosc Rev* 2022;57(1):39-76. doi:10.1080/05704928.2020.1843174
 27. Nuengmatcha P, Porrawatkul P, Chanthai S, Sricharoen P, Limchoowong N. Enhanced photocatalytic degradation of methylene blue using Fe₂O₃/graphene/CuO nanocomposites under visible light. *J Environ Chem Eng* 2019;7(6):103438. doi:10.1016/j.jece.2019.103438
 28. Goudjil MB, Dali H, Zighmi S, Mahcene Z, Bencheikh SE. Photocatalytic degradation of methylene blue dye with biosynthesized hematite α -Fe₂O₃ nanoparticles under UV-Irradiation. *Desalin Water Treat* 2024;317:100079. doi:10.1016/j.dwt.2024.100079
 29. Turkoglu S, Kepekci RA, Keskinan O. Photocatalysis of methylene blue by ZnO nanoparticles produced by green synthesis. *Int J Phytoremediation* 2026;28(3):513-23. doi:10.1080/15226514.2025.2574902
 30. Zulfa LL, Ediati R, Hidayat AR, Subagyo R, Faaizattunnisa N, Kusumawati Y, et al. Synergistic effect of modified pore and heterojunction of MOF-derived α -Fe₂O₃/ZnO for superior photocatalytic degradation of methylene blue. *RSC Adv* 2023;13(6):3818-34. doi:10.1039/d2ra07946a
 31. Hamdan S, Al-Ali K, Vega LF, Muscetta M, Yusuf AO, Palmisano G. Zinc cadmium sulphide (ZnxCd1-xS)-based photocatalysts for hydrogen production from seawater and wastewater. *J Environ Chem Eng* 2024;12(5):113937. doi:10.1016/j.jece.2024.113937
 32. Mahmood A, Zulfiqar S, Ali S, Ammara U, Mahmood K, Farrukh MA, et al. Novel Fe₂O₃-CuO-MoO₃ magnetic nanocomposite for photocatalysis of methylene blue. *J Supercond Nov Magn* 2021;34(7):1791-9. doi:10.1007/s10948-020-05725-6
 33. Iqbal S, Bahadur A, Anwer S, Shoaib M, Liu G, Li H, et al. Designing novel morphologies of L-cysteine surface capped 2D covellite (CuS) nanoplates to study the effect of CuS morphologies on dye degradation rate under visible light. *CrystEngComm* 2020;22(24):4162-73. doi:10.1039/d0ce00421a
 34. Prathap MU, Kaur B, Srivastava R. Hydrothermal synthesis of CuO micro-/nanostructures and their applications in the oxidative degradation of methylene blue and non-enzymatic sensing of glucose/H₂O₂. *J Colloid Interface Sci* 2012;370(1):144-54. doi:10.1016/j.jcis.2011.12.074
 35. Forouzani M, Mardani HR, Ziari M, Malekzadeh A, Biparva P. Comparative study of oxidation of benzyl alcohol: influence of Cu-doped metal cation on nano ZnO catalytic activity. *Chem Eng J* 2015;275:220-6. doi:10.1016/j.cej.2015.04.032
 36. Benhammada A, Trache D, Kesraoui M, Tarchoun AF, Chelouche S, Mezroua A. Synthesis and characterization of α -Fe₂O₃ nanoparticles from different precursors and their catalytic effect on the thermal decomposition of nitrocellulose. *Thermochim Acta* 2020;686:178570. doi:10.1016/j.tca.2020.178570
 37. Farahmandjou M, Soflaee F. Synthesis and characterization of α -Fe₂O₃ nanoparticles by simple co-precipitation method. *Phys Chem Res* 2015;3(3):191-6. doi:10.22036/pcr.2015.9193
 38. Ponmudi S, Sivakumar R, Sanjeeviraja C, Gopalakrishnan C, Jeyadheepan K. Tuning the morphology of Cr₂O₃: CuO (50:50) thin films by RF magnetron sputtering for room temperature sensing application. *Appl Surf Sci* 2019;466:703-14. doi:10.1016/j.apsusc.2018.10.096
 39. Sackey J, Nwanya AC, Bashir AK, Matinise N, Ngilirabanga JB, Ameh AE, et al. Electrochemical properties of *Euphorbia pulcherrima* mediated copper oxide nanoparticles. *Mater Chem Phys* 2020;244:122714. doi:10.1016/j.matchemphys.2020.122714
 40. Wang Y, Jiang T, Meng D, Yang J, Li Y, Ma Q, et al. Fabrication of nanostructured CuO films by electrodeposition and their photocatalytic properties. *Appl Surf Sci* 2014;317:414-21. doi:10.1016/j.apsusc.2014.08.144
 41. Gao Y, Zhang N, Wang C, Zhao F, Yu Y. Construction of Fe₂O₃@CuO heterojunction nanotubes for enhanced oxygen evolution reaction. *ACS Appl Energy Mater* 2020;3(1):666-74. doi:10.1021/acsaeam.9b01866
 42. Ghasemi S, Parastesh A, Padervand M, Ren H, Li X, Labidi A, et al. Recent progress on Z- and S-scheme photocatalysis: mechanistic understanding toward green applications. *Curr Opin Chem Eng* 2025;47:101059. doi:10.1016/j.coche.2024.101059
 43. Wang S, Zhang Y, Feng L, Tian Z, Huang G, Yu J, et al. Degradation of tetracycline in medical wastewater by Fe₂O₃/CuO photocatalyst under visible light. *Nano* 2026;2650060. doi:10.1142/S1793292026500608
 44. Asenath-Smith E, Ambrogi EK, Barnes E, Brame JA. CuO enhances the photocatalytic activity of Fe₂O₃ through synergistic reactive oxygen species interactions. *Colloids Surf A Physicochem Eng Asp* 2020;603:125179. doi:10.1016/j.colsurfa.2020.125179
 45. Hassaan MA, El-Nemr MA, Elkatory MR, Ragab S, Niculescu VC, El Nemr A. Principles of photocatalysts and their different applications: a review. *Top Curr Chem* 2023;381(6):31. doi:10.1007/s41061-023-00444-7
 46. Javaid R, Qazi UY. Catalytic oxidation process for the degradation of synthetic dyes: an overview. *Int J Environ Res Public Health* 2019;16(11):2066. doi:10.3390/ijerph16112066
 47. Papić S, Koprivanac N, Božić AL, Vujević D, Dragicević SK, Kusić H, et al. Advanced oxidation processes in azo dye wastewater treatment. *Water Environ Res* 2006;78(6):572-9.
 48. Barzegar MH, Ghaedi M, Madadi Avargani V, Sabzehmeidani MM, Sadeghfard F, Jannesar R. Electrochemical synthesis and efficient photocatalytic degradation of azo dye alizarin yellow R by Cu/CuO nanorods under visible LED light irradiation using experimental design methodology. *Polyhedron* 2019;158:506-14. doi:10.1016/j.poly.2018.10.040
 49. Fterich M, Ben Nasr F, Lefi R, Toumi M, Guermazi S. Effect of concentration of hexamethylenetetramine in structure, microstructure and optical properties of CuO nanoparticles synthesized by hydrothermal route. *Mater Sci Semicond Process* 2016;43:114-22. doi:10.1016/j.mssp.2015.11.023
 50. El Nahrawy AM, Abou Hammad AB, Bakr AM, Shaheen TI, Mansour AM. Sol-gel synthesis and physical characterization of high impact polystyrene nanocomposites based on Fe₂O₃ doped with ZnO. *Appl Phys A* 2020;126(8):654. doi:10.1007/

- s00339-020-03822-w
51. Kyesmen PI, Nombona N, Diale M. Heterojunction of nanostructured α -Fe₂O₃/CuO for enhancement of photoelectrochemical water splitting. *J Alloys Compd* 2021;863:158724. doi:10.1016/j.jallcom.2021.158724
 52. Baran T, Visibile A, Busch M, He X, Wojtyła S, Rondinini S, et al. Copper oxide-based photocatalysts and photocathodes: fundamentals and recent advances. *Molecules* 2021;26(23):7271. doi:10.3390/molecules26237271
 53. Jha K, Raula M, Gomey S, Kalyani C. Recent advances on catalytic potentials of copper oxides (I & II): fundamentals to applications. *Crit Rev Solid State Mater Sci* 2025;1-43. doi:10.1080/10408436.2025.2560337
 54. Dien ND, Thu Ha PT, Vu XH, Trang TT, Thanh Giang TD, Dung NT. Developing efficient CuO nanoplate/ZnO nanoparticle hybrid photocatalysts for methylene blue degradation under visible light. *RSC Adv* 2023;13(35):24505-18. doi:10.1039/d3ra03791f
 55. Ghobadifard M, Farhadi S, Mohebbi S. Sonocatalytic performance of magnetic flower-like CoFe₂O₄ nanoparticles prepared from a heterometallic oxo-centered trinuclear complex under microwave irradiation. *Polyhedron* 2018;155:66-76. doi:10.1016/j.poly.2018.08.028
 56. Saha D, Desipio MM, Hoinkis TJ, Smeltz EJ, Thorpe R, Hensley DK, et al. Influence of hydrogen peroxide in enhancing photocatalytic activity of carbon nitride under visible light: an insight into reaction intermediates. *J Environ Chem Eng* 2018;6(4):4927-36. doi:10.1016/j.jece.2018.07.030
 57. Buthiyappan A, Abdul Aziz AR, Wan Daud WM. Recent advances and prospects of catalytic advanced oxidation process in treating textile effluents. *Rev Chem Eng* 2016;32(1):1-47. doi:10.1515/revce-2015-0034
 58. Ścieżyńska D, Bury D, Marcinowski P, Bogacki J, Jakubczak M, Jastrzębska A. Two-dimensional nanostructures in the world of advanced oxidation processes. *Catalysts* 2022;12(4):358. doi:10.3390/catal12040358
 59. Ahmad W, Shah R, Ul Haq H, Khan D, Khan S, Al-Anazi A, et al. UV-activated and Fe²⁺-catalyzed H₂O₂ for the treatment of dye-contaminated water: kinetics, mechanism and toxicity investigations. *Catalysts* 2024;14(12):906. doi:10.3390/catal14120906
 60. Coleman HM, Vimonses V, Leslie G, Amal R. Degradation of 1,4-dioxane in water using TiO₂ based photocatalytic and H₂O₂/UV processes. *J Hazard Mater* 2007;146(3):496-501. doi:10.1016/j.jhazmat.2007.04.049
 61. Zyoud AH, Zyoud SH, Zyoud SH. Photodegradation of aqueous imidacloprid by CuS@TiO₂ under solar-simulated light: complete mineralization, catalyst efficiency, recovery, and reuse. *Appl Water Sci* 2025;15(7):159. doi:10.1007/s13201-025-02519-w
 62. Liu L, Gao ZY, Su XP, Chen X, Jiang L, Yao JM. Adsorption removal of dyes from single and binary solutions using a cellulose-based bioadsorbent. *ACS Sustain Chem Eng* 2015;3(3):432-42. doi:10.1021/sc500848m
 63. Khan MS, Khalid M, Ahmad MS, Shahid M, Ahmad M. Three-in-one is really better: exploring the sensing and adsorption properties in a newly designed metal-organic system incorporating a copper(II) ion. *Dalton Trans* 2019;48(34):12918-32. doi:10.1039/c9dt02578b
 64. Dariani RS, Esmaeili A, Mortezaali A, Dehghanpour S. Photocatalytic reaction and degradation of methylene blue on TiO₂ nano-sized particles. *Optik* 2016;127(18):7143-54. doi:10.1016/j.ijleo.2016.04.026
 65. Sudrajat H, Babel S. A novel visible light active N-doped ZnO for photocatalytic degradation of dyes. *J Water Process Eng* 2017;16:309-18. doi:10.1016/j.jwpe.2016.11.006
 66. Liu X, Yang Y, Shi X, Li K. Fast photocatalytic degradation of methylene blue dye using a low-power diode laser. *J Hazard Mater* 2015;283:267-75. doi:10.1016/j.jhazmat.2014.09.031
 67. Khan I, Saeed K, Zekker I, Zhang B, Hendi AH, Ahmad A, et al. Review on methylene blue: its properties, uses, toxicity and photodegradation. *Water* 2022;14(2):242. doi:10.3390/w14020242
 68. Zhou J, Li M, Tao Y, Zha L. Study on the adsorption characteristics of methylene blue by magnesium-modified fly ash. *Molecules* 2025;30(5):992. doi:10.3390/molecules30050992
 69. Kalaycıoğlu Z, Özüğür Uysal B, Pekcan Ö, Erim FB. Efficient photocatalytic degradation of methylene blue dye from aqueous solution with cerium oxide nanoparticles and graphene oxide-doped polyacrylamide. *ACS Omega* 2023;8(14):13004-15. doi:10.1021/acsomega.3c00198
 70. Maleki B, Khlaif TH, Jasim MK, Mansouri M. Preparation and utilization of Zn-La oxide nanocatalyst as a binary composite for photocatalytic degradation of methylene blue dye: optimization through RSM-BBD. *Arab J Chem* 2024;17(4):105667. doi:10.1016/j.arabjc.2024.105667
 71. Ahmad I, AlFaify S, Qasem Alfaifi M, M.Abduljawad M, Albaqi F, Alrashed YI, et al. A review of Fe₂O₃-based S-scheme heterojunction photocatalysts for pollutant removal. *J Ind Eng Chem* 2025;144:255-68. doi:10.1016/j.jiec.2024.10.018
 72. da Nóbrega Silva E, Brasileiro IL, Madeira VS, de Farias BA, Ramalho ML, Rodríguez-Aguado E, et al. Reusable CuFe₂O₄-Fe₂O₃ catalyst synthesis and application for the heterogeneous photo-Fenton degradation of methylene blue in visible light. *J Environ Chem Eng* 2020;8(5):104132. doi:10.1016/j.jece.2020.104132
 73. Ali MA, Maafa IM, Qudsieh IY. Photodegradation of methylene blue using a UV/H₂O₂ irradiation system. *Water* 2024;16(3):453. doi:10.3390/w16030453