

RESEARCH PAPER

ZnO/Fe₃O₄/Nitrogen doped Carbon Quantum Dots Nanocomposite for Efficient Visible-light Photocatalytic Removal of Methylene Blue and Acid Red 88

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ABSTRACT

Nitrogen-doped carbon quantum dots (N-CQDs) were incorporated onto ZnO/Fe₃O₄ to fabricate a magnetic nanocomposite for visible-light-driven photocatalytic degradation of methylene blue (MB) and acid red 88 (AR88). Surface modification with N-CQDs narrowed the band gap to 2.73 eV, improving visible-light absorption and charge transfer. The inclusion of Fe₃O₄ imparted superparamagnetic behavior, with a saturation magnetization of 91.14 emu g⁻¹, enabling easy magnetic separation and reuse. The prepared nanocomposite was evaluated under visible light for 120 min, achieving over 93% removal of MB and 90% removal of AR88 using 0.03 g and 0.05 g of catalyst, respectively. The material retained excellent activity across five consecutive cycles, demonstrating strong recyclability. Radical scavenging experiments identified hydroxyl radicals as the dominant reactive species governing dye degradation. These results indicate that N-CQD modification and magnetic integration synergistically enhance the photocatalytic efficiency, recovery, and operational stability of ZnO-based nanocomposites. The proposed ZnO/Fe₃O₄/N-CQDs system offers a promising, reusable photocatalyst for efficient treatment of dye-contaminated wastewater under visible-light irradiation.

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INTRODUCTION

Among the nanomaterials used for photo-sensitization of common photocatalysts (such as zinc oxide (ZnO) and titanium dioxide (TiO₂)), carbon quantum dots (CQDs) have recently received significant attention [1-3]. CQDs were first reported in 2004 and were identified as discreet and semi-spherical carbon nanoparticles with sizes below 10 nm [4, 5]. Owing to their *sp*² hybridization, quantum confinement effect, and abundant surface functional groups, CQDs exhibit remarkable electronic and optical properties

[6]. Accordingly, CQDs have unique electronic structure broad visible-light absorption, and efficient charge transfer capability, which justify their use in the visible-light sensitization of photocatalyst [7, 8]. It has been well established that CQDs act as an electron acceptors by capturing photo-generated electrons in metal oxide/CQDs systems [9, 10]. Consequently, the enhanced photocatalytic efficiency is expected as a results of the suppression in the recombination of electrons and holes [10].

Recently, metal oxide/CQDs photocatalytic

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systems have been widely studied for removal of organic pollutants from aqueous solution. For instance, M. Utami et al. studied ibuprofen degradation using CQDs/TiO₂. For this purpose, they synthesized CQDs from banana peels extract through a hydrothermal method [11]. Also, C. Wei et al. modified CdS-WO₃ by CQDs and studied its photo-activity to degrade norfloxacin and tetracycline [12].

Structural modification has been suggested to tailor the electronic properties of CQDs to attain further improvement in photocatalytic efficiency [9, 13, 14]. In this regard, CQDs doped with heteroatoms have been synthesized [15]. For example, Sulfur and nitrogen co-doped CQDs embedded on ZnO nanoflowers has been synthesized as a photocatalyst for degradation of antibiotics [16]. B. Liu et al. reported the photocatalytic removal of rhodanine B using nitrogen, sulfur, and boron doped CQDs/ZnO [17].

Herein, the synthesis and photocatalytic investigation of ZnO/Fe₃O₄/nitrogen doped CQDs nanocomposite were reported. The nitrogen doped CQDs (N-CQDs) were synthesized using hydrothermal reaction and simultaneously immobilized in situ onto the surface of ZnO/Fe₃O₄ composite. The Fe₃O₄ nanoparticles were used to impart magnetic character to the synthesized nanocomposite, which facilitates the separation of nanocomposite from the dye solution [18]. The photocatalytic experiments were performed for removal of two different nature organic dye. Methylene blue (MB) and acid red 88 (AR88) were used as cationic and anionic dye, respectively.

MATERIALS AND METHODS

Synthesis of nitrogen doped CQDs (N-CQDs)

The N-CQDs were synthesized by hydrothermal method. To this end, 0.2 mmol of urea and 1.0 mmol of citric acid were dissolved into 50 mL of deionized water. The mixture was stirred for 10 min and then poured into a Teflon lined stainless steel autoclave. The autoclave was put in an oven at temperature of 180 °C for 8 h.

Synthesis of ZnO nanoparticles

The ZnO nanoparticles were synthesized using precipitation method. To control the size of nanoparticles, polyethylene glycol (PEG) was used as a capping agent. The synthetic route was conducted as: 0.2 g of PEG (MW: 400) was dissolved in 100 mL of deionized water followed by

addition of specific amount of Zn(NO₃)₂. The pH of the solution was set to around 7 using addition of NaOH solution (5% W/W). After stirring for 30 min, the solid particles were separated and washed several times using deionized water. Finally, the solid was dried at 90 °C and calcined at 400 °C for 4 h.

Synthesis of Fe₃O₄ nanoparticles

1.0 mmol of FeCl₂·4H₂O and 2.0 mmol of FeCl₃·6H₂O were carefully weighed and dissolved in 100 mL deionized water containing 0.2 g of PEG (MW: 400) as capping agent. Then, NH₄OH solution (0.1 M) was added dropwise until reaching the pH of 7-8. The mixture was mechanically stirred for 30 min. Then, the black solid was separated and washed with deionized water to remove residual chloride ions and dried in an oven at 80 °C overnight.

Synthesis of ZnO/Fe₃O₄/N-CQDs nanocomposite

An in situ hydrothermal reaction was used to simultaneously synthesize N-CQDs and anchor them onto the surface of ZnO/Fe₃O₄ particles. Briefly, 1.0 g of as-prepared ZnO nanoparticles and 0.2 g of Fe₃O₄ nanoparticles were dispersed in 50 mL of deionized water and stirred for 1 h. Subsequently, the precursors for the synthesis of N-CQDs, prepared according to the procedure described in Section 2.1, were added to the suspension, which was then transferred into the autoclave. The temperature and reaction time were parallel to aforementioned conditions in 2.1. During the hydrothermal process, three components of the nanocomposite were well combined together, simultaneously with formation of N-CQDs nanoparticles. After completion of the hydrothermal reaction, the product were collected magnetically using a magnet, washed with deionized water, and finally dried at 80 °C for 3 h.

Photocatalytic tests

The photocatalytic experiments were carried out for removal of both methylene blue (MB) and acid red 88 (AR88) as cationic and anionic dye, respectively. The ZnO/Fe₃O₄/N-CQDs nanocomposite was activated under the visible light irradiation to perform the photocatalytic reaction. A common OSRAM lamp (100 W) was used for this intention. The MB and AR88 aqueous solution were prepared at constant concentrations

of 50 ppm. To determine the optimal conditions for maximum degradation efficiency, different parameters were investigated, including nanocomposite dosage, pH of dye solution, and addition of different radical scavenger. In each experiments, first the specific amount of the nanocomposite was added to the dye solution (50 mL, 50 ppm) under vigorous stirring for 20 min in the darkness. Then, the illumination was carried out to drive the photocatalytic reaction and removal level of dye solution was investigated at constant time interval (20 min). The absorbance of MB and AR88 was measured at their maximum absorption wavelengths of 664 and 505 nm.

Characterization

Structural characterization of the ZnO, N-CQDs, and ZnO/Fe₃O₄/N-CQDs samples was carried out using X-ray diffraction (XRD) analysis

(Rigaku Ultima IV). The morphology of the ZnO/Fe₃O₄/N-CQDs nanocomposite was studied using transmission electron microscope (TEM) (Philips EM 208S). Fourier transform infrared (FTIR) spectroscopy (FTIR 1720-X) was employed to identify the functional groups on the surface of the N-CQDs and ZnO/Fe₃O₄/N-CQDs samples. Optical properties of the samples were studied using diffuse reflectance spectroscopy (DRS) (S-4100 SCINCO). Magnetic behavior of ZnO/Fe₃O₄/N-CQDs nanocomposite was tested using vibrating sample magnetometer (VSM) (LBKFB).

RESULTS AND DISCUSSION

XRD

Fig. 1 shows the XRD patterns of the synthesized samples. As can be seen, the ZnO nanoparticles represent the diffraction planes around $2\theta = 31.8^\circ, 34.5^\circ, 36.3^\circ, 47.6^\circ, 56.7^\circ, 63.1^\circ, 66.5^\circ, 68.2^\circ$,

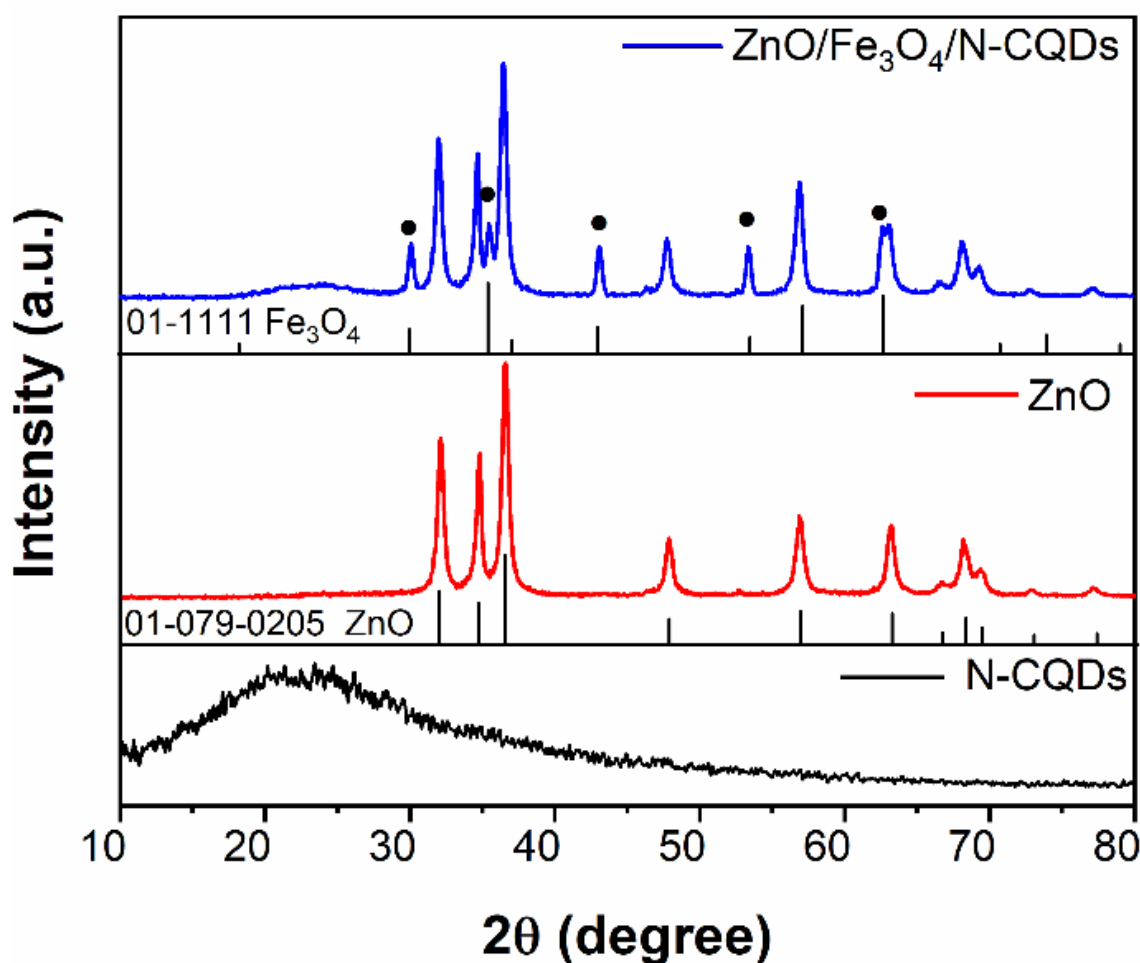


Fig. 1. XRD patterns of N-CQDs, ZnO, ZnO/Fe₃O₄/N-CQDs samples.

69.3°, 72.8°, and 77.2°. This diffraction pattern confirms the hexagonal structure (JCPDS file no. 01-079-0205) of the synthesized zinc oxide nanoparticles. The XRD pattern for the synthesized N-CQDs represents a broad peak around 26°, which is characteristic of carbon material (JCPDS file no. 02-0456) [19-21]. The ZnO/Fe₃O₄ nanocomposite shows the several reflections (marked with ●) which are assigned to (220), (311), (400), (422), and (440) diffraction planes of Fe₃O₄ phase (JCPDS file no. 01-1111). Also, no prominent peaks corresponding to the coated N-CQDs can be observed in the nanocomposite pattern due to their amorphous nature and low content. However, a weak and broad reflection can be detected around $2\theta = 26^\circ$, which is truly attributed to the presence N-CQDs.

FTIR

Chemical investigations of the synthesized samples were performed using FTIR spectroscopy. Fig. 2 represents the FTIR spectra for the pristine N-CQDs (red line) and N-CQDs coated on the surface of ZnO/Fe₃O₄ nanoparticles (blue line). The absorption bands for C=O (1718 cm⁻¹) [22], C=C (1500 cm⁻¹) [23], C-N (1404 cm⁻¹) [24], C-O (1210

cm⁻¹) [25] are clearly observed for the N-CQDs (red line). The presence of these oxygen- and nitrogen-containing functional groups is consistent with the successful synthesis of N-CQDs. Moreover, the FTIR spectrum for ZnO/Fe₃O₄/N-CQDs nanocomposite (blue line) also exhibits the stretching vibrations belonged to C=O and C-N, centered at 1743 and 1440 cm⁻¹, respectively, approving the presence of N-CQDs on the surface of the nanocomposite. In addition, the sample depicts the strong and broad absorption bands in the range of 600-400 cm⁻¹, which is attributed to the metal-oxygen bonds, including Zn-O at 484 cm⁻¹ and Fe-O at 606 cm⁻¹ [26]. Additionally, the broad absorption band around 3400 cm⁻¹ is attributed to the stretching vibration of O-H bonds [19, 27].

TEM

TEM image (Fig. 3) reveal quasi-spherical ZnO/Fe₃O₄ nanoparticles decorated with small dark spots, shown by red circles, which are attributed to the N-CQDs. The ZnO/Fe₃O₄ nanoparticles exhibit a size range of approximately 20-40 nm. The N-CQDs appear to be relatively well distributed over the surface of the ZnO/Fe₃O₄ nanoparticles, although some degree of particles aggregation can

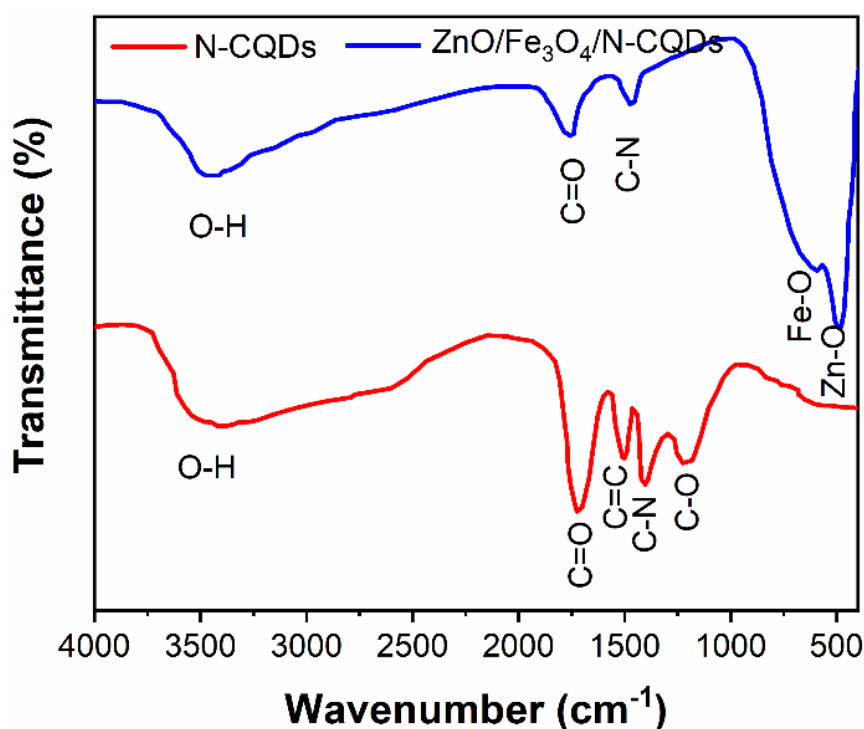


Fig. 2. FTIR spectra of ZnO/Fe₃O₄/N-CQDs (blue line) and N-CQDs (red line).

also be observed.

DRS

Fig. 4 shows the DRS spectra for the pure ZnO and ZnO/Fe₃O₄/N-CQDs nanocomposite. The pure ZnO nanoparticles exhibited negligible absorption in the visible light region (400–700 nm). The ZnO band gap was determined to be 3.24 eV using Tauc method [28]. According to this method, $(\alpha h\nu)^2$ was plotted as a function of photon energy $h\nu$ and then the linear portion of the plot was extrapolated to the energy axis [29, 30]. On the other hand, ZnO/Fe₃O₄/N-CQDs nanocomposite showed the enhanced visible light absorption. Moreover, its absorption edge shifted to the longer wavelengths, confirming a substantial reduction in the optical band gap for the ZnO/Fe₃O₄/N-CQDs. The band gap of the nanocomposite was estimated to be 2.73 eV, which means that the ZnO/Fe₃O₄/N-CQDs nanocomposite possesses the strong potential to drive the photocatalytic reactions under visible

light illumination.

VSM

The room temperature magnetic measurement is provided in Fig. 5, showing the superparamagnetic character with saturation magnetization of 91.14 emu/g [31]. This magnetic behavior facilitates the separation of photocatalyst from pollutant media using a simple magnetic field.

Photocatalytic tests

This work provides the comprehensive photocatalytic investigations for the ZnO/Fe₃O₄/N-CQDs nanocomposite. For this purpose, the photocatalytic reactions were conducted to degrade both the cationic and anionic dye solutions using the different amounts of ZnO/Fe₃O₄/N-CQDs nanocomposite.

Degradation of MB and AR88

Fig. 6 shows the photocatalytic degradation of

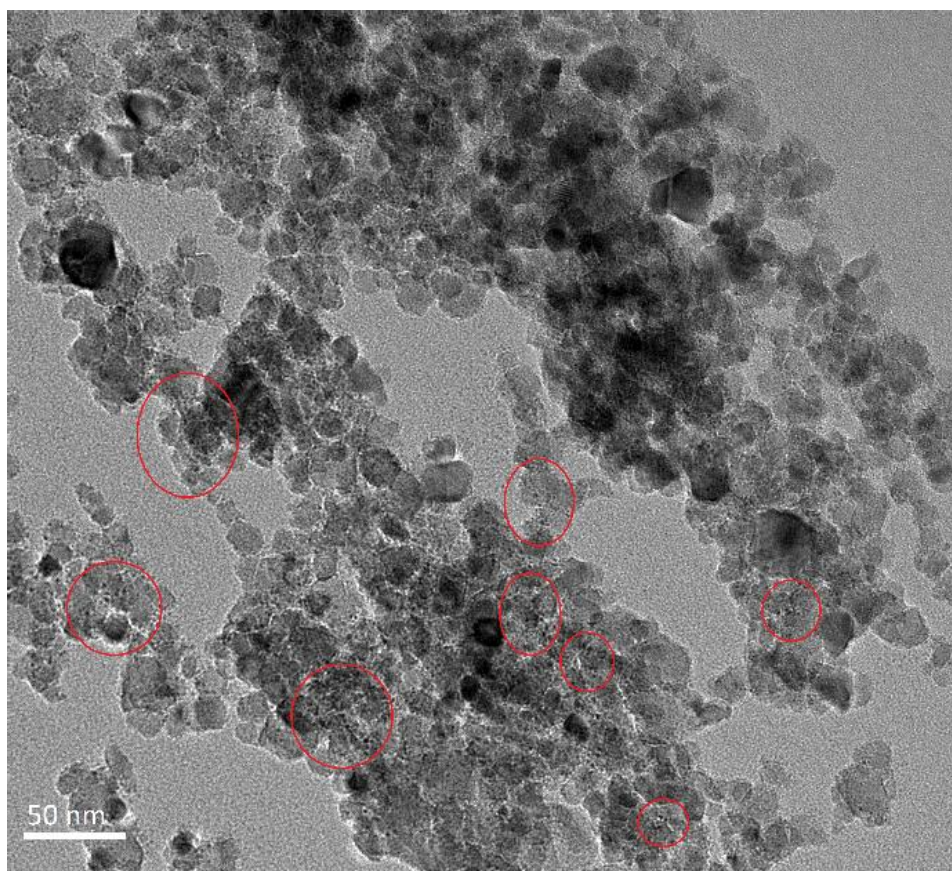


Fig. 3. TEM image of ZnO/Fe₃O₄/N-CQDs nanocomposite.

MB (cationic) and AR88 (anionic) solutions using the different loaded of the nanocomposite (0.01, 0.03, 0.05, 0.07 g). For the MB solution (Fig. 6a), the photocatalytic removal level peaked up to 93.55% using 0.03 g of the nanocomposite. By using 0.05 g of the nanocomposite, slight enhancement was attained in the degradation efficiency. However, excess in the nanocomposite amount to 0.07 g, the degradation efficiency of the MB solution was decreased to 73.91%. Fig. 6b represents the degradation efficiency of the AR88 solution under the same irradiation time. As seen, the degradation efficiency of AR88 gradually increased to reach the higher level of 90.82% using 0.05 g of the nanocomposite. This increasing trend was interrupted when 0.07 g of the nanocomposite was loaded. According to the Fig. 6, the adverse effect of the photocatalyst overloaded is observed at 0.07 g, which is attributed to the reduced light activation of the nanocomposite as the result of

hindered light transmittance through the dye solutions [32, 33].

pH effect

The MB and AR88 degradation reactions were carried out under different pH solutions (3, 5, 7, 9, 11), shown in Fig. 7. The results disclosed opposite pH-dependent photocatalytic behaviors for the nanocomposite toward MB and AR88. Fig. 7a shows that the acidic conditions (pH = 3 and 5) were more favorable for the degradation of AR88 than for that of MB [34]. The degradation level of AR88 approached 94.17% at the pH of 4. This result can be assigned to the anionic nature of AR88 solution meaning that AR88 adsorbs more on the photocatalyst surface carrying positive charges [34]. However, the MB degradation reaction (Fig. 7b) proceeded in opposite direction since the cationic molecules of MB tend to degrade on the negatively charged surface of the photocatalyst

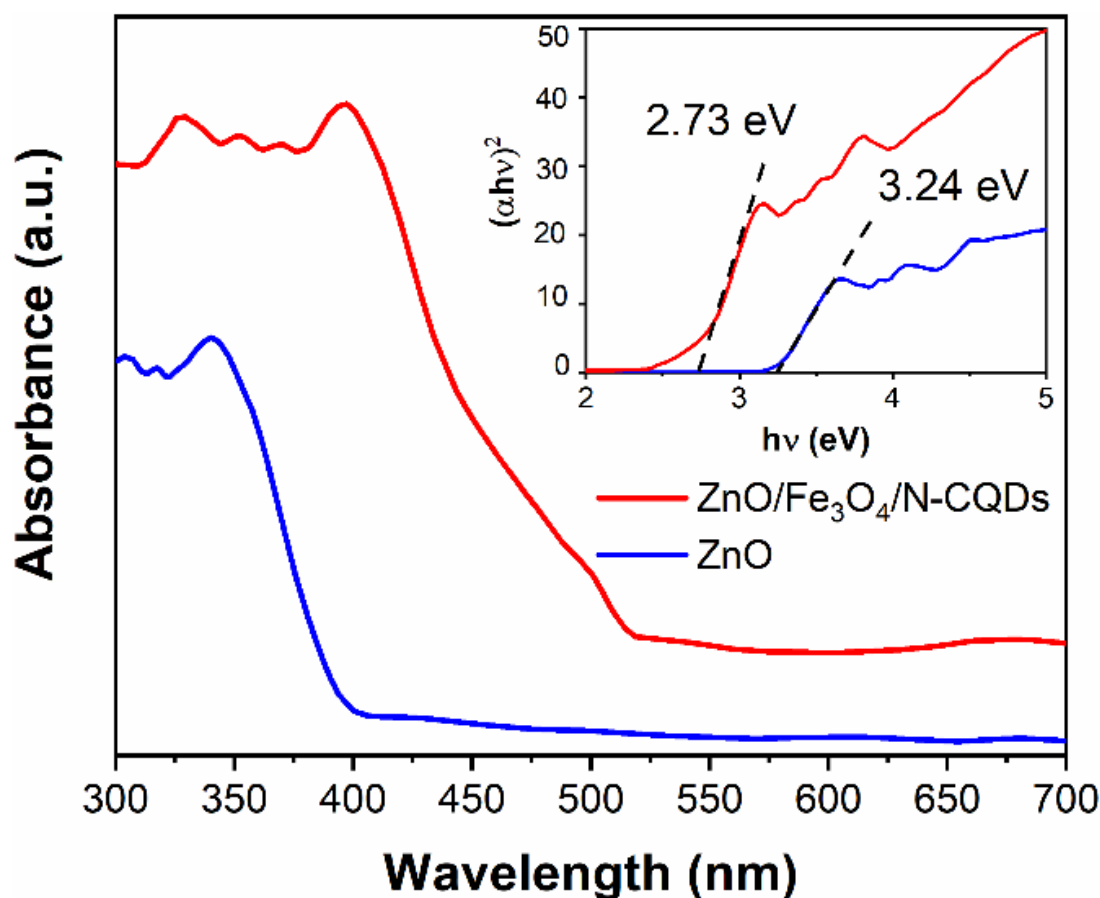


Fig. 4. UV/Vis spectra and Tauc curves for ZnO and ZnO/Fe₃O₄/N-CQDs nanocomposite.

[35]. So that, the photocatalytic removal of the MB increased to 96.07% at the pH 9. Additionally, the increased in pH of MB solution led to dramatic decrease in the photocatalytic efficiency.

Radical scavenger effect

The photocatalytic activity of the ZnO/Fe₃O₄/N-CQDs was also investigated using different radical scavenger agents, including

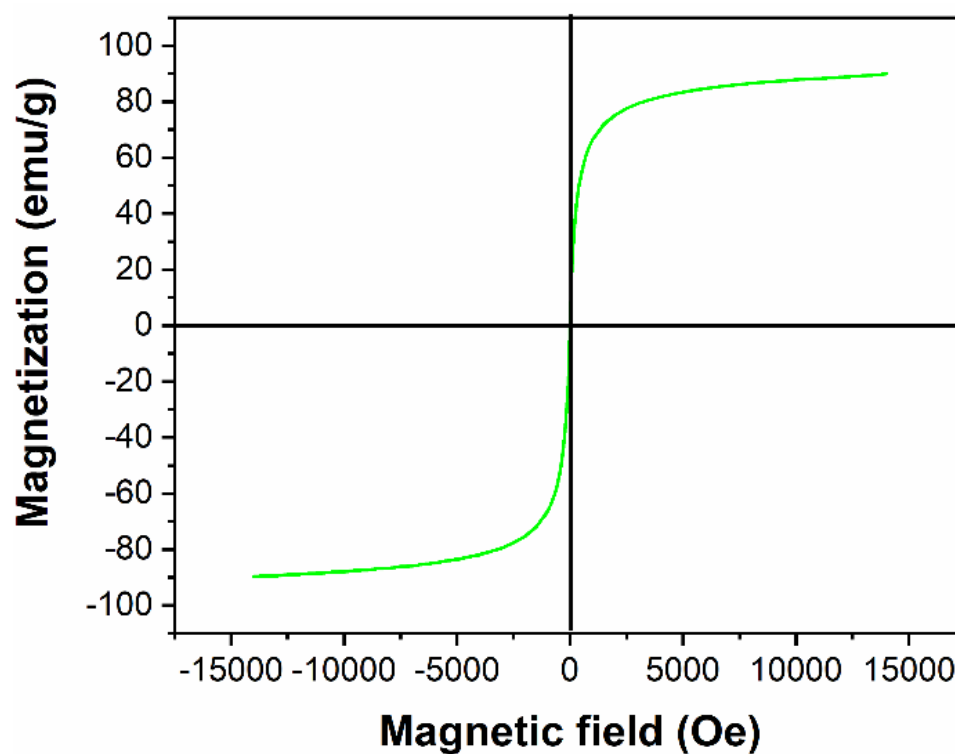


Fig. 5. *M-H* curve for the ZnO/Fe₃O₄/N-CQDs nanocomposite.

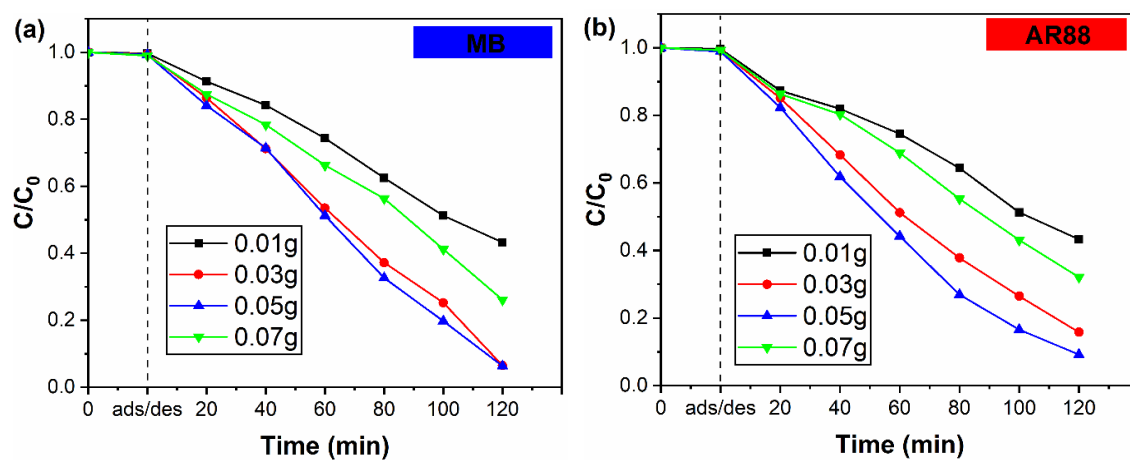


Fig. 6. Degradation of MB (a) and AR88 (b) using different loaded of ZnO/Fe₃O₄/N-CQDs nanocomposite.

ethylenediaminetetraacetic acid (EDTA) [36], isopropyl alcohol (IPA) [37], and ascorbic acid (AsA) [38] for trapping photo-generated holes, hydroxyl radicals, and superoxide radicals, respectively. Fig. 8 shows the photocatalytic degradation of MB and AR88 solution after addition of 1 mL of the scavengers. As shown, the degradation levels of both MB (Fig. 8a) and AR88 (Fig. 8b) significantly reduce by addition of IPA, suggesting that the oxidation process is likely triggered by the hydroxyl radicals.

Photocatalytic mechanism

Based on the results in Section 3.6.3, the

photocatalytic degradation mechanism is proposed, as follows (shown in Fig. 9): first, the ZnO/Fe₃O₄ nanoparticles is photo-excited by the light beam, generating the charge carriers (electrons and holes) are produced. The photo-generated electrons are transferred to the N-CQDs—serving as electron acceptor— which effectively suppresses the recombination of the charge carriers. Consequently, the charge carriers have the sufficient time to perform the redox reaction leading to the release highly reactive radicals. In this regard, the separated electrons react with dissolved oxygen to produce superoxide radicals ($\bullet\text{O}_2^-$), while the holes oxidize H_2O to

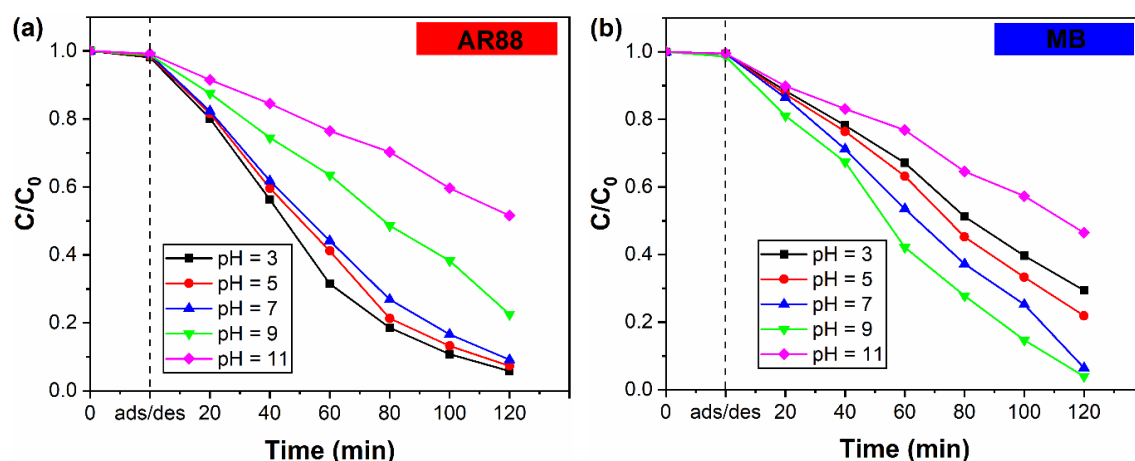


Fig. 7. Degradation of AR88 (a) and MB (b) in different pH conditions.

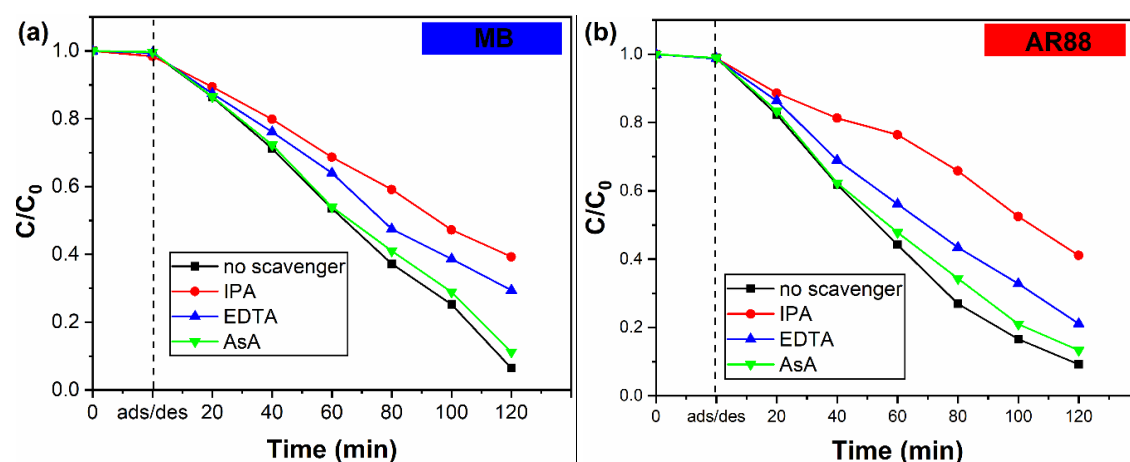


Fig. 8. Degradation of MB (a) and AR88 (b) in the presence of different radical scavengers.

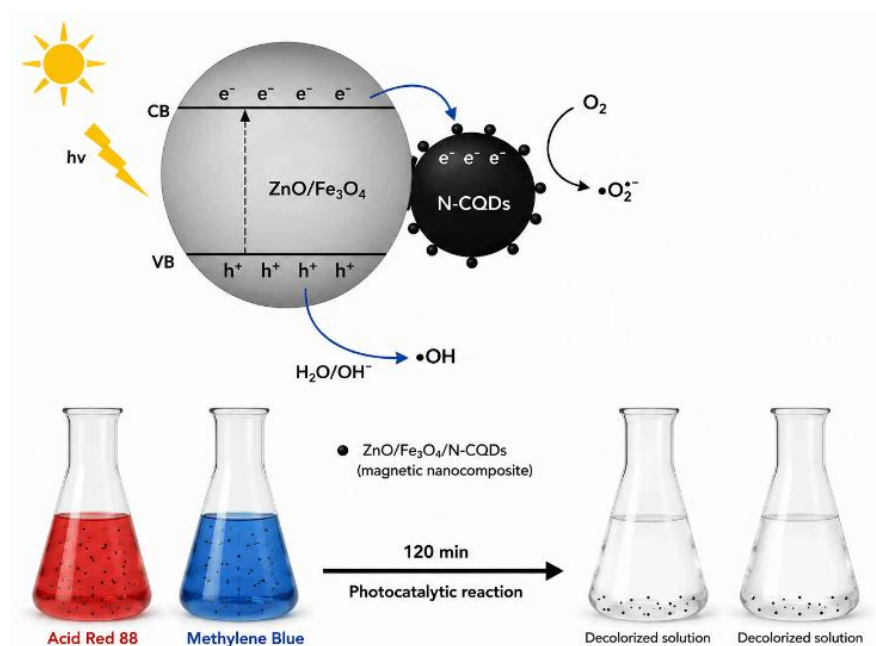


Fig. 9. Schematic representation of the suggested photocatalytic mechanism over ZnO/Fe₃O₄/N-CQDs nanocomposite.

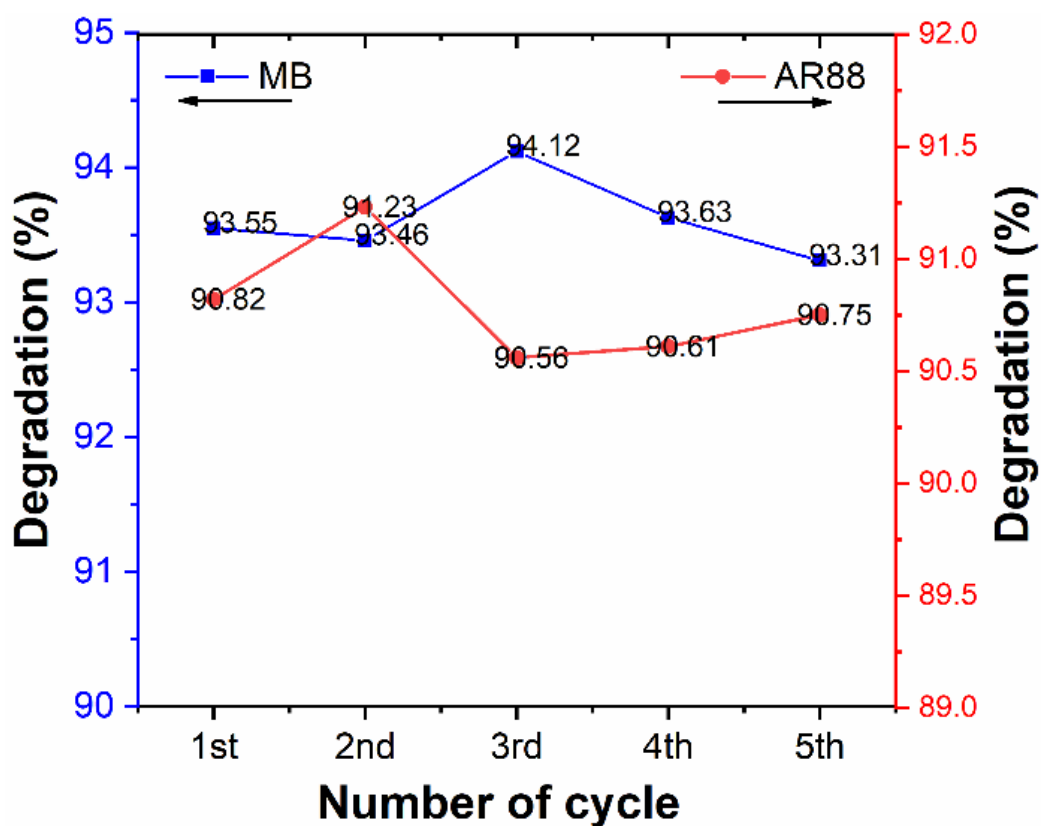


Fig. 10. Recyclability of the ZnO/Fe₃O₄/N-CQDs nanocomposite.

generate hydroxyl radicals ($\bullet\text{OH}$). Eventually, the MB and AR88 molecules are degraded by the radicals.

Reusability

The ZnO/Fe₃O₄/N-CQDs nanocomposite particles were recovered from both the MB and AR88 solutions using a magnet bar. The particles were washed using deionized water, dried at 80 °C and then reused for over 5 consecutive reaction cycles. Fig. 10 depicts that the ZnO/Fe₃O₄/N-CQDs nanocomposite retains its photocatalytic efficiency, with no serious decrease in the photo-efficiency.

CONCLUSION

In summary, the synthesis of ZnO/Fe₃O₄/N-CQDs nanocomposite was introduced to provide a prominent photocatalyst operating under visible light along with magnetic recoverability. The optical band gap was determined to be 2.73 eV, confirming the visible light photoactivity. Also, the saturation magnetization was 91.14 emu/g, which is sufficient for magnetic recovery the photocatalyst particles from pollutant media. The photocatalytic degradation was carried out over a 120-min reaction period under the visible light irradiation. The ZnO/Fe₃O₄/N-CQDs nanocomposite showed the photo-efficiency of 93% for methylene blue (MB) and 90% for acid red 88 (AR88). The pH effect was studied on the photo-efficiency of the nanocomposite, revealing that the improvements in degradation level were achieved for MB and AR88 solutions at pH of 9 and 3, respectively. In addition, the degradation levels of both dye solutions decreased after the addition of isopropanol as the radical scavenger, which confirmed the significant involvement of hydroxyl radicals in the degradation reactions. Furthermore, thanks to magnetic behavior, the nanocomposite particles were easily recovered from reaction medium and reuse for 5 successive reaction cycles without noticeable loss of the photo-efficiency.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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