

Article

Design and Multifunctional Performance of Zinc-Doped Magnesium Ferrite Nanostructures for Enhanced Electrochemical, Sensing and Photocatalytical Applications

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Abstract

In this study, zinc-doped magnesium ferrite ($\text{Zn}_x\text{-Mg}_{1-x}\text{Fe}_2\text{O}_4$) nanoparticles were synthesized using a facile combustion method and investigated for their electrochemical sensing and photocatalytic applications. The structural, morphological, and optical properties of the synthesized nanoparticles were characterized using X-ray diffraction (XRD), scanning electron microscopy (SEM), Energy-dispersive X-ray spectroscopy (EDAX), Fourier-transform infrared spectroscopy (FTIR), Energy band gap (Eg) and UV-Vis spectroscopy. The synthesized $\text{Zn-MgFe}_2\text{O}_4$ nanoparticles exhibited crystallite sizes ranging from 18.7 to 27.9 nm with an optical band gap of 1.86–1.89 eV. The catalyst achieved degradation efficiencies of 78% for Eriochrome Black T and 85% for Methyl Orange within 120 min, while the electrochemical sensor exhibited excellent linearity toward HgCl_2 detection ($R^2 = 0.99664$), demonstrating the multifunctional capability of the synthesized nanostructure. The synergistic effects of Zn doping contributed to enhanced electrical conductivity, catalytic activity, and structural stability. The novelty of this work lies in the development of combustion-synthesized $\text{Zn-MgFe}_2\text{O}_4$ nanoparticles as a multifunctional material capable of simultaneously achieving efficient photocatalytic degradation of organic dyes and sensitive electrochemical detection of mercury chloride using a simple and scalable synthesis route. These findings demonstrate that $\text{Zn-MgFe}_2\text{O}_4$ nanoparticles hold significant potential for integrated environmental remediation and electrochemical sensing applications.

Keywords: $\text{Zn-MgFe}_2\text{O}_4$; combustion method; electrochemical; sensor; photocatalytic



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1. Introduction

Spinel ferrites have garnered significant attention from researchers due to their unique structural evolution and advantageous physical properties. These materials exhibit remarkable electronic, magnetic, and catalytic characteristics, making them highly valuable for advanced technological applications, including sensors, energy storage, and environmental remediation [1,2]. The precise particle size and uniform distribution of ferrites further enhance their applicability in microwave devices and dielectric materials [3,4]. Among spinel ferrites, magnesium ferrite (MgFe_2O_4) has emerged as a promising candidate due to its high chemical stability, excellent electrical conductivity, and superior surface reactivity. However, the moderate electrical conductivity and limited active sites in pure MgFe_2O_4 necessitate further modifications to enhance its functional capabilities. One of the most

effective approaches to overcoming these limitations is doping with transition metal ions, which can significantly alter the physicochemical and electrochemical properties of the material [5–9].

Recent investigations have extensively explored Zn-substituted magnesium ferrite systems due to their tunable cation distribution and multifunctional behavior. For instance, Senthamilselvan et al. reported that Zn incorporation in MgFe_2O_4 significantly modifies structural, magnetic, and electrochemical properties, enhancing charge transport characteristics and redox activity [10]. Similarly, Zeng et al. demonstrated that Zn doping strongly influences cation redistribution in normal spinel MgFe_2O_4 , directly affecting its electronic structure and magnetic behavior [11]. In addition, Nigam et al. confirmed that Zn-doped MgFe_2O_4 nanoparticles exhibit enhanced surface reactivity and improved functional performance compared to the pristine ferrite phase [12]. The combustion method has emerged as a superior technique for synthesizing ferrite composites, offering several advantages over traditional methods such as sol–gel [13], coprecipitation [14], and hydrothermal synthesis [15]. This process involves an exothermic reaction between metal nitrates and fuels like urea or glycine, rapidly forming ferrite nanoparticles. Compared to conventional techniques, which often require longer processing times, higher energy inputs, and intricate procedural controls, the combustion method provides a more efficient pathway for producing high-quality ferrite nanoparticles. Its simplicity, cost-effectiveness, and ability to yield high-purity materials with controlled properties make it an ideal choice for synthesizing advanced ferrite composites [16,17]. Doping MgFe_2O_4 with zinc (Zn) has proven to be a highly effective strategy for enhancing its electrochemical and photocatalytic performance. Incorporating Zn^{2+} ions into the MgFe_2O_4 nanoparticle modifies the charge distribution, enhances carrier mobility, and facilitates efficient electron-hole separation [18]. These modifications significantly improve the material's electrochemical sensing capabilities, making it highly effective for detecting environmental and biological analytes with high sensitivity and selectivity. Additionally, Zn-doped MgFe_2O_4 exhibits superior photocatalytic efficiency, enabling the degradation of organic pollutants under visible-light irradiation. This advancement contributes to sustainable environmental remediation by promoting efficient pollutant degradation and waste management [19,20].

Recent studies have demonstrated that Zn-doped MgFe_2O_4 nanoparticles exhibit enhanced electrochemical performance, making them suitable for detecting environmental and biological analytes with high sensitivity and selectivity. Additionally, their photocatalytic properties have effectively degraded organic dyes and pollutants under visible-light irradiation, contributing to sustainable environmental remediation efforts. Zn substitution in MgFe_2O_4 enhances charge carrier mobility and interfacial electron transfer, making it suitable for electrochemical sensing applications. Photocatalysis was selected as a sustainable remediation strategy due to its ability to mineralize pollutants through reactive oxygen species generation, unlike conventional methods such as adsorption that only transfer contaminants between phases. Methyl Orange (MO) and Eriochrome Black T (EBT) were chosen as representative azo dyes because of their environmental relevance, structural stability, and resistance to biodegradation. Binary metal oxides such as TiO_2 , ZnO , Fe_2O_3 , and CuO have been extensively investigated as photocatalysts because of their chemical stability and catalytic activity. Nevertheless, many binary oxides suffer from rapid electron–hole recombination, limited visible-light utilization, and relatively poor electrical conductivity, which restrict their photocatalytic efficiency. In contrast, ternary spinel ferrites provide improved charge transport, tunable electronic structures through cation substitution, enhanced magnetic recoverability, and superior chemical stability. Among these materials, Zn-substituted MgFe_2O_4 has attracted considerable attention because Zn

incorporation modifies the cation distribution within the spinel lattice, thereby improving charge separation and electrochemical activity [21–24].

Recent studies have demonstrated that Zn-substituted spinel ferrites exhibit remarkable electrochemical and photocatalytic performances. For example, photocatalytic degradation efficiencies exceeding 80–95% have been reported for azo dyes under UV or visible irradiation, while ferrite-based electrochemical sensors have achieved excellent linearity and high sensitivity toward toxic heavy-metal ions. Nevertheless, simultaneous optimization of photocatalytic degradation and electrochemical sensing using combustion-synthesized Zn-doped MgFe_2O_4 remains scarcely investigated, motivating the present study. This study aims to synthesize and characterize Zn-doped MgFe_2O_4 nanoparticles and evaluate their electrochemical and photocatalytic properties. The investigation focuses on understanding the role of Zn doping in modifying the structural, optical, and electronic properties of MgFe_2O_4 , leading to enhanced functional performance. The findings of this research contribute to the ongoing advancements in nanostructured materials for energy and environmental applications.

2. Results and Discussion

2.1. X-Ray Diffraction (XRD) Analysis

The ferrite material ($\text{Zn-MgFe}_2\text{O}_4$) is depicted in the XRD pattern, and the peak positions in Figure 1A confirm that ferrites often assume a cubic spinel structure. The normal spinel cubic structure is indicated by the indexed peaks (220), (311), (222), (422), (511), (440), and (533) which are located at 30.15° , 35.80° , 43.30° , 53.86° , 57.30° , 63.63° , and 74.13° , respectively (Table 1). Since it is the most prominent reflection plane in this material, the (311) peak stands out as the most intense. This indicates good crystallinity and is typical of spinel ferrites. The existence of strong and well-defined peaks suggests a high degree of crystallinity. The absence of any additional peaks indicates that the sample is pure $\text{Zn-MgFe}_2\text{O}_4$ and devoid of impurity phases [25]. Microstrain or tiny crystallite size could be shown by small peak broadening. In addition to peak indexing, the lattice parameter (a) was estimated using the interplanar spacing values derived from Bragg's law, confirming structural consistency with the cubic spinel framework. The calculated lattice parameter falls within the typical range reported for Zn-substituted MgFe_2O_4 systems, indicating that Zn^{2+} ions are accommodated within the spinel lattice rather than forming segregated phases. The systematic preservation of peak symmetry and the absence of peak splitting further support structural homogeneity. Moreover, the agreement between the calculated d-spacing values and those observed in the SAED pattern strengthens the crystallographic validation and confirms the formation of a well-defined nanocrystalline spinel phase.

Table 1. XRD Parameters of $\text{Zn-MgFe}_2\text{O}_4$ nanoparticles.

Peak Index (hkl)	2θ (Degrees)	θ (Dadians)	D-Spacing (Å)	Estimated FWHM, β (Degrees)	β (Dadians)	Crystallite Size (nm)
(220)	30.15	0.262	2.98	0.45	0.00785	18.7
(311)	35.80	0.310	2.52	0.40	0.00698	20.8
(222)	43.30	0.327	2.40	0.38	0.00663	19.8
(422)	54.06	0.471	1.70	0.35	0.00611	22.0
(511)	57.50	0.502	1.60	0.33	0.00576	23.3
(440)	63.63	0.550	1.47	0.30	0.00524	24.7
(533)	74.13	0.654	1.27	0.28	0.00489	27.9

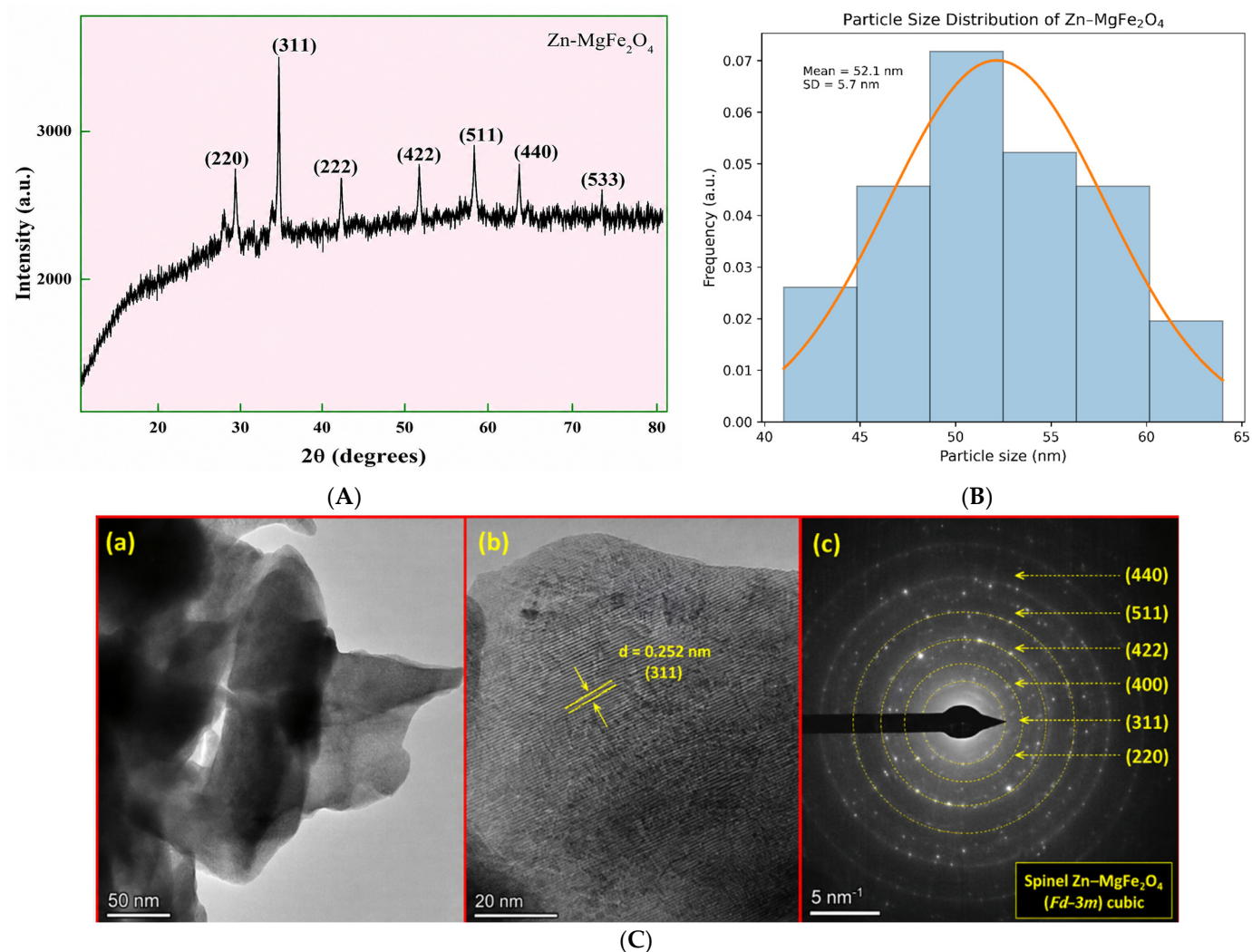


Figure 1. (A) XRD pattern, (B) particle size distribution histogram, and (C) TEM image of the synthesized Zn-MgFe₂O₄ nanoparticles. (a) Low-magnification TEM image. (b) High-resolution TEM (HRTEM) image. (c) Selected area electron diffraction (SAED) pattern.

To estimate the crystallite size of Zn-MgFe₂O₄ from the XRD pattern, we use the Scherrer Equation (1) [26]:

$$D = K \lambda / \beta \cos \theta \quad (1)$$

where D = Crystallite size (nm), K = Scherrer constant (typically 0.9 for spherical particles), λ = X-ray wavelength (for Cu-K α , λ = 0.154 nm), β = Full width at half maximum (FWHM) of the peak in radians, θ = Bragg's angle.

The Zn-MgFe₂O₄ ferrite nanoparticles are likely nanocrystalline, as their estimated crystallite size (D) is approximately 21 nm. Spinel ferrites, such as Zn-MgFe₂O₄, find extensive application in the fields of catalysis, magnetic materials, and sensors. The XRD pattern helps to confirm the formation of the desired phase and provides structural insights. Although the Zn atomic percentage is relatively high, no additional diffraction peaks corresponding to ZnO or other zinc-containing secondary phases were detected in the XRD pattern, indicating that Zn²⁺ ions are incorporated into the MgFe₂O₄ spinel lattice rather than forming segregated phases. In spinel ferrites, Zn²⁺ ions preferentially occupy tetrahedral (A) sites and can be accommodated within a wide compositional range through cation redistribution without structural phase separation. Similar behavior has been reported for Zn-substituted MgFe₂O₄ systems, where Zn incorporation modifies lattice

parameters and electronic structure while preserving the cubic spinel framework [27,28]. Therefore, the structural evidence supports that Zn acts as a dopant within the spinel lattice rather than forming a secondary phase.

2.2. Transmission Electron Microscopy (TEM)

The TEM investigation is illustrated in Figure 1B,C. Figure 1B shows the particle size distribution histogram, and Figure 1C shows the TEM image of the synthesized Zn-MgFe₂O₄ nanoparticles. In Figure 1C the low-magnification TEM image reveals agglomerated clusters of nanoparticles exhibiting irregular, plate-like morphology with interconnected structures that form a porous network, likely due to magnetic interactions and high surface energy at the nanoscale. The particles are generally within the size range of 30–70 nm [29]. Figure 1C shows a high-resolution TEM (HRTEM) image and displays clear lattice fringes that confirm the crystalline nature of the nanoparticles, with interplanar spacings consistent with the spinel ferrite structure, suggesting successful zinc incorporation without disrupting the host lattice. Figure 1C shows the selected area electron diffraction (SAED) pattern, where well-defined concentric rings and bright diffraction spots indicate a polycrystalline nature, further affirming the formation of highly crystalline Zn-MgFe₂O₄ with a spinel phase.

Collectively, the TEM observations confirm that the synthesized nanoparticles possess nanoscale dimensions, crystallinity, and a polycrystalline spinel structure with a tendency to agglomerate. The larger particle size observed by TEM compared with the crystallite size estimated from XRD is expected because individual particles are composed of multiple coherent crystallites that become agglomerated during the combustion process owing to their high surface energy and magnetic interactions.

2.3. Morphological Studies

The surface morphology of the synthesized Zn-MgFe₂O₄ (1:1) ferrite nanoparticles was analyzed using SEM. Figure 2a presents high- and low-magnification images, indicating that the sample comprises flakes with a non-uniform structure, without any visible pores or voids. Additionally, EDX analysis of the spinel nanoparticles (Zn_x-Mg_{1-x}Fe₂O₄) is displayed in Figure 2b. The proportions of Mg, Zn, Fe, and O in terms of elemental composition are shown in the table provided (Figure 2b). The presence of Mg, Fe, Zn, and O was validated, with weight percentages of 10.71%, 38.44%, 27.61% and 21.10%, respectively.

The irregular nanosheet-like morphology observed by SEM provides numerous exposed active sites and short diffusion pathways for photogenerated charge carriers. This morphology facilitates adsorption of dye molecules and enhances interfacial electron transfer, thereby improving photocatalytic degradation efficiency. Likewise, the increased electrochemically active surface area enhances the interaction between the electrode surface and mercury ions, resulting in improved sensing performance.

2.4. Fourier-Transform Infrared (FTIR) Studies

Figure 2c presents the FT-IR spectrum of the Zn²⁺-substituted MgFe₂O₄ sample, recorded within the wavenumber range of 4000–400 cm^{−1} [30]. The broad absorption band observed at 3455.36 cm^{−1} corresponds to the stretching vibrations of hydroxyl (O–H) groups, indicating the presence of adsorbed water molecules or surface hydroxyl species [31–33]. The peak at 2358.04 cm^{−1} is attributed to CO₂ molecules, likely originating from atmospheric contamination or surface-adsorbed carbonates. The band at 1631.32 cm^{−1} is associated with the bending vibrations of water molecules (H–O–H bending mode), further confirming the presence of moisture within the sample. The absorption peak at 1507.18 cm^{−1} may be linked to organic impurities or carboxylate functional group

vibrations. The characteristic absorption band at 440.67 cm^{-1} corresponds to metal-oxygen (M–O) stretching vibrations, specifically associated with Fe–O bonds within the spinel ferrite framework of $\text{Zn-MgFe}_2\text{O}_4$. The FT-IR analysis validates the successful synthesis of $\text{Zn-MgFe}_2\text{O}_4$, confirming the presence of the characteristic spinel ferrite structure. It is important to note that manganese is not part of the present composition; therefore, Mn–O vibrations are not applicable to this system. The detected absorption band in the low-frequency region is attributed to lattice metal–oxygen vibrations within the spinel structure. The absence of additional bands typically associated with segregated ZnO phases indicates that Zn^{2+} ions are structurally incorporated into the MgFe_2O_4 framework rather than forming a separate zinc oxide phase. Compared with previously reported FTIR spectra of pristine MgFe_2O_4 , the characteristic metal–oxygen vibration in the present Zn-substituted sample exhibits a slight shift toward lower wavenumbers, which can be attributed to the incorporation of Zn^{2+} ions into the spinel lattice. Such spectral shifts have been widely reported for Zn-substituted magnesium ferrites and reflect changes in bond length and cation distribution within tetrahedral and octahedral sites.

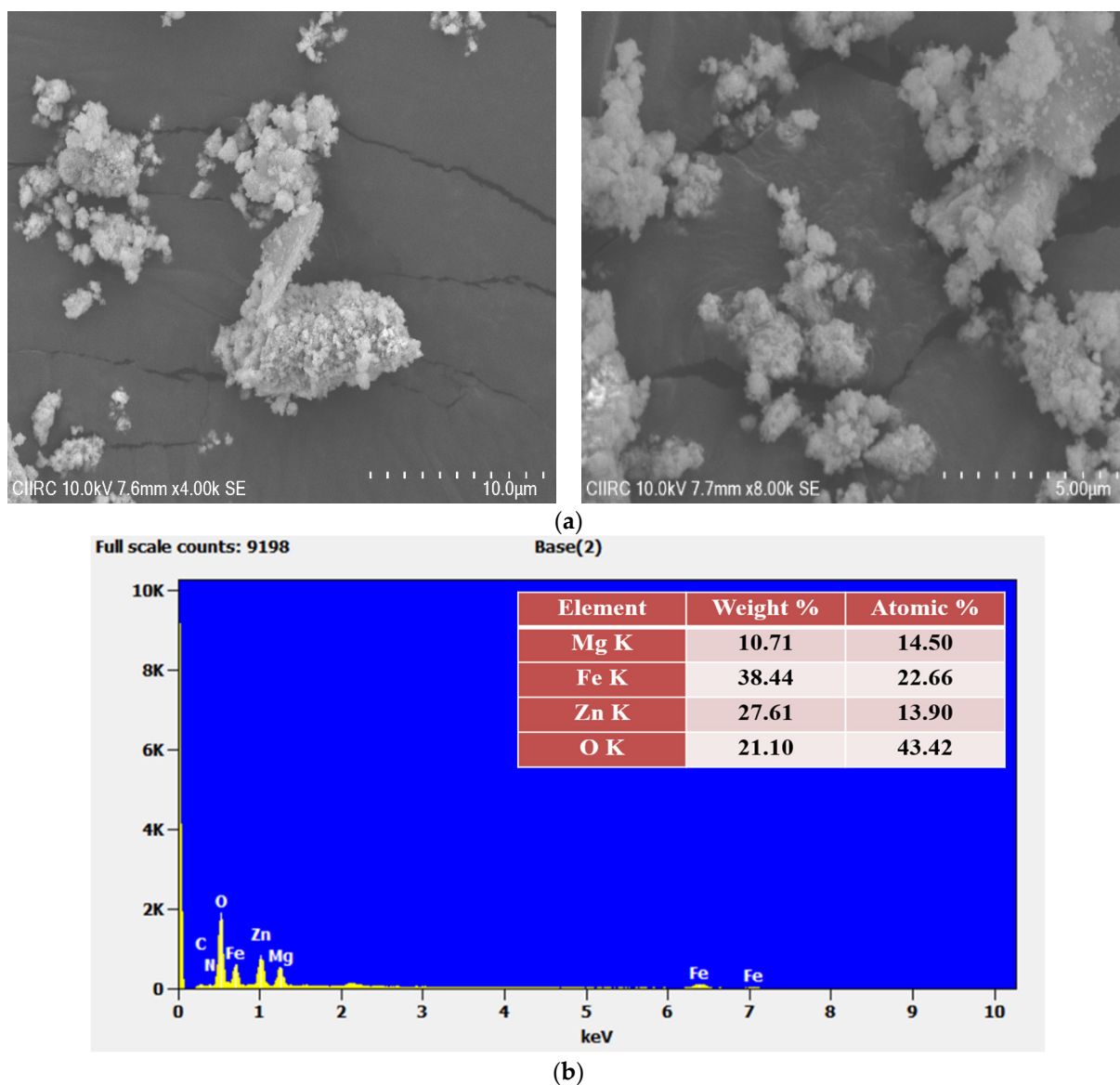


Figure 2. Cont.

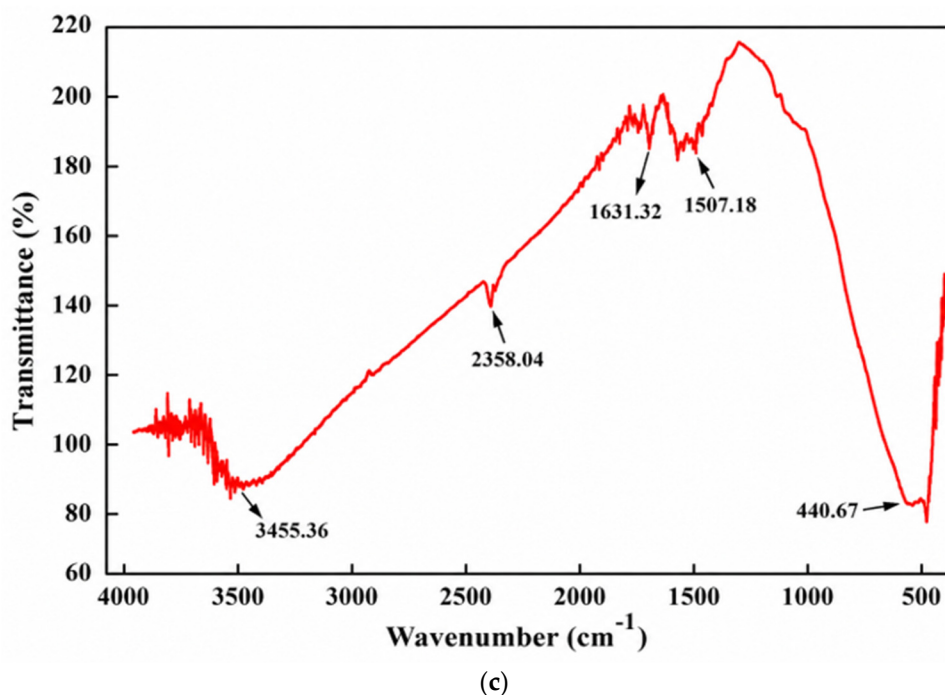


Figure 2. (a) SEM micrograph and (b) EDX spectrum, and (c) FTIR spectrum of Zn-MgFe₂O₄ nanoparticles.

3. Electrochemical Studies

3.1. Cyclic Voltammetry (CV) Analysis

Figure 3a depicts the CV curves of Zn-MgFe₂O₄ nanoparticles recorded at varying scan rates (10–50 mV/s) in a suitable electrolyte. The CV curves exhibit redox peaks, indicating the material's pseudocapacitive behavior and electrochemical activity. Notably, as the scan rate increases, the anodic and cathodic peak currents increase proportionally, suggesting a surface-controlled electrochemical process. The symmetric nature of the redox peaks and their consistent shift with increasing scan rate indicate a fast electron transfer mechanism and excellent reversibility of the redox reactions at the electrode surface [34–37].

Figure 3b shows the Nyquist plot obtained from electrochemical impedance spectroscopy (EIS) measurements. The plot consists of a linear segment with a steep slope, characteristic of a diffusion-controlled process. The absence of a prominent semicircular arc in the high-frequency region implies low charge transfer resistance (R_{ct}), further confirming the efficient electron mobility across the electrode–electrolyte interface [38–40]. The linearity in the low-frequency region demonstrates favorable ionic diffusion behavior, reinforcing the suitability of Zn-MgFe₂O₄ nanoparticles as an efficient material for electrochemical applications such as sensing or energy storage. Together, these electrochemical findings demonstrate the excellent conductivity, rapid charge transfer kinetics, and electrochemical stability of the Zn-MgFe₂O₄ nanoparticles, highlighting their potential in various electrochemical and environmental applications.

3.2. Electrochemical Sensor Studies

By employing the Zn-MgFe₂O₄ electrode, the CV measurement successfully detected mercury chloride. Figure 3c illustrates the CV response of the Zn-MgFe₂O₄-based sensor toward varying concentrations (1–5 mM) of mercury chloride in an electrochemical cell. A well-defined oxidation peak is observed, with current intensity progressively increasing as the mercury chloride concentration rises. This behavior indicates a strong electrocatalytic interaction between the analyte and the active surface of the modified electrode, facilitating

electron transfer. The consistent increase in peak current with concentration suggests a concentration-dependent electrochemical process and confirms the sensor's responsiveness to mercury chloride.

Figure 3d presents the calibration curve plotted between the peak current (i_p) and mercury chloride concentration, revealing a linear relationship within the studied range. The linear regression analysis yields a high correlation coefficient ($R^2 = 0.99664$), indicative of excellent linearity and sensor sensitivity. The slope and intercept values, as shown in Figure 3c, further validate the linear response and reproducibility of the sensor. The combined electrochemical data demonstrate that the Zn-MgFe₂O₄ nanomaterial exhibits high electrocatalytic activity and sensitivity toward mercury chloride detection. The pronounced reaction at low concentrations and excellent linearity suggest its suitability for practical sensing applications, particularly in environmental monitoring of mercury-based contaminants.

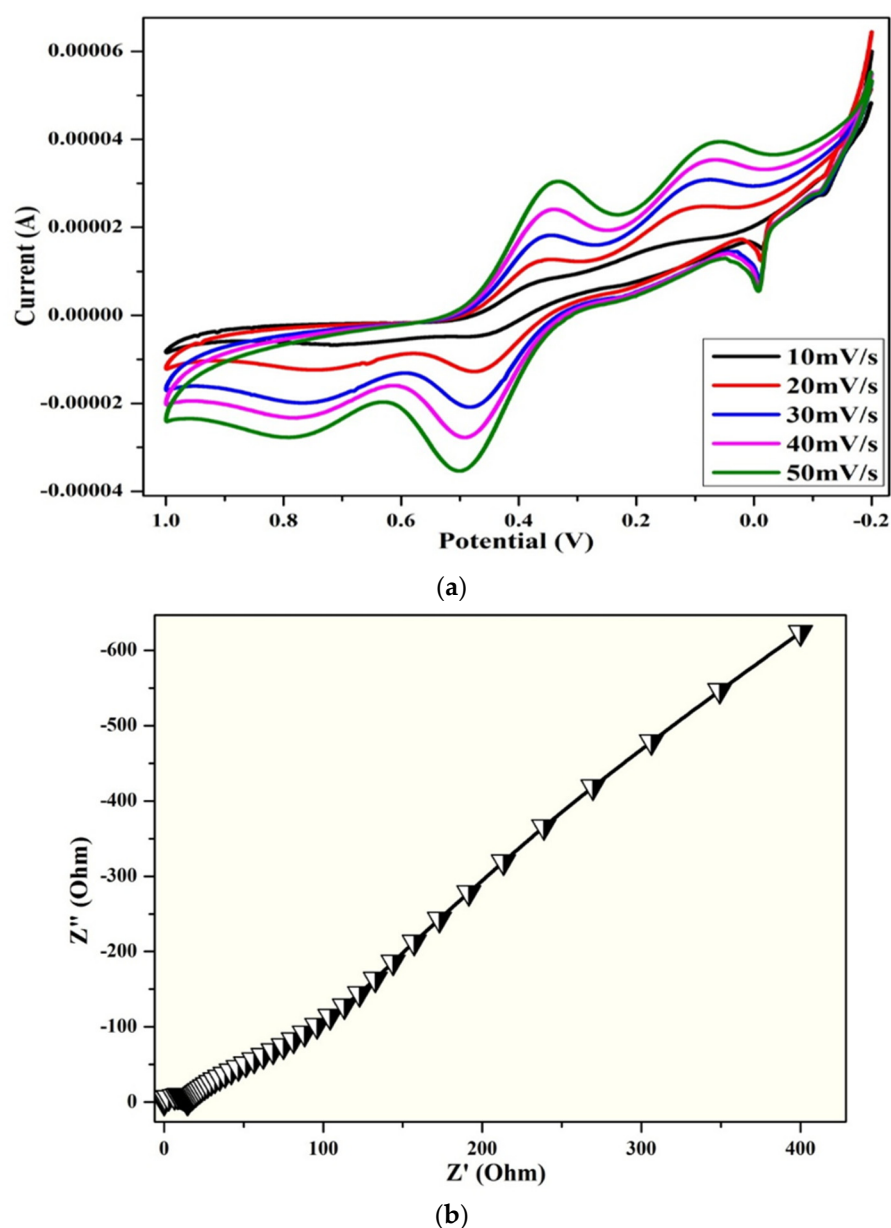


Figure 3. Cont.

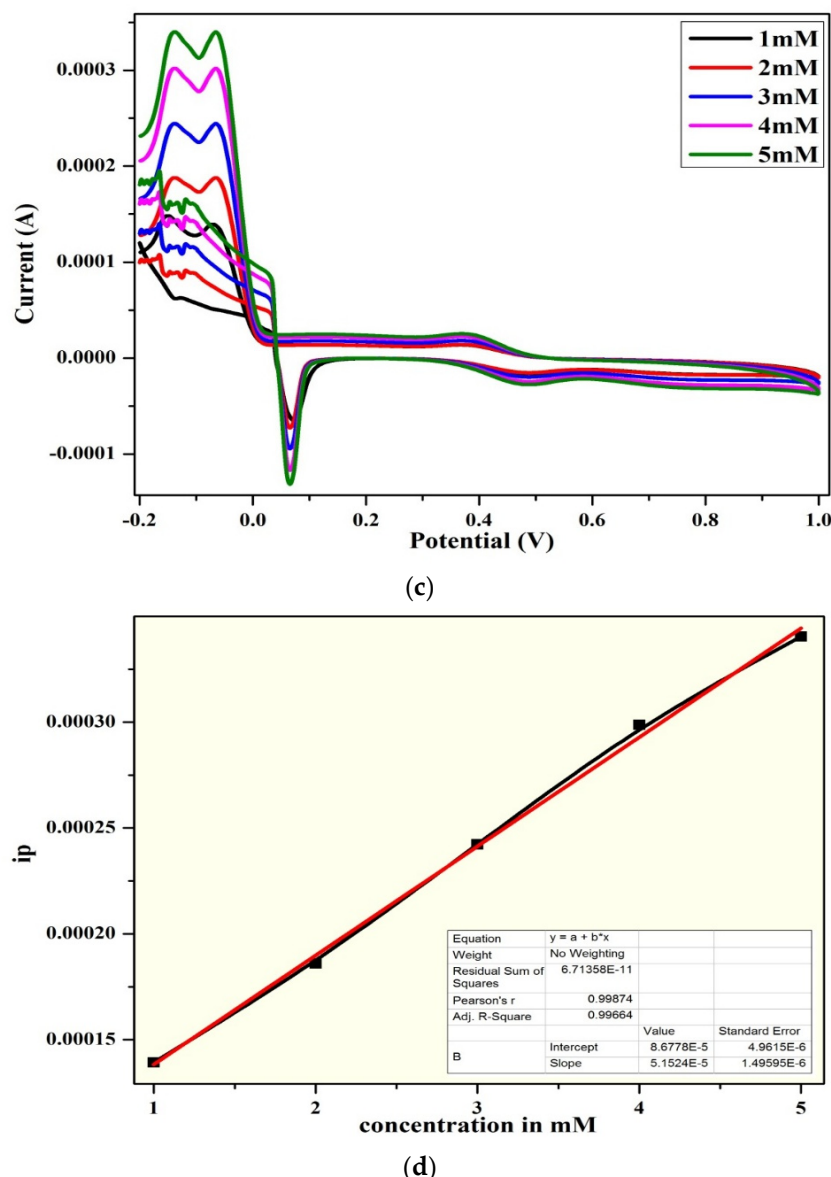


Figure 3. (a) Cyclic voltammograms recorded at different scan rates and (b) Nyquist impedance plot. (c) Cyclic voltametric and (d) corresponding calibration curve.

To further substantiate the electrochemical findings, a comparative analysis with previously reported ferrite-based sensing systems has been incorporated. The observed increase in anodic and cathodic peak currents with scan rate confirms a surface-controlled redox process and fast electron transfer kinetics, which are characteristic of well-structured spinel ferrites with improved conductivity. Similar behavior has been reported for Zn-doped MgFe_2O_4 and related ferrite nanostructures, where Zn substitution enhanced charge carrier mobility through cation redistribution between tetrahedral and octahedral sites [41]. Additionally, the low charge transfer resistance inferred from the Nyquist plot is comparable to values reported for ferrite-based electrochemical sensors targeting toxic ions, where improved interfacial charge transfer was directly linked to nanoscale crystallinity and optimized surface states [42]. Therefore, the enhanced sensing response toward mercury chloride in the present study can be attributed to the synergistic effect of nanoscale particle size, Zn-induced electronic modulation, and improved electrode–electrolyte interaction. Although the Nyquist plot qualitatively demonstrates low charge-transfer resistance, quantitative determination of R_s and R_{ct} through equivalent-circuit fitting was beyond the scope of the present study and will be addressed in future investigations using

complete impedance datasets. The electrochemical sensing parameters of Zn-MgFe₂O₄ electrode have been illustrated in Table 2. Although the sensor exhibited excellent linearity ($R^2 = 0.99664$), determination of the limit of detection (LOD) requires calibration measurements at significantly lower analyte concentrations and replicate blank measurements, which were beyond the scope of the present study.

Table 2. The electrochemical sensing parameters of Zn-MgFe₂O₄ electrode.

Parameter	Value
Linear range	1–5 mM
Correlation coefficient	0.99664
Electrolyte	0.1 M HCl
Electrode	Zn-MgFe ₂ O ₄

4. Photocatalytic Degradation

The photocatalytic decoloration of Eriochrome Black T (EBT) and Methyl Orange (MO) dye was performed for prepared Zn-MgFe₂O₄ nanoparticles under UV light irradiation. In the photocatalytic experiments, the specific amounts of optimized prepared photocatalysts (40 mg) were added to a 20 ppm EBT and MO dye solution. These experiments were conducted using a glass reactor and continuous stirring with the help of a magnetic stirrer. During the reaction, 5 mL of dye solution was taken at different intervals and subjected to centrifugation to separate the photocatalyst [43]. Absorbance measurements were carried out at the maximum wavelength (λ_{max}) of EBT, which was 535 nm (Figure 4a).

As shown in Figure 4b, the degradation of the MO solution was selected as a reference, and the characteristic absorption of the MO solution at about 460 nm (Figure 4b) was chosen for monitoring the adsorption and photocatalytic degradation process. A significant decrease in the λ_{max} of about 460 nm can be assigned to light absorption caused by the excitation of electrons from the valence band to the conduction band of MO dye solution [44,45].

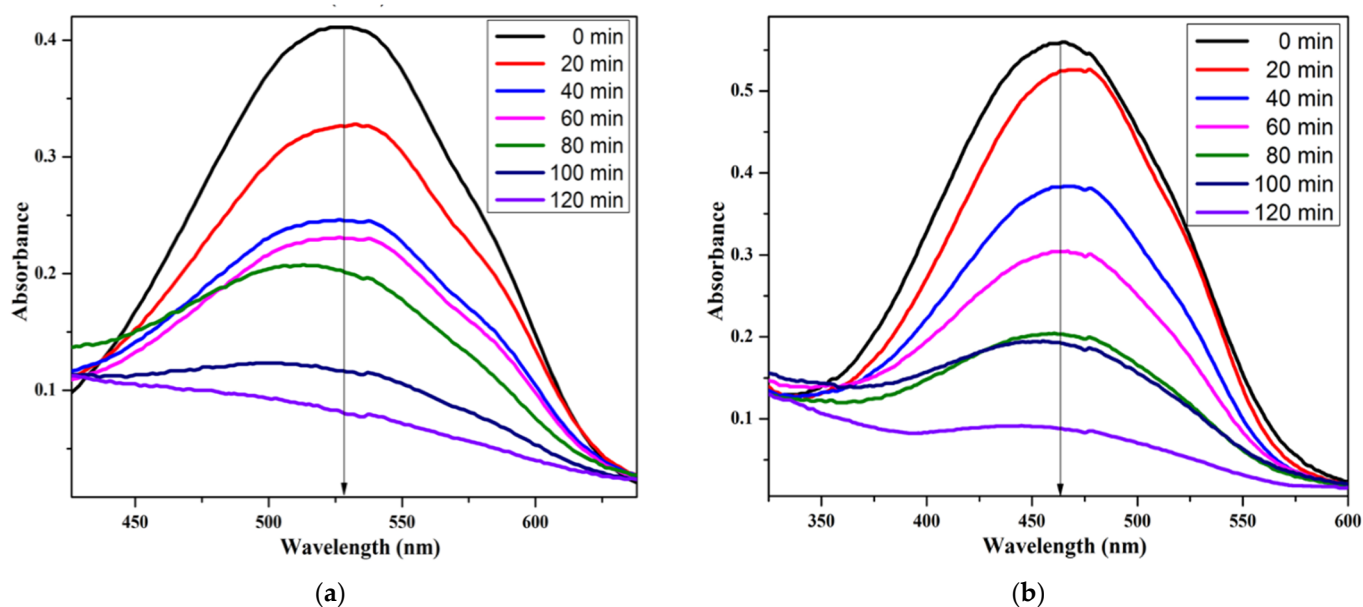


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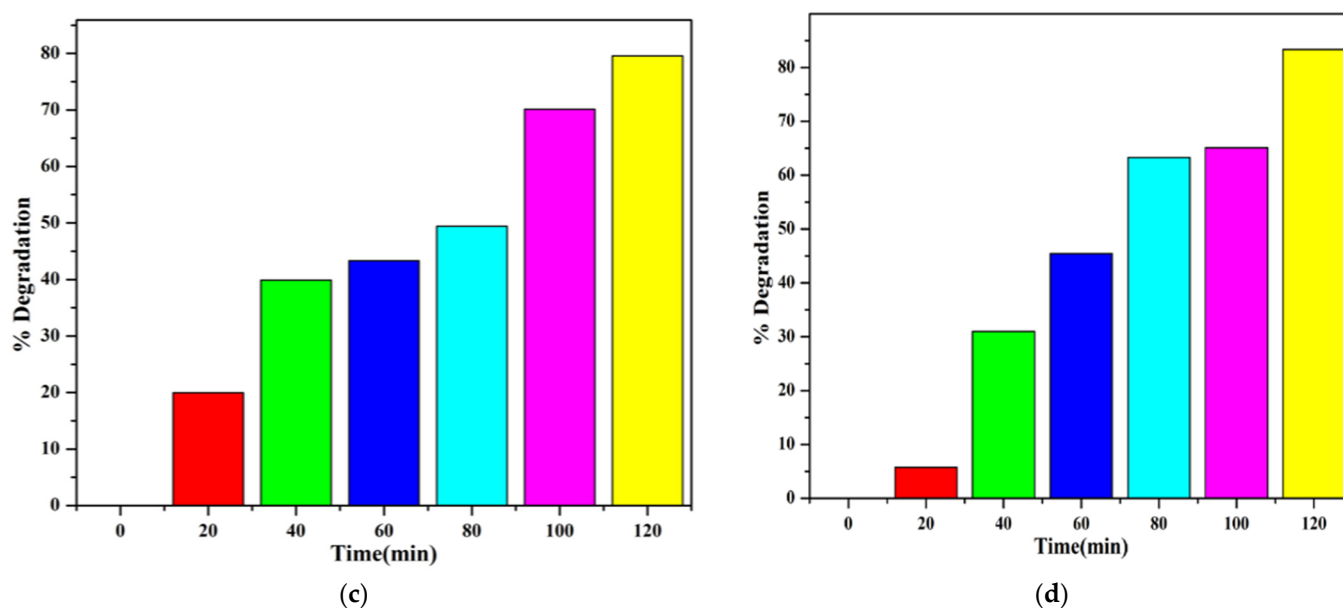


Figure 4. UV–Vis absorption spectra during the photocatalytic degradation of (a) Eriochrome Black T (EBT) and (b) Methyl Orange (MO) under UV irradiation, and the corresponding photocatalytic degradation efficiencies of (c) Methyl Orange (MO) and (d) Eriochrome Black T (EBT) as a function of irradiation time.

The data shows that the photocatalytic system represented in Figure 4c exhibits a higher photocatalytic degradation efficiency of 85% compared to Figure 4d, which showed a 78% degradation efficiency. Despite a slower onset, the system in (Figure 4c) achieves more substantial degradation with time, suggesting a potentially higher quantum efficiency or improved interaction with the target pollutant at longer exposure times. The enhanced performance in (Figure 4d) may be attributed to improved catalyst properties, such as higher surface area, better light absorption, or increased generation of reactive oxygen species.

Figure 5a illustrates the kinetic profile of the photocatalytic degradation of two organic dyes, EBT and MO, under visible light irradiation using a specified photocatalyst. The degradation kinetics were evaluated by monitoring the concentration ratio $\log(C/C_0)$ versus time (min), where C_0 and C signify the initial and time-dependent concentrations of the dyes, respectively. The degradation of both dyes adheres to an apparent pseudo-first-order kinetic model. Both dyes demonstrate a progressive decrease in concentration over 120 min of irradiation, with the slope of the curves reflecting the degradation rate. The higher degradation rate observed for MO may be attributed to its simpler molecular structure, higher photo reactivity, or better adsorption affinity toward the catalyst surface. In contrast, EBT's relatively complex azo structure or lower interaction with the catalyst might contribute to its slower degradation kinetics.

The photocatalyst's reusability performance for the degradation of EBT and MO over five cycles is illustrated in Figure 5b,c, respectively. The percentage of deterioration was documented after each cycle to assess the catalyst's stability and regeneration performance. The photocatalyst demonstrates remarkable stability in both cases by maintaining a high degradation efficiency even after several cycles. Figure 5b shows that, in the first cycle of EBT degradation, the catalyst achieves an initial degradation efficiency of about 79%, which remains relatively constant until the third cycle. There appears to be a slight decrease in activity, presumably due to catalyst surface fouling or active site deactivation, as evidenced by the fourth (~76%) and fifth (~72%) cycles. Similarly, regarding MO degradation (Figure 5c), the initial degradation efficiency was around 83%, but it fell to approximately

75% by the fifth cycle. After the third cycle, the decline is more noticeable, suggesting greater susceptibility to reusability fatigue than EBT.

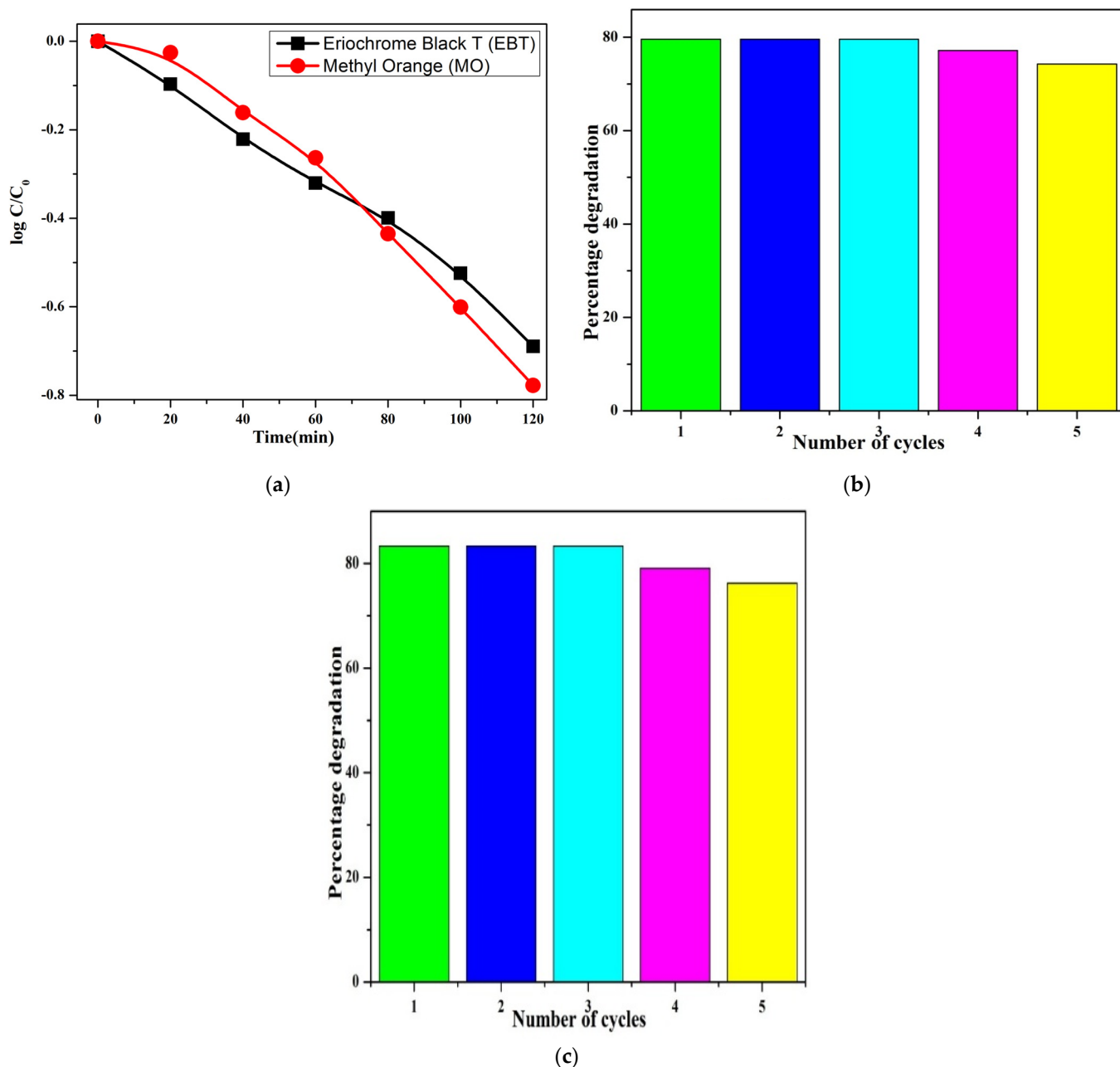


Figure 5. (a) The apparent pseudo-first-order kinetic Plot of $\log C/C_0$ versus time. (b) Reusability of Zn-MgFe₂O₄ nanoparticles for a five-cycle run EBT and (c) MO dyes.

The proposed photocatalytic mechanism for the degradation of EBT and MO dye using Zn-MgFe₂O₄ nanoparticles under UV–visible light irradiation is illustrated in Figure 6 [46,47]. The photocatalytic activity of the synthesized Zn-MgFe₂O₄ was evaluated by monitoring the dye degradation at 15 min intervals. When exposed to UV light with a photon energy of 1.89 eV, Zn-doped MgFe₂O₄ nanoparticles absorb the energy, promoting electrons (e^-) from the valence band (VB) to the conduction band (CB). This excitation generates electron–hole pairs (e^-/h^+), essential for initiating surface redox reactions on the photocatalyst.

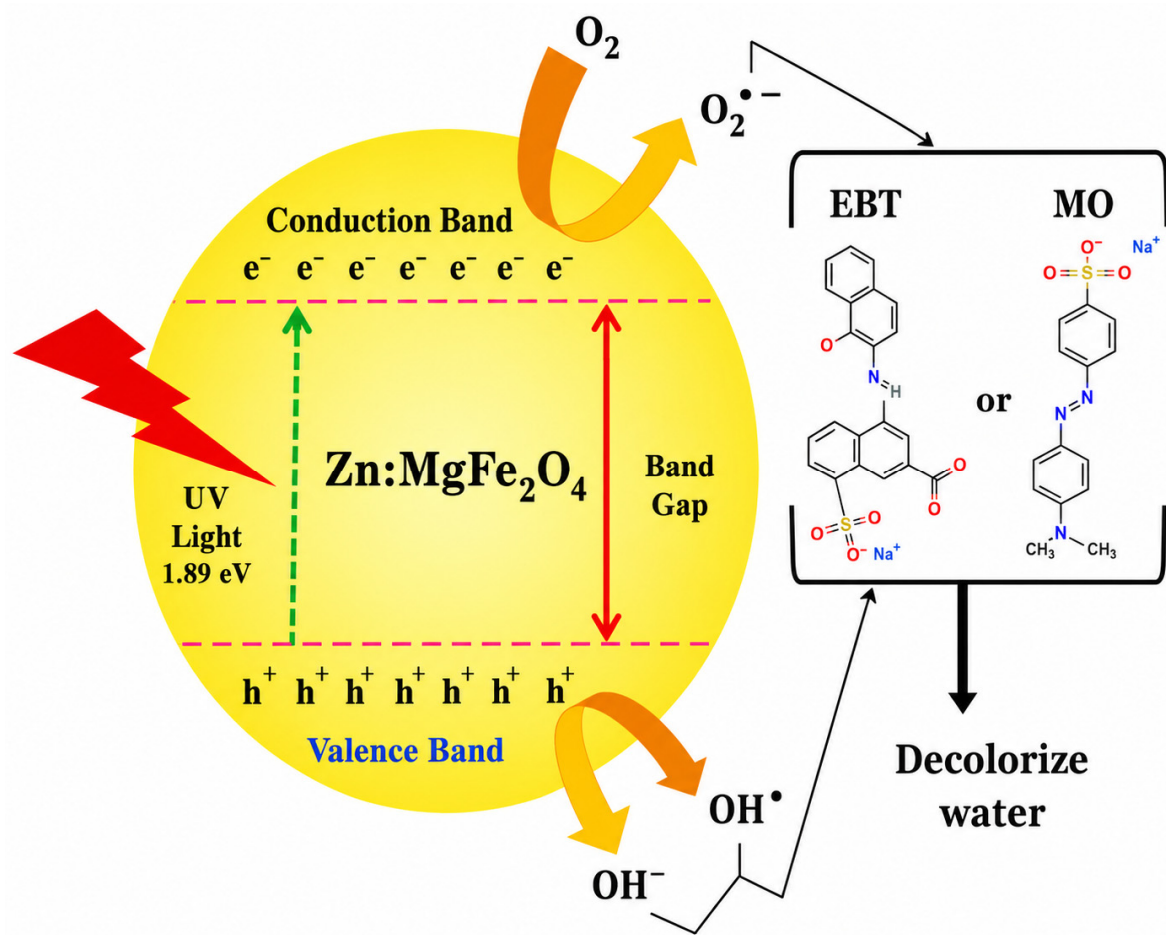


Figure 6. Mechanism of photocatalytic activity of EBT and MO dye degradation.

The excited electrons in the conduction band react with molecular oxygen (O₂) adsorbed on the surface, forming superoxide radicals ($\bullet\text{O}_2^-$). These reactive species actively contribute to the oxidative degradation of pollutants. Simultaneously, the holes remaining in the valence band oxidize water molecules (H₂O) or hydroxide ions (OH⁻), producing hydroxyl radicals ($\bullet\text{OH}$), which are highly potent in breaking down complex organic molecules. The molecular structures of EBT and MO, two commonly used azo dye pollutants, are well illustrated. Under the influence of reactive oxygen species, these dyes undergo degradation through cleavage of azo bonds (–N = N–) and disruption of aromatic structures, eventually transforming into environmentally benign products such as carbon dioxide, water, and inorganic ions [48]. This mechanistic pathway highlights the superior photocatalytic efficiency of Zn-MgFe₂O₄, which can be attributed to its relatively low band gap and efficient charge carrier separation. The combined action of electrons and holes in ROS generation facilitates fast and repeated degradation cycles, establishing this material as a strong contender for use in environmental cleanup processes. To further justify the formation of reactive oxygen species, the band edge potentials were estimated using the electronegativity-based (Mulliken) approach. According to this method, the conduction band (CB) and valence band (VB) potentials can be approximated from the absolute electronegativity of the semiconductor and its band gap energy. Based on the measured band gap (~1.86–1.89 eV), the calculated CB position is sufficiently negative relative to the O₂/ $\bullet\text{O}_2^-$ reduction potential (–0.33 V vs. NHE), enabling superoxide radical generation. Simultaneously, the VB position remains positive enough to oxidize surface hydroxyl groups or water molecules to produce hydroxyl radicals. Therefore, despite the relatively

narrow band gap, the estimated band alignment supports the proposed ROS-mediated degradation mechanism.

The photocatalytic experiments were conducted for a controlled duration of 120 min to ensure consistent comparison between the two dye systems. The degradation efficiency did not reach 100% within this period, likely due to adsorption–desorption equilibrium and the formation of intermediate degradation products that slow the apparent reaction rate. It is important to note that the objective of this study is not solely to maximize degradation percentage, but to demonstrate the multifunctional capability of Zn–MgFe₂O₄ as a structurally stable material exhibiting combined electrochemical sensing and photocatalytic performance. The achieved degradation efficiencies (78% for EBT and 85% for MO) fall within the typical range reported for spinel ferrite photocatalysts under similar conditions, confirming competitive performance while maintaining material stability.

To further clarify the photocatalytic performance, the degradation efficiencies obtained in this study (78% for EBT and 85% for MO within 120 min) were analyzed in comparison with previously reported spinel ferrite photocatalysts. Ferrite-based systems typically exhibit degradation efficiencies in the range of 60–90% under similar irradiation conditions, depending on crystallinity, surface area, and band gap tuning [49]. The relatively narrow band gap (~1.86–1.89 eV) observed in the present work promotes effective visible-light absorption while maintaining sufficient redox potential for reactive oxygen species (ROS) generation. Furthermore, Zn substitution is known to improve electron–hole separation efficiency by modifying cation distribution and reducing recombination probability [50,51]. The slightly higher degradation efficiency for MO compared to EBT may be attributed to differences in molecular structure and adsorption affinity toward the catalyst surface, as reported in azo dye degradation studies [52,53]. These comparative discussions confirm that the obtained photocatalytic activity is consistent with, and competitive among, previously reported Zn-substituted ferrite system. Upon UV irradiation, electrons are excited from the valence band to the conduction band of Zn–MgFe₂O₄, leaving holes in the valence band. The excited electrons reduce dissolved oxygen to produce superoxide radicals ($\bullet\text{O}_2^-$), while the photogenerated holes oxidize surface hydroxyl groups or water molecules to form hydroxyl radicals ($\bullet\text{OH}$). These reactive oxygen species subsequently oxidize and mineralize the dye molecules. Zn substitution enhances charge separation by reducing electron–hole recombination, thereby improving the overall photocatalytic efficiency.

To further clarify the role of Zn substitution, the present results were compared with previously reported pristine MgFe₂O₄. Undoped MgFe₂O₄ is known to exhibit limited electrochemical performance owing to its relatively low electrical conductivity and rapid recombination of photogenerated electron–hole pairs. In contrast, Zn incorporation promotes cation redistribution within the spinel lattice, enhances charge carrier mobility, and suppresses charge recombination, thereby improving both photocatalytic and electrochemical performance. Kumar et al. reported that Zn-doped MgFe₂O₄ exhibited approximately two-fold higher photocatalytic reaction kinetics and nearly three-fold higher photocurrent density than pristine MgFe₂O₄, demonstrating that Zn doping markedly accelerates interfacial charge transfer and separation [54]. These findings are consistent with the enhanced multifunctional performance observed in the present work.

Finally, Table 3 compares the photocatalytic activity of the present Zn–MgFe₂O₄ nanoparticles with recently reported ferrite-based photocatalysts. Although several composite and heterojunction photocatalysts exhibit higher degradation efficiencies, they generally require more complex synthesis procedures involving secondary semiconductors, noble metals, or carbon-based supports. In contrast, the present Zn–MgFe₂O₄ nanoparticles were synthesized via a simple combustion route and still achieved efficient degradation of both Eriochrome Black T (78%) and Methyl Orange (85%) within 120 min. These results

demonstrate that Zn substitution effectively enhances the multifunctional performance of MgFe_2O_4 while maintaining a facile and scalable synthesis process.

Table 3. Comparison of the photocatalytic performance of the present Zn– MgFe_2O_4 photocatalyst with previously reported ferrite-based photocatalysts.

Catalyst	Pollutant	Light Source	Degradation Efficiency (%)	Time (min)	Reference
MgFe_2O_4	Reactive Black 21	Visible light	93	180	[55]
$\text{MgFe}_2\text{O}_4/\text{Bi}_2\text{WO}_6$	Tetracycline	Visible light	95.8	90	[55]
Ag-functionalized Zn/Mg ferrite	Malachite Green	Visible light	99	180	[56]
Present work (Zn– MgFe_2O_4)	Eriochrome Black T	UV	78	120	This work
Present work (Zn– MgFe_2O_4)	Methyl Orange	UV	85	120	This work

5. Materials and Methods

5.1. Preparation of Precursors

Metal nitrates (e.g., zinc, magnesium, and ferric nitrate) are dissolved in deionized water to form a homogeneous solution. Urea was employed as the combustion fuel. The total oxidizer-to-fuel ratio was adjusted according to the propellant chemistry approach to achieve a near-stoichiometric combustion reaction, ensuring complete combustion and homogeneous formation of Zn– MgFe_2O_4 nanoparticles [21–23]. The solution is stirred thoroughly to ensure uniform mixing of the precursors and the fuel. The mixture is heated in a magnetic stirrer at 90 °C. The temperature is raised to the ignition point of the fuel, typically between 300 and 600 °C (Figure 7). Once ignited, the exothermic reaction proceeds rapidly, generating a large amount of heat and gas, forming a fluffy, porous ferrite powder. The resultant powder is collected and may undergo additional heat treatment (calcination) to improve crystallinity.

5.2. Characterization Techniques

High-resolution X-ray diffraction (XRD) analysis of Zn– MgFe_2O_4 nanoparticles was conducted using a Shimadzu Maxima–7000 instrument (Shimadzu Corporation, Kyoto, Japan). The measurements were performed with $\text{CuK}\alpha$ radiation ($\lambda = 1.54 \text{ \AA}$), operated at 40 kV and 30 mA, and scanned at a $2\text{--}4 \text{ min}^{-1}$ rate. Fourier-transform infrared (FTIR) spectroscopy was carried out using a Perkin Elmer FTIR 1650 spectrometer (PerkinElmer Inc., Shelton, CT, USA) at room temperature. The samples were prepared using the KBr pellet method, where 0.0010 g of the nanoparticle sample was mixed with 0.3 g of high-purity KBr and subjected to 16 scans over a spectral range of $400\text{--}4000 \text{ cm}^{-1}$. The surface morphology of the synthesized nanoparticles was examined using a scanning electron microscope (SEM, HITACHI SU-1500, Hitachi High-Technologies Corporation, Tokyo, Japan), operating at an accelerating voltage of 15 kV and a beam current of 20 mA. Elemental composition was analyzed using energy-dispersive X-ray spectroscopy (EDX) with a JEOL JSM-5600LV (JEOL Ltd., Tokyo, Japan) SEM equipped with an EDX detector. The optical properties were evaluated through UV–Visible absorption spectroscopy using an ELICO SL 159 (Elico Ltd., Telangana, India) spectrophotometer. Additionally, the electrochemical behavior of the nanoparticles was assessed using a CHI608E (CH Instruments, Inc., Austin, TX, USA) electrochemical workstation in 0.1 M KOH electrolyte at various scan rates.

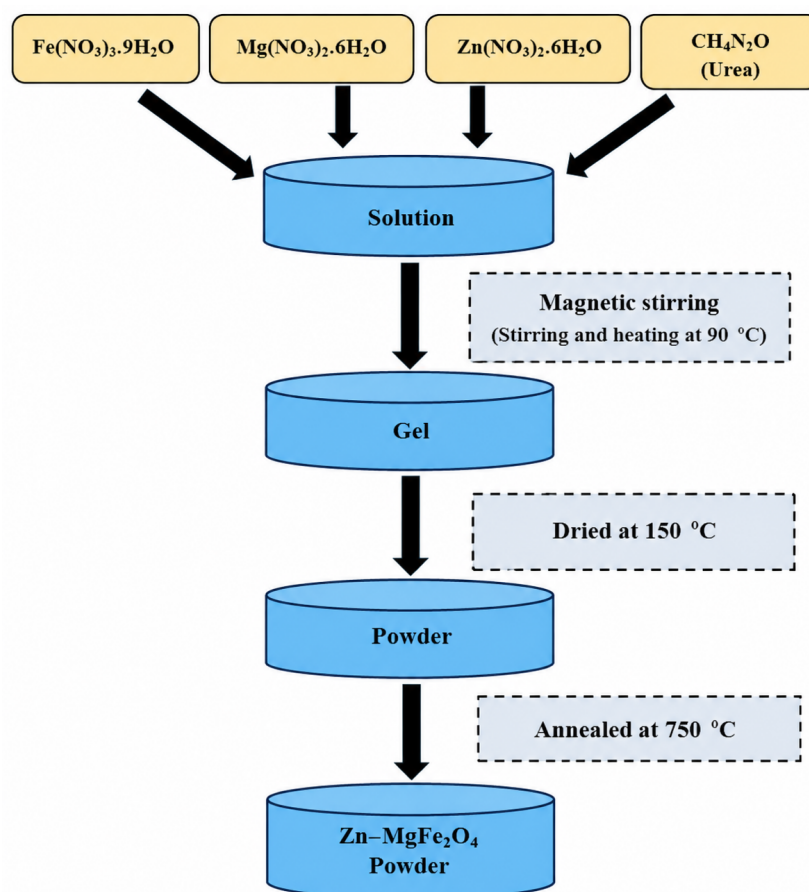


Figure 7. Schematic illustration of the combustion synthesis process for Zn-MgFe₂O₄ nanoparticles.

6. Conclusions

In this study, Zn-MgFe₂O₄ nanoparticles were successfully synthesized and characterized for their structural, morphological, optical, electrochemical, and photocatalytic properties. The XRD analysis confirmed the formation of a pure, nanocrystalline spinel ferrite phase with a crystallite size range of 18.7–27.9 nm. SEM imaging revealed a flake-like morphology, and EDX confirmed the elemental composition, verifying the successful incorporation of Zn²⁺ and Mg²⁺ into the ferrite matrix. FTIR spectroscopy further validated the formation of the spinel structure, showcasing characteristic metal–oxygen vibrations. The optical band gap of the synthesized nanoparticles, determined to be approximately 1.89 eV, indicates strong absorption in the UV region and suggests suitability for photocatalytic applications. Electrochemical analysis, including CV and EIS, demonstrated promising redox behavior and efficient charge transport, reflecting the potential of Zn-MgFe₂O₄ as an active material in sensor technologies and energy storage devices. Specifically, the modified electrode exhibited a linear and sensitive response towards mercury chloride detection, confirming its applicability in environmental monitoring. Moreover, the photocatalytic experiments highlighted the high degradation efficiency of the material against organic dyes such as EBT and MO under UV irradiation. The catalyst achieved up to 85% degradation efficiency and maintained notable stability over multiple reuse cycles. The photocatalytic mechanism was attributed to effective electron–hole pair generation and subsequent formation of reactive oxygen species, leading to the breakdown of complex dye molecules. Overall, the Zn-MgFe₂O₄ nanomaterial exhibits a versatile combination of structural integrity, optical responsiveness, electrochemical activity, and environmental remediation capability, making it a promising candidate for multifunctional applications in sensing, catalysis, and pollutant degradation.

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