

RESEARCH ARTICLE

Elucidating Visible-Light Photocatalytic Degradation of Organophosphorus Residues Over Reduced Graphene Oxide/Iron-Manganese Mixed Oxides Composite

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ABSTRACT

In this work, a ternary Fe₂O₃/Mn₂O₃/reduced graphene oxide (FM/rGO) composite was prepared via a rapid microwave-assisted coprecipitation method. The engineered composite exhibited exceptional visible-light-driven photocatalytic activity as compared to both single Fe₂O₃ and Fe₂O₃/Mn₂O₃ (FM) heterojunction, achieving nearly complete degradation of parathion, a resistant organophosphorus pollutant, within 60 min accompanied by a remarkable mineralization (~86% TOC removal). Kinetic analysis confirmed that the degradation complies with a pseudo-first-order model with a significant rate of 0.0597 min⁻¹. Comprehensive photoelectrochemical measurements revealed that the Type-I FM heterojunction and the highly conductive rGO network facilitate efficient charge transfer. Interestingly, the coexistence of mixed-valence transition metals (Fe³⁺/Fe²⁺ and Mn³⁺/Mn²⁺) alongside a high density of π bonds in rGO carriers served as dynamic electron traps, effectively accelerating spatial charge separation and suppressing electron-hole recombination. Radical scavenging and GC-MS analyses identified critical roles of hydroxyl radicals ($\bullet$ OH) for the P–O–aryl bond cleavage. Furthermore, the FM/rGO composite demonstrated practical magnetic recoverability and outstanding cyclic stability, providing a robust and highly efficient photocatalytic platform for hazardous pollutant remediation.

1 | Introduction

In contemporary agriculture, pesticides are widely used to manage pest infestations, enhance crop yields, and safeguard global food security. Nevertheless, their pervasive and frequently unregulated application has caused severe environmental and public health crises. The intrinsic persistence of these agrochemicals facilitates their extensive accumulation within

soil and aquatic ecosystems, thereby driving prolonged ecological degradation and the severe contamination of vital drinking water reservoirs [1]. Furthermore, a multitude of epidemiological and toxicological investigations have established that chronic exposure to such pesticide residues is inextricably associated with adverse human health outcomes. These health risks include neurological, endocrine, and cardiovascular issues, as well as increased cancer vulnerabilities [2–5]. Consequently, developing

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efficient and sustainable technologies to remove pesticide residues from various environments is urgent and critical.

Among various classes of pesticides, organophosphates represent a particularly hazardous group due to their high acute toxicity and widespread use. Organophosphates are phosphate ester derivatives that exist in thion ($\text{P}=\text{S}$) and oxon ($\text{P}=\text{O}$) forms, with the latter generally exhibiting higher toxicity [6, 7]. Ethyl parathion, a representative organophosphate insecticide, has been extensively applied in agricultural activities involving crops such as cotton, soybean, corn, wheat, and various fruits and vegetables [8]. Despite its effectiveness as a pesticide, its high toxicity has led to severe environmental and health risks, prompting its classification as an extremely hazardous substance and resulting in strict regulations and bans in many countries [9]. Parathion is moderately persistent in soil and can undergo oxidative desulfurization to form paraoxon, a more toxic metabolite [10]. Consequently, the removal and degradation of parathion from contaminated environments remain a critical challenge [11].

To date, diverse remediation technologies have been investigated for the elimination of organophosphate pesticides, encompassing physical adsorption, electrochemical oxidation, and various advanced oxidation processes (AOPs), notably ozonation, Fenton, and photo-Fenton systems [12–14]. Among these established methodologies, semiconductor-based photocatalysis is a promising strategy utilizing solar energy to completely mineralize organic pollutants into non-toxic inorganic end products, primarily CO_2 and H_2O , under ambient conditions [15, 16]. Mechanistically, this process is activated by the generation of electron-hole (e^-/h^+) pairs on the semiconductors during light irradiation, before the charge carriers transfer over the catalyst surface to drive a cascade of redox reactions, ultimately generating highly potent reactive oxygen species (ROS) that are fundamentally responsible for the oxidative cleavage and destruction of complex organic architectures [17].

Hematite ($\alpha\text{-Fe}_2\text{O}_3$) is highly abundant, low-cost, and extensively studied metal oxides [18]. $\alpha\text{-Fe}_2\text{O}_3$ with a visible-light-absorption-compatible band gap of 1.8–2.2 eV, allowing utilization of nearly 40% of the solar spectrum [19, 20]. In addition, $\alpha\text{-Fe}_2\text{O}_3$ exhibits excellent chemical stability in aqueous environments, particularly at pH values above 3, rendering it suitable for long-term photocatalytic and photoelectrochemical applications. The valence band (VB) edge of $\alpha\text{-Fe}_2\text{O}_3$ is sufficiently positive relative to the water oxidation potential, facilitating efficient transfer of photogenerated holes (h^+) to water molecules or adsorbed organic species. This favorable band alignment makes $\alpha\text{-Fe}_2\text{O}_3$ a promising candidate for solar-driven water oxidation and the degradation of persistent organic contaminants [21].

Despite advantages, $\alpha\text{-Fe}_2\text{O}_3$ still has several drawbacks that restrict its photocatalytic performance. These include short hole diffusion length (2–4 nm), low intrinsic electrical conductivity, and slow oxygen evolution reaction kinetics. These factors lead to rapid recombination of photogenerated charge carriers and consequently reduced photocatalytic efficiency [22, 23]. To mitigate these, extensive efforts have been devoted to tailoring the structural and electronic properties of hematite. The approaches such as nanostructure engineering, elemental

doping, surface fabrication, and the establishment of heterojunctions or composites with conductive carbon materials have been widely explored. These approaches are primarily aimed at promoting charge separation, accelerating carrier transport, and improving light-harvesting efficiency. For example, coupling hematite with secondary oxides, including TiO_2 or Mn_xO_y , has been demonstrated to modify its electronic structure, optimize band alignment, enhance charge carrier mobility, and facilitate interfacial charge transfer processes, ultimately improving overall photocatalytic activity [24–26].

Not only Fe_2O_3 but manganese oxides (Mn_xO_y), including MnO , Mn_3O_4 , Mn_2O_3 , and MnO_2 , are other promising choices as semiconductor photocatalysts for treatment of organic contaminants. Mn_xO_y exhibits its advantages of natural redox reagents, cost-effectiveness, high abundance, non-toxicity, flexible electron transfer process, and visible-light responsiveness [27]. Despite these advantages, Mn_xO_y , still face several limitations, including moderate electrical conductivity and relatively fast recombination of charge carriers [28]. To address their disadvantages, similar to Fe_2O_3 , Mn_xO_y has been engineered with various semiconductors (TiO_2 , ZnO , SiO_2 , etc.) by heterojunction construction [29–32], transition metals (Ag, Zn, etc.) by doping [33, 34], or high-surface-area carriers by formation of hybrid composites [35–38].

In addition, photocatalysts have been integrated with carbon-based materials, including activated carbon (AC), graphene oxide (GO), and reduced graphene oxide (rGO) to enhance their catalytic activities [39, 40]. With advantages of high electrical conductivity, large specific surface area, and various functional groups, such as $\text{C}=\text{C}$ or $>\text{C}=\text{O}$, these carbon nanostructures serve as efficient electron acceptors and charge transport pathways, thereby facilitating spatial separation of e^-/h^+ pairs. Specifically, GO and rGO can reserve the generated electrons from the conduction band (CB) of Fe_2O_3 and rapidly shuttle them across the composite interface, effectively suppressing electron-hole recombination within the semiconductor matrix [41, 42]. This enhanced charge carrier separation extends the lifespan of reactive species and promotes interfacial redox reactions. Consequently, the coupling between Fe_2O_3 and graphene-families significantly improves the photocatalytic performance of Fe_2O_3 -based nanocomposites.

Despite significant efforts in escalating the performance of Fe_2O_3 -based photocatalysts, several challenges remain unresolved. In particular, the application of Fe_2O_3 -based systems for the degradation of highly toxic organophosphate pesticides such as ethyl parathion is still limited. Moreover, Mn_2O_3 or $\text{Fe}_2\text{O}_3/\text{Mn}_2\text{O}_3$ heterojunction and rGO, which enhance charge separation and facilitate interfacial electron transfer, have not been systematically elucidated. In this study, the $\text{Fe}_2\text{O}_3/\text{Mn}_2\text{O}_3/\text{rGO}$ ternary photocatalyst was prepared by a coprecipitation with the assistance of microwave irradiation to improve charge separation and interfacial electron transfer. The morphological, structural, and optical properties of the prepared materials were systematically characterized. The photocatalytic performance of the composite was subsequently investigated via ethyl parathion degradation using a simulated visible-light source. Furthermore, insight into the degradation pathway and generated intermediate products were also discussed.

2 | Experimental

2.1 | Chemicals

Ferric nitrate nonahydrate ($\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, $\geq 99\%$, Xilong Scientific, China), manganese(II) nitrate solution ($\text{Mn}(\text{NO}_3)_2$, 50 wt%, Xilong Scientific, China), graphite powder ($\geq 99\%$, Xilong Scientific), potassium permanganate (KMnO_4 , $\geq 99\%$), sodium nitrate (NaNO_3 , $\geq 99\%$), hydrogen peroxide (H_2O_2 , 30 wt%), sulfuric acid (H_2SO_4 , 98%), and polyvinyl alcohol (PVA) were used as received without further purification. Ethyl parathion ($\text{C}_{10}\text{H}_{14}\text{NO}_3\text{PS}$, analytical grade) was obtained from Merck.

2.2 | Synthesis $\text{Fe}_2\text{O}_3/\text{Mn}_2\text{O}_3/\text{rGO}$ Nanocomposite Material

A modified Hummers' method was employed for GO preparation [43]. First, graphite powder and NaNO_3 were dispersed in concentrated H_2SO_4 (98%) under stirring at $\sim 0^\circ\text{C}$ for 30 min. KMnO_4 was then slowly added over 2 h while maintaining the temperature below 20°C . Subsequently, 23 mL of distilled water was added to the mixture and heated at 95°C for 30 min before being diluted with additional 120 mL of distilled water. Residual oxidants were removed by adding H_2O_2 . The suspension was then neutralized by 5% HCl and distilled water, and then dried at 60°C to obtain GO. Afterward, the obtained GO powder was homogeneously dispersed in deionized water and ultrasonicated. The suspension was then heated to 65°C , and ascorbic acid as reductant was added at a mass ratio of 10:1 (ascorbic acid: GO) [44]. The reaction was maintained for 1 h to forming rGO as the suspension changed from brown to black. Finally, the rGO powder was isolated, washed thoroughly with deionized water, and dried.

In the following step, FM/rGO was synthesized via coprecipitation with a microwave-assisted method. First, an equimolar of $\text{Fe}(\text{NO}_3)_3$ 0.5 mol/L and $\text{Mn}(\text{NO}_3)_2$ 0.5 mol/L solutions as metal precursors were gently added to as-prepared 5 wt% PVA solution as gel forming agent. The mixture was heated at 80°C under continuous stirring for the following 2 h. Subsequently, rGO (5 wt% of composite [45]) was introduced in the viscous gel before adding drops of NaOH until reaching pH 12. The resulting solid was then collected by centrifugation and dried at 60°C . Finally, the dried powder was exposed to microwave irradiation (800 W, 2450 MHz) for 5 min and re-calcined at 450°C to achieve the desired composites.

2.3 | Material Characterization

Thermal behaviors of FM/rGO sample were first characterized in LabsysEvo (SETARAM, France). The sample microstructure was captured by a JEM 2100 (Japan) transmission electron microscope (TEM) and a Hitachi S4800 (Japan) scanning electron microscope (SEM). The elements involved in the samples were also mapped by an SEM-integrated energy-dispersive x-ray spectroscopy (EDX). X-ray photoelectron spectroscopy (XPS) (ESCA-3400, Mg $K\alpha$ (1253.6 eV) source, Shimadzu, Japan) was used to examine the oxidation states of the elements in the materials. The Au (powder) $4f_{7/2}$ peak at 84.0 eV was used for

calibration while the Shirley examined subtraction was used for peak fitting. In addition, an x-ray diffractometer (D8 Advance, Cu $K\alpha$ irradiation, Bruker, Germany) was utilized to characterize the crystal structure and phase purity. Raman spectroscopy (XploRA PLUS) was used to explore the materials' vibrational modes and structural properties. A 532 nm excitation source, combined with a 1% neutral-density filter and an 1800 grooves/mm grating, was applied. The laser was focused on a $\sim 1\ \mu\text{m}$ spot using a $100\times$ objective (N.A. = 0.8), and the scattered signal was then collected. Besides, the nitrogen sorption isotherms were then conducted in an Autosorb IQ Station to derive the sample surface area (S_{BET}) and porosity. Diffuse reflectance spectroscopy (DRS) was conducted in a UV-VIS spectrophotometer (JASCO V-500 model, Japan). Magnetic behaviors over the magnetic range of ± 12000 Oe were analyzed by a magnetometer (10^{-4} emu sensitivity) at the temperature range of 77–1000 K. Lastly, a potentiostat analyzer (Biologic SP-300, French) was employed to survey photoelectrochemical characteristics of the samples.

2.4 | Photocatalysis Experiment

Parathion photodegradation was performed in a 500-mL photoreactor (Pyrex glass, ACE, USA) equipped under visible-light simulation (low-pressure Hg lamp source with UV-cutoff filter, 450 W). The catalysts, the reaction time, the pH, the initial parathion concentration, and the dosages of used materials were varied and monitored for assessment. Each photocatalyst was first dispersed into a 250-mL parathion aqueous solution. The adsorption equilibrium between the parathion molecules and the catalyst surface was settled in the darkness for 4 h under a gently magnetic stirring. During photocatalysis, samples were withdrawn at certain intervals (depending upon practical experiment) and filtered to remove suspended catalyst particles. The samples were then extracted with an acetone/n-hexane mixture (1:1, v/v) before analyzed by gas chromatography–mass spectrometry (GC–MS) (see Table S1). The degradation efficiency (DE) was determined by Equation (1).

$$H(\%) = \frac{C_0 - C_t}{C_0} \times 100\% = \frac{A_0 - A_t}{A_0} \times 100\% \quad (1)$$

where A_0 and A_t were the integrated peak areas of parathion on GC/MS at 0 and t min, and C_0 and C_t were the corresponding parathion concentrations. Additionally, the total organic content of extract aliquot was also assessed on a TOC analyzer (OI Analytical Aurora 1030).

3 | Results and Discussion

3.1 | Materials Characterization

First, the thermal behavior of FM/rGO sample is demonstrated in Figure 1a. The composite exhibits an endothermic event accompanied by a remarkable mass loss within 30°C – 200°C were attributed to the evaporation of physical surface-adsorbed and lattice-bound water [46]. A plateau in mass is observed between 300°C and 400°C , indicating thermal stability within this interval. An exothermic peak and a noticeable loss in

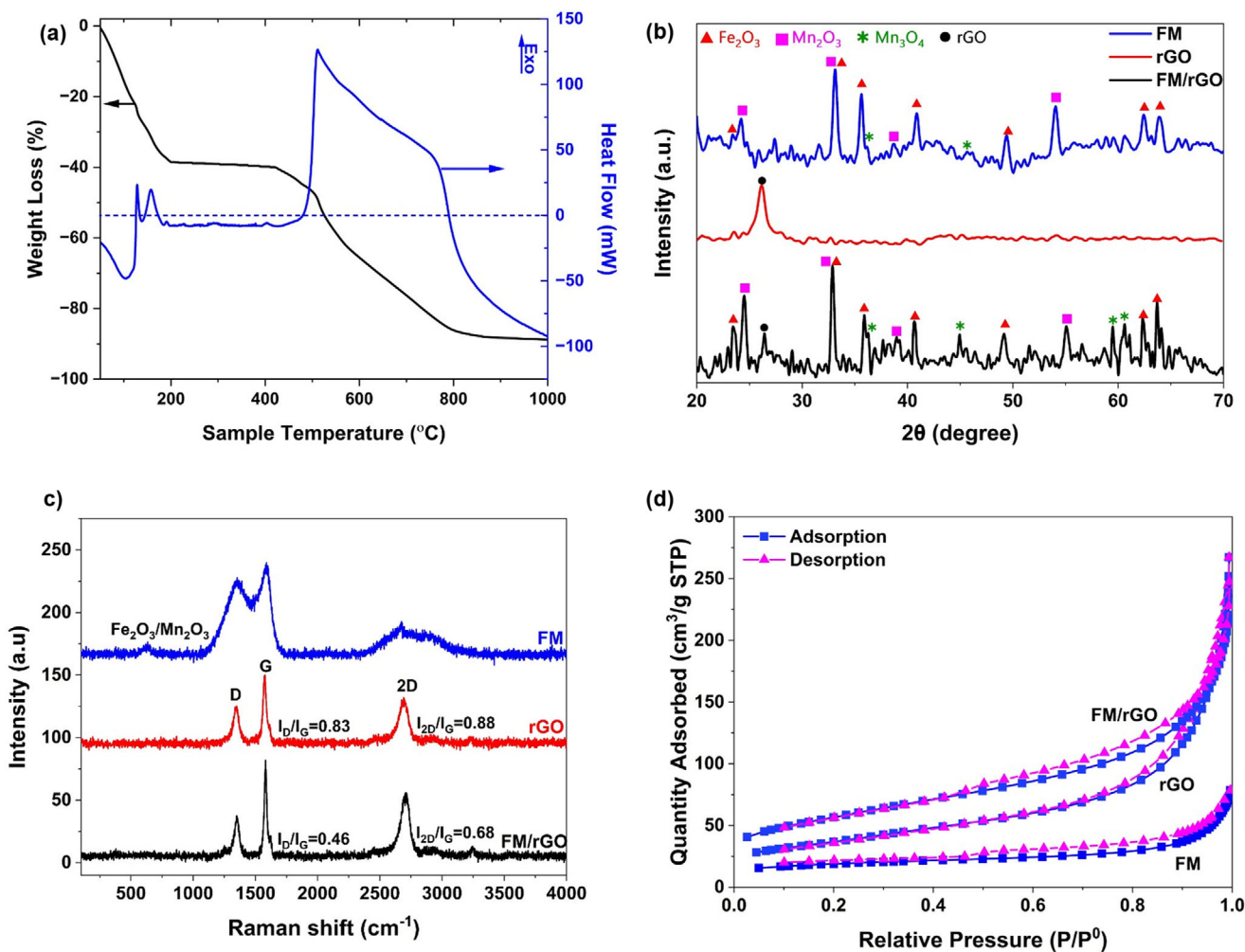


FIGURE 1 | Structural and thermal characterization of the synthesized samples. (a) Thermal analyses of FM/rGO samples. (b) XRD patterns, (c) Raman spectra, and (d) nitrogen adsorption/desorption curves of rGO, FM, and FM/rGO samples.

weight were detected from 500°C to 850°C. This significance was assigned to carbonaceous combustion, primarily the oxidation of rGO to CO₂, in the FM/rGO composition [47]. At a higher temperature beyond 800°C, the composite retained its weight loss, suggesting the formation of a thermally stable structure of attached oxides, Fe₂O₃ and Mn₂O₃.

The phase composition of FM/rGO sample were characterized by the XRD method (Figure 1b). In the FM and FM/rGO curves, while rhombohedral α -Fe₂O₃ indicated the characteristic peak at 23.4°, 32.4°, and 36.0° (JCPDS No. 33-0664, signal $\blacktriangle$), the cubic α -Mn₂O₃ also exhibited the diffraction peaks at 23.7°, 32.4°, and 38.2° (JCPDS No. 24-0508, signal $\blacksquare$) [48, 49]. Although the characteristic peaks of included components were not fully presented, the corresponding peaks of (104) and (222) diffraction planes of Fe₂O₃ and Mn₂O₃, respectively, confirmed the formation of this heterojunction with a spherical structure [49]. Otherwise, the distinctive peaks at 26° demonstrated the establishment of the π -conjugated structure of graphene-based materials of rGO in the prepared composite (JCPDS No. 01-275-2078, signal $\bullet$) [50]. In addition, the diffraction peaks related to Mn₃O₄ (JCPDS No. 24-0734, signal $*$) [51] phase were also illustrated, suggesting

a partial reduction of Mn³⁺ to Mn²⁺ during preparation stage. Moreover, according to Scherrer's equation [52], the size of integrated oxide nanoparticles on rGO ranges between 15.3 and 34.2 nm.

The Raman spectroscopy shows the bonding between oxide and graphene mono layer of FM, rGO, and FM/rGO (Figure 1c). In FM curve, a small peak at 700 cm⁻¹ indicates Fe–O and Mn–O bonds. Besides, the D band, G band and 2D bands located at 1346, 1574, and 2692 cm⁻¹, respectively, were attributed to the carbon existed in rGO. Likewise, in the spectrum of FM/rGO, the combination of FM heterojunction and rGO in FM/rGO slightly shifted the D band, G band and 2D band peaks to 1350, 1582, and 2708 cm⁻¹, respectively. rGO curve exhibits characteristic D and G bands with an intensity ratio I_D/I_G of 0.83 and an I_{2D}/I_G ratio of about 0.88, which are typical multi-layered structure containing residual oxygenated defects [53–55]. For the bare FM sample, the weak and broad bands in the carbon region indicate the presence of minor residual amorphous carbon, likely from the incomplete combustion of PVA gel during preparation stage. Notably, upon the formation of the FM/rGO composite, the I_D/I_G ratio significantly decreases to 0.46. This

marked reduction suggests that the $\text{Fe}_2\text{O}_3/\text{Mn}_2\text{O}_3$ nanoparticles preferentially anchor at the defect sites and remaining functional groups of the rGO sheets. This anchoring mechanism effectively occupies these localized defects, thereby enhancing the overall structural ordering and graphitic crystallization of the composite. Furthermore, the slightly attenuated I_{2D}/I_G ratio (0.68) in the FM/rGO sample further supports the strong interfacial electronic interaction between the metal oxides and the multi-layered rGO carrier.

Afterwards, S_{BET} and porosity of FM/rGO composite were examined using nitrogen sorption isotherms. Remarkably, the nitrogen uptake at relatively low pressure (P/P^0) signifies the filling of micropores, which serves as a recognized fingerprint of micro- and mesoporous carbon materials [56]. The S_{BET} and average pore size derived from the Brunauer-Emmett-Teller and Barrett-Joyner-Halenda model are $68 \text{ m}^2/\text{g}$ and 11.48 nm for $\text{Fe}_2\text{O}_3/\text{Mn}_2\text{O}_3$, $200.7 \text{ m}^2/\text{g}$ and 8.86 nm for rGO, and $131.98 \text{ m}^2/\text{g}$ and 14.33 nm for FM/rGO, respectively. The results suggested that the dispersion of FM on rGO enhanced both S_{BET} and pore size, which consequently facilitates the interaction and easy diffusion of the substances involved in the reactions.

The microstructure of FM, rGO, and FM/rGO was shown by FE-SEM and TEM images (Figure 2). For FM sample, uniformly sized nanoparticles (NPs) with a diameter range of $20\text{--}30 \text{ nm}$ are distinctly observable (Figure 2a). Conversely, the rGO support exhibits a disordered architecture comprised of thin, crumpled nanosheets that tend to agglomerate (Figure 2b). FE-SEM image of FM/rGO further demonstrates that the $\text{Fe}_2\text{O}_3/\text{Mn}_2\text{O}_3$ NPs are homogeneously dispersed and intimately anchored onto the rGO nanosheets (Figures 2c and S1). This tight immobilization is likely due to strong chemical interactions between the oxide nanoparticles and the oxygenated functional groups on the rGO surface. In addition, the TEM images confirm that $\text{Fe}_2\text{O}_3/\text{Mn}_2\text{O}_3$ nanoparticles are well dispersed on the rGO sheets without significant aggregation, maintaining a particle size consistent with the SEM observations ($20\text{--}30 \text{ nm}$), which is close to the size derived from XRD data (Figure 2f). Moreover, the interaction between the oxide NPs and the rGO sheets is clearly observed, indicating the successful formation of a heterostructure composite. This close interfacial interaction is expected to facilitate efficient charge transfer and suppress e^-/h^+ recombination, thereby facilitating the photocatalytic performance of the composite material. The elemental mapping displays the spatial distribution of O, C, Fe, and Mn over the FM/rGO composite. The mapping also shows that 'Mn' significantly overlapped with that of the 'Fe', which suggested a proximity of Mn_2O_3 NPs with the Fe_2O_3 NPs.

XPS method was employed to elucidate the surface chemical oxidation states of the elements in the FM/rGO composite. The survey spectrum (Figure S2) clearly illustrates the peaks of C 1s, O 1s, Mn 2p, and Fe 2p located at the binding energies (BE) of 291.38, 436.15, 659.01, and 739.77 eV, respectively. Noteworthy, the experimental Fe/Mn molar ratio in product derived from XPS (0.86) (Table S2) is close to the Fe/Mn molar ratio in precursors, which indicates Fe and Mn elements are uniformly dispersed. A high-resolution XPS spectrum in Figure 3a shows characteristic peaks of Fe_2O_3 at 711 and 725 eV, corresponds to the Fe $2p_{3/2}$ and Fe $2p_{1/2}$ respectively [57]. Furthermore, the detection of a

distinct shake satellite peak at approximately 719 eV further corroborates the predominant Fe^{3+} oxidation state in the Fe_2O_3 lattice [58]. In the Mn 2p spectra, Mn $2p_{3/2}$ and Mn $2p_{1/2}$ display the peaks at the BE of 641 and 653 eV, respectively (Figure 3b). The characteristic spin-orbit separation of approximately 10.0 eV strongly indicates Mn^{3+} [59]. Furthermore, both Mn 2p and Fe 2p display multiple valence states (+2 and +3), suggesting the possible redox reactions between Fe^{3+} (or Mn^{3+}) and the generated excited electrons during photocatalysis [60, 61]. In Figure 3c, the asymmetric O 1s profile can be resolved into three distinct peaks centered at 529, 530, and 533 eV. These peaks are appropriately assigned to the metal–oxygen lattice (M–O) bonds, the surface defect-related oxygen or hydroxyl groups, and the oxygenated functional groups of rGO (e.g., C–O/C=O) alongside chemisorbed water, respectively [62, 63]. This profile firmly corroborates the simultaneous existence of these chemical species within the FM/rGO composite. Ultimately, the deconvoluted C 1s spectrum displays a dominant peak at 284.5 eV representing the graphitic carbon framework (C–C, C=C, and C–H bonds) of the rGO support. The secondary peaks at higher BEs correspond to interfacial M–O–C linkages (285.4 eV) and adsorbed CO_2 (289 eV) [64, 65]. Therefore, the obtained data firmly indicates the formation of $\text{Fe}_2\text{O}_3/\text{Mn}_2\text{O}_3/\text{rGO}$ composite in the present work.

To further elucidate the optical properties of the prepared materials, DRS method was employed to examine their light absorption behavior (the inset of Figure 4a). Both bare Fe_2O_3 and Mn_2O_3 exhibited distinct absorption in the visible region, with Fe_2O_3 absorbing predominantly within $500\text{--}660 \text{ nm}$ and Mn_2O_3 extending further into the longer wavelength region of $600\text{--}760 \text{ nm}$ [66, 67]. Upon formation of the FM heterojunction and subsequent integration with rGO, the characteristic absorption features of both Fe_2O_3 and Mn_2O_3 were retained, indicating that the intrinsic optical properties of the individual components were largely preserved. Notably, the FM/rGO composite exhibited a near-UV absorption band ($360\text{--}410 \text{ nm}$), which can be ascribed to the light absorption of rGO arising from its $\pi\text{--}\pi^*$ transitions and the surface functional groups like C=O or $>\text{C=O}$ [68]. Furthermore, the absorption profiles corresponding to Fe_2O_3 and Mn_2O_3 in the FM/rGO composite became broader and less distinct compared to those of the FM heterojunction and the individual oxides [69]. This behavior suggests enhanced electronic interaction and overlaps between the components, leading to a more continuous absorption spectrum and improved light-harvesting capability. The direct bandgap (E_g) of semiconductors was then determined using Equation (2)

$$(\alpha h\nu)^{1/2} = A(h\nu - E_g) \quad (2)$$

where α , $h\nu$, and A are the absorption coefficient, incident photon energy, and a constant factor, respectively. Accordingly, the E_g of immobilized Mn_2O_3 and Fe_2O_3 in FM and FM/rGO has the values of 1.72 and 2.06 eV (FM), and 1.68 and 2.00 eV (FM/rGO), respectively (Figure 4a). The E_g of Mn_2O_3 and Fe_2O_3 in the fabricated systems demonstrated the slightly shifts as compared to original Mn_2O_3 (1.70 eV) and Fe_2O_3 (2.05 eV) that coincide to previous studies [19, 20, 27, 70]. The unremarkable shifts indicated an insignificant alteration in optical properties of Mn_2O_3 and Fe_2O_3 , but the electron transfer at the interface was possibly

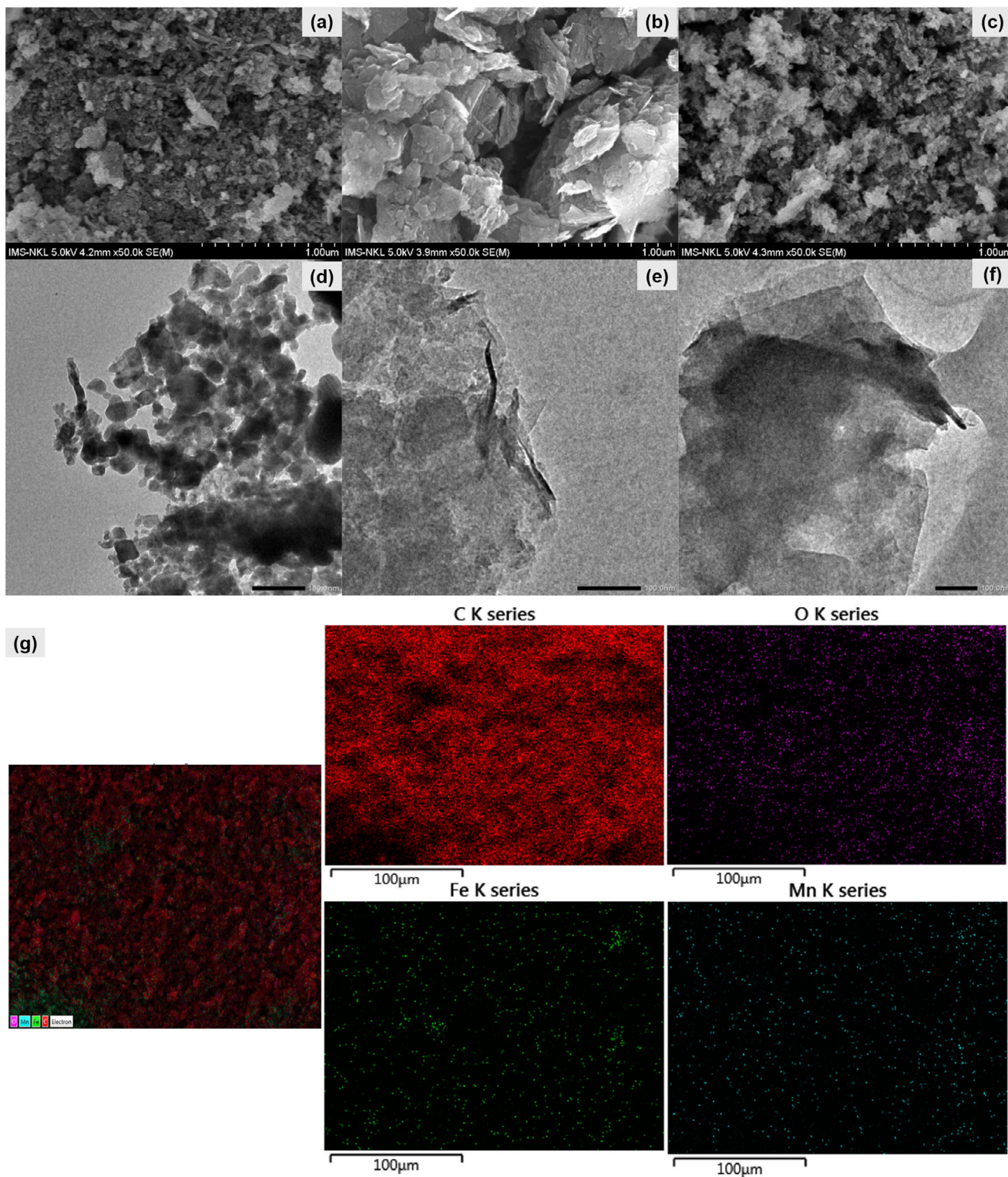


FIGURE 2 | Morphological analysis and elemental mapping of the photocatalysts. (a–c) FE-SEM and (d–f) TEM images of FM, rGO, and FM/rGO samples. (g) Elemental mapping of the FM/rGO sample.

formed between Mn_2O_3 and Fe_2O_3 (in FM heterojunction) as well as FM heterojunction and rGO support. Such synergistic effects are expected to facilitate the mobility of generated charges in the surface of the composite system, thereby improving the photocatalytic performance.

Subsequently, to understand the charge separation and transfer behavior of the prepared materials, the photocurrent response (PCR) (Figure 4b) and the electrochemical impedance spectroscopy (EIS) (Figure 4c) were conducted. These complementary techniques provide direct evidence for the charge carrier

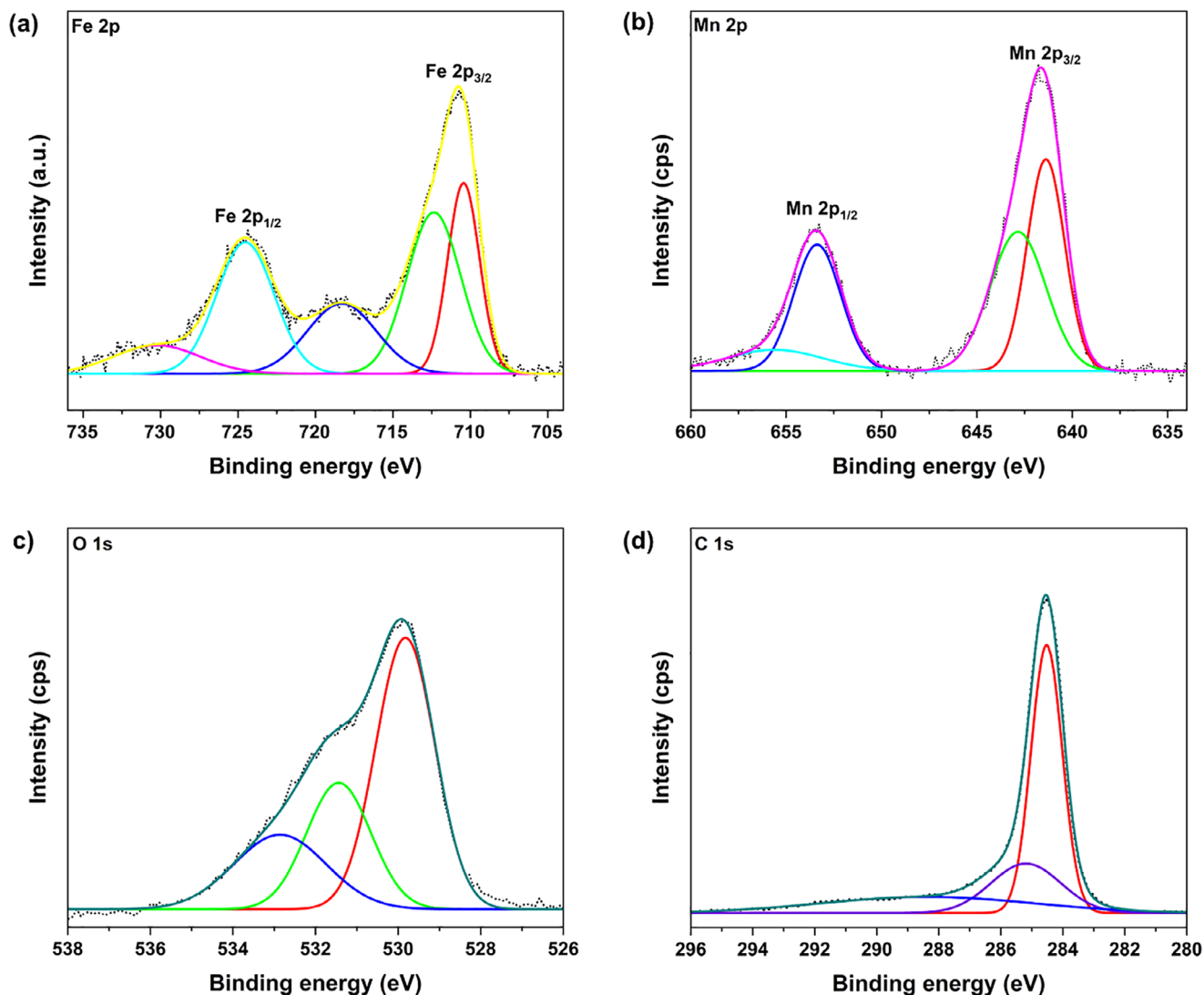


FIGURE 3 | XPS analysis of FM/rGO composite. The high-resolution spectrum of (a) Fe 2p, (b) Mn 2p, (c) O 1s, and (d) C 1s core levels.

generation, migration, and recombination of at the catalyst surface. As shown in Figure 4b, all samples exhibited reproducible and stable photocurrent responses under each irradiation period, confirming their intrinsic photoactivity. Among them, the FM/rGO composite displayed the highest photocurrent density ($\sim 6 \mu\text{A}$), which is significantly higher than those of FM, Fe_2O_3 , and rGO samples. The substantial enhancement in photocurrent for FM/rGO indicates a more efficient generation and separation of e^-/h^+ pairs. This improvement can be assigned to the synergistic effect between FM heterojunction and the conductive rGO network. Specifically, the heterojunction facilitates spatial separation of charge carriers through band alignment, while rGO serves as an electron acceptor and transport pathway, enabling rapid electron migration and suppressing recombination [71]. The Nyquist plots (Figure 4c) further corroborate these findings. Obviously, the smallest semicircle diameter of FM/rGO among all samples indicates the lowest charge transfer resistance (R_{ct}) and the most efficient interfacial charge transport. In contrast, Fe_2O_3 shows the largest semicircle, reflecting sluggish charge mobility and a higher probability of electron-hole recombination,

while the FM heterojunction presents intermediate behavior. The reduced R_{ct} of FM/rGO confirms that rGO significantly improves the electrical conductivity and facilitates faster electron transfer across the catalyst-electrolyte interface [72]. Overall, the combined PCR and EIS analyses clearly demonstrate that the FM/rGO composite possesses effective charge recombination suppression and interfacial charge transfer. The enhancement originates from the synergistic interaction between Mn_2O_3 and Fe_2O_3 in the heterojunction in combination with the rGO support, where rGO not only provides a conductive pathway for electron transport but also contributes as an electron reservoir, thereby promoting migration of charge carriers and effectively limiting their recombination. These characteristics are crucial for improving photocatalytic performance and confirm the advantage of incorporating rGO into heterojunction systems.

In addition, the magnetic properties of the FM and FM/rGO samples were investigated using the vibrating sample magnetometry, as presented in Figure 4d. The FM heterojunction exhibits a typical ferromagnetic-like behavior with a well-defined hysteresis loop, indicating strong magnetic responsiveness. The saturation

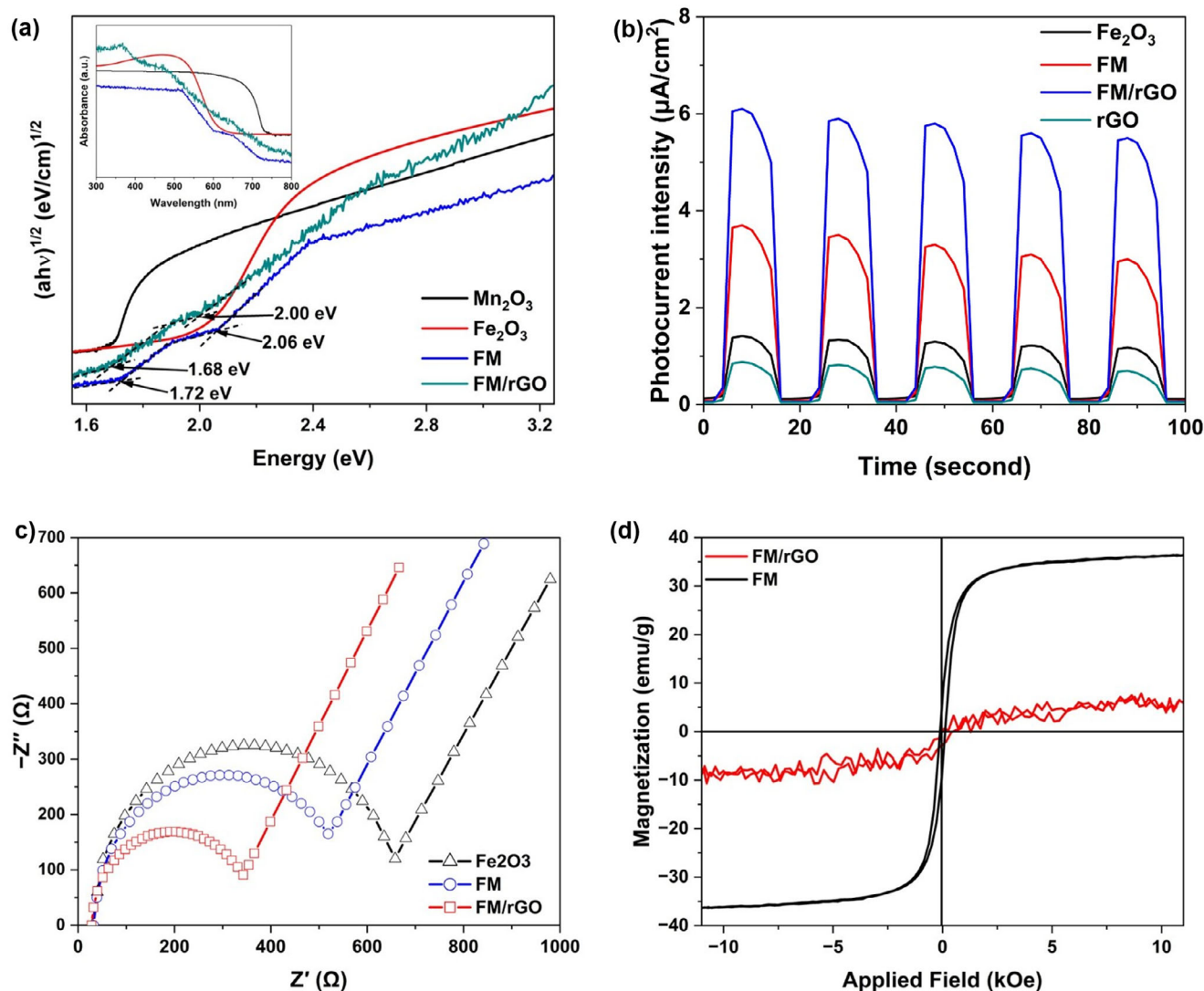


FIGURE 4 | Optical, photoelectrochemical, and magnetic properties of the samples. (a) DRS (inset figure) with the derived Tauc plots, (b) PCR, (c) Nyquist plots, and (d) VSM curves of Fe_2O_3 , FM, and FM/rGO samples.

magnetization of FM reaches approximately 37 emu/g, which is primarily attributed to the presence of iron oxide species and their intrinsic magnetic domains. In contrast, the FM/rGO composite shows a markedly reduced magnetization, fluctuating within 7–10 emu/g. This drop can be reasonably attributed to the incorporation of non-magnetic rGO sheets, which dilute the magnetic component and partially disrupt magnetic interactions between particles. Despite this reduction, the FM/rGO composite still demonstrates a clear magnetic response that is highly advantageous for practical applications for separation after use by applying an efficient magnetic field.

3.2 | Photocatalytic Performance of FM/rGO Composite for Parathion Treatment

The photocatalytic performance of FM/rGO, FM, rGO, and Fe_2O_3 materials toward parathion remediation under visible light was systematically studied (Figure 5a). The experiments illustrate time-dependent photocatalytic activity of all materials.

FM/rGO composite demonstrated the highest DE, followed by FM, Fe_2O_3 , and rGO. For the first 30 min of treatment process, FM/rGO reached 81.46% degradation, whereas FM and Fe_2O_3 only achieved 51.61% and 24.55%, respectively. The gap in photocatalytic performance further widened at longer irradiation times, with FM/rGO achieving 97.47% at 60 min and approaching nearly complete degradation ($\sim 99\%$) at 90 min. In contrast, Fe_2O_3 exhibited moderate photocatalytic activity, reaching only 65.32% degradation after 90 min, while rGO alone showed minimal DE of 11.88%, confirming that rGO is not an active photocatalyst by itself. The FM heterojunction exhibited the improvement in parathion removals as compared to Fe_2O_3 , achieving a DE of 82.66% after 90 min, highlighting the beneficial role of coupling Fe_2O_3 with Mn_2O_3 . Finally, after 2 h of photodegradation process, besides a mostly degradation using FM/rGO composite, FM, Fe_2O_3 , and rGO can correspondingly reach the DEs of 89.88%, 76.43%, and 13.92%, respectively.

Additionally, to quantitatively evaluate the photocatalytic performance, the kinetics of parathion photodegradation were

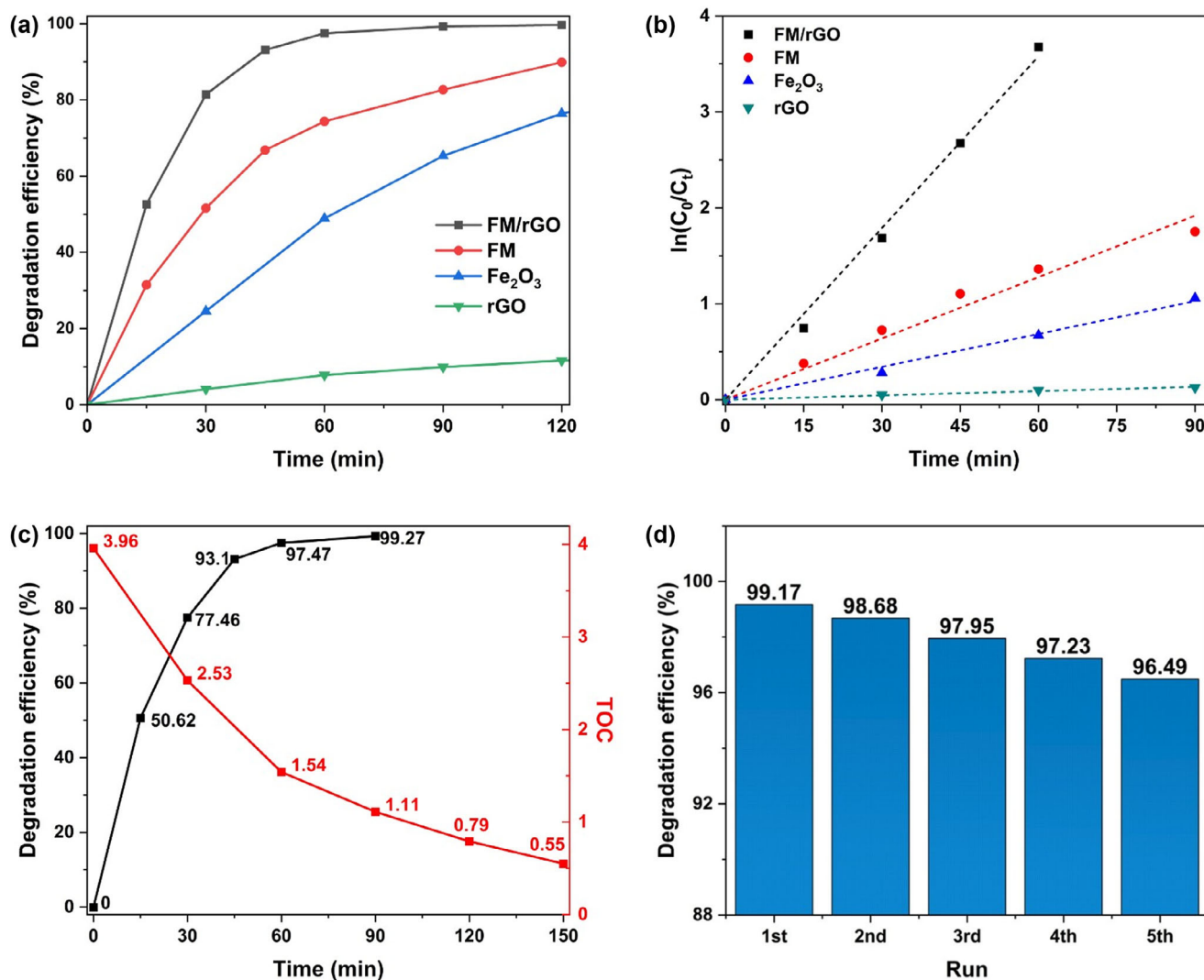


FIGURE 5 | Photocatalytic performance and reusability for parathion degradation. Parathion photodegradation (a) efficiency and (b) rates over FM/rGO, FM, rGO, and Fe_2O_3 . (c) TOC changes and (d) reusability of the FM/rGO photocatalyst for parathion treatment.

evaluated by fitting to the pseudo-first-order model (Equation 3)

$$\ln\left(\frac{C_0}{C_t}\right) = kt \quad (3)$$

where k stands the apparent reaction rate constant, C_0 and C_t represent the parathion concentration at the initial and at the t min, respectively. The linear plots of $\ln(C_0/C_t)$ against irradiation time for each sample with a linear regression $R^2 \approx 0.99$ are shown in Figure 5b. The rate constants decreased in the following order $k_{\text{FM/rGO}} (0.0597 \text{ min}^{-1}) \gg k_{\text{FM}} (0.0211 \text{ min}^{-1}) > k_{\text{Fe}_2\text{O}_3} (0.0114 \text{ min}^{-1}) > k_{\text{rGO}} (0.0015 \text{ min}^{-1})$.

Moreover, the mineralization efficiency, as evaluated by total organic carbon (TOC) removal, provides further insight into the degradation of parathion into inorganic products using FM/rGO. The TOC value shown in Figure 5c steadily decreases as prolonging the irradiation time, indicating a progressive mineralization of the organic pollutant. As observed on the graph, the TOC change showed a slower rate as compared to the DE change.

The difference between DE values and TOC removal at a certain degradation time indicates that intermediate products (smaller organic fragments) formed during the treatment process, and it required additional time to completely mineralize parathion. However, with a mineralization degree of nearly 72% at 90 min and 86% at 150 min, FM/rGO still indicated the effectiveness for not only break but also deeply mineralized parathion molecules under light irradiation.

Generally, the outstanding photocatalytic performance of FM/rGO is primarily credited to synergistic effects. First, the $\text{Fe}_2\text{O}_3/\text{Mn}_2\text{O}_3$ heterojunction promotes the spatial charge separation by driving directional migration of photogenerated charge carriers across the material interface. Second, the rGO carriers significantly enhance the specific surface area of the photocatalysts and provides numerous adsorption sites for parathion molecules through π - π interactions and surface functional groups. This results in a promotion in the interaction between generated oxidative radicals and target contaminants for treatment [73]. Third, with those specific functional groups,

rGO also acts as an effective electron sink, suppressing e^-/h^+ recombination, extending the lifespan of photogenerated charges, and enhancing the electric conductivity of the composite. Overall, these combined effects result in a higher generation rate of ROS, majorly $\bullet OH$ radicals, for the oxidative degradation of parathion [39].

In addition, the recyclability of the FM/rGO composite was evaluated over multiple cycles since the practical applicability of a photocatalyst largely depends on its stability and reusability (Figure 5d). After several cycles, DE slightly decreased from 99.17% in the first cycle to 96.49%. The minor decrease in photocatalytic activity of $\sim 2.7\%$ suggested outstanding stability and resistance to photocorrosion. Furthermore, the XRD curves of FM/rGO samples before and after several uses of parathion photodegradation display the analogous patterns without any major changes, suggesting the good catalyst's durability under investigation conditions. Generally, the slight reduction in DE was possibly attributed to the residues or generated intermediate adsorbed on catalytic centers, the possible aggregation particles, and minimal fouling by environments or photocorrosion.

The photocatalytic DE of parathion is strongly influenced by several operational parameters. Amongst them, the following experiments reported the change of DE for parathion photodegradation using FM/rGO by varying the pH of the solution, the initial concentrations of pollutants, and catalyst dosages (Figure 6). The influence of initial solution pH on the DE of parathion was investigated within a range of 3–11, and the results clearly demonstrate that pH directly affects the photocatalytic performance of FM/rGO. In Figure 6a, DE increases sharply from 61.86% at pH 3 to reach the peak of 98.54% at pH 5, followed by a slight decrease to 97.06% at pH 6. Beyond this range, the DE drops significantly to 72.21% at pH 7 and gradually recovers to 80.78% at pH 11. At acidic media (pH 3–4), the catalytic sites (Fe_2O_3 and Mn_2O_3) on FM/rGO is positively charged (pH < IEP) while parathion ($pK_a \approx 5.2$ [74]) is protonated. This leads to a less effective adsorption of pollutants on the composite due to electrostatic repulsion, subsequently limiting the interaction between them for photodegradation. As the media is less acidic to nearly neutral (pH 5–6), the charge at Fe_2O_3 and Mn_2O_3 sites change to suitably attract either parathion cation (pH < pK_a) or anion (pH > pK_a), that facilitates the adsorption of parathion on the composites. Consequently, catalytic sites and pollutants are well-interacted, promoting the parathion photodegradation. At the neutral to alkaline media (pH ≥ 7 > $IEP_{Fe_2O_3}$ > $IEP_{Mn_2O_3}$), while Fe_2O_3 and Mn_2O_3 are negatively charged and parathion is also in anionic form, the catalytic sites and parathion anions ineffectively interacted, resulting in a significant drop in DE value. However, interestingly, in a slight alkaline environment, a trivial recovery in DE is observed. This enhancement can be ascribed to the increased hydroxide ion (OH^-) concentration that possibly react with h^+ to produce more $\bullet OH$ for parathion degradation [75]. The effect is also described in parathion photodegradation without FM/rGO photocatalyst. However, the efficiency does not reach the level observed at pH 5–6 (Equation 4)



The relationship between initial parathion concentration and the parathion DE was examined within the parathion con-

centration of 7–15 ppm (Figure 6c). The results reveal a clear decreasing trend in DE with increasing parathion concentration, from 99.34% at 7 ppm to 86.72% at 15 ppm. At low concentrations, the active sites on the FM/rGO surface are abundantly sufficient to accommodate most of the parathion molecules, leading to efficient adsorption and degradation. However, when increasing the parathion concentration, the multitude of pollutant molecules noticeably exceeds the maximal capacity of the active sites, resulting in saturated adsorption and intensified competitive binding among the molecules. In addition, at a high concentration of parathion, both the generated and the exceed parathion in the solution with aromatic rings or π -bonds possibly absorb the energy of incident light like a “shield” effect that limits the photoexcitation of the semiconductor composites [76, 77].

The effect of the FM/rGO amounts on parathion degradation was assessed with the catalyst dosage of 0.6–1.2 g/L (Figure 6d). The DE value exhibited an ascending trend from 79.65% at 0.6 g/L to a maximum of 98.24% at an optimal dosage of 1.0 g/L, followed by a marginal decline to 95.77% at 1.2 g/L. Initially, the increment in catalyst dosage provides additional abundance of accessible catalytic active sites. This significantly accelerates the production of reactive radicals, consequently enhancing the overall effectiveness of parathion treatment. However, increasing the FM/rGO content beyond the optimal threshold (1.2 g/L) adversely affected the photocatalytic performance, resulting in a slight reduction in DE. This inverse relationship at excessive catalyst loadings is a well-documented phenomenon governed by physical constraints. Primarily, the elevated particle concentration drastically increases the suspension opacity and turbidity. The increased turbidity causes light scattering, which reduces light penetration and prevents uniform photoactivation of the catalyst [78]. Furthermore, an overloading of the catalyst inevitably exacerbates particle agglomeration in the aqueous phase. This aggregation not only shields the inner active sites from light irradiation but also geometrically diminishes the specific surface area available for target molecule adsorption. Consequently, these detrimental physical shielding effects outweigh the kinetic benefits of added catalytic sites, leading to the observed decline in DE [76].

According to obtained data, in the context of this study, the optimal conditions for parathion photodegradation using FM/rGO should be pH 5–6, a parathion concentration of 7–10 ppm, and a catalyst content of ~ 1.0 g/L. These optimal conditions were also applied for evaluation of parathion photodegradation mechanism for the following experiments.

3.3 | Study on Parathion Photodegradation Mechanism Using FM/rGO Composite

Radical scavenging experiments were performed to identify the primary reactive species responsible for the degradation of parathion (Figure 7a). In the control experiment lacking scavengers, the FM/rGO system demonstrated superior photocatalytic performance, attaining a DE of 98.36%. The decrease in DE after adding selective scavengers helped identify the primary ROS involved in the process. Specifically, IPA nearly interrupted the parathion photodegradation using FM/rGO as

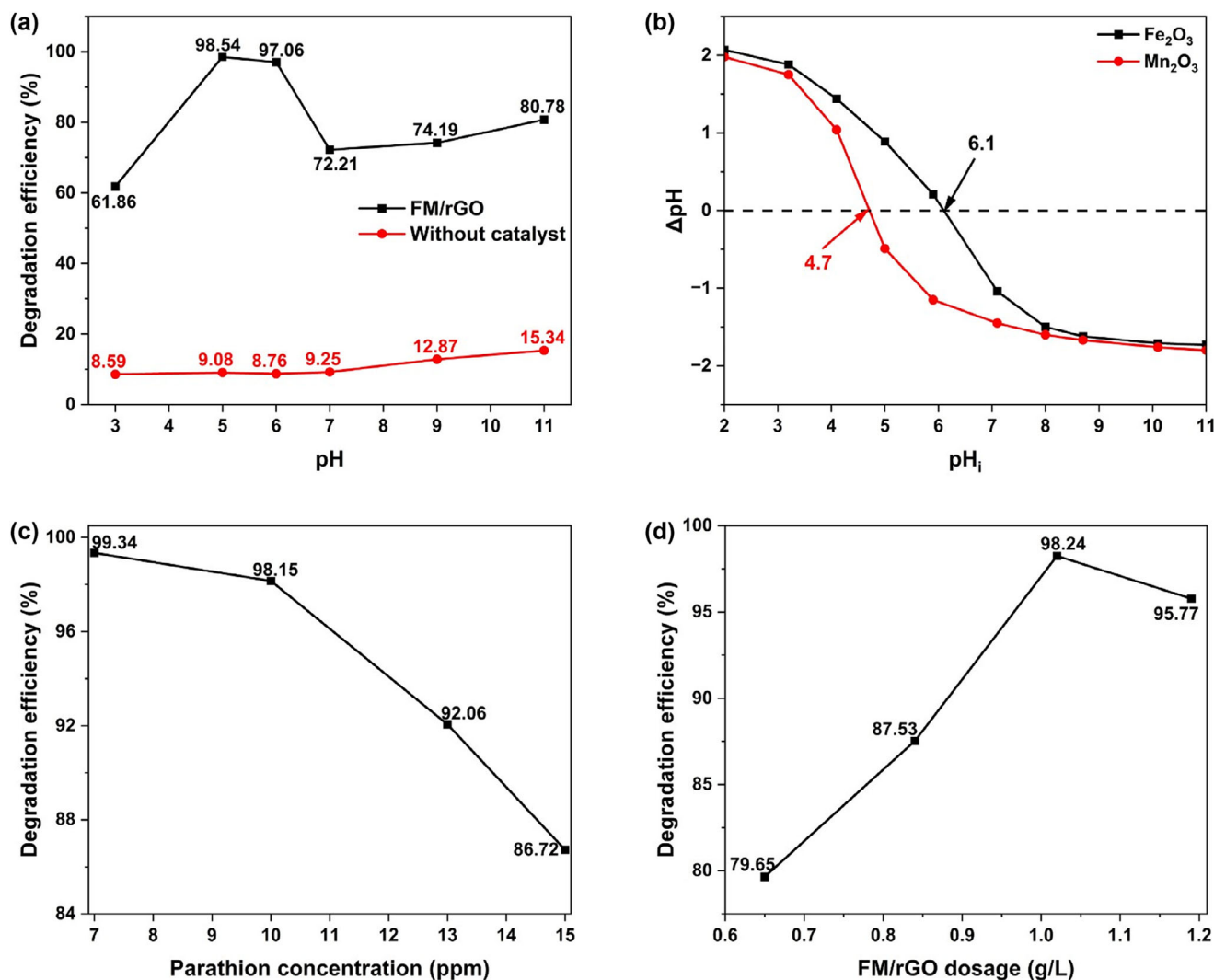


FIGURE 6 | Influences of operational parameters on parathion photodegradation efficiency. Effects of (a) pH, (c) initial parathion concentration, and (d) catalyst dosage on parathion photodegradation using FM/rGO. (b) Isoelectric points of Fe_2O_3 and Mn_2O_3 determined by the drift method.

the DE dropped to 12.74%, indicating a dominant role of $\bullet OH$. Similarly, the hole scavenger, EDTA, significantly suppressed the DE to 39.81%, suggesting that h^+ are also crucial reactive species, either directly oxidizing parathion or indirectly generating $\bullet OH$ radicals. The presence of $AgNO_3$, an electron (e^-) scavenger, resulted in a moderate decrease in DE to 70.28%, implying that electrons contribute to the photocatalytic process but are not the primary active species. Interestingly, contrary to previous study, benzoquinone (BQ), a superoxide radical scavenger, reduced the DE to 68.59%, indicating that $\bullet O_2^-$ also participate in parathion degradation. This observation indicated the feasible formation of $\bullet O_2^-$ is feasible in the FM/rGO composite, which differs from conventional Fe_2O_3 - or Mn_2O_3 -based systems.

The potentials for the CBs and VBs of the semiconductors were typically determined using Equations (5) and (6) [79]

$$E_{VB} = \chi - E_f + 0.5E_g \quad (5)$$

$$E_{CB} = E_{VB} - E_g \quad (6)$$

where χ represents the electronegativity of the materials ($\chi(Fe_2O_3) \approx 5.88$ eV; $\chi(Mn_2O_3) \approx 5.94$ eV) and $E_f = 4.5$ eV. According to the bandgap energies determined by DRS study, the CB and VB positions were defined to be +0.38 and +2.38 eV for Fe_2O_3 , and +0.60 and +2.28 eV for Mn_2O_3 , respectively. The results revealed the formation of a type-I (straddling) heterogeneous photocatalyst system on the prepared composite [80]. Because the VB potential of Fe_2O_3 (+2.38 eV) is more positive than the $H_2O/\bullet OH$ standard potential, hydroxyl radicals are readily generated through the reaction $h^+ + H_2O \rightarrow \bullet OH$. Conversely, the calculated CB potentials of both Fe_2O_3 and Mn_2O_3 are theoretically less negative than the standard single-electron reduction potential of $O_2/\bullet O_2^-$ (-0.33 V vs. NHE). Hence, it is proposed that the excited electrons rapidly migrate to the highly conductive rGO layers, where the unique electronic microenvironment and the presence of localized defect sites lower the thermodynamic barrier, thereby facilitating the subsequent multi-electron reduction of dissolved oxygen to generate $\bullet O_2^-$ radicals [81]. Overall, the parathion photodegradation can be described as follow (Equations 7–13) (Figure 7b)

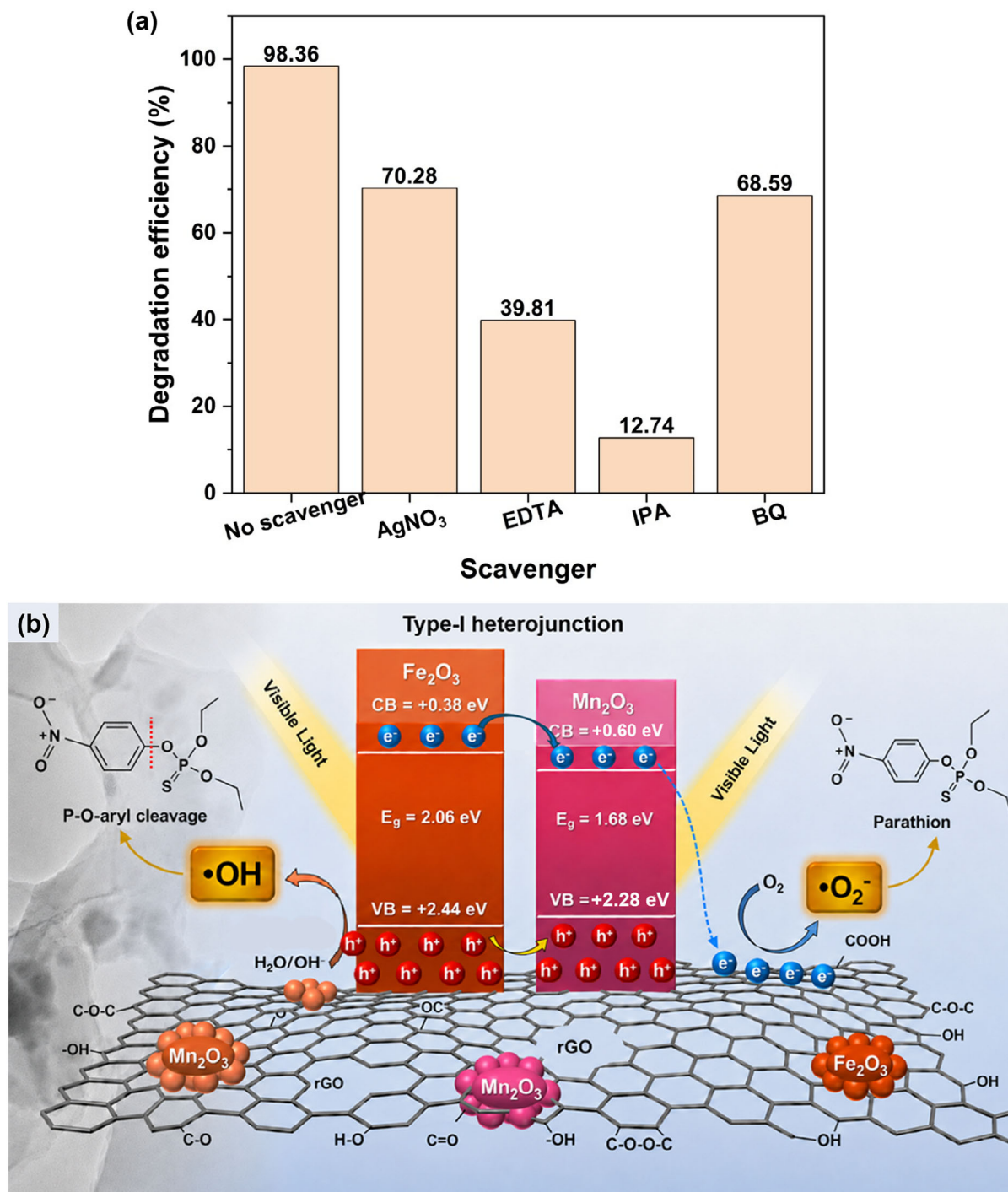
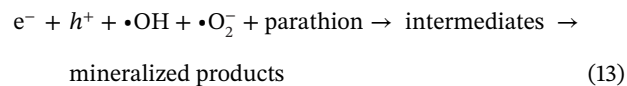
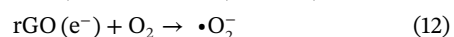
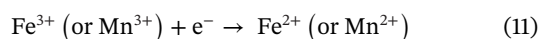
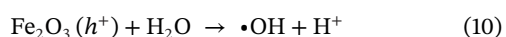
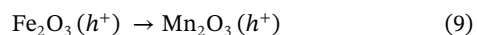
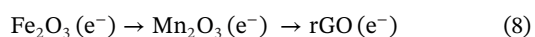
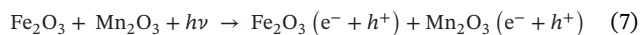


FIGURE 7 | Mechanistic investigation of parathion photodegradation under solar light. (a) Parathion photodegradation under solar light using the FM/rGO composite in the presence of scavengers. (b) Plausible degradation mechanisms.



The photocatalytic degradation of parathion over FM/rGO proceeds through a multistep transformation pathway rather than direct mineralization, as evidenced by the slower reduction in TOC compared to the DE. GC/MS techniques would be once

TABLE 1 | Identified intermediates of parathion photocatalytic degradation using FM/rGO.

Order	Possible generated intermediates		
	Compounds	Retention time R_t (min.)	Characteristic ions (m/z)
I	4 – ethyl – 4 – heptanol	5.640	115, 101, 83
II	1-Norvaline, N-methoxycarbonyl-, hexyl ester	6.173	131, 87
III	2-Methylpentan-3-yl propyl succinate	6.543	143, 101, 85
IV	2,6-Dimethyl-3,5- heptanedione	7.417	113, 89, 85
V	Ethyl 3-ethoxyacrylate	8.004	99, 87, 71, 58
VI	2-Methylhept-6-en-1-ol	8.318	128, 110, 95, 71, 58
VII	5-Methyl-4-octanone	8.701	85, 71
VIII	1-(3 4-Dimethoxyphenyl) ethanone	9.043	180, 165, 123
IX	4,4-Dimethyl heptanedioic acid	9.493	115, 97
X	Butyl dimethyl phosphate	12.309	127, 153, 109, 97, 71
XI	Butyl neopentyl butylphosphonate	14.960	153, 95
XII	3,8-Dimethyldecane,	21.111	85, 71, 57
XIII	5,5-Dimethylnonane	21.905	99, 85, 71, 57
XIV	3-Methyl heneicosane	22.665	85, 71, 57, 43

employed to verify the formation of those fragments. Figure S4 presents the GC diagrams and of photoproducts at 0, 15, 30, and 60 min of parathion photodegradation using FM/rGO. According to the chromatograms, at the retention time of 17.7 min, the peaks are assigned to parathion, which exhibits a significant decrease in integrated peak areas over time. As previously mentioned, hydroxyl radicals predominate in the photodegradation of parathion on FM/rGO, indicating that oxon derivatives are the primary transformation products [82]. It is well established that photocatalytic oxidation preferentially cleaves the P–O–aryl bond, producing *p*-nitrophenol intermediate [83]. In addition, besides the major fragments with m/z of 109, 209, and 291 units (Figure S5), several intermediates prior to the subsequent oxidation of aromatic species through hydroxylated intermediates such as hydroquinone and benzenetriol, followed by ring-opening reactions to generate low-molecular-weight aliphatic compounds [9, 84] (Table 1). A proposed degradation pathway is reported in Figure 8.

4 | Conclusion

The FM/rGO nanocomposite was successfully synthesized via a microwave-assisted coprecipitation method to enhance the photocatalytic degradation of ethyl parathion. Structural and morphological analyses confirmed the uniform distribution of

Fe₂O₃/Mn₂O₃ NPs on rGO sheets, leading to increased surface area and improved adsorption capability. XPS analysis revealed mixed valence states (Fe³⁺/Fe²⁺ and Mn³⁺/Mn²⁺), indicating potential redox cycling, while optical and electrochemical measurements demonstrated enhanced visible-light absorption, improved photocurrent response, and reduced charge transfer resistance, confirming efficient charge separation and migration. The FM/rGO composite exhibited outstanding photocatalytic performance, delivering nearly 100% degradation of ethyl parathion within just 60 min of visible-light irradiation and achieving a high mineralization degree of ~86%. The reaction kinetics were well-fitted with a pseudo-first-order model ($k = 0.0597 \text{ min}^{-1}$), significantly outperforming the pristine oxides and binary counterparts. The superior activity is attributed to the synergistic interaction between the FM heterojunction and the conductive rGO layers. The heterojunction facilitates directional charge transfer, while rGO acts as an electron reservoir and transport pathway, efficiently suppressing recombination of excited electrons and holes, and enhancing ROS generation. Mechanistic studies revealed that hydroxyl radicals ($\bullet\text{OH}$) are the dominant species responsible for parathion degradation, with additional contributions from photogenerated holes and superoxide radicals. The photocatalytic performance was influenced by operational parameters, with optimal conditions identified at pH 5–6, pollutant concentration of 7–10 ppm, and catalyst dosage of ~1.0 g/L. Furthermore, the FM/rGO composite demonstrated

writing – original draft. **Vu Ngoc Mai Nguyen:** investigation, formal analysis. **Quang Khieu Dinh:** formal analysis, data curation. **Minh Phuong Nguyen:** supervision, validation. **Quang Bac Nguyen:** software, visualization. **Thi Ha Chi Nguyen:** investigation, formal analysis. **Minh Tri Truong:** resource, software. **Ngoc Nhiem Dao:** resource, data curation, methodology. **Trung Kien Nguyen:** investigation, formal analysis, writing – review & editing.

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Data Availability Statement

The datasets generated and/or analyzed during the current study are available within the published article and its [Supporting Information](#) files. Additional raw data is available from the corresponding author upon reasonable request.

Conflicts of Interest

The authors declare no conflicts of interest.

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Supporting Information

Additional supporting information can be found online in the Supporting Information section.

Supporting File 1: slct74435-sup-0001-SuppMat.docx.