



Original Article

Nanocomposite of TiO_2 and Phase-transition-derived $\alpha\text{-Fe}_2\text{O}_3$ for Enhancing Photocatalytic Dye Degradation

Dinh Tan Muon^{1,2}, La Phan Phuong Ha³, Dang Vinh Quang³, Nguyen Thien Trang^{1,2},
Nguyen Hoang Hung^{1,2}, Tran Viet Cuong^{1,2}, Huynh Tran My Hoa^{1,2,*}

¹VKTECH Research Center, NTT Hi-Tech Institute, Nguyen Tat Thanh University,
Vo Chi Cong Street, Tang Nhon Phu Ward, Ho Chi Minh City, Vietnam

²Nguyen Tat Thanh University Center for Hi-Tech Development, Saigon Hi-Tech Park,
Vo Chi Cong Street, Tang Nhon Phu Ward, Ho Chi Minh City, Vietnam

³VNU-HCM University of Science, Nguyen Van Cu, Cho Quan, Ho Chi Minh City, Vietnam

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Abstract: Although $\alpha\text{-Fe}_2\text{O}_3$ is a promising photocatalyst for Advanced Oxidation Processes in dye-polluted wastewater treatment, its practical efficiency is limited by inherent drawbacks including rapid electron-hole recombination and poor electron mobility. This study proposes a $\text{TiO}_2/\alpha\text{-Fe}_2\text{O}_3$ nanocomposite system to enhance dye removal efficiency of $\alpha\text{-Fe}_2\text{O}_3$, specifically Methylene Blue (MB), in which hierarchical porous, coral-like $\alpha\text{-Fe}_2\text{O}_3$ was prepared via a thermal phase transition process from $\gamma\text{-Fe}_2\text{O}_3$. X-ray diffraction analysis and peak deconvolution were used to confirm that the phase transition to $\alpha\text{-Fe}_2\text{O}_3$ completed after 3 hours of annealing at 800 °C. With the combination of TiO_2 and $\alpha\text{-Fe}_2\text{O}_3$, the dye degradation efficiency increased significantly, demonstrating superior photocatalytic performance under visible light compared to the conventional photo-Fenton process observed for pure $\alpha\text{-Fe}_2\text{O}_3$. In detail, this nanocomposite system achieved nearly 100% degradation of 20 ppm MB within 2 hours, reaching the degradation rate constant of 1.5595 h^{-1} which was considerably higher than that of $\alpha\text{-Fe}_2\text{O}_3$ ($k = 0.4563 \text{ h}^{-1}$). Furthermore, $\text{TiO}_2/\alpha\text{-Fe}_2\text{O}_3$ proved to be durable, maintaining over 90% efficiency after five reuse cycles with MB concentration of 40 ppm with negligible structural degradation. This enhanced performance is proposed due to the synergistic photocatalytic effect, including photo-Fenton and photocatalytic pathways, which comes from the favorable band alignment of TiO_2 and $\alpha\text{-Fe}_2\text{O}_3$. These findings contributed that the $\text{TiO}_2/\alpha\text{-Fe}_2\text{O}_3$ is promising catalyst with great potential for practical applications in solar-driven treatment of dye-polluted wastewater.

Keywords: phase transition; $\alpha\text{-Fe}_2\text{O}_3$; TiO_2 ; methylene blue, dye degradation.

* Corresponding author.

E-mail address: htmhoa@ntt.edu.vn

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1. Introduction

Rapid industrialization has led to the discharge of large volumes of wastewater containing hazardous pollutants, which threatens both human health and environmental sustainability. Among these, synthetic organic dyes from industries, especially in textiles and paper, are of primary concern [1-3]. These dyes are engineered for stability, making them resistant to biodegradation and allowing them to persist in aquatic ecosystems. To address this, various eco-friendly remediation strategies have been explored such as the use of activated carbon and carbon nanofiber combined with cerium oxide (CeO_2) for pollutant removal [4]. Concurrently, recent research has also highlighted advanced systems for water remediation through the tuning of magnetic properties in Gd-doped CeO_2 and the development of multiferroic BiFeO_3 nanoparticles for the efficient and rapid sunlight-driven degradation of Methylene Blue (MB) [5, 6]. Furthermore, general strategies for enhancing catalytic efficiency, including modification of electronic structure and interface engineering, have been successfully demonstrated in nickel-based heterostructures for energy-related applications [7]. However, to ensure complete mineralization rather than simple phase transfer, Advanced Oxidation Processes (AOPs) have emerged as promising candidates for wastewater treatment [8-11]. AOPs utilize catalytic nanomaterials to generate highly reactive oxygen species (ROS) like hydroxyl radicals ($\cdot\text{OH}$), singlet oxygen ($^1\text{O}_2$), superoxide anions ($\text{O}_2^{\cdot-}$). These ROS are powerful oxidizing agents which can degrade complex organic dyes into simpler, less toxic compounds. The use of nanomaterials in AOPs is driven by their exceptional properties, such as high specific surface area, tunable morphology, and excellent reusability, leading to the development of highly efficient systems. For example, Kumar *et al.* reported a Z-scheme system of $\text{CeO}_2\text{-TiO}_2\text{@CNT}$ that achieved over 99% removal of mixed antibiotics and pollutants [12]. Similarly, Wang *et al.* introduced a MOF-on-MOF membrane which could degrade rhodamine B (RhB) selectively with the efficiency exceeding 90%, primarily through the generation of $^1\text{O}_2$. These studies highlight the significant potential of nanomaterial-based AOPs for practical water remediation.

Within AOPs, the Fenton reaction is a well-established method for wastewater treatment [13, 14]. With the presence of hydrogen peroxide (H_2O_2) and Fe^{2+} ions, $\cdot\text{OH}$ free radicals are generated and then participate in the pollutants' decomposition. Moreover, the efficiency of this process is significantly enhanced by light, in a method known as the photo-Fenton process. For these applications, iron(III) oxide (Fe_2O_3) has emerged as a promising catalyst due to its low cost, natural abundance, and stability. It has four crystalline phases, including $\alpha\text{-Fe}_2\text{O}_3$, $\beta\text{-Fe}_2\text{O}_3$, $\varepsilon\text{-Fe}_2\text{O}_3$ and $\gamma\text{-Fe}_2\text{O}_3$ [15]. However, $\beta\text{-Fe}_2\text{O}_3$ and $\varepsilon\text{-Fe}_2\text{O}_3$ are rare and mostly exist in the laboratory [16, 17]. Likewise, $\gamma\text{-Fe}_2\text{O}_3$, known as maghemite, is easily magnetized, making it responsive to external magnetic fields [18]. Compared to other forms, $\alpha\text{-Fe}_2\text{O}_3$ has been shown to be a structurally stable material with available sources [19, 20], helping it attract more attention and research. In addition, its favorable electronic band structure compared to $\gamma\text{-Fe}_2\text{O}_3$ facilitates more efficient charge carrier generation, enabling them to participate in photocatalytic reactions and increase overall efficiency [21, 22]. Notably, $\alpha\text{-Fe}_2\text{O}_3$ can be synthesized by many methods such as sol-gel [23, 24], hydrothermal [25-27], solvothermal [28], thermal decomposition [29, 30], and phase transition [31, 32]. This allows the synthesis of $\alpha\text{-Fe}_2\text{O}_3$ with different morphologies and sizes, leading to tunable bandgap properties [33]. This feature is crucial for practical applications, as it is able to utilize the energy from natural sunlight. Among those synthesis methods, the thermally induced phase transition is a unique and simple way to produce $\alpha\text{-Fe}_2\text{O}_3$ from $\gamma\text{-Fe}_2\text{O}_3$. This offers a promising strategy for the valorization of $\gamma\text{-Fe}_2\text{O}_3$, which is abundantly available in natural maghemite deposits and industrial metal scrap [18, 34, 35]. However, understanding how this transition pathway influences the catalytic performance is crucial for valorizing $\gamma\text{-Fe}_2\text{O}_3$ sources but remains less explored compared to other chemical methods.

Despite these advantages, Fe-based oxides including α -Fe₂O₃ generally suffer from two major drawbacks that limit their photocatalytic performance, including rapid recombination of photogenerated charges and low mobility of electrons [36, 37]. A common strategy is to form heterojunctions by combining Fe-based oxides with other functional materials, such as magnetic Fe₂O₃/CNT nanocomposites and core-shell CeO₂@Fe₂O₃ architectures, which show improved efficiency in the photocatalytic degradation of organic dyes [38, 39]. Furthermore, specialized α -Fe₂O₃ heterostructures have been proven to enhance charge separation and overall efficiency specifically for the photocatalytic process. For instance, Bi et al. created a biphasic α - γ -Fe₂O₃ heterostructure that improved the efficiency of RhB degradation up to 78% [21]. In another approach, Alp et al. reported a CuO/ α -Fe₂O₃ composite that completely degraded RhB within an hour under visible light [40]. Similarly, Azimi et al. designed an α -Fe₂O₃@TiO₂@SO₃H catalyst that demonstrated enhanced photo-Fenton activity, removing over 94.78% of chemical oxygen demand and 99.9% of turbidity from wood wastewater [41]. These studies demonstrate that engineering α -Fe₂O₃-based heterojunctions is a highly effective approach for boosting catalytic activity.

In this work, a TiO₂/ α -Fe₂O₃ nanocomposite was successfully synthesized in which α -Fe₂O₃ was derived from γ -Fe₂O₃ via a thermal phase transition. Although the combination of TiO₂ with Fe-based oxides has been extensively explored due to its stability, low toxicity, and favorable band alignment especially with α -Fe₂O₃ [42, 43], previous reports have primarily utilized complex modifications such as utilizing Z-scheme models or noble metal doping strategies [44-47]. Here, we employed a straightforward phase transition method to obtain α -Fe₂O₃ from a high-purity γ -Fe₂O₃ synthesized via the co-precipitation method. This process resulted in a unique hierarchical porous, coral-like α -Fe₂O₃ that serves as a scaffold for the uniform distribution of TiO₂ nanoparticles, facilitating the effective interfacial interaction between both materials. The resulting system demonstrated significantly enhanced photocatalytic activity for MB degradation under visible light, potentially attributed to the synergistic effect of photo-Fenton and photocatalytic processes of TiO₂/ α -Fe₂O₃ nanocomposite.

2. Experimental Methods

2.1. Investigation of Phase Transition Process of α -Fe₂O₃ from γ -Fe₂O₃ by Thermal Treatment

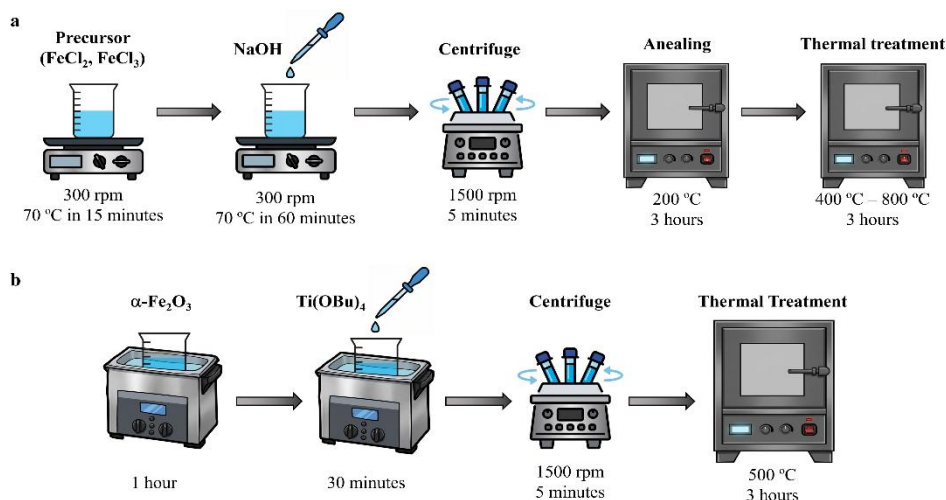


Figure 1. The schematic illustration of the phase transition synthesis of α -Fe₂O₃ using thermal treatment a); and the synthesis process of the TiO₂/ α -Fe₂O₃ nanocomposite b).

This study investigated the effect of annealing temperature on the phase transition of γ -Fe₂O₃ to α -Fe₂O₃. The γ -Fe₂O₃ was first prepared via a co-precipitation method, as illustrated in Fig. 1a. An 80 mL aqueous solution containing 0.125 M FeCl₂ (Sigma-Aldrich, 98%) and 0.25 M FeCl₃ (Sigma-Aldrich, 97%) was stirred at 300 rpm and heated to 70 °C. After 15 minutes, 5 M NaOH was slowly added to the solution, and stirring was continued for an additional 60 minutes. The resulting precipitate was collected by centrifugation (1500 rpm for 15 minutes) and washed thoroughly with distilled water before being annealed at 200 °C for 3 hours to obtain γ -Fe₂O₃. Finally, to induce the formation of α -Fe₂O₃, the prepared γ -Fe₂O₃ was subjected to thermal treatment at temperatures ranging from 400 °C to 800 °C for 3 hours.

2.2. TiO₂/α-Fe₂O₃ Nanocomposite Preparation

The synthesis procedure for the TiO₂/α-Fe₂O₃ nanocomposite is illustrated in Fig. 1b. Firstly, 0.15 g of α-Fe₂O₃ was dispersed in 50 mL of distilled water by ultrasonication for 1 hour to form a stable suspension. Subsequently, 1 mL of titanium(IV) butoxide (Ti(OBu)₄, Sigma-Aldrich, 97%) was added dropwise into the α-Fe₂O₃ suspension at room temperature to form TiO₂ via hydrolysis. To ensure uniform coating of TiO₂ onto the α-Fe₂O₃ surface, the mixture was sonicated for an additional 30 minutes. Finally, the resulting precipitate was collected via centrifugation (1500 rpm for 15 minutes) and washed thoroughly with distilled water, followed by annealing at 500 °C for 1 hour.

2.3. Material Characterizations

The crystalline structure of the synthesized materials was investigated by XRD patterns using a D8 Advance-Bruker diffractometer (Cu-Kα radiation of $\lambda = 0.154$ nm, voltage of 40 kV and current of 100 mA). The surface morphology and elemental composition were analyzed by scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDX), respectively, using a Hitachi S-4800 instrument. Regarding the optical properties of the materials, JASCO V550 UV-Vis and HORIBA XploRA One Raman Spectrophotometers were utilized in this study. Additionally, nitrogen adsorption-desorption isotherms were measured using a BELSORP MAX instrument, which helps to further understand the properties of α-Fe₂O₃. Finally, Raman and XRD analyses were also used to investigate the structural integrity of the material after dye degradation experiments.

The phase transition from γ -Fe₂O₃ to α -Fe₂O₃ was analyzed using X-ray diffraction (XRD) patterns, specifically in the region where an overlap occurs at the (311) facet of γ -Fe₂O₃ and the (110) facet of α-Fe₂O₃. This allows quantifying both phases of Fe₂O₃ based on the deconvolution technique as previously reported by Bi et al., [21]. This semi-quantitative analysis was based on the key assumption that the X-ray scattering factors for these specific facets of both materials are equivalent. Under this assumption, the relative fraction of α-Fe₂O₃ was determined using the following equation:

$$\% \alpha - \text{Fe}_2\text{O}_3 = \frac{I_\alpha}{I_\alpha + I_\gamma} \times 100\% \quad (1)$$

where I_γ and I_α represent the diffraction intensities of γ -Fe₂O₃ (311) facet and α-Fe₂O₃ (110) facet, respectively. These facets were also utilized for crystallite size (D) calculation, following the Scherrer formula [48]:

$$D = \frac{0.9 \times \lambda}{\beta \times \cos(\theta)} \quad (2)$$

where λ is the wavelength of XRD source (1.541 Å), β is the full width at half maximum of the γ -Fe₂O₃ (311) and α -Fe₂O₃ (110) peaks, and θ is the Bragg angle.

2.4. Dye Degradation Experiments

The dye degradation experiments of TiO₂/ α -Fe₂O₃ nanocomposite were conducted using MB through the photocatalytic process under the irradiation of visible light (Osram 64260, 30W). Briefly, 50 mg of synthesized TiO₂/ α -Fe₂O₃ nanocomposite was added to 20 mL of MB solution with the concentration of 20 ppm. This mixture was adjusted to pH = 3.0 with H₂SO₄ before being placed in the dark for 2 hours to establish adsorption-desorption equilibrium. After this step, 0.1 mL H₂O₂ (30 %wt., Sigma-Aldrich) was added to the mixture, which was then irradiated for 4 hours. The samples were also analyzed hourly to monitor the reaction. The remaining concentration of MB was determined by measuring the absorbance of its characteristic peak at 664 nm using a JASCO V670 UV-Vis spectrometer. From this analysis data, the degradation efficiency was calculated using Eq. 3, and the pseudo-first-order rate constant (k) was determined following the Langmuir-Hinshelwood (L-H) model, using Eq. 4.

$$\% \text{Degradation} = \frac{C_0 - C}{C_0} \times 100\% \quad (3)$$

$$\ln \left(\frac{C}{C_0} \right) = -k \times t \quad (4)$$

where C_0 and C are the initial MB concentration and the MB concentration at time “ t ”, respectively.

For comparison, control experiments were conducted in the same procedure, including a blank test for photolysis, and experiments using H₂O₂ with and without α -Fe₂O₃ to investigate the effect of H₂O₂ and the efficiency of conventional photo-Fenton process, respectively. Moreover, experiments were conducted at different initial MB concentrations (40 ppm and 60 ppm) to investigate the dye degradation efficiency of TiO₂/ α -Fe₂O₃ nanocomposite according to the initial concentration. Additionally, the removal of MB in 5 cycles of this catalyst was also studied for the reusability assessment, an important factor for practical applications.

3. Results and Discussions

3.1. Phase Transition Process from γ -Fe₂O₃ to α -Fe₂O₃

The effect of annealing temperature on the phase transition from γ -Fe₂O₃ to α -Fe₂O₃ is shown in the XRD patterns in Fig. 2a. Before the thermal treatment stage, γ -Fe₂O₃ was completely synthesized after annealing at 200 °C, with diffraction peaks corresponding to the (220), (311), (400), (511), and (440) planes of the γ -Fe₂O₃ cubic crystal structure referenced to the Crystallography Open Database (COD: 9006316). After the thermal treatment, while only minor changes were apparent at 400 °C, signs of the phase transition emerged at intermediate temperatures from 500 °C to 700 °C. At 800 °C, γ -Fe₂O₃ was fully converted to the rhombohedral α -Fe₂O₃ phase, identified by its characteristic peaks including (012), (104), (110), (013), (024), (116), (214), and (300) (COD: 9000139). This transition is highlighted in Fig. 2b, which focuses on the 2θ region around 35.75° as it shows the overlapping peaks from the γ -Fe₂O₃ (311) facet and the α -Fe₂O₃ (110) facet.

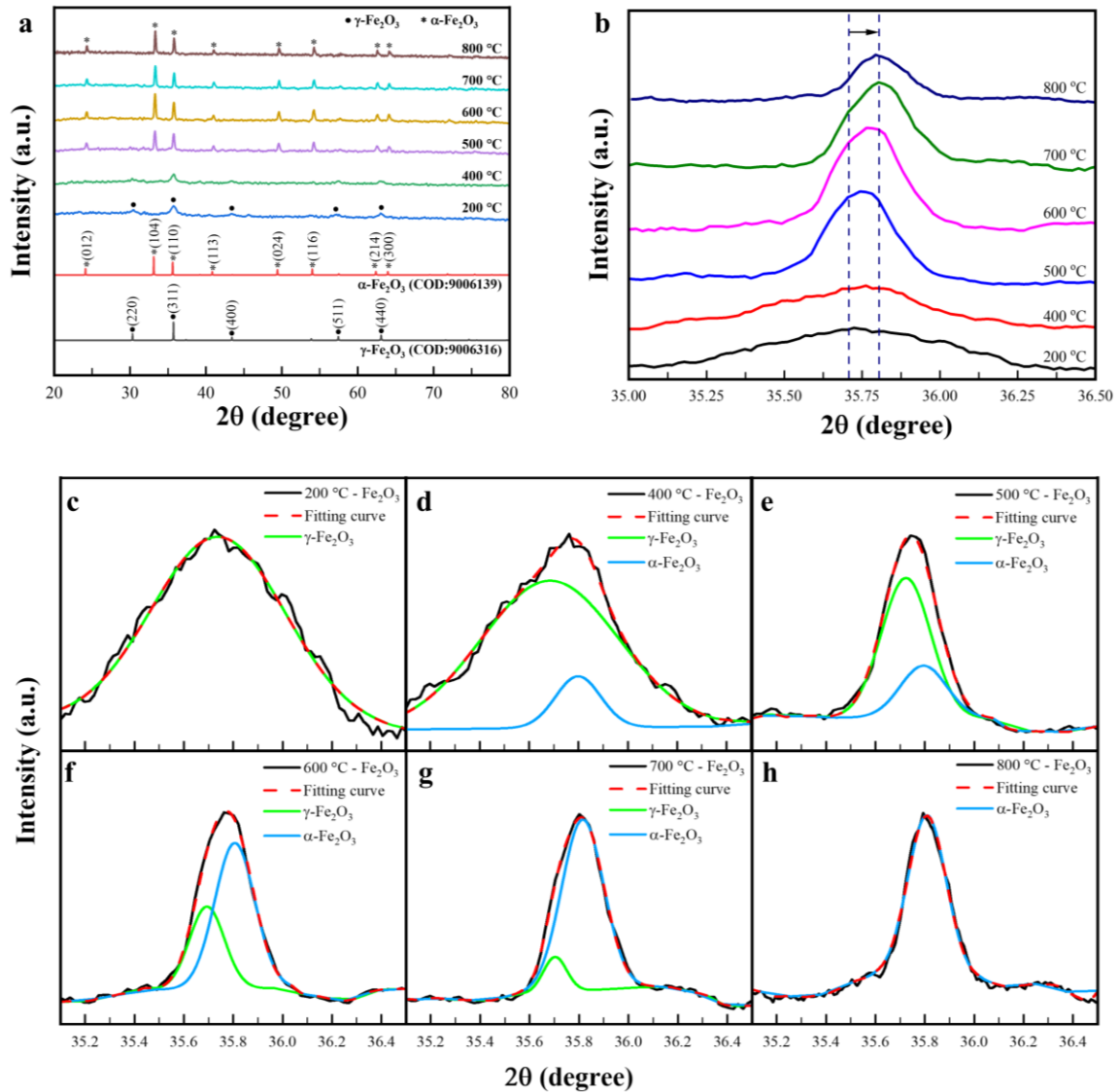


Figure 2. XRD patterns of Fe_2O_3 synthesized at different temperatures (a); XRD peak shifts of $\gamma\text{-Fe}_2\text{O}_3$ (311) to $\alpha\text{-Fe}_2\text{O}_3$ (110) with increasing temperature (b); the deconvolution of XRD peaks at around 35.75° to identify $\alpha\text{-Fe}_2\text{O}_3$ (110) and $\gamma\text{-Fe}_2\text{O}_3$ (311) (c-h).

The crystallite sizes of the $\gamma\text{-Fe}_2\text{O}_3$ and $\alpha\text{-Fe}_2\text{O}_3$ phases calculated using their respective characteristic (311) and (110) facets are summarized in Table 1. The results indicate that the $\gamma\text{-Fe}_2\text{O}_3$ crystallite size increases continuously with rising annealing temperature. This trend is attributed to thermal aggregation and crystal growth prior to the complete transformation into $\alpha\text{-Fe}_2\text{O}_3$ [49]. In contrast, the $\alpha\text{-Fe}_2\text{O}_3$ crystallite size fluctuates at intermediate temperatures before stabilizing at 700°C, leading to the complete structural transition at 800°C. This behavior is likely attributed to the structural reorganization of the $\alpha\text{-Fe}_2\text{O}_3$ lattice during the ongoing phase transition from $\gamma\text{-Fe}_2\text{O}_3$. The completion of this transformation is evidenced by the progressive disappearance of the $\gamma\text{-Fe}_2\text{O}_3$ characteristic peaks and the concurrent enhancement of the $\alpha\text{-Fe}_2\text{O}_3$ diffraction signals.

Table 1. Crystallite size derived from the Scherrer equation for the γ -Fe₂O₃ (311) facet and the α -Fe₂O₃ (110) facet.

Annealing Temperature (°C)	Crystallite size (nm)	
	γ -Fe ₂ O ₃	α -Fe ₂ O ₃
200	11.54	-
400	12.87	38.80
500	34.86	34.84
600	48.01	47.21
700	68.88	42.13
800	-	42.73

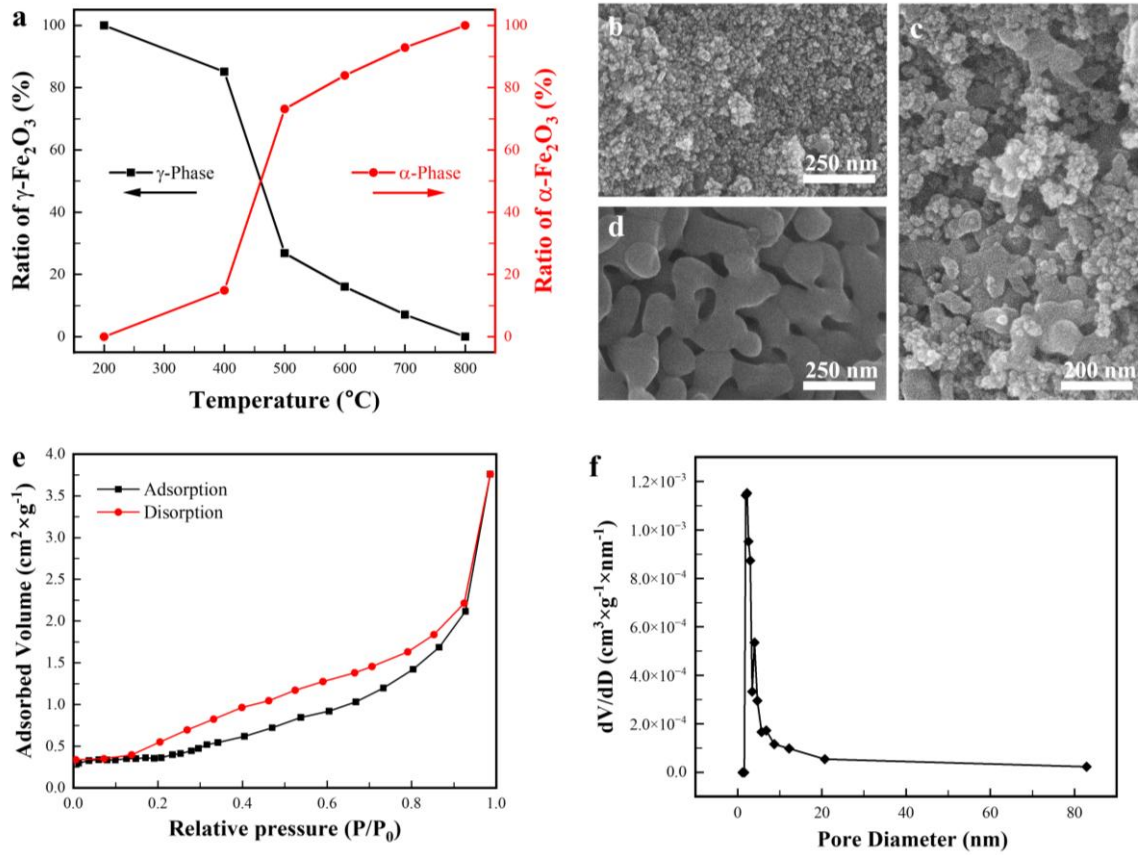


Figure 3. The relative ratio of γ -Fe₂O₃ and α -Fe₂O₃ as a function of synthesis temperature (a); SEM images of γ -Fe₂O₃ synthesized at 200 °C (b), biphasic γ - α -Fe₂O₃ at 500 °C (c), and α -Fe₂O₃ at 800 °C (d); Nitrogen adsorption-desorption isotherm (e), and the pore size distributions (f) of α -Fe₂O₃.

To semi-quantitatively estimate the relative phase evolution, the overlapping XRD peak around 35.75° was analyzed using Gaussian deconvolution, as shown in Fig. 2c-h. This process resolved the individual contributions from the γ -Fe₂O₃ (311) facet and the α -Fe₂O₃ (110) facet. As the annealing temperature increased, the intensity of the γ -Fe₂O₃ peak diminished while the α -Fe₂O₃ peak emerged and grew, clearly illustrating the phase conversion. Based on the intensities of these deconvoluted peaks, the relative fraction of each phase was calculated and plotted as a function of temperature in Fig. 3a.

The results reveal that the transition from $\gamma\text{-Fe}_2\text{O}_3$ to $\alpha\text{-Fe}_2\text{O}_3$ accelerates significantly above 500 °C. This conclusion is further supported by the changes in morphology observed in the SEM images (Fig. 3b-d). At 200 °C, the $\gamma\text{-Fe}_2\text{O}_3$ material consisted of uniform nanoparticles with a size range of 15–20 nm (Fig. 3b). As the annealing temperature increased, these particles began to aggregate. At 500 °C, a bi-phasic morphology was evident (Fig. 3c), where larger, more crystalline structures of $\alpha\text{-Fe}_2\text{O}_3$ appeared alongside the original nanoparticles, consistent with the XRD results. Finally, after annealing at 800 °C, the morphology had completely transformed into a porous, coral-like shape (Fig. 3d). This morphological evolution aligns with the XRD crystallite size calculations (Table 1), where the particle aggregation observed at 500 °C validates the thermal growth trend, and the morphology at 800 °C confirms the complete phase transition.

In addition, the N_2 adsorption-desorption isotherm of $\alpha\text{-Fe}_2\text{O}_3$ shown in Fig. 3e is classified as a Type V isotherm with the H3 hysteresis loop according to IUPAC [50]. In the range of P/P_0 below 0.15, the adsorption process mostly takes place in a monolayer form, indicating a weak interaction between the adsorbate and the sample surface. The process changes to multilayer adsorption when P/P_0 ranges from 0.15 to 0.9, evidenced by the appearance of the H3 hysteresis loop. This reflects the existence of mesopores, which is confirmed by the pore size distribution graph calculated from the Barrett-Joyner-Halenda method (Fig. 3f), with the pore diameter ranging from 2 nm to 5 nm. Finally, when P/P_0 is higher than 0.9, an unsaturated rise is observed, indicating the presence of macropores with size above 50 nm. These macropores likely originate from the coral-like structure of $\alpha\text{-Fe}_2\text{O}_3$, which can be easily observed in the SEM image (Fig. 3d), revealing a hierarchical porous structure of the synthesized $\alpha\text{-Fe}_2\text{O}_3$ material.

3.2. $\text{TiO}_2/\alpha\text{-Fe}_2\text{O}_3$ Nanocomposite

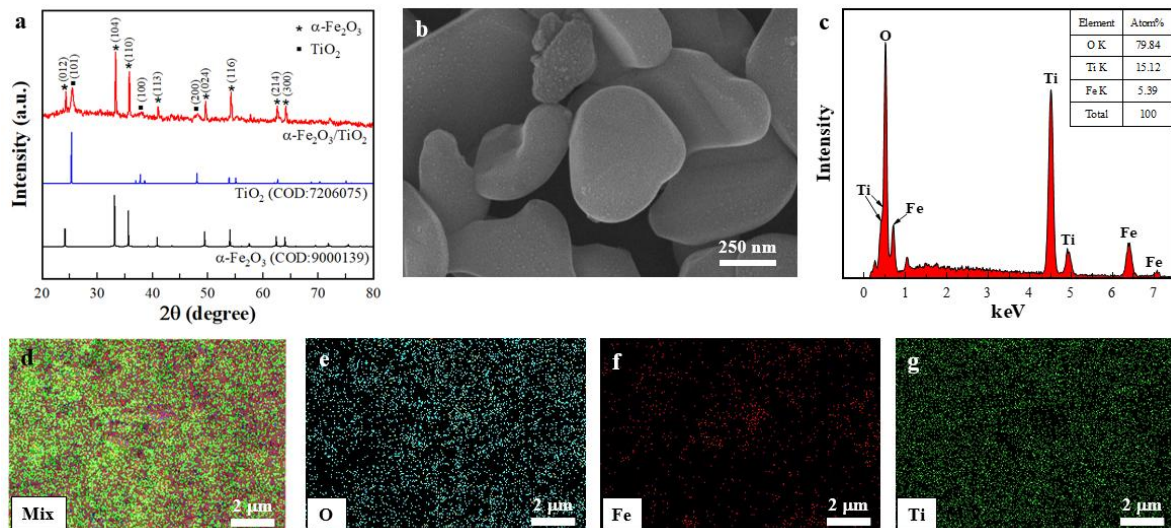


Figure 4. XRD pattern (a), SEM image (b), EDX spectrum (c), and EDX elemental mapping (d-g) of $\text{TiO}_2/\alpha\text{-Fe}_2\text{O}_3$ nanocomposite.

The successful synthesis of the $\text{TiO}_2/\alpha\text{-Fe}_2\text{O}_3$ nanocomposite was confirmed by the characterization results shown in Fig. 4. The XRD pattern (Fig. 4a) displays peaks for both $\alpha\text{-Fe}_2\text{O}_3$ and the anatase TiO_2 phase, with the latter identified by diffraction peaks at 2θ angles of 25.4° (101), 38.2° (004), and 48.1°

(200) (COD: 7206075). The crystallite sizes of the TiO_2 and $\alpha\text{-Fe}_2\text{O}_3$ in their nanocomposite were also estimated using the Scherrer equation. Based on the full width at half maximum (FWHM) of characteristic TiO_2 (101) and $\alpha\text{-Fe}_2\text{O}_3$ (110) diffraction peaks, the average crystallite sizes were calculated to be 17.0 and 42.8 nm, respectively. This is visually supported by the SEM image (Fig. 4b), which shows small TiO_2 nanoparticles distributed on the surface of the $\alpha\text{-Fe}_2\text{O}_3$ structure. Furthermore, EDX analysis (Fig. 4c) and elemental mapping (Fig. 4d-g) confirmed the presence and uniform distribution of Ti, Fe, and O elements throughout the nanocomposite. These results demonstrate the successful formation of a $\text{TiO}_2/\alpha\text{-Fe}_2\text{O}_3$ nanocomposite, which is expected to enhance the photocatalytic performance of the material.

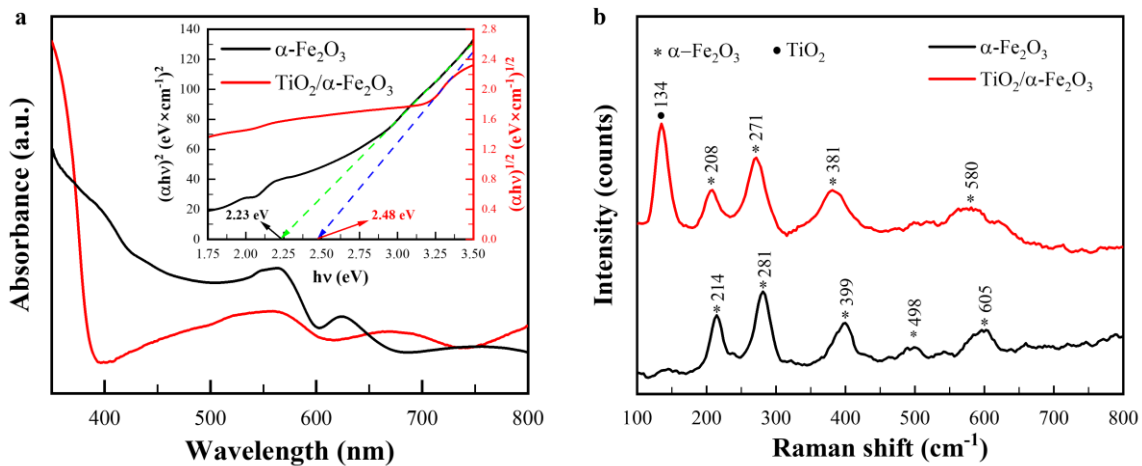


Figure 5. The UV-Vis absorption spectra with an inset of their Tauc plots (a), and the Raman spectra of $\alpha\text{-Fe}_2\text{O}_3$ and the $\text{TiO}_2/\alpha\text{-Fe}_2\text{O}_3$ nanocomposite (b).

The optical analyses of the $\alpha\text{-Fe}_2\text{O}_3$ and $\text{TiO}_2/\alpha\text{-Fe}_2\text{O}_3$ samples are presented in Fig. 5, consisting of the UV-Vis spectra with the inset of their Tauc plots (Fig. 5a) and their Raman spectra (Fig. 5b). The absorbance spectrum of pure $\alpha\text{-Fe}_2\text{O}_3$ shows several characteristic absorption features, including a shoulder near 400 nm, peaks at approximately 565 nm and 624 nm, and 750 nm. This broad absorption range of $\alpha\text{-Fe}_2\text{O}_3$ can be attributed to the intrinsic electronic transitions of Fe^{3+} , arising from the d-d transition, ligand-to-metal charge transfer, and pair excitations [51]. Meanwhile, The UV-Vis spectrum of the $\text{TiO}_2/\alpha\text{-Fe}_2\text{O}_3$ nanocomposite, together with the characteristic absorption of $\alpha\text{-Fe}_2\text{O}_3$, shows a distinct absorption edge near 390 nm from anatase TiO_2 . From the Tauc plots analysis inserted in Fig. 5a, the direct band gap of the $\alpha\text{-Fe}_2\text{O}_3$ is 2.23 eV, which is consistent with other reports [52, 53], while the indirect bandgap of the $\text{TiO}_2/\alpha\text{-Fe}_2\text{O}_3$ nanocomposite is at 2.44 eV. Moreover, as TiO_2 is known for its transparency to visible light [54], $\alpha\text{-Fe}_2\text{O}_3$ component acts as the primary photosensitizer governing the nanocomposite's optical behavior in the visible region. Theoretically, the interaction between TiO_2 and $\alpha\text{-Fe}_2\text{O}_3$ leads to the hybridization of Fe 3d and Ti 3d orbitals near their CBs [55-57], thus modifying the local density of states at the interface. This electronic environment facilitates visible light transitions, where the Fe 3d states determine the dominant optical response due to the role of $\alpha\text{-Fe}_2\text{O}_3$ as the primary photosensitizer. Consequently, the observed optical bandgap of $\text{TiO}_2/\alpha\text{-Fe}_2\text{O}_3$ reflects this modified electronic environment rather than the intrinsic value of isolated $\alpha\text{-Fe}_2\text{O}_3$. This explains why the bandgap of $\alpha\text{-Fe}_2\text{O}_3/\text{TiO}_2$ derived from Tauc plot analysis is near the fundamental absorption edge of

α -Fe₂O₃, despite the wide bandgap of the TiO₂ layer. The indirect bandgap estimation of TiO₂/ α -Fe₂O₃ is attributed not merely to the isolated materials but to the absorption edge in the heterostructure as well as the interfacial charge transfer between the CBs of α -Fe₂O₃ and TiO₂. The complex electronic environment at the interface as well as the difference between two materials makes this indirect transition the dominant optical pathway. This optical bandgap estimation suggests an electronic interaction between α -Fe₂O₃ and TiO₂, which is structurally confirmed by the Raman spectra (Fig. 5b), where the characteristic peaks of both materials are present [58-60]. Crucially, all the α -Fe₂O₃ vibrational modes (A_{1g} and E_g) in the nanocomposite exhibit a slight redshift, indicating the interfacial interaction between α -Fe₂O₃ and TiO₂ [61]. These combined features suggest the formation of heterojunctions between TiO₂ and α -Fe₂O₃ rather than a simple physical mixture.

3.3. Photocatalyst Efficiency in Dye Degradation

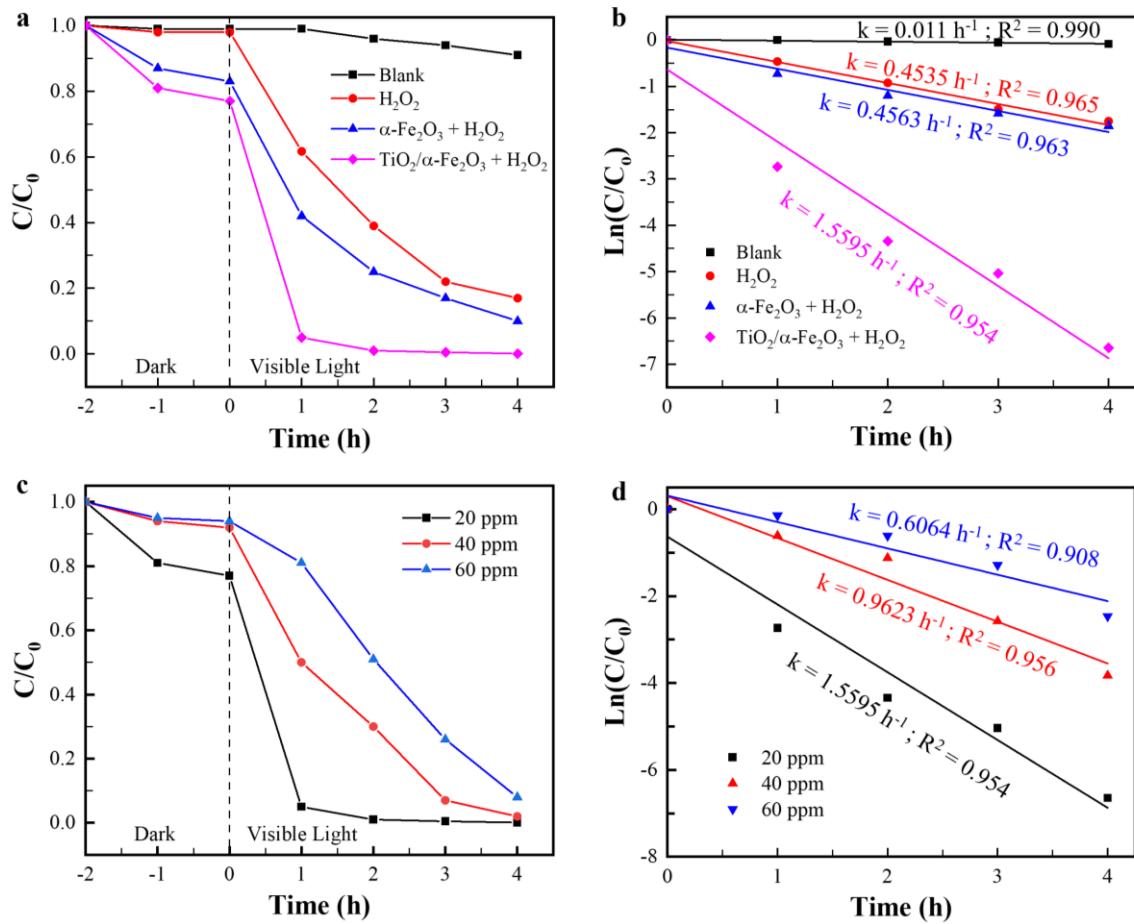


Figure 6. The degradation of 20 ppm MB under different photocatalytic conditions (a) with their pseudo-first-order kinetics following the L-H model (b); the degradation of MB by the TiO₂/ α -Fe₂O₃ nanocomposite based on the photocatalytic process at different MB concentrations (c) with their pseudo-first-order kinetics following the L-H model (d). (Experimental conditions: $C_{\text{catalyst}} = 2.5 \text{ mg/mL}$, $\text{pH} = 3.0$; H₂O₂ = 0.1 mL (30 wt.%); Light source = 30W visible light.).

The results of the MB degradation experiments are presented in Fig. 6. At the MB concentration of 20 ppm (Fig. 6a), there is a slight initial decrease in MB concentration for both the $\alpha\text{-Fe}_2\text{O}_3$ and $\text{TiO}_2/\alpha\text{-Fe}_2\text{O}_3$ samples during the 2-hour dark period. This is attributed to the adsorption of MB molecules onto the catalyst surfaces to establish adsorption-desorption equilibrium. Upon visible light irradiation, the degradation accelerated significantly in all active samples. The superior performance of the $\text{TiO}_2/\alpha\text{-Fe}_2\text{O}_3$ nanocomposite in MB removal, as derived from the pseudo-first-order equation, is demonstrated in Fig. 6b. This nanocomposite catalyst exhibited a remarkably high degradation rate constant of 1.5595 h^{-1} , achieving 95% MB removal within the first hour and nearly complete degradation thereafter. In contrast, the respective rate constants for the control samples were significantly lower: 0.4563 h^{-1} for $\alpha\text{-Fe}_2\text{O}_3$, 0.4535 h^{-1} for H_2O_2 alone, and 0.011 h^{-1} for the blank. The validity of these high degradation activities is ensured by control experiments shown in Fig. 6a, where the “Blank” line and “Dark” phase confirm that photolysis and adsorption alone are not responsible for the observed removal. Furthermore, the effect of initial MB concentration on the degradation rate of photocatalytic process is displayed in Fig. 6c. As expected, the degradation rate constant decreased at higher initial concentrations, with k values of 0.9623 h^{-1} and 0.6064 h^{-1} for 40 ppm and 60 ppm MB (as shown in Fig. 6d), respectively, while still achieving high removal efficiencies (98% for 40 ppm MB and 92% for 60 ppm MB in 4 hours). Additionally, the high efficiency in dye degradation of active samples can be attributed to the sufficient catalyst dosage in the experiments, which ensures a high availability of surface sites in a photocatalytic system [45]. These results confirm the superior and robust photocatalytic activity of the $\text{TiO}_2/\alpha\text{-Fe}_2\text{O}_3$ nanocomposite compared to the conventional photo-Fenton reaction of $\alpha\text{-Fe}_2\text{O}_3$.

The MB degradation results indicate the superior efficiency of the $\text{TiO}_2/\alpha\text{-Fe}_2\text{O}_3$ nanocomposite compared to pure $\alpha\text{-Fe}_2\text{O}_3$, which is well-known for its photo-Fenton activity. Crucially, these experiments were conducted under visible light, which does not have enough energy to excite wide-bandgap TiO_2 directly. This suggests a mechanism where $\alpha\text{-Fe}_2\text{O}_3$ acts as the primary photosensitizer, absorbing visible light to generate electron-hole pairs. These charge carriers are then efficiently separated at the $\text{TiO}_2/\alpha\text{-Fe}_2\text{O}_3$ heterojunction, which minimizes recombination. This enhanced charge separation boosts the production of $\cdot\text{OH}$, leading to a photocatalytic efficiency that significantly surpasses the conventional process with pure $\alpha\text{-Fe}_2\text{O}_3$.

The proposed photocatalytic mechanism of this process is shown in Fig. 7a, which describes the formation of type-II heterojunctions due to the arrangement of valence bands (VBs) and CBs of $\alpha\text{-Fe}_2\text{O}_3$ and TiO_2 based on absolute electronegativity [62, 63]. Specifically, CB energy levels (E_{CB}) and VB energy levels (E_{VB}) of $\alpha\text{-Fe}_2\text{O}_3$ and TiO_2 are determined using the following equations:

$$E_{\text{CB}} = \chi - E_{\text{e}} - 0.5 \times E_{\text{g}} \quad (5)$$

$$E_{\text{VB}} = E_{\text{CB}} + E_{\text{g}} \quad (6)$$

where E_{e} (4.5 eV) represents the energy of free electrons on the hydrogen scale, E_{g} is the bandgap energy, and χ is the absolute electronegativity of $\alpha\text{-Fe}_2\text{O}_3$ (4.87 eV) and TiO_2 (5.81 eV) [64, 65]. In this case, the bandgap energy of anatase- TiO_2 is approximately 3.2 eV, while that of $\alpha\text{-Fe}_2\text{O}_3$ in the present study is 2.23 eV. Based on the above calculations, the CB and VB energy levels of TiO_2 are estimated to be -0.29 eV and 2.91 eV (vs. NHE), respectively, whereas those of $\alpha\text{-Fe}_2\text{O}_3$ are -0.745 eV and 1.485 eV (vs. NHE).

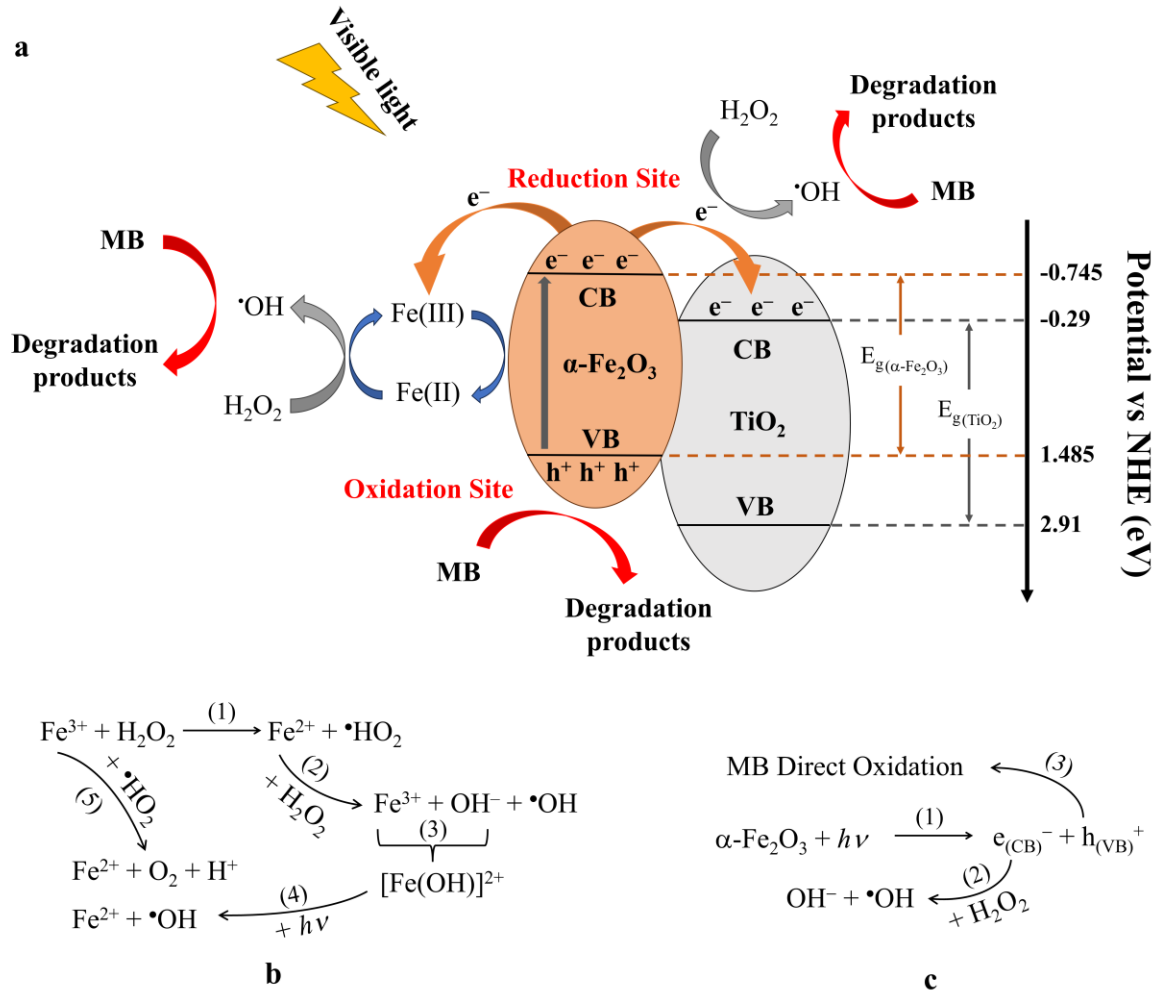


Figure 7. Photocatalytic mechanism of MB degradation using the $\text{TiO}_2/\alpha\text{-Fe}_2\text{O}_3$ nanocomposite (a); pathways of $\cdot\text{OH}$ formation: bidirectional conversion cycle of Fe^{2+} and Fe^{3+} in the photo-Fenton process (b) and the photocatalytic process (c).

The primary oxidant $\cdot\text{OH}$ formation, as illustrated in Figs. 7b and 7c, is demonstrated through two main pathways: the conventional photo-Fenton reaction and photocatalytic pathway. Fig. 7b presents the reactions occurring during the photo-Fenton process, with the reaction between Fe^{3+} and H_2O_2 to produce Fe^{2+} and hydroperoxyl species ($\cdot\text{HO}_2$) (Fig. 7b, reaction 1) [66, 67]. This produced Fe^{2+} is able to be oxidized by H_2O_2 (Fig. 7b, reaction 2), returning to Fe^{3+} state and releasing the $\cdot\text{OH}$ and hydroxyl ions (OH^-) simultaneously [66]. Under low pH condition, the complex $[\text{Fe}(\text{OH})]^{2+}$ is formed, as reaction 3 in Fig. 7b, which is then reduced to Fe^{2+} and $\cdot\text{OH}$ in the presence of excitation light (Fig. 7b, reaction 4) [68]. Additionally, Fe^{3+} can also interact with $\cdot\text{HO}_2$ to form Fe^{2+} , O_2 and H^+ (Fig. 7b, reaction 5) [66]. These reactions form a bidirectional conversion cycle between Fe^{2+} and Fe^{3+} , simultaneously releasing $\cdot\text{OH}$ to facilitate MB removal. In terms of the photocatalytic pathway, this process is initiated when $\alpha\text{-Fe}_2\text{O}_3$ absorbs visible light, exciting electrons from its VB to its CB, creating photogenerated electron-hole pairs ($e_{(\text{CB})}^-$ and $h_{(\text{VB})}^+$) (Fig. 7c, reaction 1). Crucially, $e_{(\text{CB})}^-$ is capable of transferring to the CB of TiO_2 at the heterojunction interface of this structure due to the favorable band alignment. This spatial

separation of charges minimizes the recombination of photogenerated electron-hole pairs, thereby enhancing photocatalytic efficiency. Moreover, these excited electrons take part in the reduction of H_2O_2 directly, forming $\cdot\text{OH}$ and OH^- (Fig. 7c, reaction 2) [68, 69]. Additionally, owing to the relatively low VB potential of $\alpha\text{-Fe}_2\text{O}_3$ (1.485 eV vs. NHE), direct $\cdot\text{OH}$ generation from H_2O or OH^- is thermodynamically unfavorable [70]. Instead, h_{VB}^+ remaining in the VB of $\alpha\text{-Fe}_2\text{O}_3$ mainly contributes to degradation by directly oxidizing organic pollutants. Therefore, the $\text{TiO}_2/\alpha\text{-Fe}_2\text{O}_3$ nanocomposite is a highly efficient system that enhances charge separation and boosts radical production while the h_{VB}^+ contributes to the photocatalytic activity, leading to a significant improvement in photocatalytic activity.

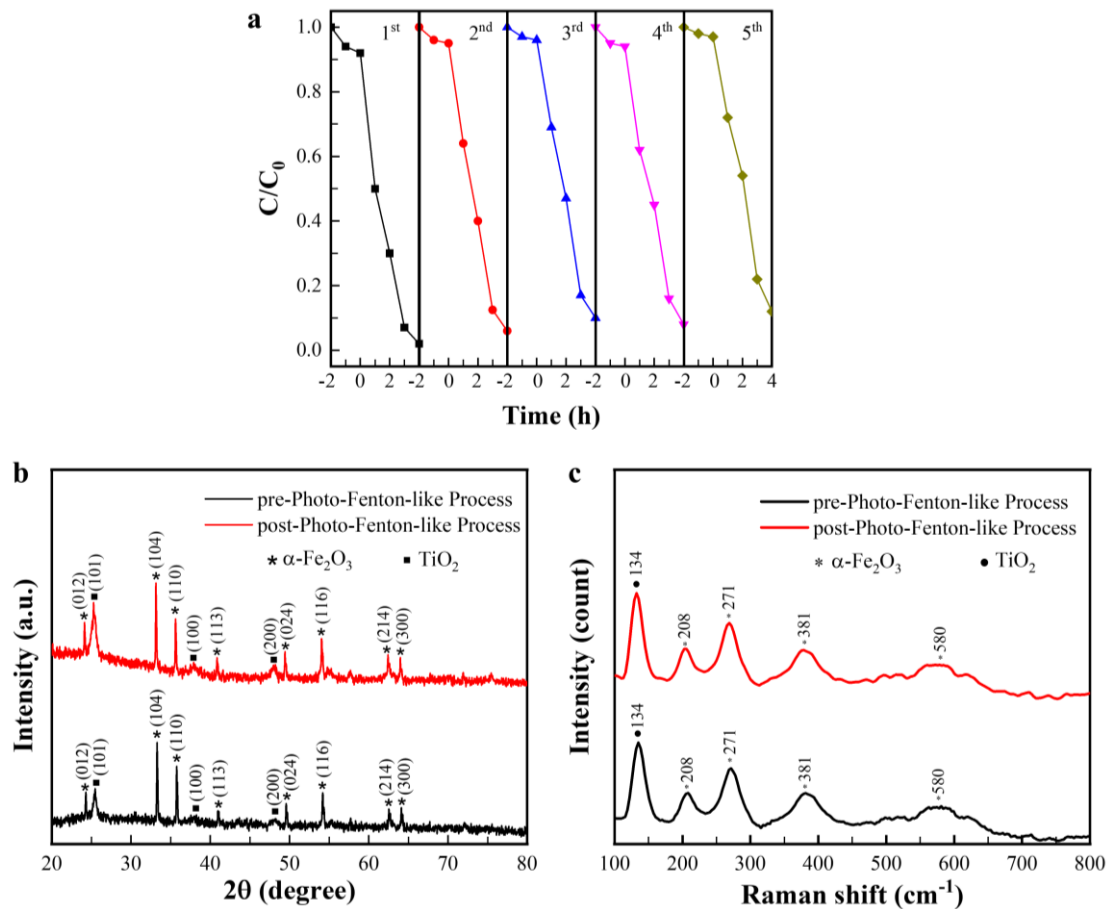


Figure 8. The reusability of the $\text{TiO}_2/\alpha\text{-Fe}_2\text{O}_3$ nanocomposite over 5 cycles of MB removal at 40 ppm concentration (a); XRD patterns (b) and Raman spectra (c) of the $\text{TiO}_2/\alpha\text{-Fe}_2\text{O}_3$ nanocomposite before and after 5 cycles of MB degradation based on the photocatalytic process.

In this study, the reusability and stability of the $\text{TiO}_2/\alpha\text{-Fe}_2\text{O}_3$ nanocomposite were evaluated through recycling experiments. Specifically, the photocatalyst's performance was evaluated over five cycles for the degradation of 40 ppm MB solution. The results show that the degradation efficiency was well-maintained at over 90% even after the fifth cycle (Fig. 8a), confirming the reversible redox cycles of $\text{Fe}^{2+}/\text{Fe}^{3+}$. Furthermore, the structural integrity of the nanocomposite was confirmed by XRD and Raman spectroscopy. The XRD patterns (Fig. 8b) and Raman spectra (Fig. 8c) of the catalyst remained almost unchanged compared to the fresh catalyst, indicating its high stability with negligible phase

degradation. This confirms not only the structure's resistance to phase transformation during the photocatalytic process, but also the maintenance of interfacial interaction of TiO_2 and $\alpha\text{-Fe}_2\text{O}_3$ after 5 degradation cycles.

When quantitatively compared to other recent photocatalyst systems, including photocatalytic, $\text{TiO}_2/\text{Fe}_2\text{O}_3$ -based, and advanced Z-scheme structures as summarized in Table 2, the $\text{TiO}_2/\alpha\text{-Fe}_2\text{O}_3$ nanocomposite heterostructure demonstrates highly competitive performance. Notably, our structure achieves high removal efficiencies within similar timeframes and initial MB concentrations compared to other complex systems. Most importantly, this study suggests that the proposed phase transition strategy from $\gamma\text{-Fe}_2\text{O}_3$ serves as a feasible and effective approach to construct high-performance system, indicating that simple structural engineering can achieve catalytic results comparable to complex chemical synthesis methods. However, while the current work establishes the high efficiency of this phase-transition-derived heterostructure for organic model pollutants, future studies such as total organic carbon mineralization, precise quantification of trace metal leaching and ROS, and the performance assessment in real or simulated wastewater are required to comprehensively validate the mechanism and its practical robustness.

Table 2. Comparison of the MB removal efficiency obtained in this study with several advanced photocatalyst systems

Material system	MB concentration (ppm)	Time (h)	Efficiency (%)
Ag-SiO ₂ @ $\alpha\text{-Fe}_2\text{O}_3$ [71]	10	1,5	~ 100
ZnFe ₂ O ₄ /Fe ₂ O ₃ [72]	10	2	~84.3
VO ₂ @Fe ₂ O ₃ -TiO ₂ [73]	10	2	~88.81
ZnO/Fe ₂ O ₃ [74]	10	2	~ 100
$\alpha\text{-Fe}_2\text{O}_3$ NPs/g-C ₃ N ₄ [75]	15	1	~100
N-doped KBiFe ₂ O ₅ [76]	20	2	~ 93
FeWO ₄ /Bi ₂ MoO ₆ [77]	20	1,5	~ 97
Fe ₂ O ₃ @TiO ₂ [78]	20	3	~100
MnFe ₂ O ₄ /g-C ₃ N ₄ [79]	80	4	~ 100
$\text{TiO}_2/\alpha\text{-Fe}_2\text{O}_3$ (<i>This work</i>)	20	2	~ 100
	40	4	~ 98
	60	4	~ 92

4. Conclusion

In this study, the $\text{TiO}_2/\alpha\text{-Fe}_2\text{O}_3$ nanocomposite was successfully synthesized using $\alpha\text{-Fe}_2\text{O}_3$ fabricated by the thermal phase transition from $\gamma\text{-Fe}_2\text{O}_3$ with the complete conversion at 800 °C, converting the $\gamma\text{-Fe}_2\text{O}_3$ nanoparticles into $\alpha\text{-Fe}_2\text{O}_3$ with the hierarchical porous, coral-like structure. The physical formation of the $\text{TiO}_2/\alpha\text{-Fe}_2\text{O}_3$ nanocomposite was confirmed by structural analyses, where XRD and SEM demonstrated the coexistence of both components and their distinct morphologies, while EDX elemental mapping revealed the uniform distribution of TiO_2 on the $\alpha\text{-Fe}_2\text{O}_3$ surface. Furthermore, the modification of the optical bandgap from 2.23 eV ($\alpha\text{-Fe}_2\text{O}_3$) to 2.44 eV ($\text{TiO}_2/\alpha\text{-Fe}_2\text{O}_3$), coupled with the redshift observed in the Raman spectra, provides an effective interfacial interaction between TiO_2 and $\alpha\text{-Fe}_2\text{O}_3$. Moreover, this nanocomposite demonstrated a superior photocatalytic activity compared to the conventional photo-Fenton process, evidenced by the degradation rate constant of 1.5595 h⁻¹, which is more than three times higher than that of pure $\alpha\text{-Fe}_2\text{O}_3$ (0.4563 h⁻¹) and reaching nearly 100% removal efficiency of 20 ppm MB after 2-hour irradiation. This superior performance can

be explained by the synergistic effects between photo-Fenton and photocatalytic pathways, theoretically driven by the suppression of electron-hole recombination at the $\text{TiO}_2/\alpha\text{-Fe}_2\text{O}_3$ heterojunction. Furthermore, the $\text{TiO}_2/\alpha\text{-Fe}_2\text{O}_3$ nanocomposite proved to be a robust and reusable catalyst. It maintained high degradation efficiency (>92%) even at increased initial MB concentrations of 60 ppm after 4-hour irradiation. Crucially, the material exhibited excellent stability, retaining over 90% of its initial activity after 5 cycles without structural changes. These findings confirm that the $\text{TiO}_2/\alpha\text{-Fe}_2\text{O}_3$ nanocomposite is a promising candidate, which is visible-light-driven and high performance, for the advanced treatment of dye-polluted wastewater.

Author contributions

Conceptualization: H. T. M. Hoa, D. T. Muon, D. V. Quang; *Data curation:* D. T. Muon, N. T. Trang, L. P. P. Ha; *Formal analysis:* D. T. Muon, T. V. Cuong; *Funding acquisition:* H. T. M. Hoa, N. H. Hung; *Methodology:* D. T. Muon; *Validation:* H. T. M. Hoa, D. V. Quang, L. P. P. Ha; *visualization:* D. T. Muon, N. T. Trang; *writing – original draft:* D. T. Muon, N. T. Trang; *writing – review and editing:* H. T. M. Hoa, T. V. Cuong, D. T. Muon. All authors reviewed the results and approved the final version of the manuscript.

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