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Dye-Selective Photocatalytic Degradation by Hydrothermally Synthesized α-FeOOH: Preferential Activity Toward Malachite Green

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ABSTRACT

In this study, α-FeOOH was successfully synthesized via a hydrothermal method, and its photocatalytic activity was evaluated against five organic dyes, namely Eosin Y, Methylene Blue, Malachite Green, Methyl Orange, and Rhodamine B, under illumination. X-ray diffraction confirmed α-FeOOH to be the only crystalline phase detectable, with crystallite sizes in the nanoscale range. UV–Vis absorption spectra showed that α-FeOOH exhibited excellent photocatalytic selectivity: MG was substantially degraded, with a removal efficiency of >90% after 240 min of illumination, while the other four dyes showed negligible degradation under the same conditions. This selectivity may be associated with differences in the molecular structures, adsorption behaviors, and intrinsic susceptibility of the dye chromophores to oxidative degradation. In particular, the structural characteristics of MG may facilitate oxidative attack on its chromophore compared with the other dyes investigated. These results indicate that α-FeOOH is a promising material for selective photocatalytic systems in the treatment of wastewater containing organic dyes.

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1. Introduction

The rapid growth of textile, printing and chemical industries has resulted in the release of large amount of wastewater containing organic dyes into the environment [1]–[3]. Some of the most common pollutants are different compounds such as Malachite Green (MG), Methyl Orange (MO), Rhodamine B (RhB), Eosin Y and Methylene Blue (MB). These dyes have aromatic ring structures that are stable, but are resistant to biodegradation, and are acutely toxic, mutagenic and persistent in aquatic environments [4]–[7]. The presence of these dyes in water not only leads to a decrease in light transmittance, affecting the photosynthesis of aquatic ecosystems, but also causes serious risks to human health [8]. Thus, the development of effective treatment methods for these recalcitrant organic compounds has become an urgent requirement in the field of environmental protection.

In the last decades, heterogeneous photocatalysis has been identified as a promising Advanced Oxidation Process (AOP) for the treatment of organic pollutants under light irradiation [9]. This process is based on the excitation of a semiconductor material to generate electron-hole pairs (e^-/h^+) that can produce strong oxidizing radicals such as •OH and $•O_2^-$ that could decompose the dye molecules into less toxic products. In recent years, metal oxide and oxyhydroxide materials, especially iron based ones, have attracted considerable attention owing to their high stability, low cost, environmental friendliness and their ability to operate under visible light.

Goethite (α-FeOOH) is an iron oxyhydroxide which is one of the photo catalytic materials that has received attention because of its low cost, non-toxicity and high chemical stability. α-FeOOH is an n-type semiconductor with a relatively narrow band gap (~ 2.1–2.3 eV) which in principle allows for efficient absorption of the visible light, an abundant natural energy source [10], [11]. Moreover, the hydroxyl-rich surface of α-FeOOH is conducive to the adsorption and interaction with dye molecules in

solution. In contrast, the traditional photocatalysts such as TiO_2 work mainly in the UV range, which is only a small part of the solar irradiation, limiting their effective use in wastewater treatment under natural light conditions [10], [12] α-FeOOH is therefore better at harvesting visible light, but the actual efficiency achieved is dependent on the particular light conditions used in each study.

With its favorable visible-light response and low cost relative to traditional photocatalysts, α-FeOOH is considered a promising candidate for practical wastewater treatment. Such potential, however, has been demonstrated mostly for the degradation of a single dye under controlled conditions [1], [13] hereas systematic comparisons of α-FeOOH performance across dyes with different molecular configurations and surface charges remain rare. The gap represents an important limitation, since real wastewater usually comprises complex mixtures of cationic dyes (MG, RhB, MB) and anionic dyes (MO, Eosin Y) whose degradation behavior cannot be reliably predicted from single-dye systems. In addition, surface charge may not be the only governing factor, as the degradation of each dye also depends on the stability of its molecular framework and other physicochemical characteristics. To clarify the issue, the photocatalytic response of α-FeOOH toward dyes of contrasting charge and structure is compared directly under identical conditions.

In the present work, α-FeOOH was synthesized by a hydrothermal method, and its photocatalytic activity was systematically investigated against five representative dyes, namely MG, RhB, MB (cationic dyes), MO, and Eosin Y (anionic dyes) under identical light conditions. The novelty lies in comparing five dyes over a single α-FeOOH catalyst, which reveals the photocatalytic selectivity of the material and clarifies whether charge, molecular structure, or both govern its performance. The results are expected to provide a more solid scientific basis for the development of photocatalytic materials for wastewater treatment with complex mixtures of organic dyes.

2. Research Methods

2.1. Synthesis Procedure of α-FeOOH Powder

By using a hydrothermal method, α-FeOOH was prepared with $FeCl_3.6H_2O$ and NaOH as precursors (as shown in Figure 1). First, 0.5 M NaOH solution and 0.075 M $FeCl_3.6H_2O$ solution were prepared by dissolving NaOH and $FeCl_3.6H_2O$ precursors in distilled water. Then, the NaOH solution was added dropwise to the $FeCl_3.6H_2O$ solution and precipitated with stirring constantly. The addition was continued till the pH of the system attained the desired value of 10. The resulting solution was transferred into a hydrothermal reaction vessel (autoclave) and thermally treated at 200°C for 3 hour. When the hydrothermal process was complete, the product was recovered by centrifugation. The washed samples were dried at 70°C to get α-FeOOH powder. Finally, the powder was calcined at 200 °C for 1 hour and ground into fine powder for later experiments.

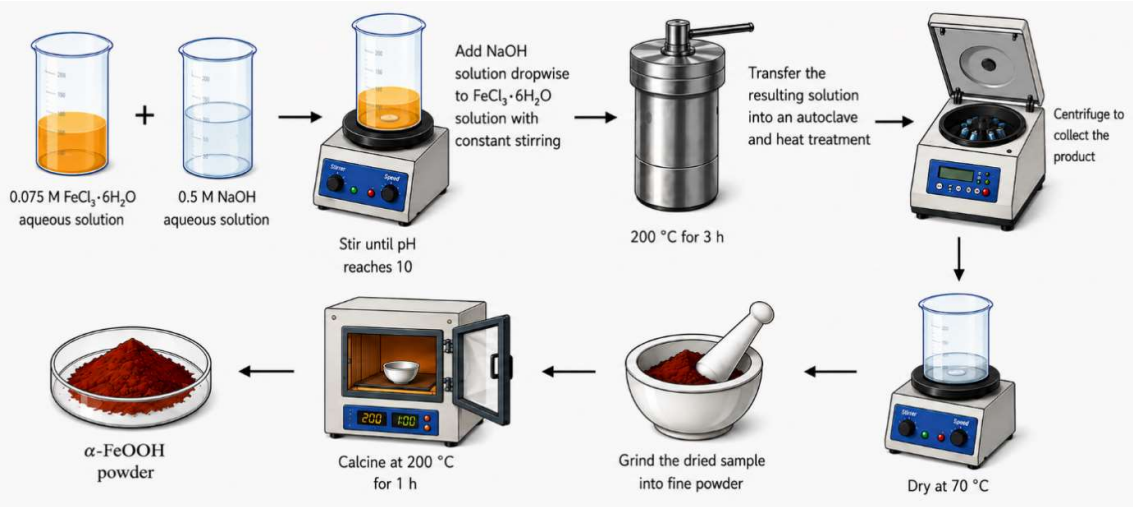


Figure 1. *Synthesis procedure of FeOOH.*

2.2. Dye Degradation Photocatalytic Procedure Using α-FeOOH Powder

The photocatalytic activity of α-FeOOH was evaluated based on its ability to degrade organic dye solutions under illumination. First, accurate quantities of α-FeOOH powder and dye were weighed. The dye solution was prepared by dissolving in distilled water to the desired concentration and stirred with magnetic stirrer until homogeneous as shown in Figure 2. Then, the α-FeOOH powder was dispersed into the dye solution, and the resulting suspension was stirred in the dark for 30 min to establish adsorption-desorption equilibrium between the catalyst surface and the dye molecules prior to illumination. The step ensures that the decrease in dye concentration observed after illumination is mainly attributable to photocatalytic degradation rather than to simple adsorption on the catalyst surface. After the dark equilibration period, the suspension was irradiated with a halogen lamp emitting light (λ = 380-780 nm) for 4 h. Before adding the α-FeOOH photocatalyst, the dye solution was adjusted to an initial pH of 7.5-10. Solution samples were periodically taken every 30 min during illumination to follow the change in the dye concentration with time. The collected samples were centrifuged to achieve the complete separation of the solid phase (α-FeOOH) from the solution. The clear supernatant was collected in sample tubes and analyzed, usually by UV–Vis absorption spectroscopy, to determine the reduction in dye concentration. For kinetic and degradation evaluation, C denotes the dye concentration measured at irradiation time in the presence of α-FeOOH (0.1 g), whereas C_0 refers to the corresponding dye concentration in the catalyst-free control experiment under identical illumination (0–240 min, Figure 4) prior to catalyst addition.

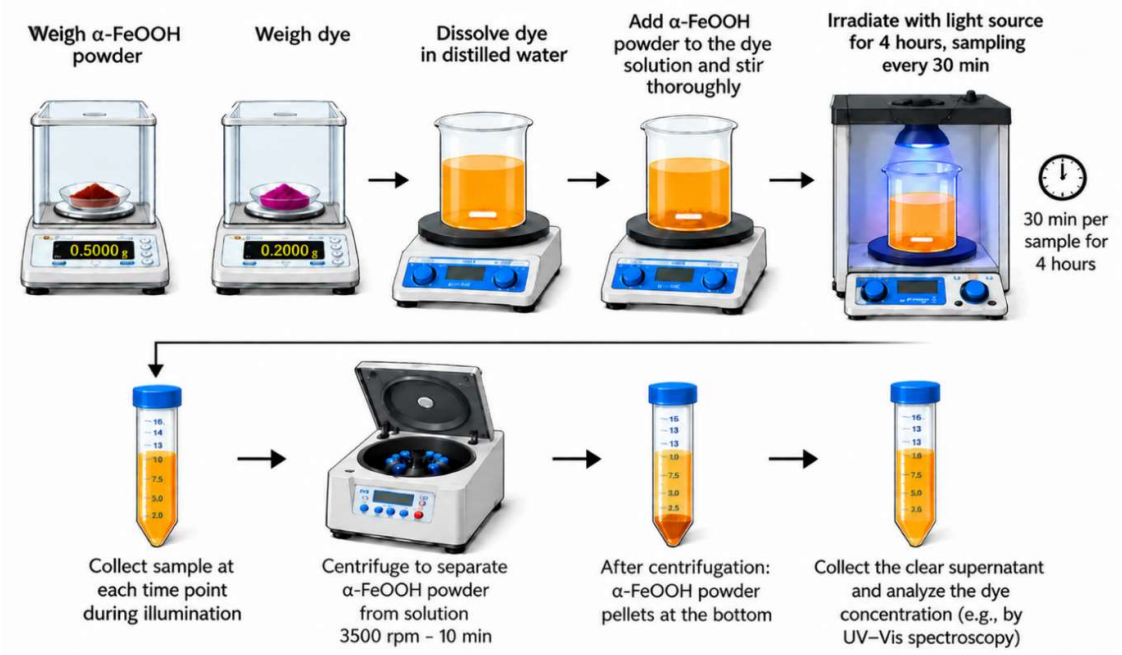


Figure 2. *Photocatalytic procedure of FeOOH for dye degradation.*

2.3. Characterization

The phase composition of the samples was characterized using a D2 PHASER X-ray diffractometer (Bruker, Germany) with Cu Kα radiation (λ = 1.5406 Å). The diffraction patterns were recorded over a 2θ range of 20°–65°, with a step size of 0.02°. The optical properties of sample were measured using a JASCO V-730 UV–Vis spectrophotometer.

The average crystallite size was estimated using the Debye–Scherrer equation:

$$D = \frac{K\lambda}{\beta cos\theta} \quad (1)$$

where D is the average crystallite size (nm), K is the Scherrer shape factor (typically taken as 0.9), λ is the X-ray wavelength (0.15406 nm for Cu Kα radiation), β is the full width at half maximum (FWHM) of the diffraction peak corrected for instrumental broadening (in radians), and θ is the Bragg diffraction angle.

3. Results and Discussions

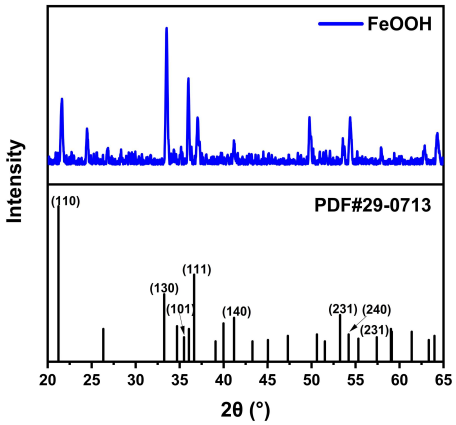


Figure 3. *XRD pattern of α-FeOOH.*

Table 1. *Diffraction parameters and crystallite size of the α-FeOOH.*

FeOOH peaks (hkl)	2θ (°)	FWHM (°)	D(nm)
(110)	21.625	0.25025	32.31
(130)	33.525	0.23777	34.88
(101)	35.998	0.20713	40.31
(140)	41.168	0.19923	42.58
(231)	53.354	0.12944	68.66
(240)	54.385	0.29977	29.78
(231)	57.892	0.12978	69.92

Figure 3 shows the X-ray diffraction (XRD) pattern of the α-FeOOH sample. The characteristic diffraction peaks are observed at 2θ positions coresponding to the crystal planes (110), (130), (101), (140), (231), (240), and (231). The peaks show a good agreement with the standard data of the goethite structure (α-FeOOH, PDF#29-0713), which reveals the polycrystalline structure of the obtained sample. This result is in agreement with previous reports, where α-FeOOH is observed for the range of $2\theta \approx 20$–65°, which confirms the crystalline structure of the material and the formation of a pure goethite phase [14]–[17]. The diffraction parameters and crystallite sizes are given in Table 1. The full width at half maximum (FWHM) of the diffraction peaks is in the range of ~0.12° to 0.29°, which is a normal peak broadening in nanomaterials. The crystallite size calculated by using the Scherrer equation [18] varies from ~29.78 to 69.92 nm. Most of the crystallites are in the range of 30–70 nm, which indicates a relatively uniform particle size at the nanoscale. The larger size values of the crystallites grown on the (121) and (061) planes can be explained by the preferential growth. In addition, the broadening of the diffraction peaks indicates the existence of microdefects and grain boundaries in the crystal lattice, which is a common phenomenon in nanomaterials, and it could influence the physical and chemical properties and photocatalytic activity of the material. The XRD results confirm the successful synthesis of α-FeOOH, with goethite as only crystalline phase detectable by XRD, nanoscale crystallite size, and good crystallinity.

Figure 4 shows the UV–Vis absorption spectra of (a) Eosin Y, (b) Methylene Blue (MB), (c) Malachite Green (MG), (d) Methyl Orange (MO) and (e) Rhodamine B (RhB) dye solutions (20 ppm)

with illumination time in the absence of catalyst. All dyes showed typical absorption peaks in the visible region, at about 515 nm (Eosin Y) [19], [20], 664 nm (MB) [8], [21], 616–617 nm (MG) [22], 463–464 nm (MO [23], [24] and 553–554 nm (RhB) [25], [26], corresponding to the π–π* electronic transitions [19], [27], [28] in the conjugated systems of the molecular structures.

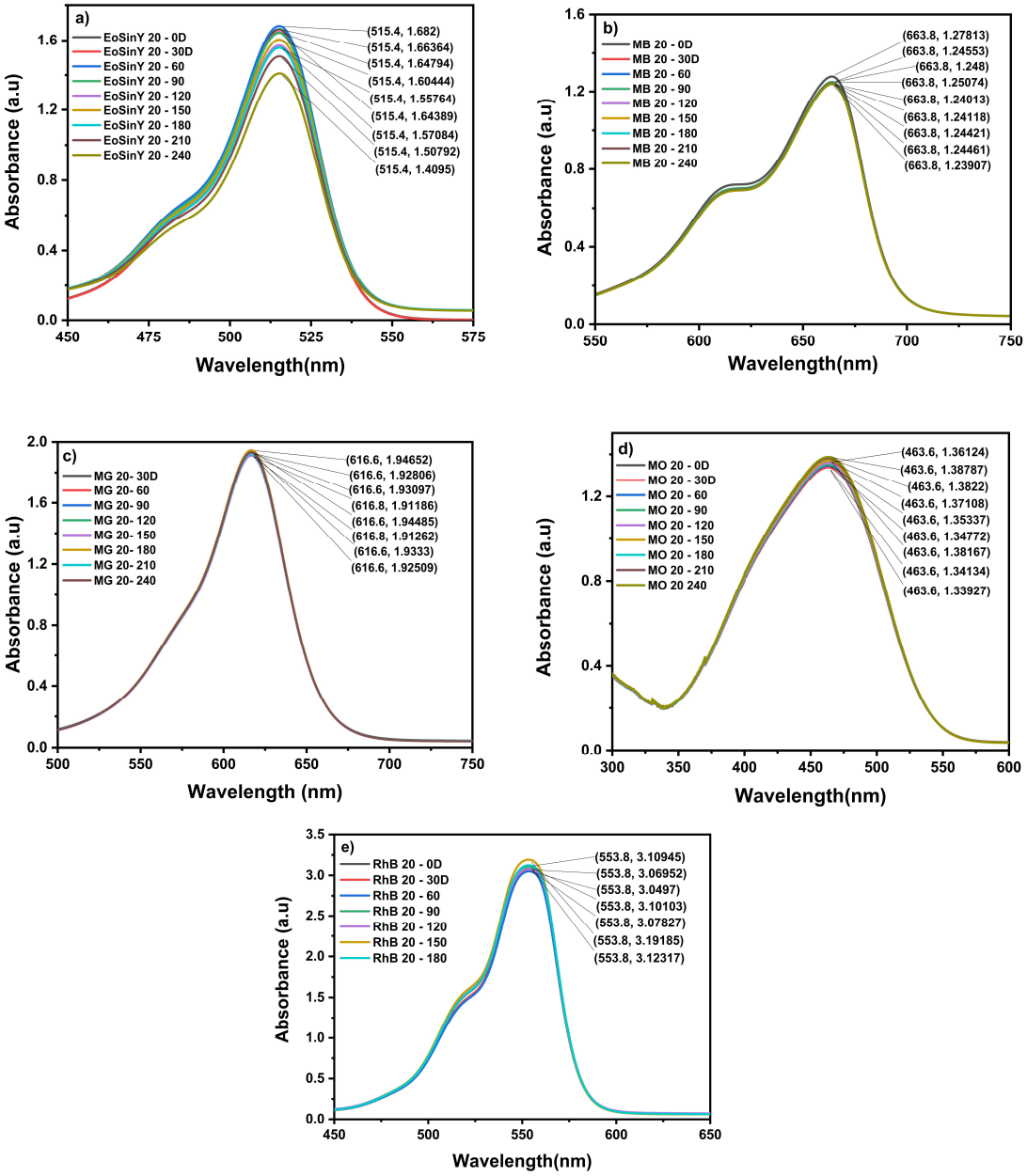


Figure 4. *UV–Vis spectra of all dyes over time in the absence of catalyst: (a) Eosin Y, (b) MB, (c) MG, (d) MO, and (e) RhB.*

The absorption intensity at the characteristic wavelengths of all the dyes did not change significantly during the illumination period (30–240 min) and the peak positions and spectral shapes were stable [20], [28], [29]. No spectral distortion or appearance of new absorption peaks was observed, suggesting that the molecular structures were not damaged during illumination. These results demonstrate that the dyes investigated have a relatively high photostability and the self-photolysis is negligible under the

experimental conditions. Thus, the decrease of dye concentration in the presence of α-FeOOH can be mainly attributed to the photocatalytic activity of the material and not to direct light effects. This result is very important to establish a valid control and provide a reliable basis for the accurate evaluation of the photocatalytic performance of α-FeOOH in the following sections.

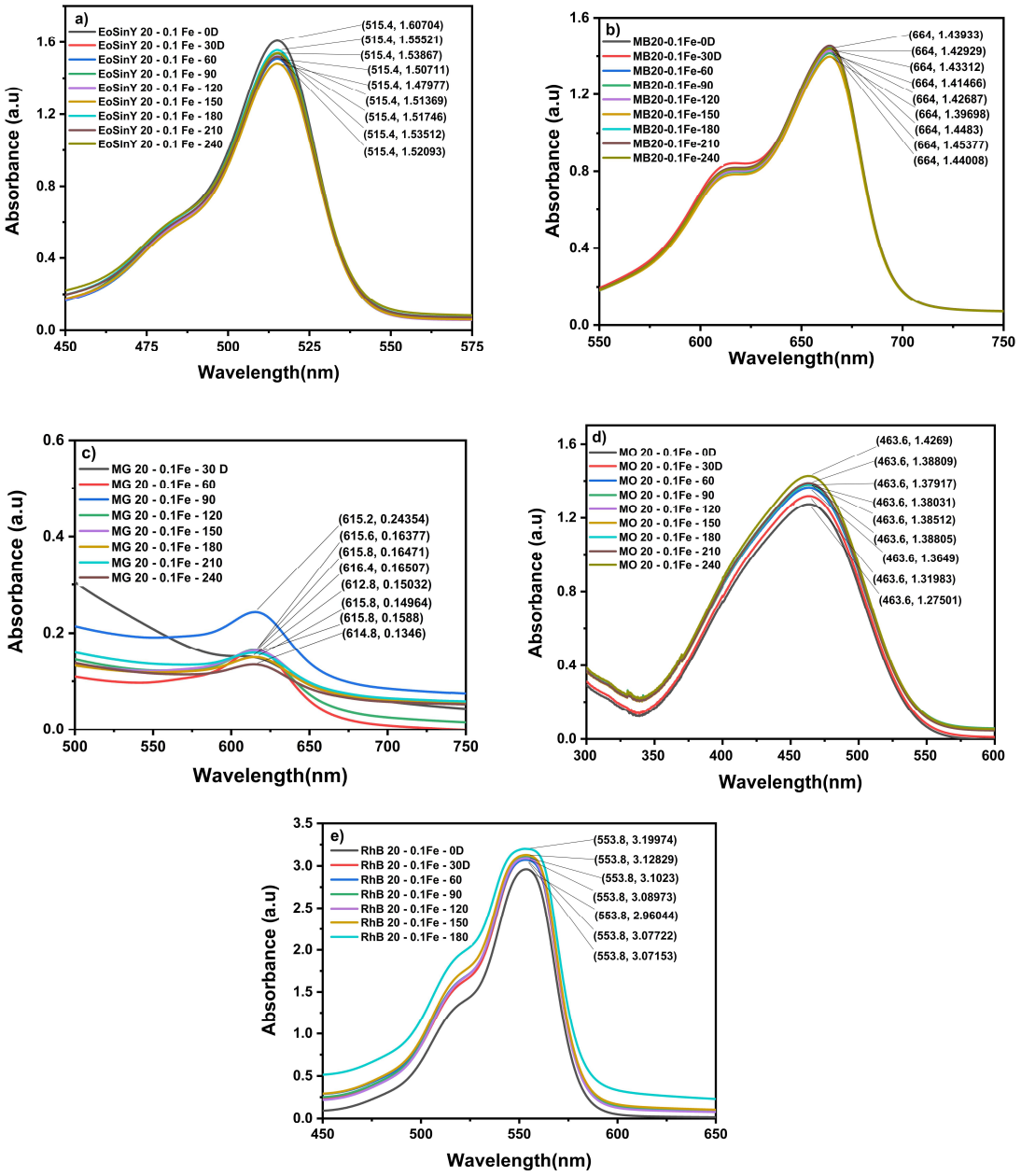


Figure 5. *UV–Vis spectra of the dyes in the presence of 0.1 g α-FeOOH: (a) Eosin Y, (b) MB, (c) MG, (d) MO, and (e) RhB.*

Figure 5 shows the UV–Vis absorption spectra of (a) Eosin Y, (b) Methylene Blue (MB), (c) Malachite Green (MG), (d) Methyl Orange (MO) and (e) Rhodamine B (RhB) dye solutions with α-FeOOH catalyst (0.1 g) under illumination. The photocatalytic degradation efficiency of the material was determined by the variations in the absorption intensity at the characteristic maximum wavelengths, which were used to assess the reduction of the dye concentration [27]–[31].

Eosin Y is an anionic dye, and the point of zero charge (PZC) of α-FeOOH has been reported to lie in the pH range 7.5–10 [32]–[34]. The pH of the experimental system was neutral to slightly alkaline (pH = 7.5-10), so the surface of the α-FeOOH may have been almost uncharged or negatively charged. This would have created an electrostatic repulsion between the negatively charged Eosin Y molecules and the α-FeOOH surface, thus decreasing the possibility of the dye reaching the sites where the oxidizing radicals •OH and $•O_2^-$ were being generated. However, the actual surface charge state under the specific pH used in this study should be verified by direct zeta-potential measurement in the future because the PZC of α-FeOOH has been reported to have a relatively wide range. Moreover, Eosin Y has a relatively stable molecular structure and can participate in the photogenerated charge trapping, which promotes the electron–hole recombination [35] and reduces the efficiency of free radical generation.

The characteristic absorption peak of MB at about 664 nm was also almost unchanged in the presence of α-FeOOH. After illumination only a slight decrease of the absorption intensity was found and no spectral distortion or peak shift was observed. This observation showed that the MB conjugated structure was relatively intact [36], and the photocatalytic degradation was minimal. MB is a cationic dye and in principle could interact favorably with the negatively charged α-FeOOH surface through electrostatic attraction. The low degradation efficiency indicates that in practice adsorption and surface charge transfer were not sufficiently favorable. Indeed, part of the adsorbed MB may have desorbed back to the solution due to the effect of the light which would explain even more the limited net degradation observed, as will be discussed later with the C/C_0 data (Table 2). Moreover, the relatively high electron-hole recombination rate of α-FeOOH may reduce the number of photogenerated charges for the formation of •OH and $•O_2^-$ radicals [37] , which results in low oxidation efficiency.

For MO, the characteristic absorption peak at approximately 463–464 nm remained largely unchanged throughout the reaction. This absorption region corresponds to the azo conjugated system (–N=N–) responsible for the MO molecule colour. No new peaks are observed and only a slight change in absorption intensity is observed, which indicates that the azo bond was not broken to a great extent [38]. This suggested that the oxidative radicals generated on the surface of α-FeOOH were not powerful enough and not sufficient to cleave the azo group. In addition, the weak adsorptive interaction between MO and the catalyst surface [39], [40] further limited the contact of MO molecules with reactive sites.

For RhB, the absorption peak at approximately 553–554 nm was maintained in the presence of α-FeOOH. The intensity of absorption was only weakly reduced and no significant blue shift of the peak was observed. In many efficient photocatalytic systems, the stepwise N-deethylation and degradation of the xanthene structure leads to a peak shift to shorter wavelength for RhB. However, the phenomenon was only slightly observed in the present results, indicating that the molecular structure of RhB was relative stable. This means that α-FeOOH was not effective in promoting the oxidation and degradation of RhB under the tested conditions.

In contrast to the above four dyes, MG showed a significant decrease in the absorption intensity at ca. 616-617 nm with the illumination time. This suggests that the presence of α-FeOOH results in a significant decrease in MG concentration [41]. Along with the decrease in absorption intensity, a small blue shift of the peak was observed, pointing to the gradual breakdown of the conjugated electronic system of the MG molecule. This phenomenon may be due to stepwise N-demethylation and cleavage of bonds in the triphenylmethane structure of MG [42] to produce oxidative intermediate products. These results suggest that not only was MG adsorbed on the surface of the material, but it was also photocatalytically degraded.

The mechanism of photocatalytic degradation of MG is described as follows: under irradiation, α-FeOOH absorbs photons to produce photogenerated electron-hole pairs. The electrons in the conduction band can reduce the dissolved oxygen in the solution to produce superoxide radicals ($•O_2^-$), and the holes in the valence band can oxidize H_2O or OH^- to form hydroxyl radicals (•OH) [37]. Then, these strong oxidizing radicals attack the conjugated system of the MG molecule [43] reducing the characteristic absorption intensity and causing decolorization of the solution. Compared with the other dyes, MG may have a better interaction with the α-FeOOH surface, which may provide better accessibility to the active sites and make it more susceptible to oxidation under illumination.

To gain insight into the structural origin of the observed selectivity, the susceptibility of each dye framework to attack by reactive oxygen species ($^{\bullet}OH$ and $^{\bullet}O^{2-}$) generated at the α-FeOOH surface was examined.

Malachite Green (MG) has a triphenylmethane skeleton in which the three phenyl rings are linked via a single sp^2-hybridized carbon atom in the center, not fused into a rigid polycyclic core. The positive charge in the cationic MG^+ species is mainly distributed over the terminal N,N-dimethylaminophenyl groups resulting in a central carbon atom that is both electron-deficient and sterically accessible. The electron-poor central site readily attacks the surface-generated superoxide radicals ($^{\bullet}O^{2-}$) nucleophilically, while the photogenerated holes (h^+) and $^{\bullet}OH$ radicals promote concurrent N-demethylation (as evidenced by the characteristic blue shift in Figure 5c). Decolorization occurs rapidly as one cleavage at this central bridging carbon permanently collapses the extended π-conjugation of the chromophore.

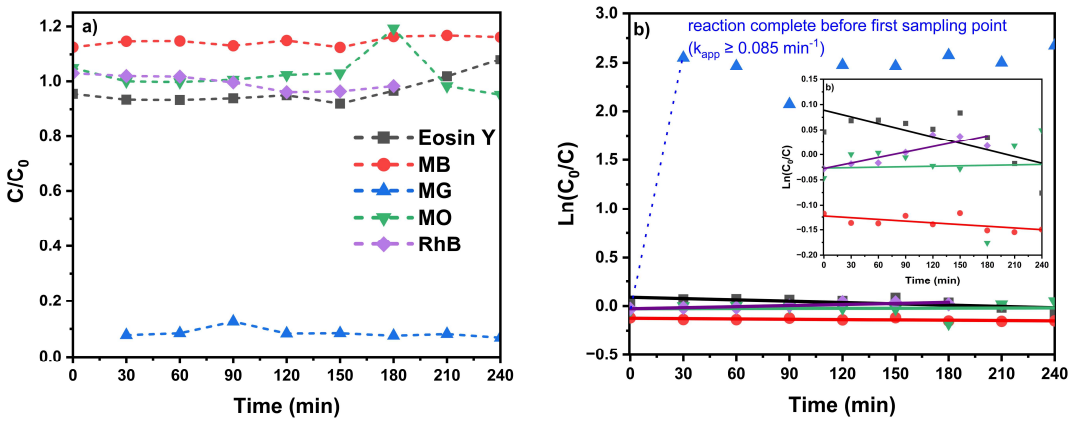
However, Methylene Blue (MB) has a planar fused phenothiazine ring and Rhodamine B (RhB) and Eosin Y are comprised of fused xanthene systems. These rigid aromatic extended structures do not have a single-bonded central carbon bridge exposed. The weaker surface radical densities mainly provide the first oxidative attacks on the peripheral alkylamino groups or heteroatoms (e.g. S or N in MB), thus keeping the central conjugated core intact and maintaining the optical absorption in the visible region. Combined with the moderate radical generation rate of single phase α-FeOOH, this results in the high resistance of these robust heterocyclic frameworks to photocatalytic cleavage under the same conditions, leading to the observed high structural selectivity.

MG differs from the other two cationic dyes in one important respect. The three aryl rings are not fused together, but are connected at a single sp^2 hybridized carbon which carries little of the positive charge, most of which resides on the two N,N-dimethylaminophenyl rings. The carbon is electron-deficient and sterically accessible, so the nucleophilic $\bullet O_2^-$ generated at the α-FeOOH surface attacks it readily, and one such attack yields the colorless carbinol and destroys the chromophore. The dimethylamino groups are oxidized in parallel by the photogenerated holes, in agreement with the N-demethylation indicated by the blue shift in Figure 5(c). There is no other site for MB and RhB. Their fused and planar phenothiazine and xanthene cores start oxidation at the sulfur or at the alkylamino substituents, leaving the conjugated core and its absorption band unchanged.

Figure 5 indicate that α-FeOOH has great photocatalytic selectivity towards the dyes tested. The material did not significantly affect the degradation of Eosin Y, MB, MO or RhB, but it strongly promoted the degradation of Malachite Green (MG). The different degradation efficiencies for the different dyes may be attributed to their adsorption behavior on the α-FeOOH surface, the molecular structural stability and the generation and utilization efficiency of reactive oxidizing radicals under illumination. The absorption intensity of the MG dye decreased significantly with time, indicating effective photocatalytic degradation, whereas the other dyes remained almost unchanged under the same experimental conditions.

Table 2. *C/C_0 values of the dyes at a concentration of 20 ppm over time under illumination with 0.1 g of α-FeOOH catalyst.*

Time	Eosin Y	MB	MG	MO	RhB
0	0.955	1.126	-	1.048	1.029
30	0.934	1.147	0.078	1.000	1.019
60	0.933	1.148	0.085	0.997	1.017
90	0.939	1.131	0.126	1.006	0.996
120	0.950	1.150	0.084	1.023	0.961
150	0.920	1.125	0.085	1.029	0.964
180	0.966	1.164	0.076	1.193	0.983
210	1.018	1.168	0.082	0.983	-
240	1.079	1.162	0.069	0.952	-



(a) *(b)*

***Figure 6**. (a) C/C₀ and (b) ln(C₀/C) versus irradiation time for the five dyes (20 ppm) over 0.1 g of α-FeOOH. Solid lines in (b) are linear fits over 30–240 min; the dotted segment for MG marks the unresolved 0–30 min interval, giving a lower bound only. Inset: the four non-degraded dyes, magnified.*

***Table 3.** Apparent pseudo-first-order rate constants (K_{app}), coefficients of determination and removal efficiencies at 240 min for the five dyes over α-FeOOH under illumination.*

Dye	K_{app} ($\times 10^{-3}$ min^{-1})	R^2	Removal at 240 min (%)
MG	≥ 85	-	93.1%
Eosin Y	-0.44	0.51	≈0
MB	-0.11	0.42	≈0
MO	0.03	0.00	≈0
RhB	0.36	0.73	≈0 Mesured up to 180 min

The photocatalytic performances of the α-FeOOH for five dyes (Eosin Y, MB, MG, MO and RhB) are compared by the changes of the C/C_0 ratio during the illumination as shown in Figure 6. The decreased C/C_0 value represents the improved degradation efficiency (Figure 6a). The experimental results indicated that the catalytic efficiency of α-FeOOH was quite different for the dyes, MG showed an excellent degradation efficiency, while Eosin Y, MO, MB and RhB showed negligible degradation during the experiment (Table 3 and Figure 6b).

The C/C_0 values obtained for Eosin Y and MO (anionic dyes) showed little variation which is in agreement with the electrostatic repulsion between these compounds and the negatively charged α-FeOOH surface described in the section of Figure 5 which limits the access of the dye molecules to the oxidizing radicals. Although electrostatic interactions may theoretically favor the adsorption for MB and RhB (cationic dyes), the degradation efficiency was still rather low. This indicates that electrostatic attraction is a necessary but not a sufficient condition for degradation. In particular, the C/C_0 value for MB even slightly increased over time, which further corroborates the light-induced desorption phenomenon discussed above. In addition, the structural stability of the phenothiazine (MB) and xanthene (RhB) frameworks also impeded the oxidation process.

Because C_0 is benchmarked against the catalyst-free illuminated control rather than normalized to a single initial point of the catalytic run, C/C_0 at $t = 0$ is not identically unity and minor fluctuations around or slightly above 1.000 arise from experimental variability, minor volume, evaporation changes during the 4 h lamp exposure, desorption. In addition, because RhB measurements were collected up to 180 min, its kinetic fit and degradation assessment are limited to the available data window 0–180 min.

In contrast, for MG the C/C_0 value dropped substantially during the first 30 minutes and afterwards oscillated between 0.069 and 0.126 without any constant downward trend in the following time intervals. This result shows that α-FeOOH possesses excellent photocatalytic activity for MG, which is in agreement with the good electrostatic attraction between the cationic MG^+ species and the negative surface of the catalyst and and with the triphenylmethane structure of MG, which is more prone to oxidation than those of the other dyes.

4. Conclusions

α-FeOOH was successfully prepared by the hydrothermal method. The XRD analysis confirmed that the obtained material presents characteristic single-phase goethite crystal structure with nanoscale crystallite size and relatively good crystallinity. The results of the photocatalytic activity showed that α-FeOOH had good selectivity to the studied dyes. Eosin Y, methylene blue (MB), methyl orange (MO) and rhodamine B (RhB) showed no significant degradation. This was attributed to electrostatic repulsion, which hindered the adsorption of anionic dyes Eosin Y and MO. On the contrary, for the cationic dyes MB and RhB, it was attributed to the high molecular structural stability with light induced desorption. The planar phenothiazine (MB) and xanthene (RhB) cores provide no electron-deficient bridging carbon comparable to that of the triphenylmethane framework of MG, so that their chromophores cannot be opened in a single oxidative step. The material's relatively high electron-hole recombination rate also contributed to this limited activity. On the contrary, the degradation of Malachite Green (MG) was efficient with removal efficiency of > 90%. This was attributed to the good electrostatic attraction between cationic MG species and the negatively charged α-FeOOH surface. This attraction assisted efficient access to the oxidizing radicals generated on the catalyst surface. The sterically exposed, electron-poor central carbon of its non-fused triphenylmethane structure further makes MG intrinsically more vulnerable to radical attack than the other dyes examined. α-FeOOH is a promising material for use in selective photocatalytic systems for treating wastewater containing organic dyes. The efficiency and practical applicability can be further enhanced by coupling with semiconductor heterojunctions or photo-Fenton systems.

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Conflict of Interest

The authors declare no conflict of interest.

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