

Green synthesis of nickel ferrite nanoparticles using starch for visible-light photocatalytic degradation of crystal violet

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Abstract. This study presents the green synthesis of NiFe_2O_4 nanoparticles with starch as a reducing and stabilising agent. The material was characterised with XRD, FT-IR, SEM, TEM, BET, and UV-Vis DRS, confirming a spinel structure, mesoporosity ($58.3 \text{ m}^2/\text{g}$), uniform spherical morphology (10–20 nm), and a band gap of 2.54 eV. Photocatalytic degradation of crystal violet under visible light achieved a 76.0% efficiency within 120 minutes, following pseudo-first-order kinetics. Compared with other photocatalysts, NiFe_2O_4 exhibits superior activity. The starch-mediated green synthesis improved dispersion and photocatalytic performance, offering a sustainable route for efficient visible-light-driven photocatalysts in environmental remediation.

Keywords: NiFe_2O_4 , photocatalysis, crystal violet, degradation, visible light

1 Introduction

The increasing contamination of water sources due to the discharge of industrial effluents, particularly from textile, food, and paper industries, has raised significant concerns regarding environmental and human health risks [1–3]. Among the most prevalent contaminants are synthetic dyes, especially crystal violet (CV), which are widely used in various industrial applications. These dyes are known for their stability and persistence in the environment and toxicity, making their removal from wastewater an urgent environmental challenge [4]. Traditional methods for dye removal, such as adsorption and coagulation, often lack efficiency and are not suitable for large-scale applications. Therefore, advanced techniques, particularly photocatalysis, have gained attention because of their capacity to degrade organic pollutants under light irradiation [5, 6].

Photocatalysis has proven to be an effective and sustainable approach for the degradation of organic contaminants in wastewater. Metal oxide semiconductors, such as TiO_2 , ZnO , and NiFe_2O_4 , have been extensively studied because of their capacity to generate reactive oxygen species (ROS) under light irradiation, which can break down organic pollutants into non-toxic by-products [7, 8]. Among these materials, NiFe_2O_4 emerged as a promising photocatalyst because of its unique properties, such as a narrow bandgap (2.0 to 2.5 eV), high stability, and magnetic properties that allow for easy recovery from solutions [9, 10]. Moreover, NiFe_2O_4 's capacity to absorb visible light makes it particularly attractive for photocatalytic applications, as it can be activated by sunlight, which is a readily available and renewable energy source [11–13].

The photocatalytic activity of NiFe_2O_4 can be significantly influenced by its synthesis method, particle size, and surface area. Materials

with mesoporous structures have been shown to exhibit enhanced photocatalytic performance because of the increased number of active sites available for adsorption and reaction [5, 9]. In particular, NiFe_2O_4 nanoparticles with a large surface area have been reported to demonstrate excellent photocatalytic degradation of various organic dyes, including CV, under visible light. Additionally, the use of green synthesis methods, such as utilising renewable resources like starch as a reducing agent, aligns with the growing demand for eco-friendly and sustainable production techniques [14–16].

Recent research has also focused on improving the photocatalytic efficiency of NiFe_2O_4 by modifying its morphology and crystallinity. Studies have shown that calcination at various temperatures can alter the crystallite size and surface properties of NiFe_2O_4 , affecting its photocatalytic efficiency. The synthesis of NiFe_2O_4 with starch has been reported to result in nanoparticles with favourable properties for photocatalysis, including high dispersion, smaller particle sizes, and larger surface areas [13, 17].

This study aims to synthesise NiFe_2O_4 nanoparticles with a green approach with starch and evaluate their photocatalytic activity for the degradation of CV under visible light. The synthesis, characterisation, and photocatalytic performance of NiFe_2O_4 will be compared with that of other commonly used photocatalysts, highlighting its potential for environmental applications in wastewater treatment.

2 Experimental

2.1 Chemical

The chemicals used in this study include $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (99.99%), $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ (99.99%), starch ($\geq 99\%$, China), and crystal violet ($\geq 99\%$, Merck, Germany).

2.2 Instrument

The equipment used in this study includes an ultrasonic machine, an oven, a furnace, a Precisa XB 220 analytical balance (Switzerland), a magnetic stirrer, a centrifuge, and an Osram lamp (160 W, 240 V).

The crystal phase composition of the material was determined by using X-ray diffraction (XRD) on a Bruker D8-Advance diffractometer (Germany), with a Cu anode X-ray tube, $\lambda(\text{Cu-K}\alpha) = 1.540 \text{ \AA}$, 30 kV, 0.01 A, scan step of $0.1^\circ/\text{s}$, and scan angle range of $10\text{--}60^\circ$. The morphology, structure, and relative size of the material were observed by using transmission electron microscopy (TEM) on a JEOL JEM-1010 instrument (Japan). The surface morphology of the photocatalyst was observed on a field emission scanning electron microscope (FESEM, Hitachi S-4800, Japan). The specific surface area (BET) of the material was determined by using N_2 adsorption-desorption at 77 K on a Micromeritics TriStar II Plus 3030 apparatus (USA). The band gap energy of the material was determined by using ultraviolet-visible diffuse reflectance spectroscopy (UV-Vis DRS) recorded on a BaSO_4 substrate using a UV2600 spectrophotometer (Shimadzu, Japan). UV-Vis spectra were recorded with a UViline 9400 and an Analytik Jena AG - SPECORD 50 UV-VIS spectrophotometer with a quartz cuvette.

2.3 Synthesis of nickel ferrite

$\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, starch (PA, China), and H_2O were mixed in a 100 mL round-bottom flask at a $\text{Ni}^{2+}/\text{Fe}^{3+}$ ratio of 2.0, a molar ratio of starch/ $(\text{Ni}^{2+} + \text{Fe}^{3+})$ of 1.5, and a molar ratio of $\text{H}_2\text{O}/\text{starch}$ of 120. The reaction mixture was stirred and heated to 80°C for one hour to dissolve the starch. Subsequently, the entire mixture was transferred into a hydrothermal reactor and treated at 180°C for 24 hours. The product was washed several times with distilled

water, then dried at 100 °C until a constant weight was achieved, resulting in NiFe_2O_4 . The sample obtained after the hydrothermal treatment was calcined at 700 °C for three hours to promote the crystallisation of the material, designated as NiFe_2O_4 -700 [9, 18, 19].

2.4 Photocatalytic activity investigation

The photocatalytic reactor was composed of a 250 mL Erlenmeyer flask, which was positioned on a magnetic stirrer inside a wooden chamber with dimensions of 24 × 40 × 40 cm. A tungsten filament lamp (Osram, 160 W, Germany) was employed as a simulated sunlight source and was fixed at a distance of 7 cm above the reaction vessel. An opening was incorporated at the top of the chamber to allow light irradiation, while a ventilation fan was installed on the side to maintain a stable reaction temperature of approximately 27 °C. For each photocatalytic experiment, 100 mL of CV solution at various initial concentrations (10, 15, 20, and 25 ppm) was introduced into the flask, followed by the addition of 0.05 g of the synthesised NiFe_2O_4 photocatalyst. Prior to exposure to light, the suspension was stirred in the dark for 60 minutes to establish adsorption-desorption equilibrium. Throughout both the dark and irradiation phases, 5 mL aliquots of the reaction mixture were withdrawn at predetermined intervals and subjected to centrifugation for catalyst separation. The concentration of CV in the obtained supernatant was subsequently determined UV-Vis spectroscopically at a wavelength of 585 nm.

The photocatalytic degradation efficiency of CV was calculated with the following formula

$$F(\%) = \frac{(C_0 - C_t) \times 100}{C_0}$$

where C_0 and C_t represent the initial concentration of CV and the concentration of CV at time t .

3 Results and discussion

3.1 Characterisation of spinel nickel ferrite

The X-ray diffraction (XRD) patterns of both uncalcined NiFe_2O_4 and NiFe_2O_4 -700 (calcined at 700 °C) exhibit characteristic peaks at 18.6, 30.3, 35.7, 37.2, 43.4, 54.1, and 57.4°, corresponding to the (111), (220), (311), (222), (400), (422), and (511) planes (JCPDS Card No. 10-0325), indicative of the spinel cubic structure (Figure 1) [11, 14]. The crystallite size of the uncalcined NiFe_2O_4 is 12.3 nm, while the calcined NiFe_2O_4 -700 exhibits a larger crystallite size of 18.9 nm. The broader and less intense diffraction peaks of the uncalcined material suggest a more amorphous structure. These findings are consistent with those from recent studies, reporting similar XRD patterns and crystallite sizes for NiFe_2O_4 nanoparticles [12, 17]. NiFe_2O_4 underwent further analysis because of its favourable structure, well-suited for the subsequent photocatalytic studies [20].

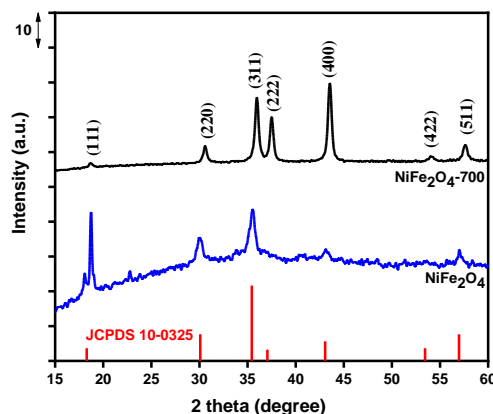


Fig. 1. XRD pattern of NiFe_2O_4

The FT-IR spectrum of NiFe_2O_4 exhibits characteristic peaks at 3381, 2987, 1610, 1392, 1319, and 590 cm^{-1} (Figure 2). These bands are consistent with the typical vibrational modes of NiFe_2O_4 , including the stretching vibration of O-H groups at 3381 cm^{-1} , the stretching vibration of C-H bonds at 2987 cm^{-1} , and the Fe-O stretching modes at 590 cm^{-1} . The observed peaks align with those reported in recent studies,

supporting the identification of the material as NiFe_2O_4 [21, 22].

The nitrogen adsorption/desorption isotherms of NiFe_2O_4 reveal a type IV isotherm with a hysteresis loop, characteristic of mesoporous materials (Figure 3). The specific surface area (S_{BET}) of the NiFe_2O_4 material was calculated to be $58.3 \text{ m}^2/\text{g}$. These results indicate that the material possesses a significant surface area, which is consistent with the findings in recent studies on NiFe_2O_4 nanoparticles, where similar nitrogen adsorption behaviour and surface areas were observed. These findings are in agreement with those in recent publications, which reported similar surface areas and mesoporous structures for NiFe_2O_4 , further confirming its potential as a suitable material for photocatalytic applications [19, 23, 24].

The scanning electron microscopy (SEM) image of NiFe_2O_4 shows well-dispersed nanoparticles with a relatively uniform size distribution (Figure 4). The morphology observed suggests the presence of spherical particles, which is consistent with recent studies on NiFe_2O_4 nanoparticles. This structural characteristic is often associated with improved photocatalytic activity because of enhanced surface area and active site availability. These results are in agreement with similar findings in the literature, where NiFe_2O_4 nanoparticles with spherical morphology have been reported for photocatalytic applications [9, 10, 18].

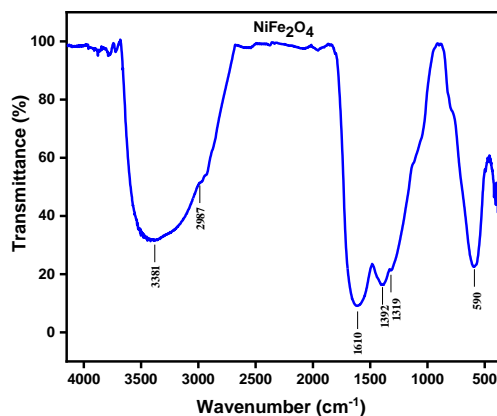


Fig. 2. FT-IR spectrum of NiFe_2O_4

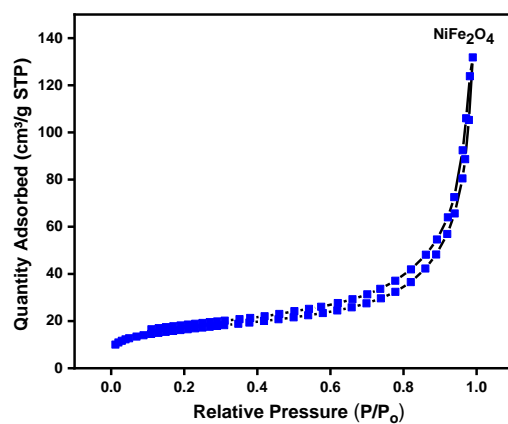


Fig. 3. Nitrogen adsorption/desorption isotherms of NiFe_2O_4

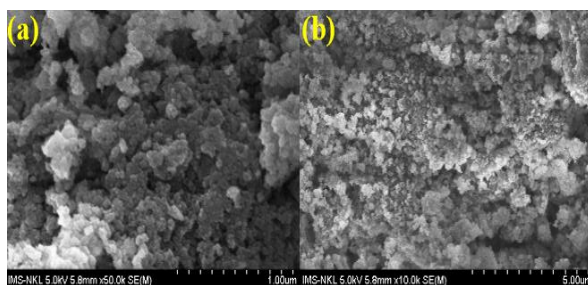


Fig. 4. SEM image of NiFe_2O_4

The transmission electron microscopy (TEM) analysis of NiFe_2O_4 reveals the nanoparticles' size and confirms the uniform distribution of particles, with an average size in the range of 10–20 nm (Figure 5). This observation supports the crystallite size obtained from XRD analysis. The TEM results align with recent

reports on NiFe_2O_4 , indicating that the nanoparticles are of appropriate size for photocatalytic processes, where small particle size is often advantageous for increased reactivity. The results are consistent with those in recent studies on NiFe_2O_4 nanoparticles for photocatalytic degradation [14, 24, 25].

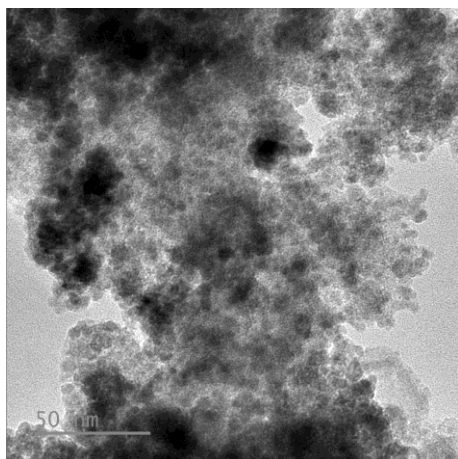


Fig. 5. TEM image of NiFe_2O_4

The UV-Vis diffuse reflectance spectrum of NiFe_2O_4 was recorded and analysed to elucidate its optical absorption characteristics, which are critical for its photocatalytic behaviour. As shown in Figure 6, a distinct absorption edge was observed in the visible light region, confirming the material's capability to harvest visible photons. This absorption onset was attributed to the electronic transitions associated with the intrinsic band structure of NiFe_2O_4 [16, 23].

The band gap energy (E_g) was estimated by applying the Tauc plot method to the UV-Vis data, where the $(\alpha h\nu)^2$ values versus photon energy ($h\nu$) was plotted, assuming a direct allowed transition. From the extrapolation of the linear portion of the Tauc plot, the band gap was determined to be approximately 2.54 eV. This value is consistent with previously reported band gaps for NiFe_2O_4 nanoparticles synthesised via similar routes [16, 23].

Such a band gap enables effective excitation under visible light irradiation, promoting the generation of electron-hole pairs, essential for photocatalytic activity. The absorption in the visible region is mainly governed by the d-d transitions of the Ni^{2+} and Fe^{3+} ions in the spinel lattice and charge transfer between metal ions and oxygen ligands [16, 23].

Furthermore, the significant light absorption capacity within the visible range explains the high photocatalytic degradation efficiency observed for CV under simulated sunlight. The broad absorption spectrum ensures that a substantial fraction of incident photons can be utilised for reactive oxygen species generation, thereby enhancing the degradation rates [16, 23].

Therefore, the UV-Vis spectrum presented in Figure 6 substantiates the suitability of NiFe_2O_4 as a visible-light-driven photocatalyst. The experimentally obtained band gap supports the material's capacity to harness visible-light energy, which correlates directly with its demonstrated photocatalytic performance [16, 23].

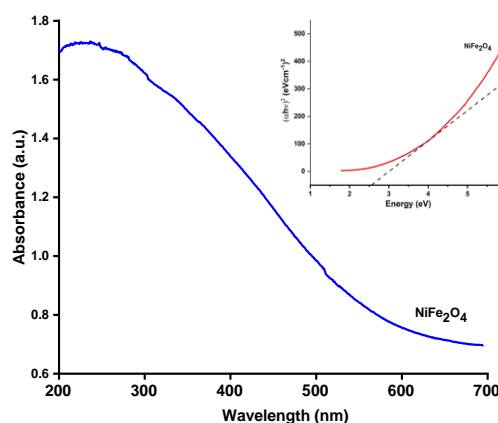


Fig. 6. UV-Vis DRS spectra and Tauc plot of NiFe_2O_4

The energy-dispersive X-ray (EDX) spectrum of NiFe_2O_4 confirms the presence of the nickel (Ni) and iron (Fe) elements, with a Ni/Fe ratio of approximately 2:1 (Figure 7). The element distribution images show a uniform distribution of Ni and Fe in the sample, supporting the

homogeneous composition of the material. These results are consistent with the expected composition of NiFe_2O_4 and are in agreement with those of recent studies, reporting similar elemental distribution and compositions in NiFe_2O_4 nanoparticles. These findings align with the results from recent studies on NiFe_2O_4 , where similar elemental distributions and ratios were observed [9, 18, 19].

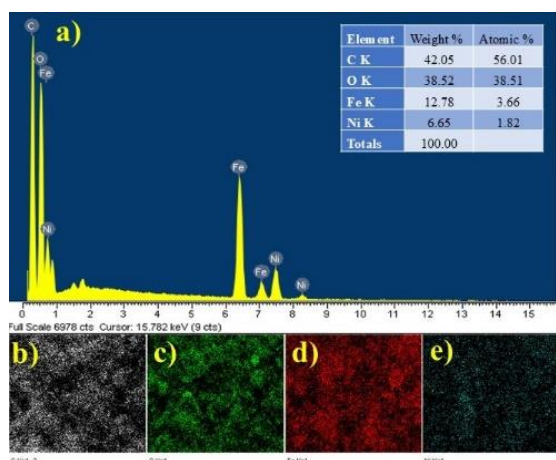


Fig. 7. (a) EDX spectra and (b-e) element distribution of NiFe_2O_4

The graph shows the photocatalytic degradation of CV under visible light, where NiFe_2O_4 exhibits significant dye degradation compared with the blank sample (Figure 8). The data were obtained by monitoring the ratio of the concentration of CV (C/C_0) over time.

During the first 60 minutes, a dark adsorption phase was observed for both NiFe_2O_4 and the blank sample, where no significant change in the CV concentration occurred. However, once the light irradiation began (after 60 minutes), the photocatalytic degradation of CV on NiFe_2O_4 was activated, resulting in a significant decrease in the concentration of CV. After 120 minutes of light exposure, a degradation efficiency of 76.0% was obtained, whereas the blank sample exhibited negligible degradation, with an efficiency of 20.1%. This behaviour is consistent with that in recent studies, which have

also reported a significant photocatalytic degradation performance of NiFe_2O_4 under visible light. The results highlight the effective photocatalytic properties of NiFe_2O_4 , which are in agreement with those in previous works where similar photocatalysts demonstrated high efficiencies for dye degradation under visible light.

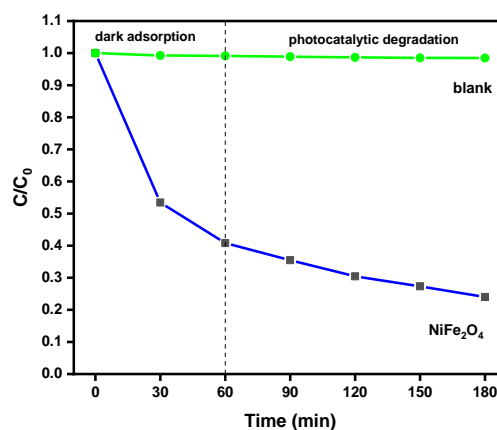


Fig. 8. Photocatalytic degradation activity of CV on NiFe_2O_4 and blank sample over time ($V_{\text{solution}} = 100 \text{ mL}$; $C_0(\text{CV}) = 10 \text{ ppm}$; $m_{\text{material}} = 0.05 \text{ g}$; $t_{\text{dark}} = 60 \text{ min}$; $t_{\text{light}} = 120 \text{ min}$, $\lambda_{\text{max}} = 585 \text{ nm}$).

The UV-Vis spectra of CV and the treated solution after photocatalytic degradation show a significant decrease in the absorption peak of CV at 585 nm, indicating the successful degradation of the dye (Figure 9). This observation is consistent with the photocatalytic results, where NiFe_2O_4 effectively degrades CV under visible light. The decrease in absorbance is in line with the results obtained in similar studies, confirming the effectiveness of NiFe_2O_4 for dye degradation. These findings are supported by similar studies in the literature, where the photocatalytic degradation of CV using NiFe_2O_4 was confirmed by UV-Vis spectral analysis [16, 20].

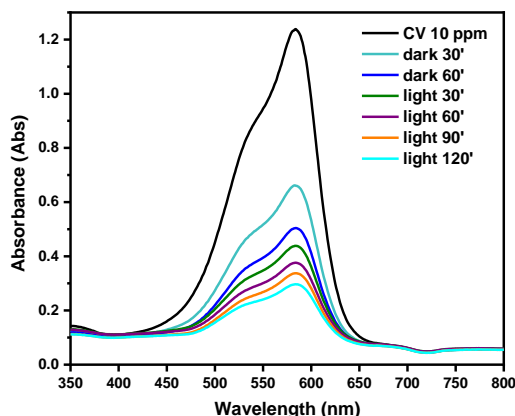


Fig. 9. UV-Vis spectra of CV and treated solution ($V_{\text{solution}} = 100 \text{ mL}$; $C_{0(\text{CV})} = 10 \text{ ppm}$; $m_{\text{material}} = 0,05 \text{ g}$; $t_{\text{dark}} = 60 \text{ min}$; $t_{\text{light}} = 120 \text{ min}$, $\lambda_{\text{max}} = 585 \text{ nm}$).

3.2 Photocatalytic degradation activity of the synthesised material

In Figure 10a, the kinetics of CV degradation on NiFe_2O_4 at different initial concentrations (10, 15, 20, and 25 ppm) are presented. The data clearly show that as the initial concentration of CV increases, the degradation rate decreases. The CV concentration decreases with time under visible-light irradiation, with the most significant reduction observed at the lowest initial concentration (10 ppm). After 120 minutes, the CV concentration for the 10 ppm sample decreased by 76.0%, as indicated in Table 1, while the degradation at higher concentrations, such as 25 ppm, was less efficient. This trend is consistent with the expected behaviour for photocatalytic processes, where lower concentrations typically result in higher degradation rates because of the higher availability of active sites for adsorption and reaction.

In Figure 10b, the natural logarithm of the concentration ratio ($\ln(C_t/C_0)$) is plotted against time for each initial concentration, showing the characteristic linear relationship expected for a pseudo-first-order reaction. The slopes of the lines correspond to the rate constant (k_{app}), and the R^2 values confirm a good fit for the first-order kinetic model. The calculated rate constants (k_{app}) for each

concentration (10, 15, 20, and 25 ppm) are 4.5×10^{-3} ; 1.3×10^{-3} ; 1.2×10^{-3} and $0.5 \times 10^{-3} \text{ min}^{-1}$, respectively, with the R^2 values ranging from 0.993 to 0.996, demonstrating excellent agreement with the first-order model.

These findings indicate that NiFe_2O_4 exhibits high photocatalytic activity for the degradation of the CV dye, and the efficiency decreases as the initial concentration increases. This is likely because of the saturation of available active sites and reduced penetration of light into the solution. The kinetic parameters derived from the data further support the suitability of NiFe_2O_4 as an efficient photocatalyst for dye degradation under visible light.

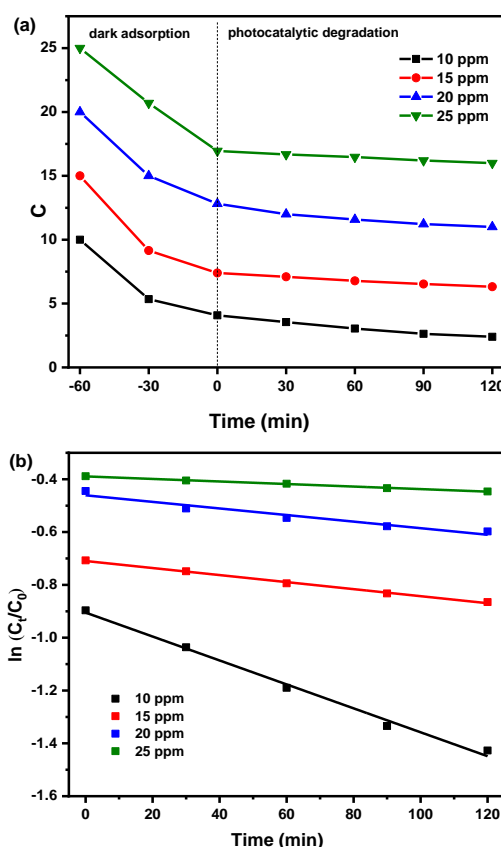


Fig. 10. (a) Kinetics of CV degradation on NiFe_2O_4 at several initial concentrations, (b) Plot of $\ln(C/C_0)$ vs. time. ($V_{\text{solution}} = 100 \text{ mL}$; $C_{0(\text{CV})} = 10\text{--}25 \text{ ppm}$; $m_{\text{material}} = 0,05 \text{ g}$; $t_{\text{dark}} = 60 \text{ min}$; $t_{\text{light}} = 120 \text{ min}$, $\lambda_{\text{max}} = 585 \text{ nm}$)

Table 1. Kinetic parameters of CV degradation on NiFe₂O₄ material

C (ppm)	F%	k _{app} (min ⁻¹)	R ²
10	76.0	4.5.10 ⁻³	0.993
15	57.9	1.3.10 ⁻³	0.996
20	45.0	1.2.10 ⁻³	0.951
25	36.0	0.5.10 ⁻³	0.996

Table 2 presents a comparison of the photocatalytic degradation efficiency of CV on various photocatalytic materials. NiFe₂O₄ demonstrates a degradation efficiency of 76.0% under visible-light irradiation, significantly outperforming other materials, such as magnetic chitosan, ZrO₂-CdZrO₃, and graphene-FeTiO₂. For example, magnetic chitosan achieves a degradation efficiency of 72%; ZrO₂-CdZrO₃ under sunlight shows a degradation efficiency of 19%, and graphene-FeTiO₂ achieves only 14.3% under visible light. These findings are in agreement with those in recent studies, which have consistently shown that NiFe₂O₄ is one of the most efficient photocatalysts for organic dye degradation under visible-light conditions.

Table 2. Comparison of CV degradation capacity on various materials

Material	Light Source	Time (min)	F %	Ref.
Magnetic-Chitosan	Visible	140	72.0	[22]
ZrO ₂ -CdZrO ₃	Sun	30	19.0	[26]
Graphene-FeTiO ₂	Visible	50	14.3	[4]
NiFe ₂ O ₄	160 W Osram bulb	120	76.0	This work

4 Conclusions

In this study, NiFe₂O₄ nanoparticles were successfully synthesised with a green approach, with starch as the reducing agent. The material

was extensively characterised, and its photocatalytic performance was evaluated through the degradation of CV under visible light. The results indicate that NiFe₂O₄ exhibited a high degradation efficiency of 76.0% after 120 minutes, making it a highly effective photocatalyst for dye degradation. The photocatalytic process followed pseudo-first-order kinetics, with the rate constants calculated for different initial CV concentrations.

Furthermore, a comparison with other photocatalysts revealed that NiFe₂O₄ outperformed several materials, including magnetic chitosan and ZrO₂-CdZrO₃, in terms of degradation efficiency, thus highlighting its potential for environmental applications. The excellent photocatalytic performance of NiFe₂O₄ was attributed to its mesoporous structure, relatively large surface area, and the availability of active sites, which enhanced its reactivity under visible light. These findings were consistent with those in recent studies on NiFe₂O₄ and similar photocatalytic materials, confirming the effectiveness of NiFe₂O₄ for environmental remediation, particularly in the degradation of organic dyes. Future studies may focus on optimising the synthesis process, exploring the recyclability of NiFe₂O₄, and evaluating its performance in real wastewater treatment scenarios.

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Conflict of interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

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