

*Research Article*

Structural, Optical, and Magnetic Characteristics of NiFe₂O₄ Nanoparticles Synthesized from Natural Iron Sand for Solar-Driven Photocatalysis

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Abstract: NiFe₂O₄ nanoparticles were successfully synthesized via a coprecipitation method using natural iron sand from Kata Beach, Pariaman, as the Fe source. The effect of synthesis pH on the crystal structure, morphology, optical, magnetic, electrical, and photocatalytic properties of the materials was systematically explored. X-ray diffraction (XRD) confirmed a cubic spinel structure for all samples, while Fourier transform infrared (FTIR) spectra revealed characteristic tetrahedral and octahedral lattice vibrations between 400–600 cm⁻¹. Scanning electron microscopy (SEM) showed significant morphological variations with pH adjustment. The nanoparticles exhibited bandgap energies of 1.52–1.54 eV, enabling strong visible-light absorption, and displayed soft magnetic behavior with saturation magnetization (Ms) of 4.4–7.5 emu/g and remanent magnetization (Mr) of 0.16–0.188 emu/g. A slight variation in bandgap energy from 1.54 to 1.52 eV was observed across the pH range investigated. Notably, NiFe₂O₄ exhibited excellent photocatalytic performance under sunlight, achieving degradation efficiencies of 98%, 84%, and 86% for Congo Red, Methylene Blue, and tetracycline antibiotic, respectively. These findings highlight NiFe₂O₄ as a highly versatile and efficient material for environmental remediation, particularly for treating water contaminated with organic pollutants.

Keywords: NiFe₂O₄ nanoparticles; Iron sand; Magnetic; Organic pollutant; Photocatalyst

1. Introduction

The rapid expansion of industrial activities in the globalization era has significantly affected the discharge of untreated effluents on environmental quality, particularly aquatic ecosystems. Industrial and other anthropogenic liquid wastes have become major environmental concerns that require effective and sustainable management to prevent secondary pollution (Bhaduri et al., 2022). Congo red and MB are aromatic heterocyclic dyes widely used in textile coloration due to their strong chromophoric properties and chemical stability (Sukandar et al., 2024). However, the fibres absorb only about 5% of the dyes, while the remaining portion is released into the environment. This process contributes to severe environmental pollution, particularly in aquatic systems, and poses health risks to humans, including bleeding, nausea, and skin irritation (Kurniasari et al., 2025; Ramu & Choi, 2021). Similarly, antibiotics, such as tetracycline, are widely used in medical and veterinary practices, leading to antibiotic-contaminated wastewater, which contributes to the emergence of resistant bacteria. Tetracycline is resistant to degradation by conventional treatment methods due to its high chemical stability and water solubility, making it a persistent contaminant in aquatic environments (Jasrotia et al., 2024). Conventional wastewater treatment methods, including chemical coagulation and biological degradation, are

often insufficient, energy-intensive, and environmentally unfriendly. Therefore, developing green, efficient, and sustainable approaches to the removal of such pollutants is an urgent necessity.

Photocatalysis has emerged as a sustainable and efficient technique for degrading organic pollutants under solar irradiation. This process has attracted increasing attention because of its ability to decompose organic compounds in aqueous systems without the production of secondary contaminants. In recent years, various low-cost and low-toxicity photocatalysts have been developed for this purpose. Spinel ferrites (MFe_2O_4 ; $\text{M} = \text{Ni}^{2+}$, Co^{2+} , Cu^{2+} , Mn^{2+} , or Zn^{2+} , or Fe^{2+}) have been widely explored owing to their narrow band gap, high chemical stability, and magnetic recoverability, which enable easy separation from treated water (Al-Tohamy et al., 2022; Chakraborty et al., 2025). Nickel ferrite (NiFe_2O_4) has gained prominence as a soft magnetic material with exceptional biocompatibility, recyclability, catalytic efficiency, and environmental benignity (Biswas et al., 2024; Cheng et al., 2023; Salih & Mahmood, 2023). NiFe_2O_4 possesses an inverse spinel structure, where Fe^{3+} ions occupy both tetrahedral (A) and octahedral (B) sites, while Ni^{2+} ions reside in the octahedral sites (Rahmayeni et al., 2019; Roberta et al., 2024). Controlling particle size and crystallinity enables the tuning of magnetic behavior from paramagnetic to ferrimagnetic, thereby enhancing the material's applicability in diverse fields (Yildirim et al., 2025). At the nanoscale, NiFe_2O_4 exhibits superior physicochemical properties compared to its bulk form, supporting its wide-ranging use in environmental remediation, catalysis, biosensing, drug delivery, and magnetic separation. The structural, magnetic, and dielectric properties of green-synthesized Ag-doped NiFe_2O_4 spinel ferrite (Malik et al., 2022; Saha et al., 2024) can be investigated.

Various methods have been developed for synthesizing NiFe_2O_4 using different precursors, with the coprecipitation technique being among the most widely employed (Tripta & Rana, 2023). Most previous studies used iron salts as the primary iron source (Aggarwal et al., 2025). Natural materials, such as iron sand, offer a sustainable, environmentally friendly alternative for ferrite nanoparticle synthesis (Nengsih et al., 2024; Saputra et al., 2020). Iron sand, which is abundant in many coastal and lowland regions, is particularly abundant in Indonesia, where large deposits are distributed across Sumatra, Java, Kalimantan, Sulawesi, and Papua (Saputra et al., 2020). It is widely used in various industries, including cement, steel, and ink manufacturing, and typically exhibits a dark gray to black color that intensifies with increasing iron (Fe) content. Iron sand is composed mainly of magnetite (Fe_3O_4) and hematite (Fe_2O_3) along with trace elements such as titanium, silica, manganese, calcium, and vanadium (Kristina et al., 2024), iron sand possesses a high iron concentration suitable for ferrite synthesis. Several studies have successfully used iron sand as a raw material for producing ferrite materials. For example, iron sand from Pariaman Beach, West Sumatra, characterized by its lustrous black appearance, contains abundant magnetite (Fe_3O_4), hematite ($\alpha\text{-Fe}_2\text{O}_3$), and maghemite ($\gamma\text{-Fe}_2\text{O}_3$) phases (Mardana et al., 2024; Salomo et al., 2018). This natural resource has been employed in cement production and ferrite fabrication (Kurniasari et al., 2025). Overall, the utilization of iron sand as a precursor offers a cost-effective, abundant, and eco-friendly alternative to conventional chemical reagents, supporting the sustainable development of ferrite-based materials (Wulandhari & Erwin, 2020).

Unlike previous studies that primarily employed chemically derived Fe precursors or focused on individual photocatalytic parameters, this study integrates naturally extracted Fe_3O_4 from Pariaman iron sand with the systematic pH-controlled synthesis of NiFe_2O_4 nanoparticles. The influence of the synthesis pH on the structural, optical, magnetic, and photocatalytic properties of the materials was comprehensively investigated. The photocatalytic performance of the NiFe_2O_4 nanoparticles was evaluated under natural sunlight irradiation for the degradation of Congo red and methylene blue individually, followed by their binary mixture, and further extended to the degradation of tetracycline. In addition, the photocatalyst's magnetic recoverability was evaluated to facilitate catalyst separation and potential reuse.

2. Methods

2.1 Materials

Iron sand from Kata Beach, Pariaman, West Sumatra Province, Indonesia; $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, NaOH (Merck); 37% HCl, polyvinyl alcohol (PVA, Aldrich), silver paste, nichrome wire, distilled water, pH indicator paper, and filter paper (Whatman) were used. The dyes (Congo Red (CR) and Methylene Blue (MB)), and tetracycline were used as target pollutants.

2.2 Extraction of Fe_3O_4 from the Iron Sand

The iron-bearing beach sand used in this study was collected from Kata Beach, Padang Pariaman City, West Sumatra Province, Indonesia. Before use, the sand was thoroughly washed with distilled water to remove surface impurities and dried at 110 °C for 1 h. The magnetic separation process was performed several times using a permanent magnet to isolate the magnetic fraction, which was then finely ground with a mortar and pestle and passed through a 160-mesh sieve. The obtained fine fraction was washed with distilled water and dried at 110 °C for 2 h to obtain purified iron sand. The iron content of the magnetic sand was determined using X-ray fluorescence (XRF). Subsequently, Fe_3O_4 was extracted from this material as the iron source for the synthesis of NiFe_2O_4 . In this process, 20 g of the purified sand was dissolved in 50 mL of 37% HCl at 80 °C under continuous stirring at 800 rpm for 30 min. The resulting solution was filtered, and 2 M NaOH was added dropwise under stirring at 500 rpm until a black Fe_3O_4 precipitate formed. The precipitate was filtered, repeatedly washed with hot distilled water until the pH reached neutrality, and dried at 110 °C for 120 min. Subsequently, the resulting Fe_3O_4 was characterized to confirm its structural and morphological properties (Mardana et al., 2024; Nengsih et al., 2024).

2.3 Synthesis of NiFe_2O_4 Nanoparticles using Fe_3O_4 from Iron Sand

NiFe_2O_4 nanoparticles were synthesized via the coprecipitation method using Fe_3O_4 extracted from natural iron sand as the Fe source, and $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ as the Ni source. The Fe_3O_4 from iron sand and nickel nitrate salt was weighed stoichiometrically. Subsequently, Fe_3O_4 was dissolved in 37% (30 mL) HCl and stirred until homogeneous for 20 min at room temperature. The mixture was then filtered using filter paper, and the filtrate was added to $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, previously dissolved in 30 mL of distilled water. The resulting mixture was stirred at 80 °C. Ferrite properties are markedly affected by synthesis conditions, particularly pH (Jabbar et al., 2024). To investigate the effect of pH on NiFe_2O_4 formation, the pH of the solution was adjusted to 11–13 by dropwise addition of 2 M NaOH, followed by stirring for 3 h. The precipitate obtained was aged for 24 h and filtered. The precipitate was repeatedly washed with distilled water until the wash solution reached neutral pH (7) and dried at 110 °C for 8 h. The dried powder was ground and subsequently calcined at 600 °C for 8 h. The samples synthesized at pH 11, 12, and 13 were designated NiF11, NiF12, and NiF13, respectively, and then stored for further characterization (Rahmayeni et al., 2019). A detailed flowchart of the experimental procedure is provided in the Supplementary Data (Figure S1).

2.4 Characterization of NiFe_2O_4

The composition of the elements in the iron sand extract was analyzed by X-ray fluorescence (XRF) spectroscopy. Crystal structure and size information were obtained using X-ray diffraction (XRD) (PANalytical). Interactions between molecules and functional groups were observed using Fourier transform infrared (FT-IR) spectroscopy (PerkinElmer Universal ATR sampling Accessory). The surface morphology and elemental content of the nanoparticles were observed using Scanning Electron Microscopy Energy Dispersive X-Ray (SEM-EDX) (Hitachi Flex Sem). More detailed analysis of the structure, particle shape, and crystallinity was performed using transmission electron microscopy (TEM), high-resolution TEM (HRTEM), and

selected area electron diffraction (SAED) (TEM, JEM-1400). The magnetic properties of the nanoparticles were observed using a vibrating sample magnetometer (VSM) (VSM-250 Electromagnetic). The optical properties of the resulting nanoparticles were observed using Diffuse Reflectance Spectroscopy UV-Vis (DRS UV-Vis, FE Inspect S50). The elemental oxidation states and chemical composition of ferrites were examined via X-ray photoelectron spectroscopy (XPS) using the XPS-Kratos Analytical Axis Supra technique. Additionally, the adsorption-desorption isotherms were obtained using a surface area and pore size analyzer (SAA: Quantachrome Nova 4200e).

2.5 Photocatalytic Performance of NiFe_2O_4

The photocatalytic performance of NiFe_2O_4 was assessed using Congo red, methylene blue, and tetracycline as representative organic pollutants. Stock solutions (1000 ppm) of each compound were prepared by dissolving 1 g of each compound in distilled water and diluting to 1000 mL. Subsequently, these stock solutions were diluted to obtain working concentrations of 20, 30, 40, 50, and 60 mg/L for the dyes and 15, 20, 25, 30, and 35 mg/L for the antibiotic. Working solution aliquots were transferred into Petri dishes, and 20 mg of NiFe_2O_4 nanoparticles was added to each. The suspensions were magnetically stirred in the dark for 30 min to establish adsorption-desorption equilibrium. The mixtures were then exposed to natural sunlight for 120 min between 11:00 a.m. and 1:00 p.m. in the chemistry laboratory yard of Andalas University. As sunny and rainy weather alternates throughout the year in Padang, West Sumatra, all photocatalytic experiments were performed under comparable clear and sunny conditions. The average sunlight intensity, measured using a mobile application, was 47,314 lux. The sunlight intensity was measured consistently using the same smartphone application throughout all experiments to monitor the relative consistency of illumination conditions between experiments. The temperature of the solution during the photocatalytic reaction was maintained at 29 ± 2 °C. The solution volumes were readjusted to their initial levels with distilled water, and the catalyst was separated by centrifugation. The absorbance of the resulting supernatants was measured using a UV-Vis spectrophotometer at wavelengths of 498 nm for Congo red, 665 nm for methylene blue, and 270 nm for tetracycline. The degradation efficiency was calculated using Equation (1):

$$\text{Degradation (\%)} = \frac{A_0 - A_t}{A_0} \times 100\% \quad (1)$$

where A_0 is the initial concentration, and A_t is the concentration at time t . Control experiments without a catalyst (blank samples) were also performed under identical irradiation conditions. Several parameters related to the photocatalytic activity test, including catalyst type, concentration, irradiation time, catalyst dosage, reusability, and degradation kinetics, were investigated (Rahmayeni et al., 2021; Zulhadjri et al., 2025). The two dyes were mixed in equal volumes and concentrations. The reusability test was conducted under the optimum photocatalytic degradation conditions obtained. Each experiment was repeated three times to evaluate degradation efficiency.

3. Results and Discussion

3.1 Characteristics of Fe_3O_4 Extracted from Padang Pariaman Beach Sand

Iron sand was collected from Kata Beach, Pariaman, West Sumatra, Indonesia, and is characterized by its black and lustrous appearance. Iron sand was used as a source of Fe for the synthesis of Fe_3O_4 compounds. Elemental composition analysis using X-ray fluorescence (XRF) revealed that the Fe_3O_4 contained 83.91% Fe. Surface morphology analysis using scanning electron microscopy (SEM) and elemental composition analysis using energy-dispersive X-ray spectroscopy (EDX) (Figures 1a and 1b) showed that Fe_3O_4 consists of irregularly sized, agglomerated grains, attributed to its strong magnetic nature. Furthermore, the EDX spectrum

confirmed the proportional composition of iron (Fe) and oxygen (O) atoms. The diffraction pattern of the Fe_3O_4 sample corresponds well with the standard ICSD pattern #159976. The characteristic diffraction peaks observed at $2\theta = 30.18^\circ$, 35.62° , 43.25° , 53.68° , 57.27° , and 62.81° correspond to the (220), (311), (400), (422), (511), and (440) Miller indices, respectively, confirming the ferrite structure (Figure 1c). The crystallite size of the synthesized magnetite (Fe_3O_4) sample was calculated to be 14.8 nm using the Debye–Scherrer equation (Zulhadjri et al., 2025). The FTIR spectrum of Fe_3O_4 (Figure 1d) exhibits two main absorption bands characteristic of the spinel structure, namely the high-frequency band (ν_1) at 533.7 cm^{-1} and the low-frequency band (ν_2) at 460.1 cm^{-1} , confirming the formation of Fe_3O_4 . The magnetic properties of Fe_3O_4 were analyzed using vibrating sample magnetometry (VSM), as shown in the hysteresis curve (Figure 1e), which indicates a ferrimagnetic behavior with a saturation magnetization of 57.2 emu/g, a remanent magnetization of 13.72 emu/g, and a coercivity of approximately 20.9 Oe. The saturation magnetization of Fe_3O_4 is higher than that reported in a previous study (27.36 emu/g), in which Fe_3O_4 was synthesized from iron sand collected from the Gelar River (Mardana et al., 2024). All characterization results confirm that the Fe_3O_4 phase was successfully extracted from iron sand. These findings confirm that Fe_3O_4 can be effectively used as a precursor for the synthesis of NiFe_2O_4 .

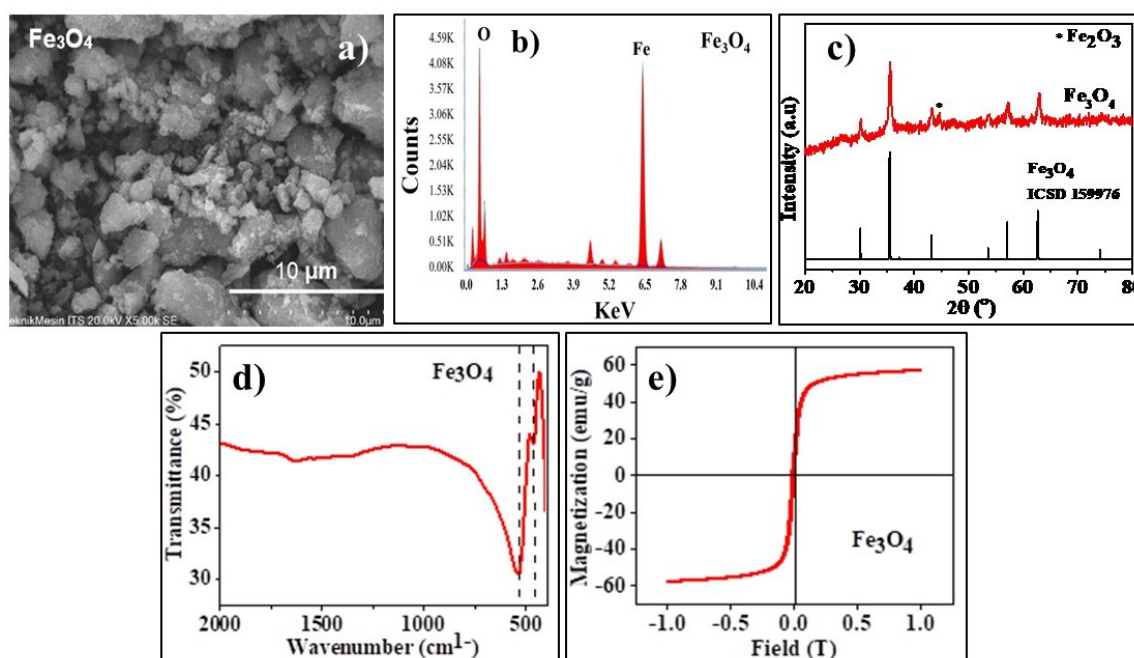


Figure 1 (a) SEM image, (b) EDX spectrum, (c) XRD pattern, (d) FTIR spectrum, and (e) magnetization hysteresis (M–H) loop of Fe_3O_4

3.2 Characterization of NiFe_2O_4

3.2.1 Crystal structure

The crystal structure of the synthesized NiFe_2O_4 nanoparticles was investigated to evaluate their phase formation, crystallinity, and structural properties. The XRD pattern of the NiFe_2O_4 sample (Figure 2a) matched the standard diffraction data (ICSD #158834). The diffraction peaks observed at $2\theta = 30.5^\circ$, 35.7° , 43.5° , 53.7° , 57.4° , and 63.1° are indexed to the (220), (311), (400), (422), (511), and (440) planes, respectively. These indices confirm the successful formation of crystalline NiFe_2O_4 with no detectable impurity phases in the NiF13 sample. The other two samples (NiF11 and NiF12) exhibited minor impurity phases corresponding to $\gamma\text{-Fe}_2\text{O}_3$ (maghemite) at $2\theta = 44.6^\circ$ (Alshehri et al., 2024). Scherrer's equation, Equation (2), was used to estimate the average crystallite size of the nanoparticles (Kurniawan et al., 2025;

Rahmayeni et al., 2025):

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

where D is the crystallite size, k is the shape factor ($k = 0.89$), λ is the wavelength of the incident X-ray radiation (Cu-K α) ($\lambda = 1.542 \text{ \AA}$), β is the full width at half maximum in radians (FWHM), and θ is Bragg's angle in radians. The crystallite sizes of NiFe₂O₄ were determined to be 5.78, 5.16, and 4.63 nm for samples synthesized at pH 11, 12, and 13, respectively. The decrease in crystallite size with increasing pH is attributed to faster nucleation at higher pH, leading to a greater number of nuclei. Consequently, the crystallites formed are smaller due to the rapid and short nucleation process (Jabbar et al., 2024).

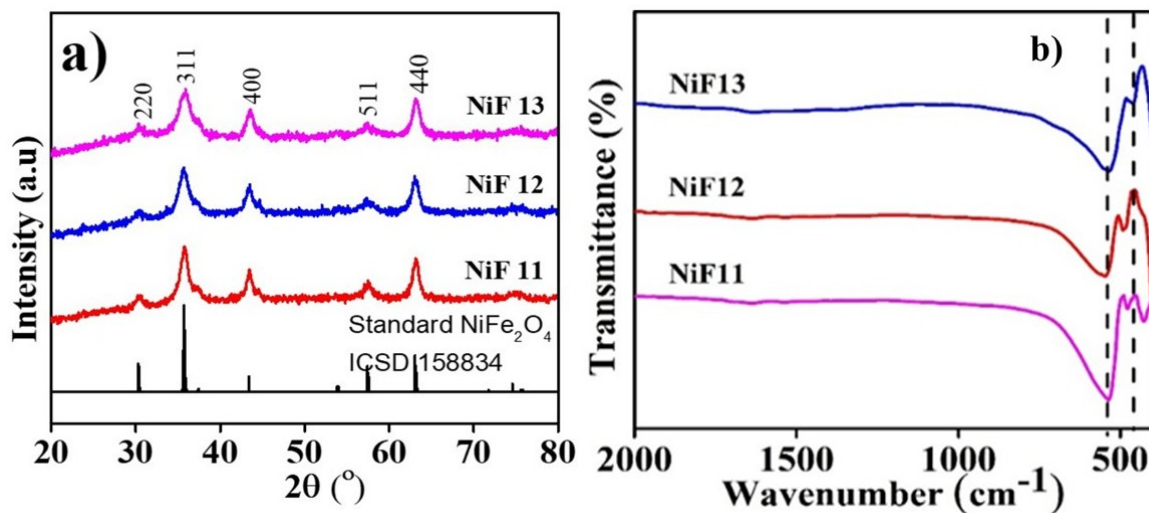


Figure 2 a) XRD pattern and b) FT-IR spectra of NiF11, NiF12, and NiF13

3.2.2 Chemical bonding and functional groups

Chemical bonding and metal–oxygen vibrations were examined to confirm the formation of the spinel ferrite structure. Figure 2b shows the FT-IR spectra of NiFe₂O₄ synthesized under different pH conditions. The observed absorption bands are attributed to the lattice vibrations of metal–oxygen bonds involving nickel and iron cations within the spinel structure. The absorption bands observed at 539, 545, and 536 cm^{−1} for samples NiF11, NiF12, and NiF13, respectively, correspond to the M–O stretching vibrations at tetrahedral sites, while those appearing at 479, 488, and 460 cm^{−1} are attributed to M–O stretching vibrations at octahedral sites (Majid et al., 2021). The obtained FT-IR spectra are in good agreement with previously reported data, thereby confirming the successful formation of NiFe₂O₄ NPs using iron sand as the Fe source. The slight variation in band positions can be attributed to differences in the bond lengths between metal ions and oxygen atoms: shorter M–O bonds at tetrahedral sites result in higher vibrational frequencies, whereas longer M–O bonds at octahedral sites lead to lower vibrational frequencies (Riyanti et al., 2023).

3.2.3 Morphology and composition

The morphology of the NiFe₂O₄ samples analyzed by SEM is presented in Figure 3(a–c). Figures 3a and 3b show the morphologies of the NiF11 and NiF12 samples, respectively, which appear as aggregated granular structures with non-uniform particle sizes. This heterogeneity can be attributed to particle agglomeration occurring during the synthesis process, a common phenomenon in the formation of magnetic nanomaterials due to the attractive forces between

particles arising from their inherent magnetic properties (Saputra et al., 2020). The NiF13 sample synthesized at pH 13 exhibited a more uniform morphology with a narrow particle size distribution, as shown in Figure 3c. The solubility of metal ions is enhanced, resulting in a higher nucleation rate relative to particle growth. This favors the rapid formation of smaller particles, thereby minimizing aggregation into larger clusters (Ghahfarokhi & Shobegar, 2020). The micrograph further reveals that the surface of the NiF13 sample is porous and evenly distributed, which can significantly enhance its performance as a photocatalyst for the degradation of organic pollutants in water. Elemental analysis of the NiFe_2O_4 samples using EDX confirmed the presence of Ni, Fe, and O, with compositions remaining consistent across the samples (Supplementary Data, Figure S2 and Table S1).

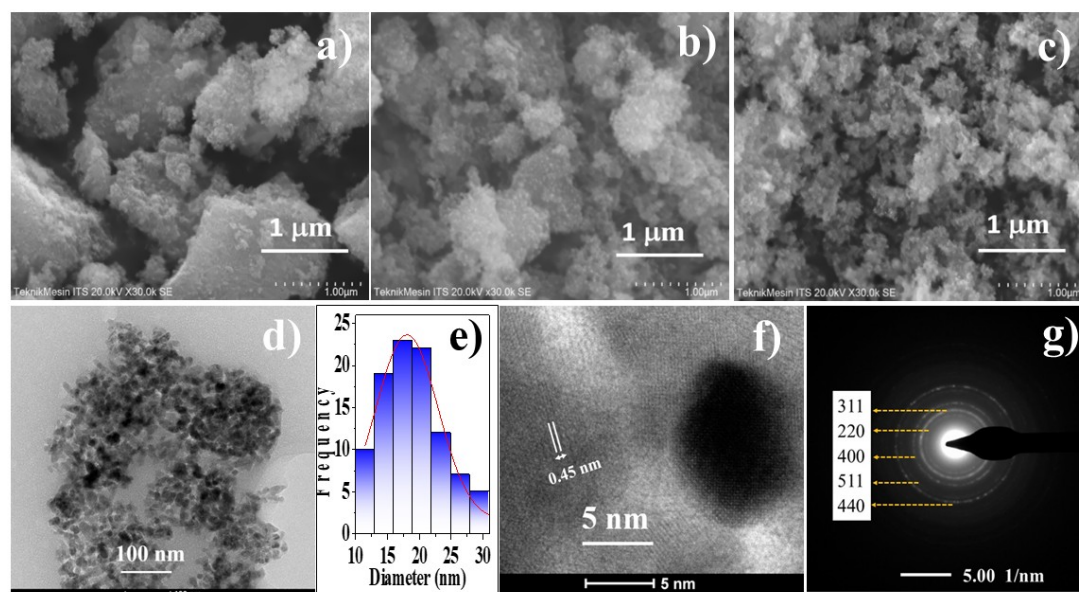


Figure 3 SEM (a–c) images of NiF11, NiF12, and NiF13 samples, and TEM (d), particle size distribution histogram (e), HRTEM (f), and SAED (g) images of the NiF13 sample

To gain a deeper understanding of the NiFe_2O_4 microstructure, the NiF13 sample was analyzed using TEM, HRTEM, and SAED techniques. The TEM image (Figure 3d) revealed that the NiF13 nanoparticles exhibited a relatively uniform granular morphology with particle sizes ranging from approximately 10 to 30 nm. Particle size analysis of 100 randomly selected nanoparticles (Figure 3e) yielded an average particle size of 18.07 nm, indicating a relatively narrow size distribution and good particle uniformity. The observed particle size is larger than the crystallite size determined from XRD, suggesting that each particle consists of one or more aggregated crystallites, which is commonly observed in ferrite nanoparticles due to magnetic interactions and high surface energy. The HRTEM image showed crystal lattice fringes, although they were not clearly resolved (Figure 3f). The selected area electron diffraction (SAED) pattern further confirmed this observation (Figure 3g). The separated dotted rings in the SAED patterns revealed the particles' crystalline structure. These rings were indexed to the (220), (311), (400), (511), and (440) crystallographic planes, consistent with the cubic spinel structure (Abouhaswa et al., 2024; Rahmayeni et al., 2025). Overall, the nanoscale particle size of NiF13 may enhance its photocatalytic performance by providing more accessible active sites for pollutant adsorption and photocatalytic reactions, while its relatively low crystallinity may facilitate charge trapping and suppress electron–hole recombination.

3.2.4 Magnetic properties

The magnetic properties of NiFe_2O_4 nanoparticles synthesized under different pH conditions were systematically investigated using a VSM instrument at room temperature, with a maximum applied magnetic field of 1000 Oe. As depicted in the hysteresis curves in Figure 4a, the saturation magnetization (M_s) of NiFe_2O_4 decreases with increasing synthesis pH, registering values of 7.55, 6.97, and 4.40 emu/g for NiF11, NiF12, and NiF13 synthesized at pH 11, 12, and 13, respectively. This reduction in M_s is closely associated with particle-size reduction, as smaller ferrite nanoparticles typically exhibit lower saturation magnetization due to finite size and surface effects.

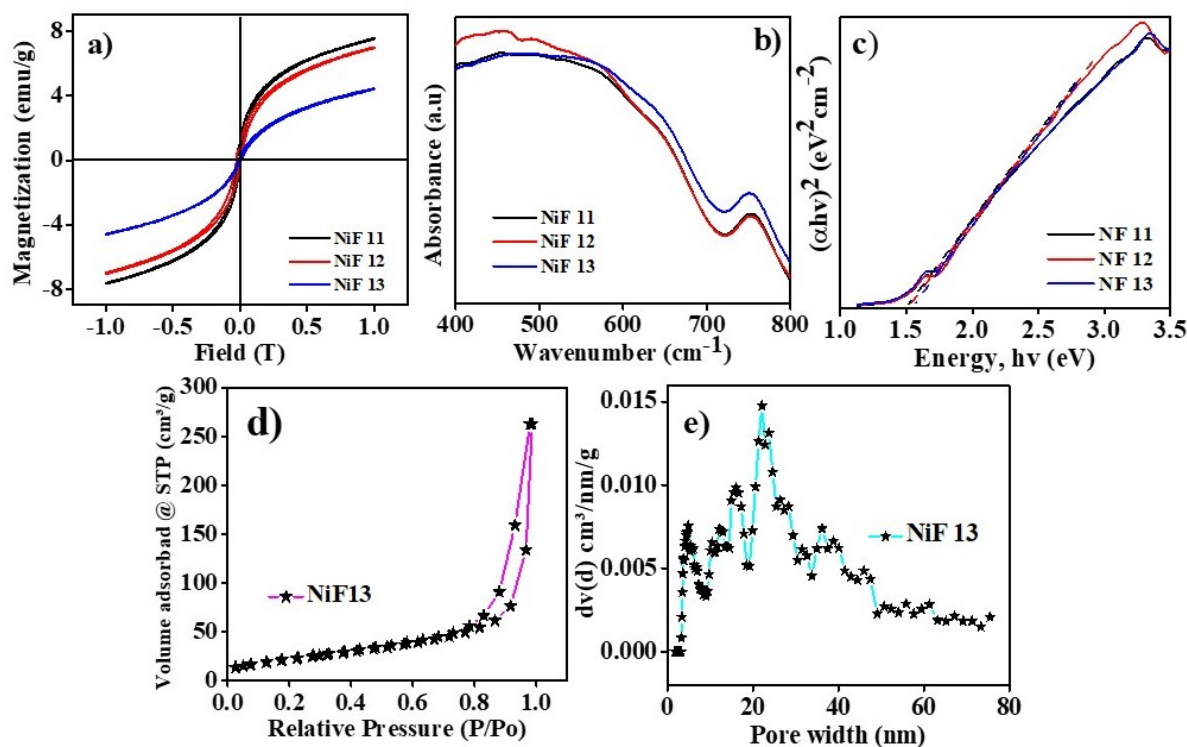


Figure 4 Hysteresis curve of NiFe_2O_4 with different synthesis pH values (a), UV-Vis DRS spectra (b), Tauc plot $[(\alpha h\nu)^2 \text{ versus } h\nu]$ curves of NiF11, NiF12, and NiF13 samples (c), N_2 adsorption-desorption isotherms (d), and pore size distribution of the NiF13 sample (e)

The crystallization process can be controlled by adjusting the pH, which influences the cation distribution in the octahedral and tetrahedral sites. Such redistribution alters the superexchange interactions, thereby modifying the magnetic response. Furthermore, elevated pH conditions affect the nanoparticle growth kinetics, which further contributes to the observed decrease in the saturation magnetization (Ghahfarokhi & Shobegar, 2020). The hysteresis curve profiles indicate that the synthesized NiFe_2O_4 nanoparticles exhibit soft magnetic behavior. These results are consistent with previous reports of NiFe_2O_4 synthesized at pH 9 and 11 via the coprecipitation method, although the observed saturation magnetization is lower than the values obtained through spray pyrolysis. The inherent magnetic properties of NiFe_2O_4 render it highly suitable for photocatalytic applications, as the material can be efficiently retrieved from aqueous media using an external magnetic field, facilitating catalyst reuse and operational sustainability (Biswas et al., 2024).

The NiFe_2O_4 sample exhibited soft magnetic behavior with relatively low coercivity (H_c). The increase in thermal energy reduces the energy barrier for domain motion, resulting in nearly zero coercivity. The magnetic properties facilitate the efficient recovery of the photocatalyst using an external magnetic field, enabling its reuse in subsequent photocatalytic cycles. The Supplementary Data (Table S2) summarizes the corresponding magnetic parameters, including

saturation magnetization (Ms), remnant magnetization (Mr), and coercivity (Hc).

3.2.5 Optical properties

Among the various factors influencing the photocatalytic activity, the most critical is the light absorption capability, which is inherently governed by the electronic structure of the material. UV-Vis spectroscopy was employed in the wavelength range of 400–800 nm to investigate the optical properties of the synthesized samples. As shown in Figure 4b, the NiFe₂O₄ samples exhibited strong absorption in the wavelength range of 400–800 nm, corresponding to the visible region, indicating their suitability as visible-light photocatalysts under sunlight irradiation. The optical bandgap energy of the NiFe₂O₄ samples was determined using the Tauc relation, as expressed by Equation (3):

$$(\alpha h\nu)^{1/n} = A(h\nu - E_g) \quad (3)$$

This equation describes the relationship between the material's optical bandgap energy (E_g) and the photon energy ($h\nu$), where h denotes Planck's constant and α is the absorption coefficient (Rasheed et al., 2025). The exponent n in the Tauc relation depends on the nature of the electronic transition; typically, $n = 1/2$ for a direct allowed transition. The proportionality constant A is specific to each material and is associated with the transition probability and the type of electronic excitation (Chakraborty et al., 2025; Debnath & Das, 2020). The bandgap energies of NiF11 and NiF12 are identical at 1.54 eV due to their overlapping absorption spectra, while NiF13 shows a slightly lower value of 1.52 eV (Figure 4c). The bandgap energy of NiFe₂O₄ decreases with increasing synthesis pH, which is consistent with previous studies on NiFe₂O₄ synthesized via the coprecipitation method (Kristina et al., 2024).

Several factors, including crystallite size, lattice parameters, and impurity concentration, influence a material's bandgap. A reduction in particle size typically results in a narrower band gap. The slight variation in bandgap energy with increasing synthesis pH may arise from the combined effects of crystallite size, structural defects, cation distribution, and impurities. The spatial restriction of electron and hole movement limits charge carrier mobility, thereby narrowing the energy separation between the valence and conduction bands. Consequently, electrons require less energy to transition between these bands, enhancing the material's potential for the photocatalytic degradation of diverse pollutants (Khatun et al., 2025).

3.2.6 Surface area and porous structure

The surface area and porous structure were evaluated to determine the textural properties that influence adsorption and photocatalytic performance. The BET analysis of the NiF13 sample, presented in the nitrogen adsorption-desorption isotherms (Figure 4d–e), shows a type IV curve with an H3 hysteresis loop, indicating a mesoporous structure (Zulhadjri et al., 2025). The NiF13 sample exhibits a specific surface area of 95.7 m²/g, a total pore volume of 0.4437 cm³/g, and a pore width in the range of 5–50 nm, consistent with the SEM observations. The mesoporous structure enhances the material's efficiency in adsorbing dye molecules and antibiotics, thereby accelerating the degradation process. An increase in relative pressure results in a higher volume of adsorbed N₂ gas. In the initial stage of adsorption, represented by the branch preceding the hysteresis loop, monolayer adsorption occurs. Once monolayer adsorption is complete, the isotherm curve exhibits a distinct hysteresis loop, indicating that the gas becomes confined within the pores of the material, leading to capillary condensation (Lee et al., 2025).

3.2.7 Surface chemical composition and electronic states

X-ray photoelectron spectroscopy (XPS) was conducted to quantitatively analyze the elemental composition and valence states of metal ions in the synthesized NiF13 sample (Figure 5). The survey spectrum over the binding energy range of 0–1400 eV (Figure 5a) displays distinct

peaks corresponding to Ni, Fe, O, and C (Baby et al., 2020; Fang et al., 2022). High-resolution spectra of O 1s, Fe 2p, and Ni 2p are shown in Figure 5b–d. The spin–orbit coupling in Ni and Fe leads to the splitting of the 2p states into $2p_{3/2}$ and $2p_{1/2}$ components. The O 1s peak at 529 eV is attributed to lattice oxygen, while the Fe $2p_{3/2}$ and $2p_{1/2}$ peaks at 710 and 723 eV indicate the presence of Fe^{3+} . Peak deconvolution reveals tetrahedral and octahedral site occupancy, consistent with previous reports. The Ni 2p spectrum exhibited peaks at 854 and 872 eV, confirming the formation of NiFe_2O_4 . Further analysis of Ni $2p_{3/2}$ shows two components corresponding to Ni^{2+} in tetrahedral sites and Ni^{3+} in octahedral sites. The detected C 1s peak is ascribed to adventitious carbon from the XPS instrument (Rahmayeni et al., 2019; Yildirim et al., 2025).

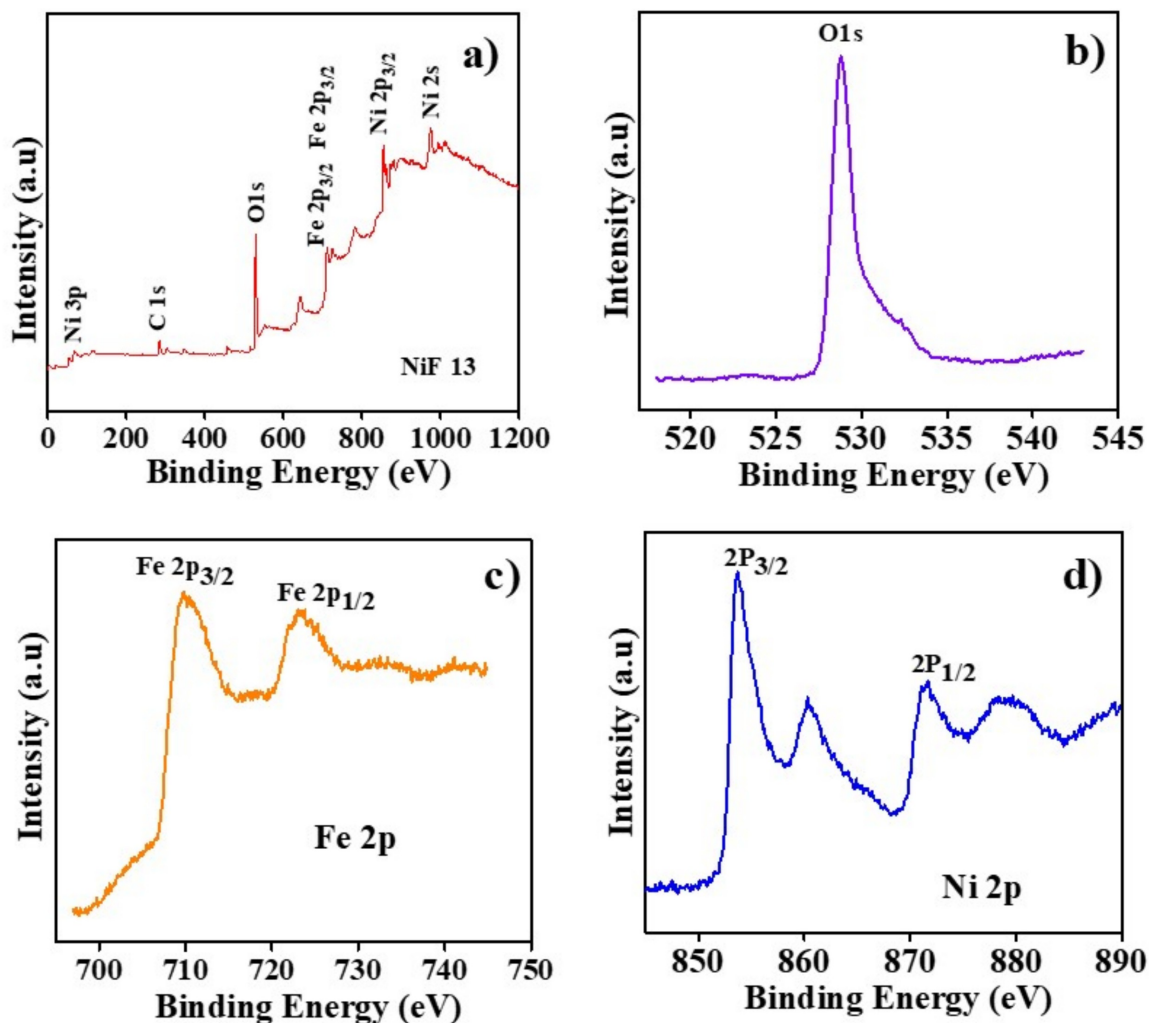


Figure 5 XPS analysis of NiF13 nanoparticles with (a) survey scan, (b) O 1s spectrum, (c) Fe 2p spectrum, and (d) Ni 2p spectrum

3.3 Photocatalytic Performance

The photocatalytic performance of NiFe_2O_4 was evaluated to investigate its ability to degrade organic pollutants under natural sunlight irradiation. The photocatalytic activity was assessed using two representative textile dyes, Congo Red (CR) and Methylene Blue (MB), together with the antibiotic tetracycline, under direct sunlight between 11:00 a.m. and 1:00 p.m. CR is an anionic azo dye ($\text{C}_{32}\text{H}_{22}\text{N}_6\text{Na}_2\text{O}_6\text{S}_2$) containing azo ($-\text{N}=\text{N}-$) and sulfonate ($-\text{SO}_3^-$) functional groups that impart a negative charge to the molecule. In contrast, MB is a cationic dye ($\text{C}_{16}\text{H}_{18}\text{ClN}_3\text{S}$) with a heterocyclic aromatic structure containing positively charged nitro-

gen atoms. Both dyes are widely used as colorants in the textile, paper, and biological industries, as well as redox indicators in analytical chemistry (Vadivel et al., 2024).

A dark adsorption experiment was conducted using Congo Red only to distinguish the contribution of adsorption from the photocatalytic process. Under dark conditions, the NiF13 catalyst exhibited only 11% Congo Red removal, whereas direct photolysis in the absence of the catalyst resulted in 22% degradation after 120 min under otherwise identical experimental conditions. These results demonstrate that adsorption only marginally contributed to pollutant removal, confirming that the enhanced degradation observed under sunlight irradiation mainly originated from the catalyst's photocatalytic activity.

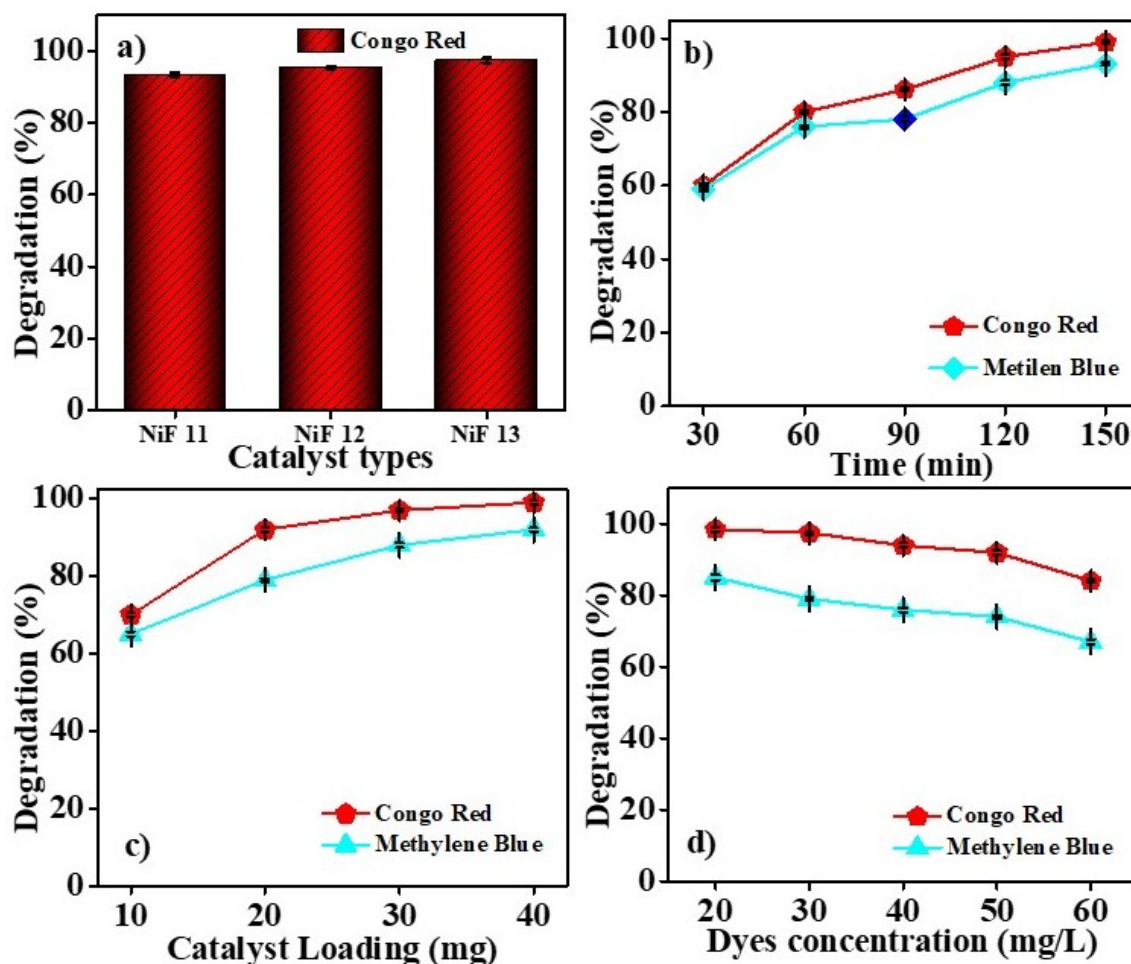


Figure 6 Catalytic performance of NiFe₂O₄ in the degradation of Congo Red (CR) and Methylene Blue (MB) dyes: (a) effect of catalyst type (NF11, NF12, and NF13), (b) effect of irradiation time, (c) effect of catalyst dosage, and (d) effect of initial dye concentration using NF13 as catalyst

Figure 6 illustrates the photocatalytic performance of the NiF series catalysts toward the degradation of CR and MB under natural sunlight irradiation. Figure 6a shows the photocatalytic activities of NiF11, NiF12, and NiF13. NiF13 exhibited the highest mean photocatalytic degradation efficiency for both Congo red and methylene blue at an initial concentration of 50 mg L⁻¹ using 20 mg of catalyst in 20 mL of dye solution after 120 min of sunlight irradiation. One-way ANOVA revealed a statistically significant difference in the photocatalytic degradation efficiencies among the three catalysts ($F = 35.64$, $p < 0.001$). Therefore, NiF13 was selected for subsequent optimization experiments. The enhanced photocatalytic activity of NiF13 can be attributed to its smoother, more homogeneous, and porous surface morphology, which provides abundant active sites for dye adsorption and degradation. Conversely, the relatively high magnetic properties of NiF11 and NiF12 may promote particle agglomeration, thereby reducing the

effective surface area and photocatalytic activity.

On the basis of these results, NiF13 was selected as the optimum catalyst for subsequent experiments. Therefore, the effect of irradiation time was investigated by varying the exposure period from 30 to 120 min. As shown in Figure 6b, the degradation efficiency gradually increased with increasing irradiation time because prolonged sunlight exposure promoted the generation of reactive oxygen species, particularly hydroxyl radicals ($\cdot\text{OH}$), which accelerated the degradation of dye molecules (Aggarwal et al., 2025). However, extending the irradiation time beyond 120 min only marginally improved the degradation efficiency. Therefore, 120 min was selected as the optimum irradiation time for all subsequent photocatalytic experiments.

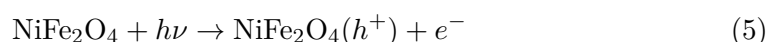
The dosage of the catalyst significantly influences the photocatalytic degradation efficiency. To evaluate this effect, various amounts of NiF13 catalyst (10, 20, 30, and 40 mg) were tested under identical conditions: dye concentration of 50 mg L⁻¹, dye volume of 20 mL, and sunlight irradiation for 120 min (Figure 6c). The degradation rate increased with catalyst loading due to the greater number of accessible active sites and enhanced surface area, which promote the generation of reactive $\cdot\text{OH}$ radicals (Rahmayeni et al., 2021). Additionally, the improved light penetration within the suspension facilitates efficient photon use. However, excessive catalyst loading decreases the photocatalytic efficiency because increased solution turbidity and particle agglomeration reduce light penetration and limit photon absorption (Chavan et al., 2024; Ullah et al., 2023). Therefore, 20 mg was identified as the optimum catalyst dosage for subsequent analyses.

The effect of the initial CR concentration (20–60 mg/L) on the photocatalytic performance was investigated to determine the optimum concentration (Figure 6d). The optimal value was obtained at 50 mg/L for both CR and MB using 20 mL of dye solution, 20 mg of NF13 catalyst, and a reaction time of 120 min. As shown in the graph, CR exhibited a higher degradation efficiency (98%) than MB (84%). This indicates that CR is more susceptible to degradation, likely due to its molecular structure, which facilitates attack by $\cdot\text{OH}$ radicals. These radicals are the active species responsible for photocatalytic degradation under sunlight irradiation. The UV–Vis absorption spectra of both dyes at different concentrations before and after irradiation are provided in the Supplementary Data (Figure S3). The reaction kinetics of the CR and MB dye degradation processes were determined using Equation (4):

$$\ln\left(\frac{[A]_t}{[A]_0}\right) = -kt \quad (4)$$

where A_0 and A_t represent the absorbance of the dye at the initial time and time t , respectively, and k is the apparent rate constant (Mohammed et al., 2024). The linear plots indicate that the degradation of CR and MB dyes follows a pseudo-first-order kinetic model, with correlation coefficients (R^2) of 0.968 and 0.931 for CR and MB, respectively (Figure 7a). Pseudo-first-order kinetic analysis is presented as evidence for the photocatalytic performance. However, the uncertainty associated with the kinetic parameters was not determined in this study and should therefore be considered when interpreting the kinetic results.

The photocatalytic process can be described using the following reaction equations:



Scavenger testing is a crucial technique used in photodegradation studies to identify the dominant reactive species responsible for pollutant degradation. Various scavengers were introduced into the reaction system under optimal conditions to elucidate the roles of charge carriers

and reactive species in dye degradation. These reagents selectively trap target species, thereby suppressing their interaction with dye molecules. Figure 7b shows the effect of different scavengers on CR degradation. Trapping experiments using NaCl, MgCl₂, ammonium acetate, H₂O₂, and EDTA were performed to clarify the photocatalytic mechanism of the synthesized nanoparticles, as these compounds are known scavengers for $\cdot\text{OH}$, h^+ , and $\cdot\text{O}_2^-$ radicals (Coromelci et al., 2022; Vargas-Bustamante et al., 2025). The addition of scavengers significantly reduced the degradation efficiency of CR over NiF13. Ammonium acetate and EDTA showed the strongest inhibitory effects, decreasing the degradation efficiency from 97% to 65%, while NaCl, H₂O₂, and MgCl₂ reduced the degradation efficiency to 71%, 71%, and 83%, respectively. Although the scavenger experiments provide valuable insight into the dominant reactive species involved in the photocatalytic process, these results indirectly infer the proposed mechanism. Direct confirmation of reactive oxygen species generation and pollutant mineralization would require complementary analyses, such as total organic carbon (TOC) measurements.

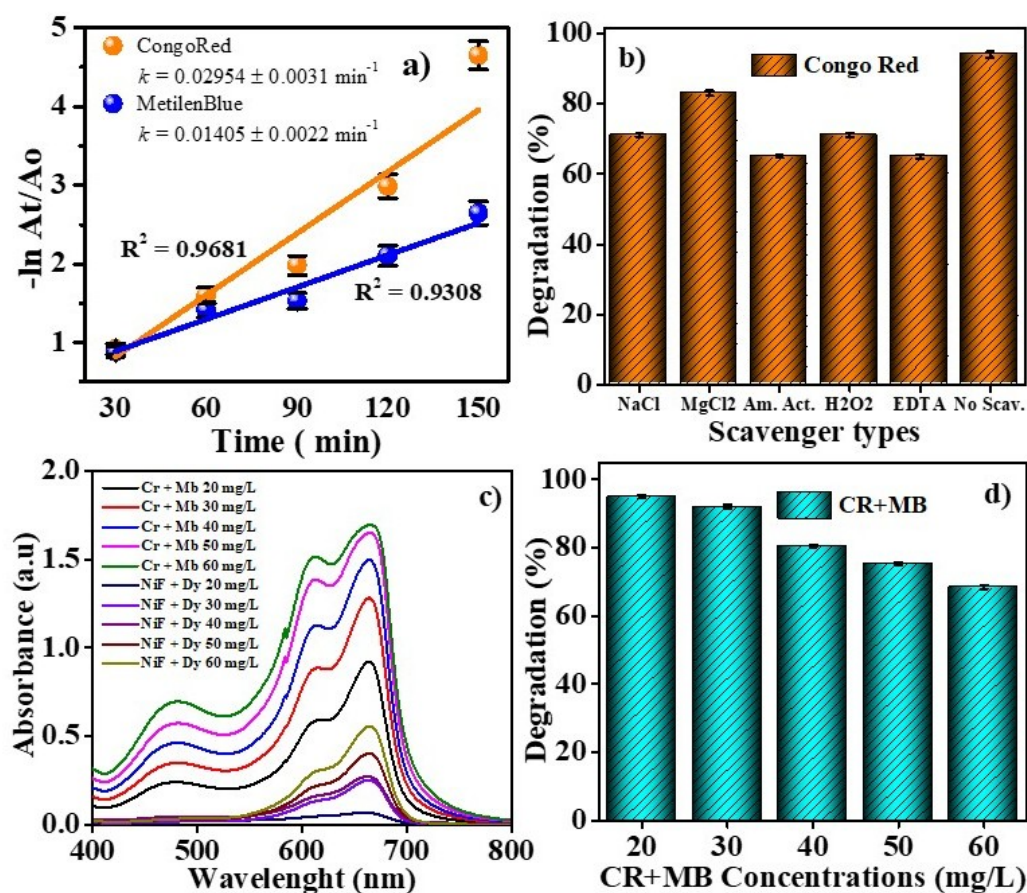


Figure 7 (a) Pseudo-first-order kinetic model of CR and MB, (b) effect of different scavengers on CR degradation, (c) absorption spectra of the mixed dye solution (CR+MB) before and after the degradation process, and (d) percentage of dye degradation at different concentrations using NF13 as the catalyst

Therefore, the proposed mechanism should be interpreted within the scope of this experimental evidence, and the reported results represent photocatalytic degradation efficiency rather than the complete mineralization of the pollutants.

The photocatalytic performance of NiF13 was further evaluated for the simultaneous degradation of CR and MB dyes. Equal concentrations of both dyes (20–60 mg/L) were mixed in a total volume of 20 mL (10 mL CR and 10 mL MB) and treated with 20 mg of NiF13 under 120 min of sunlight irradiation. The UV–Vis spectra (Figure 7c) show a significant decrease

in absorbance after 120 min, indicating effective degradation of the dye mixture. However, the degradation efficiency decreased with increasing dye concentration. This reduction is attributed to the higher number of CR and MB molecules, which hinder light penetration to the catalyst's active sites (Figure 7d), leading to a reduced generation of hydroxyl radicals during the photocatalytic process (Chavan et al., 2024). After 120 min of irradiation, the degradation percentage reached 95% at a dye mixture concentration of 20 mg/L.

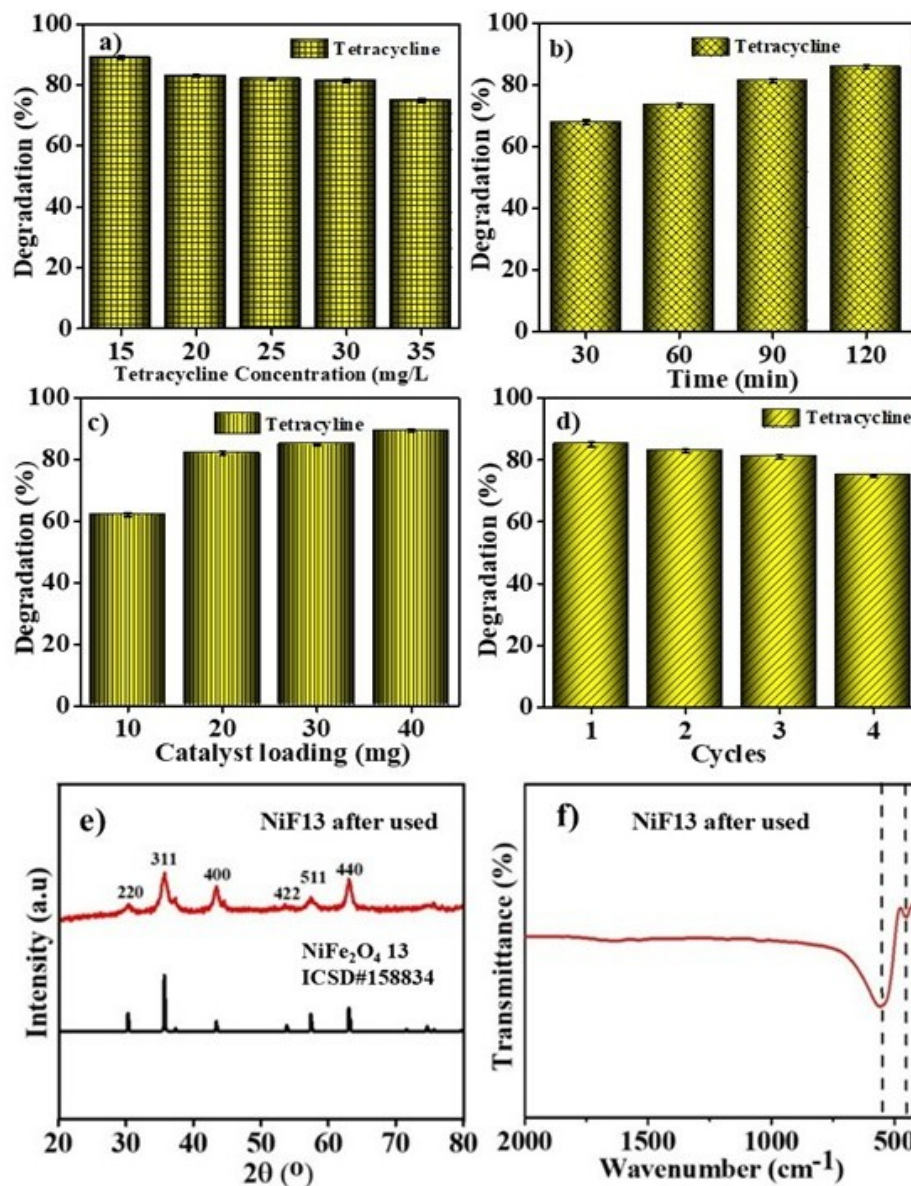


Figure 8 Photocatalytic activity of NiFe₂O₄ (NF13) toward tetracycline degradation: (a) effect of tetracycline concentration, (b) irradiation time, (c) catalyst loading, (d) reusability tests, (e) XRD pattern, and (f) FTIR spectrum of NF13 after use

The photocatalytic performance of NiF13 was further evaluated for tetracycline degradation in water, examining the tetracycline concentration, irradiation time, and catalyst dosage (Figure 8a–c). Tetracycline concentrations of 15, 20, 25, 30, and 35 mg/L were tested because tetracycline is relatively resistant to degradation. The optimum concentration was found to be 30 mg/L, which was then used for subsequent irradiation time, catalyst dosage, and reusability tests. Under the optimal conditions of 30 mg/L tetracycline, 20 mg catalyst, and 120 min irradiation, the degradation efficiency reached 86%. The stability of NiFe₂O₄ was assessed by reusing the catalyst under the same conditions for four consecutive cycles. Reusability tests over

four cycles demonstrated that the catalyst remained stable, with a slight decrease in the fourth cycle attributed to material loss during the experiments (Figure 8d).

The stability of the synthesized NiFe_2O_4 (NiF13) was assessed after photocatalytic experiments using X-ray diffraction (XRD) and Fourier transform infrared (FTIR) analyses (Figure 8e–f). Both the diffraction patterns and spectral features confirm that the material retains its structural integrity and chemical functionality, demonstrating good stability and suitability for repeated use in the photocatalytic degradation of organic pollutants in water. A comparison of the synthesis methods, precursor materials, and photocatalytic performance of NiFe_2O_4 nanoparticles reported in previous studies is provided in the Supplementary Data (Table S3). The comparison indicates that the synthesis route, precursor materials, and operating conditions influence the photocatalytic performance. In the present study, NiFe_2O_4 synthesized from natural iron sand achieved degradation efficiencies of 98%, 84%, and 86% for Congo Red, Methylene Blue, and tetracycline, respectively, and 95% for the Congo Red–Methylene Blue mixed solution under solar light irradiation. The performance falls within the range reported for NiFe_2O_4 photocatalysts under different experimental conditions, while demonstrating that natural iron sand can serve as an effective and sustainable Fe precursor for the synthesis of magnetic NiFe_2O_4 photocatalysts.

The proposed photocatalytic degradation mechanisms of Congo Red, Methylene Blue, and tetracycline are illustrated in the Supplementary Data (Figure S4). In each case, the process involves the generation of reactive species such as hydroxyl radicals ($\cdot\text{OH}$), superoxide radicals ($\cdot\text{O}_2^-$), and photogenerated holes (h^+), which attack the dye or antibiotic molecules, leading to their successive breakdown into proposed lower-molecular-weight degradation products (Biswas et al., 2024; Nugrahaningtyas et al., 2025).

Overall, increasing the synthesis pH systematically tailored the structure and properties of NiFe_2O_4 , resulting in a smaller crystallite size, a more homogeneous morphology, slight variation in bandgap energy, and enhanced photocatalytic performance. NiF13 exhibited the best overall balance between material properties and photocatalytic activity.

The structural characterization and photocatalytic results demonstrate that the optimized NiF13 possesses a favorable combination of structural, optical, magnetic, and surface properties that contribute to its superior photocatalytic performance. The BET analysis revealed a porous structure with a relatively high specific surface area, providing abundant active sites for photocatalytic reactions. The optimized magnetic NiFe_2O_4 exhibited remarkable multifunctionality as a recyclable photocatalyst for the degradation of dyes and antibiotics under solar irradiation, highlighting its significant potential for environmental remediation. Further mechanistic investigations and extended stability evaluations would provide additional insight into its photocatalytic behavior and practical implementation in wastewater treatment.

4. Conclusions

NiFe_2O_4 nanoparticles were successfully synthesized via a co-precipitation method using natural iron sand as the Fe source. The synthesis pH played a decisive role in tailoring the crystallite size, morphology, optical properties, magnetic behavior, and photocatalytic performance of the obtained NiFe_2O_4 nanoparticles. Increasing the synthesis pH from 11 to 13 reduced the crystallite size from 5.78 to 4.63 nm while preserving the spinel crystal structure, resulting in enhanced photocatalytic activity. Among the investigated samples, NiF13 exhibited the best photocatalytic performance, with degradation efficiencies of 98%, 84%, and 86% for Congo Red, Methylene Blue, and tetracycline, respectively, and 95% for the Congo Red–Methylene Blue mixed solution under solar irradiation. Furthermore, the soft magnetic properties of NiFe_2O_4 enabled rapid magnetic recovery after the photocatalytic process, demonstrating its potential as a recyclable photocatalyst for environmental remediation. Overall, these findings demonstrate that pH optimization is an effective strategy for tailoring the structure–property–performance relationship of NiFe_2O_4 synthesized from natural iron sand. Future studies employing complementary mechanistic analyses and extended stability evaluations will further strengthen the

understanding of the photocatalytic process and support the practical application of NiFe_2O_4 in sustainable wastewater treatment.

Acknowledgements

Thank you to the Ministry of Higher Education, Science and Technology and LPPM Andalas University for supporting this research in the form of PFR funds under contract number: 060/C3/DT.05.00/PL/2025.

Author Contributions

RY – conceptualization, supervision, experiment and analysis; ARD – experiment & investigation; DRS – analysis, writing & review; TPW – editing & review; YS – analysis, editing & review; ZH – formal analysis & review.

Conflict of Interest

The authors declare no competing interests.

References

- Abouhaswa, A. S., Badr, M. H., El Komy, G. M., & Abomostafa, H. M. (2024). Influence of the Mn/Fe ratio on structural, optical, electrical and magnetic characteristics of Mn-FeMgNiO spinel ferrites. *Inorganic Chemistry Communications*, 166, 112647. <https://doi.org/10.1016/j.inoche.2024.112647>
- Aggarwal, D., Bhavika, Kaur, M., Bansal, S., & Singhal, S. (2025). Fabrication of eco-friendly NiFe_2O_4 nanocatalysts via plant extract-mediated synthesis: Kinetic and mechanistic insights into photocatalytic degradation of an anthraquinone dye and tetracycline antibiotic. *Journal of Molecular Structure*, 1319. <https://doi.org/10.1016/j.molstruc.2024.139365>
- Alshehri, A., Alharbi, L., Wani, A. A., & Malik, M. A. (2024). Biogenic *punica granatum* flower extract assisted ZnFe_2O_4 and ZnFe_2O_4 -Cu composites for excellent photocatalytic degradation of RhB dye. *Toxics*, 12(1), Article 77. <https://doi.org/10.3390/toxics12010077>
- Al-Tohamy, R., Ali, S. S., Li, F., Okasha, K. M., Mahmoud, Y. A. G., Elsamahy, T., Jiao, H., Fu, Y., & Sun, J. (2022). A critical review on the treatment of dye-containing wastewater: Ecotoxicological and health concerns of textile dyes and possible remediation approaches for environmental safety. *Ecotoxicology and Environmental Safety*, 231, 113160. <https://doi.org/10.1016/j.ecoenv.2021.113160>
- Baby, J. N., Sriram, B., Wang, S. F., & George, M. (2020). Effect of various deep eutectic solvents on the sustainable synthesis of MgFe_2O_4 nanoparticles for simultaneous electrochemical determination of nitrofurantoin and 4-nitrophenol. *ACS Sustainable Chemistry & Engineering*, 8, 1479–1486. <https://doi.org/10.1021/acssuschemeng.9b05755>
- Bhaduri, B., Dikshit, A. K., Kim, T. Y., & Tripathi, K. M. (2022). Research progress and prospects of spinel ferrite nanostructures for the removal of nitroaromatics from wastewater. *ACS Applied Nano Materials*, 5, 16000–16026. <https://doi.org/10.1021/acsanm.2c02684>
- Biswas, B., Ahmed, M. F., Rahman, M. L., Khanam, J., Bhuiyan, M. H. R., & Sharmin, N. (2024). Investigation of structural, optical, and magnetic properties of NiFe_2O_4 for efficient photocatalytic degradation of organic pollutants through photo-Fenton reactions. *Heliyon*, 10, e37199. <https://doi.org/10.1016/j.heliyon.2024.e37199>
- Chakraborty, A., Bhattacharjee, B., & Ahmaruzzaman, M. (2025). A sustainable pathway for the photodegradation of rhodamine b dye using Mn-doped CaFe_2O_4 anchored on citrus fruit peel-derived biochar matrix. *Inorganic Chemistry Communications*, 171. <https://doi.org/10.1016/j.inoche.2024.113444>

- Chavan, R., Mujawar, S.P. of M.B.U.R.C.F.N., Dawkar, V., More, V., Pawar, N., Patil, R., Jadhav, J., Mustafa, J., Jameel, B., Muhaisen, H. M. H., Mohammed, A. Y. A., & Chougale, A. (2024). Enhanced photodegradation of methylene blue using reusable cobalt ferrite nanocomposites. *Science of Advanced Materials*, 16, 589–595. <https://doi.org/10.1166/sam.2024.4689>
- Cheng, Y., Zhang, S., Wang, Z., Wang, B., You, J., Guo, R., & Zhang, H. (2023). Review on spinel ferrites-based materials (MFe_2O_4) as photo-Fenton catalysts for degradation of organic pollutants. *Separation and Purification Technology*, 318, 123971. <https://doi.org/10.1016/j.seppur.2023.123971>
- Coromelci, C., Neamtu, M., Ignat, M., Samoila, P., Zaltariov, M. F., & Palamaru, M. (2022). Ultrasound assisted synthesis of heterostructured $\text{TiO}_2/\text{ZnFe}_2\text{O}_4$ and $\text{TiO}_2/\text{ZnFe}_{1.98}\text{La}_{0.02}\text{O}_4$ systems as tunable photocatalysts for efficient organic pollutants removal. *Ceramics International*, 48, 4829–4840. <https://doi.org/10.1016/j.ceramint.2021.11.019>
- Debnath, S., & Das, R. (2020). Study of the optical properties of Zn doped Mn spinel ferrite nanocrystals shows multiple emission peaks in the visible range — a promising soft ferrite nanomaterial for deep blue LED. *Journal of Molecular Structure*, 1199. <https://doi.org/10.1016/j.molstruc.2019.127044>
- Fang, K., Zhang, Y., Yin, J., Yang, T., Li, K., Wei, L., Li, J., & He, W. (2022). Hydrogel beads based on carboxymethyl cassava starch/alginate enriched with MgFe_2O_4 nanoparticles for controlling drug release. *International Journal of Biological Macromolecules*, 220, 573–588. <https://doi.org/10.1016/j.ijbiomac.2022.08.081>
- Ghahfarokhi, S. E. M., & Shobegar, E. M. (2020). Influence of pH on the structural, magnetic and optical properties of SrFe_2O_4 nanoparticles. *Journal of Materials Research and Technology*, 9, 12177–12186. <https://doi.org/10.1016/j.jmrt.2020.08.063>
- Jabbar, R., Shahatha, S. H., Kadhim, N., Magid, B., & Showard, A. F. (2024). Preparation and study of the effect of pH value on structural, morphological, electrical and magnetic properties of CoFe_2O_4 nanoparticles prepared by sol-gel precipitation method. *Ceramics International*, 50, 31114–31123. <https://doi.org/10.1016/j.ceramint.2024.05.417>
- Jasrotia, R., Jaswal, N., Prakash, J., Kit, C. C., Singh, J., & Kandwal, A. (2024). Photocatalytic application of magnesium spinel ferrite in wastewater remediation: A review. *Journal of Magnesium and Alloys*, 12, 490–505. <https://doi.org/10.1016/j.jma.2024.02.006>
- Khatun, N., Osman Goni, M., Islam, M. S., Aftab Ali Shaikh, M., Uddin Farhad, S. F., Al-Mamun, M., Alam, M. S., Islam Tanvir, N., Sajjad Hossain, M., Paul, T., & Islam, S. (2025). Impact of lanthanum ions on structural, morphological, magnetic, optical and electrical properties of cobalt-zinc ferrites. *Journal of Magnetism and Magnetic Materials*, 614. <https://doi.org/10.1016/j.jmmm.2024.172740>
- Kristina, N. P. D., Arjana, I. G., & Yasa, P. (2024). Synthesis and characterization of magnetite nanomaterials in Tianyar iron sand using co-precipitation method. *Indonesian Physical Review*, 7(3), 398–413. <https://doi.org/10.29303/ipr.v7i3.328>
- Kurniasari, M., Tri, H., Murti, B., Nurul, S., Jenie, A., & Kusumastuti, Y. (2025). Facile synthesis and characterization of magnetite mesoporous silica nanoparticles from local iron. *International Journal of Technology*, 16(2), 448–457. <https://doi.org/10.14716/ijtech.v16i2.6477>
- Kurniawan, D., Saputera, W. H., & Sasongko, D. (2025). Photocatalytic NO_x reduction to ammonia using zirconium incorporated g- C_3N_4 catalyst. *International Journal of Technology*, 16(6), 2043–2061. <https://doi.org/10.14716/ijtech.v16i6.7654>
- Lee, Y. S., Lee, K. M., & Ong, B. H. (2025). Magnetic recoverable cobalt ferrite nanophotocatalyst for crystal violet dye degradation. *Ceramics International*, 51, 39630–39638. <https://doi.org/10.1016/j.ceramint.2025.06.198>
- Majid, F., Rauf, J., Ata, S., Bibi, I., Malik, A., Ibrahim, S. M., Ali, A., & Iqbal, M. (2021). Synthesis and characterization of NiFe_2O_4 ferrite: Sol-gel and hydrothermal synthesis

- routes effect on magnetic, structural and dielectric characteristics. *Materials Chemistry and Physics*, 258, 123888. <https://doi.org/10.1016/j.matchemphys.2020.123888>
- Malik, A. R., Aziz, M. H., Atif, M., Irshad, M. S., Ullah, H., Gia, T. N., Ahmed, H., Ahmad, S., & Botmart, T. (2022). Lime peel extract induced NiFe₂O₄ NPs: Synthesis to applications and oxidative stress mechanism for anticancer, antibiotic activity. *Journal of Saudi Chemical Society*, 26, 101422. <https://doi.org/10.1016/j.jscs.2022.101422>
- Mardana, I. B. P., Nur Lutfiyah, Y., Yasa, P., & Agung Widianara, G. K. (2024). Synthesis and characterization of magnetite Fe₃O₄ nanoparticles from natural iron sand in Gelar river. *Indonesian Physical Review*, 7(1), 166–174. <https://doi.org/10.29303/ipr.v7i1.296>
- Mohammed, N. A. H., Shamma, R. N., Elagroudy, S., & Adewuyi, A. (2024). Chitosan incorporated nickel ferrite photocatalyst for complete photocatalytic degradation of ciprofloxacin, ampicillin and erythromycin in water. *Results in Chemistry*, 7. <https://doi.org/10.1016/j.rechem.2024.101307>
- Nengsih, S., Nur Abdulmadjid, S., Mursal, M., & Jalil, Z. (2024). Photocatalytic performance of Fe₃O₄-TiO₂ in the degradation of methylene blue dye: Optimizing the usability of natural iron sand. *Materials Science for Energy Technologies*, 7, 374–380. <https://doi.org/10.1016/j.mset.2024.06.001>
- Nugrahaningtyas, K. D., Nurcahyo, I. F., Putri, D. Y., & Santoso, S. J. (2025). Kaolinite-based photocatalysts for azo dyes degradation under xenon and sunlight irradiations: The effects of electro-deionization and impregnation methods. *International Journal of Technology*, 16(5), 1716–1730. <https://doi.org/10.14716/ijtech.v16i5.7624>
- Rahmayeni, Farhan, R., Wendari, T. P., & Stiadi, Y. (2025). Magnetic nanocomposite of hydroxyapatite/Co-doped ZnFe₂O₄ synthesized by green technology: Structure, character, and application. *International Journal of Technology*, 16(5), 1682–1699. <https://doi.org/10.14716/ijtech.v16i5.7291>
- Rahmayeni, Putri, J., Stiadi, Y., Zilfa, & Zulhadjri. (2019). Green synthesis of NiFe₂O₄ spinel ferrites magnetic in the presence of *hibiscus rosa-sinensis* leaves extract: Morphology, structure and activity. *Rasayan Journal of Chemistry*, 12, 1942–1949. <https://doi.org/10.31788/RJC.2019.1245304>
- Rahmayeni, R., Oktavia, Y., Stiadi, Y., Arief, S., & Zulhadjri, Z. (2021). Spinel ferrite of MnFe₂O₄ synthesized in *piper betle* linn extract media and its application as photocatalysts and antibacterial. *Journal of Dispersion Science and Technology*, 42. <https://doi.org/10.1080/01932691.2020.1721011>
- Ramu, A. G., & Choi, D. (2021). Highly efficient and simultaneous catalytic reduction of multiple toxic dyes and nitrophenols waste water using highly active bimetallic PdO–NiO nanocomposite. *Scientific Reports*, 11. <https://doi.org/10.1038/s41598-021-01989-7>
- Rasheed, S., Nadeem, S., Nawaz, R., Razzaque, S., Mahmood, A., Zidan, A., Al-Masry, W., Javed, M., Ditta, N. A., Bahadur, A., Iqbal, S., Mahmood, S., & Akhter, T. (2025). Revolutionizing textile wastewater treatment: Enhanced degradation of dyes using bimetallic zinc ferrite-GO nanocomposites. *Journal of Molecular Structure*, 1320, 139480. <https://doi.org/10.1016/j.molstruc.2024.139480>
- Riyanti, F., Nurhidayah, Purwaningrum, W., Yuliasari, N., & Hariani, P. L. (2023). MgFe₂O₄ magnetic catalyst for photocatalytic degradation of Congo red dye in aqueous solution under visible light irradiation. *Environment and Natural Resources Journal*, 21, 322–332. <https://doi.org/10.32526/ennrj/21/20230002>
- Roberta, I., Jos, A., Sara, I., Garcia, D. G., Lenzi, G., Antonio, M., Cambraia, O., Fidelis, Z., Dalla, R., & Brackmann, R. (2024). Magnetic NiFe₂O₄/TiO₂ heterostructures for the photocatalytic decontamination of glyphosate in water. *Materials Science in Semiconductor Processing*, 174. <https://doi.org/10.1016/j.mssp.2024.108205>
- Saha, S., Routray, K. L., Hota, P., Dash, B., Yoshimura, S., Ratha, S., & Nayak, T. K. (2024). Structural, magnetic and dielectric properties of green synthesized Ag doped NiFe₂O₄

- spinel ferrite. *Journal of Molecular Structure*, 1302. <https://doi.org/10.1016/j.molstruc.2023.137409>
- Salih, S. J., & Mahmood, W. M. (2023). Review on magnetic spinel ferrite (MFe_2O_4) nanoparticles: From synthesis to application. *Heliyon*, 9, e16601. <https://doi.org/10.1016/j.heliyon.2023.e16601>
- Salomo, S., Erwin, E., Malik, U., & Putra, S. U. (2018). Sifat magnetik dan ukuran partikel magnetik serta komposisi material pasir besi Pantai Kata Pariaman Sumatera Barat di sintesa dengan iron sand separator dan ball milling. *Jurnal Online Fisika*, 3(2), 11–14. <https://doi.org/10.22437/jop.v3i2.5730>
- Saputra, C., Cahyoutomo, H., Sendong, J., & Togibasa, O. (2020). Sintesis magnesium ferit berbasis pasir besi dari Sarmi menggunakan metode kopresipitasi. *Jurnal Fisika Flux: Jurnal Ilmiah Fisika FMIPA Universitas Lambung Mangkurat*, 17(2), 94. <https://doi.org/10.20527/flux.v17i2.6932>
- Sukandar, D., Adawiah, A., Rohman, S., Fitria, S., & Saridewi, N. (2024). Synthesis of MgFe_2O_4 nanoparticles and its application for photodegradation of methylene blue. *Bulletin of Chemical Reaction Engineering & Catalysis*, 19, 548–559. <https://doi.org/10.9767/bcrec.20222>
- Tripta & Rana, P. S. (2023). Structural, optical, electrical, and photocatalytic application of $\text{NiFe}_2\text{O}_4@\text{NiO}$ nanocomposites for methylene blue dye. *Ceramics International*, 49, 13520–13530. <https://doi.org/10.1016/j.ceramint.2022.12.227>
- Ullah, R., Khitab, F., Gul, H., Khattak, R., Ihsan, J., Khan, M., Khan, A., Vincevica-Gaile, Z., & Aouissi, H. A. (2023). Superparamagnetic zinc ferrite nanoparticles as visible-light active photocatalyst for efficient degradation of selected textile dye in water. *Catalysts*, 13(7), Article 1061. <https://doi.org/10.3390/catal13071061>
- Vadivel, S., Maaouni, N., Karim, M. R., Alnaser, I. A., Niyitanga, T., Kim, H., & Roy, S. (2024). Investigation of visible and UV light-induced photocatalysis properties of oleic acid-ligated cobalt-mixed magnesium ferrite nanoparticles for photodegradation of cationic and anionic dyes. *International Journal of Energy Research*, 2024, Article 5510976. <https://doi.org/10.1155/2024/5510976>
- Vargas-Bustamante, J., Benítez-Flores, E., Durán-Álvarez, J. C., Cortés-Lagunes, S., Arévalo-López, E. P., Pilo, J., Antonio, J. E., Escamilla, R., & Romero, M. (2025). Comparative study of $(\text{NiFe}_2\text{O}_4)$ photocatalysts for oxytetracycline degradation: Effect of synthesis method on performance stability. *Materials Chemistry and Physics*, 346, 131242. <https://doi.org/10.1016/j.matchemphys.2025.131242>
- Wulandhari, A., & Erwin, E. (2020). Penentuan sifat magnetik dan morfologi partikel magnetik pasir besi Pantai Arta Pariaman Sumatera Barat. *Jurnal Komunikasi Fisika Indonesia*, 17(1), 14–18. <https://doi.org/10.31258/jkfi.17.1.14-18>
- Yildirim, S., Ozler, B., Ozdemir, E. T., & Erol, M. (2025). The structural, magnetic, and optical properties of flame spray pyrolysis-derived spinel NiFe_2O_4 nanoparticles. *Journal of the American Ceramic Society*, 1–13. <https://doi.org/10.1111/jace.20220>
- Zulhadjri, Pratiwi, N., Putri, Y. E., Rahmayeni, Mufti, N., Ramli, Saraswaty, V., & Sufian, S. (2025). La^{3+} doped ZnFe_2O_4 synthesized via green chemistry approach using *uncaria gambir* roxb: A study on structural, optical, magnetic, and photocatalytic properties. *Journal of Photochemistry and Photobiology A: Chemistry*, 461. <https://doi.org/10.1016/j.jphotochem.2024.116168>