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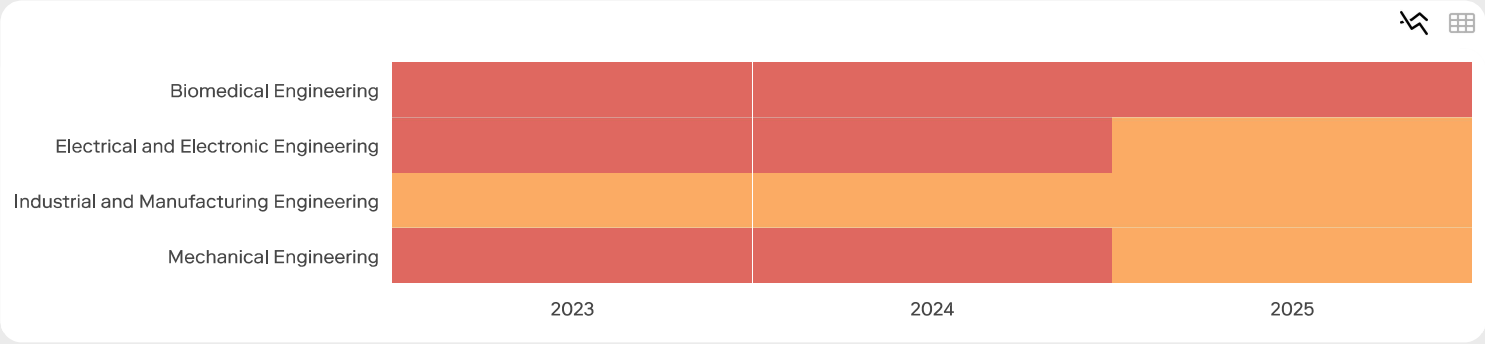
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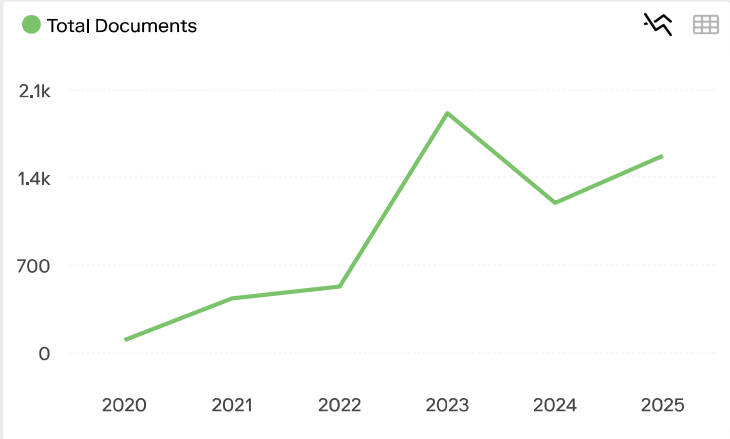
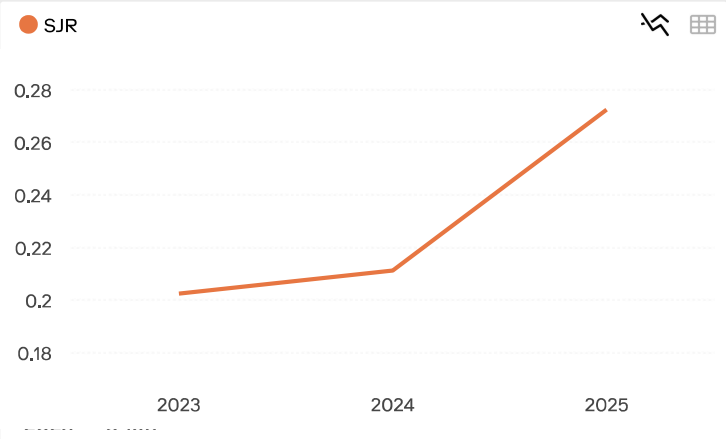
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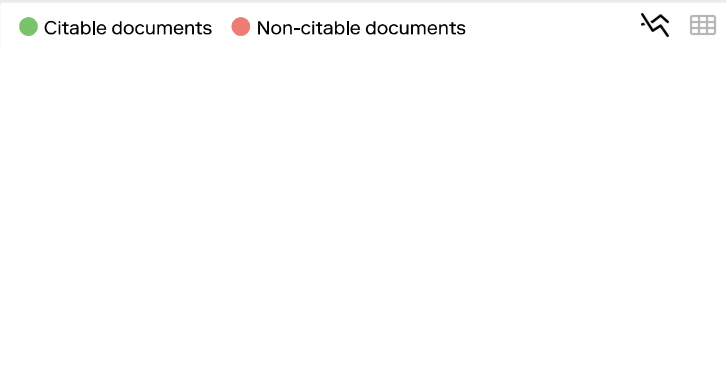
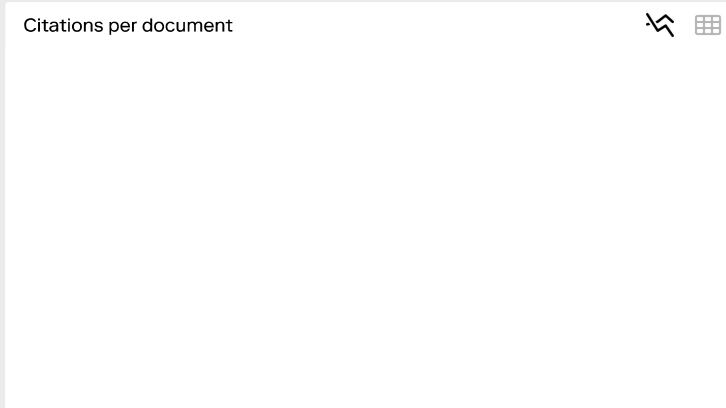
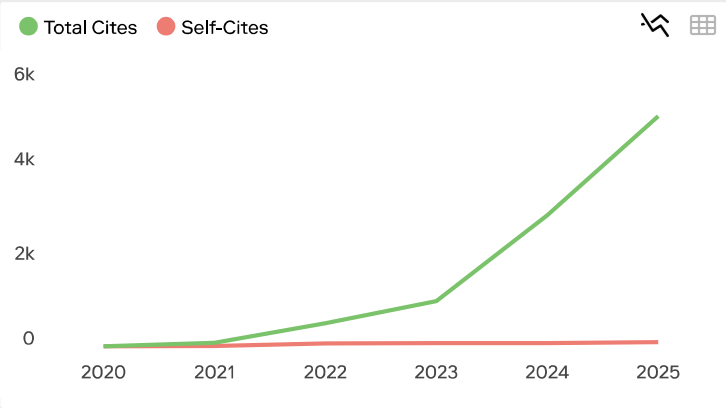
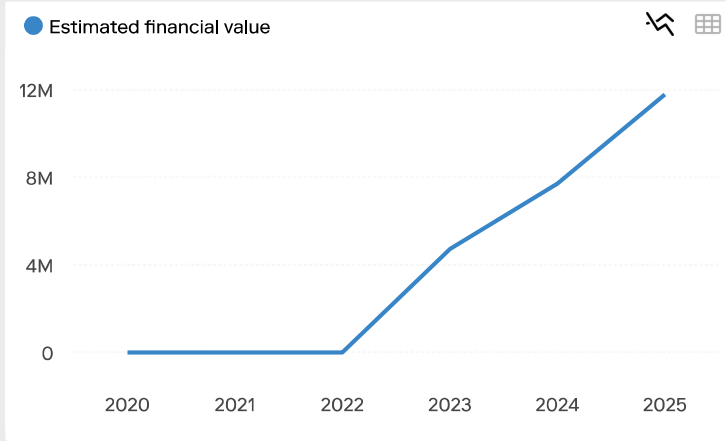
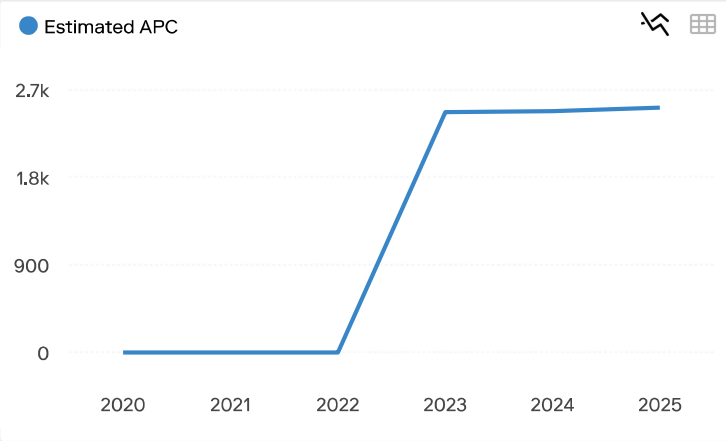
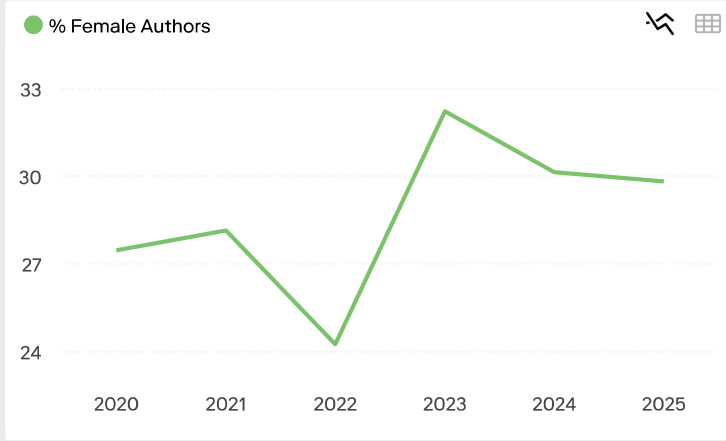
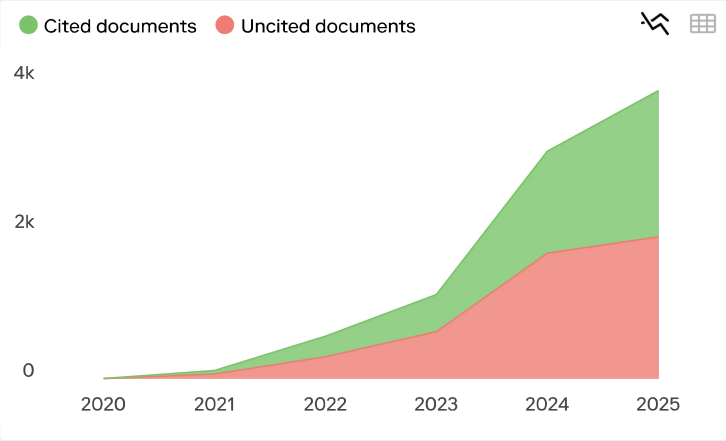
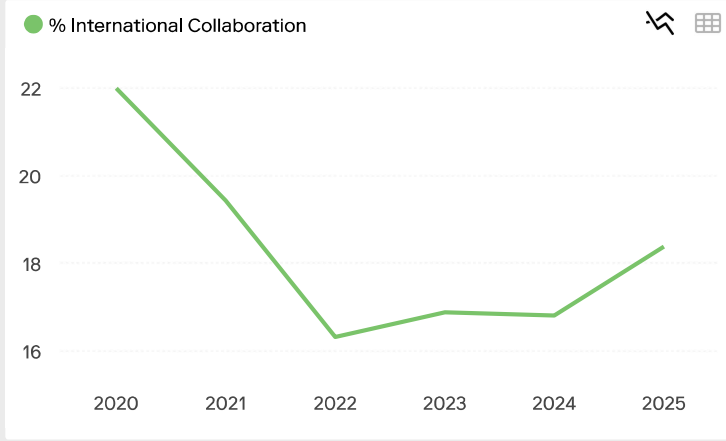
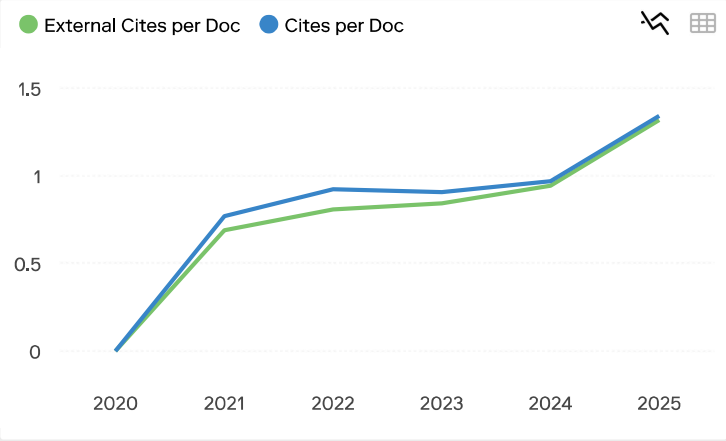
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Photocatalytic Activity of ZnFe_2O_4 for the Degradation of Fast Green FCF and Orange II [†]

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Abstract

In recent years, photocatalysis using semiconductor materials has gained significant attention as an effective strategy for dye degradation under mild conditions. Among various metal oxide photocatalysts, zinc ferrite (ZnFe_2O_4) has gained attention due to its narrow band gap, good stability, low cost, and activation under visible light. ZnFe_2O_4 nanoparticles (NPs) were synthesized using a co-precipitation process and tested for their photocatalytic effectiveness in degrading synthetic dyes Fast Green FCF and Orange II Sodium Salt under visible light. This study emphasizes the benefits of utilizing ZnFe_2O_4 as a visible light-activated, cost-effective, and environmentally friendly photocatalyst. These findings add to the growing research on wastewater treatment options.

Keywords: nanoparticles; photocatalysts; dye degradation; fast green FCF; orange II sodium salt; water management; pollution remediation; pollutant removal



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1. Introduction

Rapid industrialization, urbanization, and insufficient waste management systems continue to be major causes of environmental pollution on Earth [1]. The depletion of natural resources and environmental degradation brought on by industrial growth and environmental crises are currently the main causes of ecosystem damage. Water pollution is a serious environmental problem that puts water, the main component of life on Earth, at serious risk, highlighting the vital role that water plays in sustaining life [2]. Industrial wastewater has numerous negative impacts on ecology and is extremely detrimental to our environment. The textile, cosmetics, printing, paper, and rubber industry sectors produce the majority of wastewater. Massive amounts of extremely hazardous chemicals are produced by the textile industry and discharged at different processing stages [3]. Dyes are the most common and challenging to handle of all the hazardous substances and contaminants. Textiles, food, rubber, cosmetics, paint, pharmaceuticals, paper and pulp, tannery materials, hair, and other materials can all be colored with dye. Dyes are intended to bond with the fibers or surfaces of the materials they are applied to, producing a durable and vivid color. They are usually soluble in water or other solvents. Aquatic life is impacted by these dyes because they color the water and prevent sunlight from reaching the bottom layers [4].

Furthermore, dyes are known to be carcinogenic, mutagenic, and teratogenic, impacting a variety of bacteria and fish species. Human exposure to dyes can cause serious health problems, such as liver, renal, reproductive, brain, and central nervous system disorders [5]. For dye removal, a variety of physicochemical and biological treatment methods have been widely used, including coagulation–flocculation, adsorption, membrane filtering, and microbial degradation. However, these traditional techniques frequently have drawbacks such as a poor performance for stable or non-biodegradable dyes, high operating costs, partial breakdown, and the production of secondary waste [6]. Photocatalytic degradation has drawn a lot of interest lately as an effective and sustainable substitute [7]. This method uses light-irradiated semiconductor catalysts to produce reactive radicals that can mineralize dyes into safe byproducts like CO_2 and H_2O . Photocatalysis is now regarded as one of the most promising techniques for wastewater purification because of its high efficiency, catalyst reusability, and operation under ambient settings [8]. For example, a recent study described the unique green synthesis of CuFe_2O_4 nanoparticles (NPs) using plant extracts from *Cissus rotundifolia*. The photocatalytic dye degradation efficiency of the nanomaterial was evaluated against methylene blue (MB). The results demonstrated the photocatalyst's effectiveness in water cleanup by showing 82% breakdown under UV–visible light [9]. Sb_2O_3 NPs were created via the solvothermal procedure in a related investigation carried out by the same research team. The MB dye was used to test the photocatalytic potential. In 60 min, the NPs demonstrated 60% dye degradation. It was discovered that the dye was adsorbed onto the surface of the Sb_2O_3 nanoball, and degradation was linked to the production of reactive oxygen species (ROS). According to the study's findings, photocatalysts can be applied practically in industry to remediate wastewater [10]. By adding different transition metals to create composites based on binary and ternary metal oxides and heterojunctions, the photocatalytic efficacy of the catalysts for the destruction of pollutants under visible light is increased [11]. Among these are zinc ferrite nanoparticles, which are regarded as one of the most promising photocatalysts because of their intriguing physiochemical characteristics, superior charge carrier separation features, and high degrading efficiency in visible light. Because ZnFe_2O_4 NPs have a small energy band gap and Fe^{3+} ions, they are a powerful photocatalyst. They also possess remarkable optical and magnetic characteristics. Their strong reactivity is a result of both their small particle size and large surface area. As a result, a number of recent studies have concentrated on the synthesis of zinc ferrite [12]. In our present work, the dye degradation efficiency of ZnFe_2O_4 NPs was tested against the Fast Green FCF and Orange II dyes.

2. Materials and Methods

This study used only analytical-grade chemicals; therefore, no further purification was necessary before usage. Zinc nitrate hexahydrate ($\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$), ferric nitrate nonahydrate ($\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$), Sodium hydroxide (NaOH), ethanol, Fast Green FCF and Orange II dyes were procured from Sisco Research Laboratories Pvt. Ltd., Maharashtra, India. A NEUATION iFUGE D08 centrifuge machine was used for centrifuging the reaction mixtures and recovering the precipitates. UV–visible absorption spectra were recorded between 200 and 800 nm using the Agilent Cary UV–Vis Compact Peltier R3.15AL250V spectrophotometer, California, USA. The crystallinity and phase identification purity of the synthesized precursors and the binary nanocomposite were detected using a diffractometer (model No.: D8 Advance Eco, make: Bruker, Germany). FT-IR spectra were obtained using a spectrophotometer (model no.: Nicole 6700, make: Thermo-Scientific, Waltham, MA, USA). The morphology of the nanostructures was analyzed using scanning electron microscope (model: JSM 6490 LV, make: JEOL, Tokyo, Japan).

3. Experiments

3.1. Synthesis of ZnFe_2O_4 NPs

ZnFe_2O_4 NPs were synthesized by co-precipitation. Under constant stirring, a precursor solution comprising 0.1 mol $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ and 0.2 mol $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ was made in 100 mL of distilled water. To cause precipitation, a 10% NaOH solution was added dropwise until pH 9 was reached. After centrifuging and washing with ethanol and water, the precipitate was dried for 5–6 h at 110 °C. To create crystalline ZnFe_2O_4 NPs, the dried powder was crushed and calcined for four hours at 500 °C.

3.2. Photocatalytic Activity Evaluation

Using visible light, the photocatalytic activity of the NPs was assessed against the Fast Green FCF and Orange II dyes. For this, 50 mL of dye solution (10 ppm) was mixed with 25 mg of NPs. To achieve desorption/absorption equilibrium, the mixture was magnetically stirred for 30 min in the dark before the solution was exposed to light. Every 15 min, 3 mL of the sample was taken out, and the concentration of the degraded dye solution was measured using a UV–visible spectrophotometer (200 nm to 800 nm). Equation (1) was used to determine the dye's % degradation.

$$\% \text{Degradation} = (C_0 - C_t) / C_0 \quad (1)$$

where C_0 represents the dye absorbance at the beginning, and C_t represents the dye absorbance at time t . Additionally, the kinetics of Fast Green FCF degradation were examined. Plotting $\ln(C_t)$ versus time allowed for the evaluation of the degradation kinetics. The kinetic study was performed using a pseudo-first-order kinetic equation.

$$\ln(C_0/C_t) = kt \quad (2)$$

where C_0 represents the initial concentration, C_t denotes the concentration at time t , and k is the rate constant. The slope of the plot of $\ln(C_0/C_t)$ against time gives a rate constant, k .

4. Results and Discussion

4.1. Characterization

Two strong peaks were visible in the ZnFe_2O_4 NPs UV–vis absorption spectra (Figure 1a) at around 221 and 342 nm. The sharp absorption at 221 nm is assigned to $\text{O}^{2-} \rightarrow \text{Fe}^{3+}$ charge transfer transitions within the spinel lattice, while the absorption at 342 nm corresponds to band gap excitation. The measured band gap was 4.6 eV which was calculated through the Tauc plot (Figure 1b) [13]. The FT-IR spectra (Figure 2a) of synthesized ZnFe_2O_4 NPs, observed between 4000 and 400 cm^{-1} , indicates the presence of distinctive functional groups and the spinel ferrite structure. A wide absorption band at 3386 cm^{-1} and 3230 cm^{-1} indicates the presence of adsorbed water or surface hydroxyl groups. The bands at 1695 cm^{-1} and 1378 cm^{-1} correspond to H-O-H bending and C-H deformation vibrations, respectively. Sharp bands at 769 cm^{-1} , 543 cm^{-1} , and 455 cm^{-1} indicate metal–oxygen stretching vibrations in the spinel lattice [14]. The XRD pattern of synthesized ZnFe_2O_4 NPs revealed (Figure 2b) distinct and sharp peaks at 2θ values of approximately 29.77°, 35.04°, 42.63°, 56.44°, and 62.07°, which correspond to the (220), (311), (400), (511), and (440) crystal planes of the cubic spinel structure, respectively, as per JCPDS card no. 22-1012. The peak at 35.04° shows the presence of the typical (311) plane of ZnFe_2O_4 , indicating the successful development of the spinel ferrite phase. The average crystallite size came out to be 10.206 nm [15]. The SEM image (Figure 3) of the ZnFe_2O_4 NPs showed a needle-like morphology with a network-like arrangement. The particles

appeared elongated and intertwined, forming clusters rather than discrete spherical grains. The surface texture appeared rough and porous, which is characteristic of ferrite nanomaterials synthesized through chemical routes. Similar agglomerated morphologies have been reported for ZnFe_2O_4 NPs synthesized via green and hydrothermal methods [16].

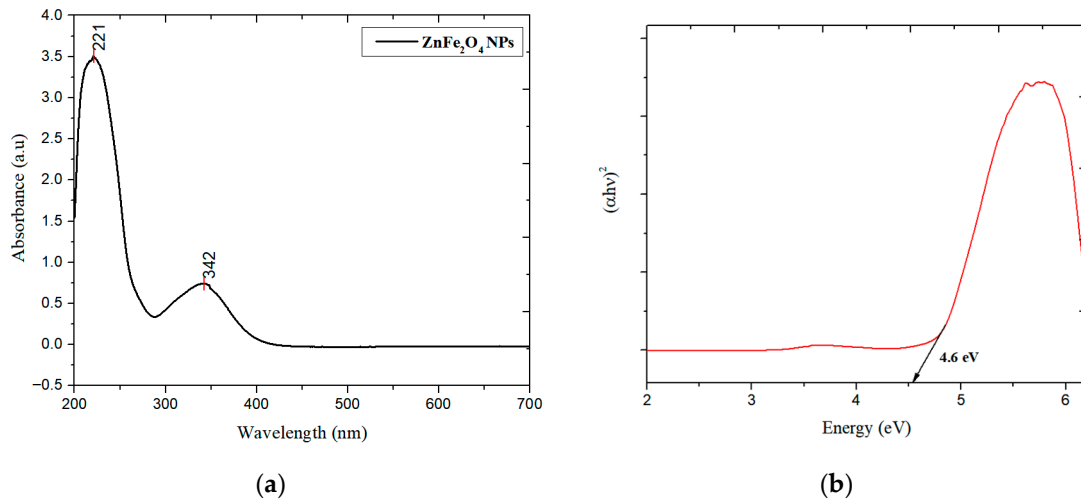


Figure 1. (a) UV-vis spectra and (b) Tauc plot of ZnFe_2O_4 NPs.

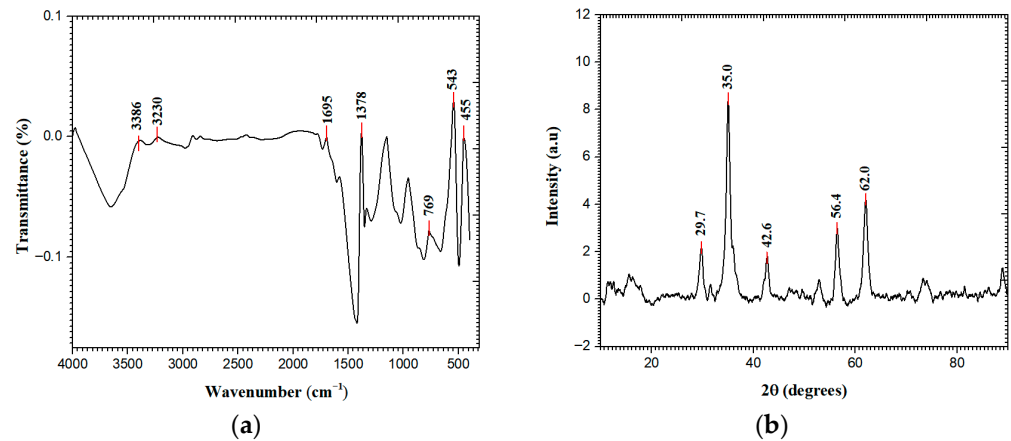


Figure 2. (a) FT-IR and (b) XRD spectra of ZnFe_2O_4 NPs.

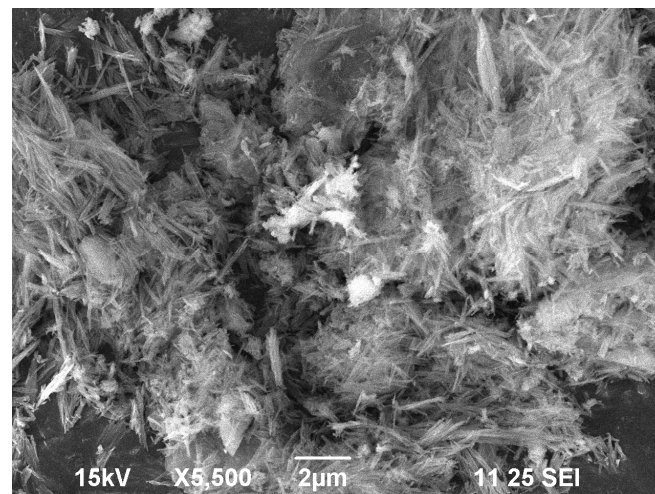


Figure 3. SEM image of ZnFe_2O_4 NPs.

4.2. Photocatalytic Dye Degradation

The widespread utilization of synthetic dyes in the food, paper, cosmetics, and textile sectors contributes to the discharge of massive quantities of colored wastewater into bodies of water. Even at low quantities, these dyes can have serious negative effects on the environment and human health since they are chemically stable and non-biodegradable [17]. Traditional treatment techniques, such as adsorption or biological degradation, are less effective due to the complex aromatic nature of these compounds. As a result, photocatalytic degradation involving semiconductor materials has become a viable and environmentally responsible method for dye removal from structures [18]. Two renowned anionic dyes that are acknowledged for their durability in aquatic settings are Orange II and Fast Green FCF [19]. Orange II ($C_{16}H_{11}N_2NaO_4S$), with a molecular weight of 248.71 g/mol, is an azo dye used in the leather, paper, and cosmetics sectors, whereas Fast Green FCF ($C_{37}H_{34}N_2Na_2O_{10}S_3$), with a molecular weight of 808.84 g/mol, is a triphenylmethane dye that is widely used as a food colorant and in textiles [20]. The structures of the dyes are illustrated in Figure 4a,b.

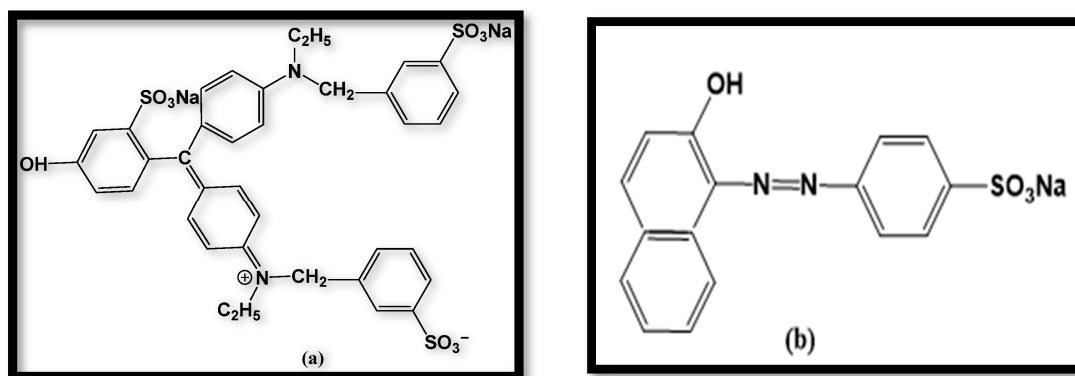


Figure 4. Structure of dyes: (a) Fast Green FCF; (b) Orange II.

The absorbance of the untreated dye solution was recorded before treatment with the NPs. In the case of Fast Green FCF, the maximum absorbance was obtained at 623 nm, and the peak area was 170.299, whereas for Orange II the $\lambda_{\max}$ was 485 nm (Figure 5) and the peak area was 84.925.

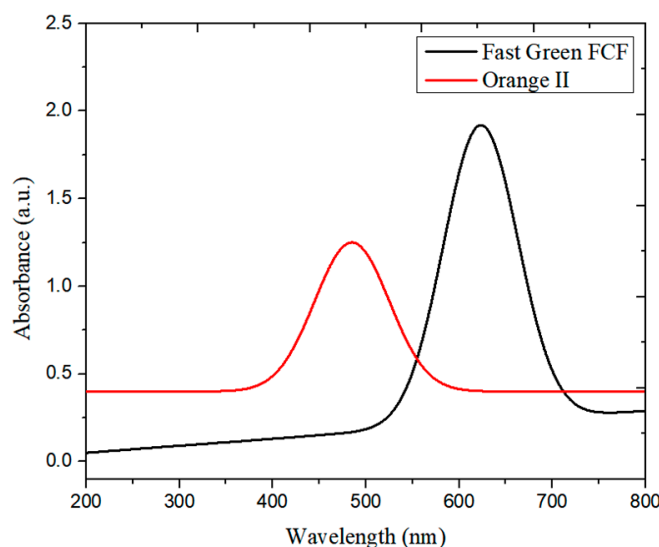


Figure 5. Time-dependent UV-vis spectra of untreated Fast Green FCF and Orange II dyes.

In the present study, the photocatalytic degradation of Fast Green FCF and Orange II was studied using ZnFe_2O_4 NPs (Figure 6a,b) under visible light irradiation in 90 min. The dye degradation of Fast Green FCF with ZnFe_2O_4 NPs was found to be 60% and follows pseudo-first-order kinetics with a rate constant of $8.01 \times 10^{-3} \text{ min}^{-1}$ ($R^2 = 0.98737$), as given in Figure 7a, while the photodegradation of Orange II with ZnFe_2O_4 NPs was found to be 73%, also following pseudo-first-order kinetics and the rate constant was found to be $6.08 \times 10^{-3} \text{ min}^{-1}$ ($R^2 = 0.94432$), as given in Figure 7b. Figure 8 shows the % degradation versus time plot for both dyes.

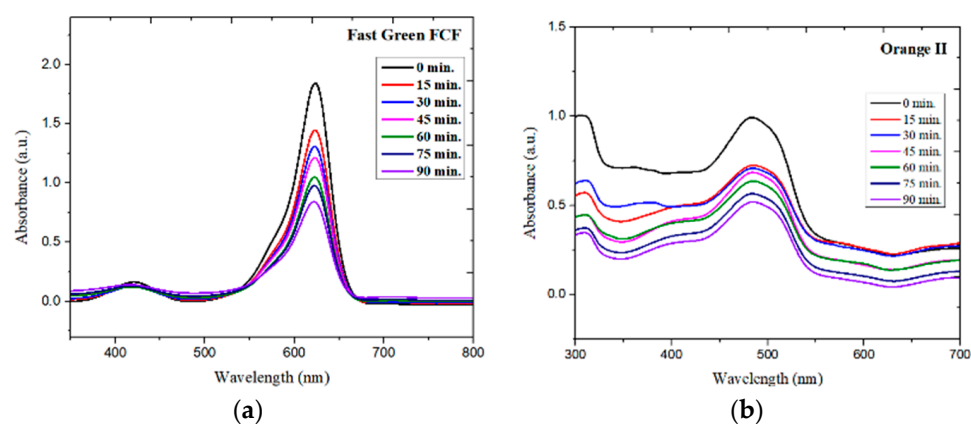


Figure 6. Time-dependent UV-vis spectra of (a) Fast Green FCF and (b) Orange II treated with ZnFe_2O_4 NPs.

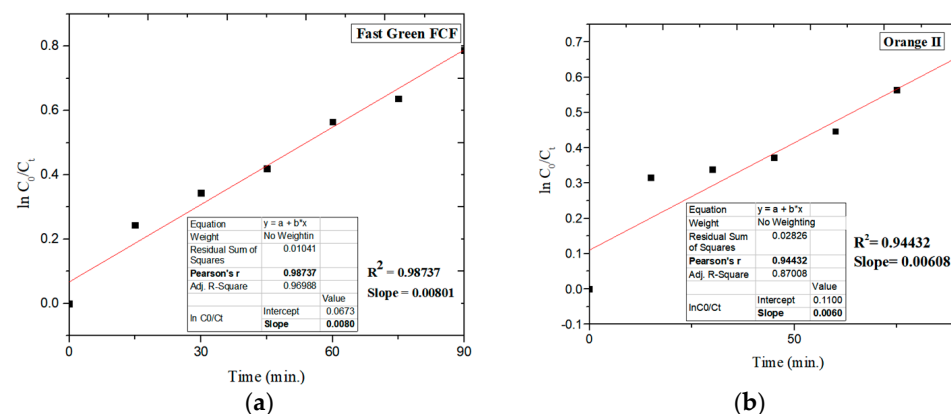


Figure 7. Pseudo-first-order kinetic plot of $\ln(C_0/C_t)$ vs. time for (a) Fast Green FCF and (b) Orange II treated with ZnFe_2O_4 NPs.

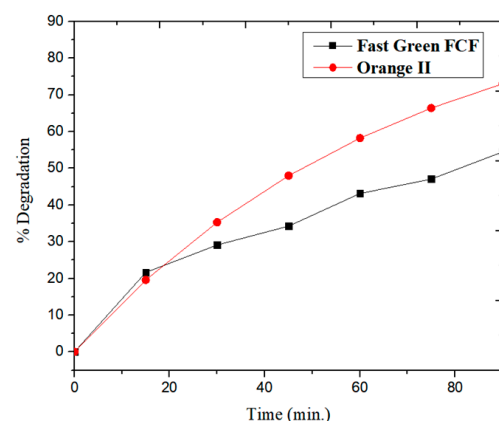


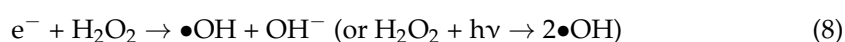
Figure 8. Degradation (%) of Fast Green FCF and Orange II.

ZnFe₂O₄ has a narrow band gap that is easily stimulated by visible and UV light [21]. Because of its paramagnetic properties, adsorption capacity, narrow band gap, photocatalytic response, stability, reusability, and sensitivity to visible light, zinc ferrite is an important photocatalyst [22]. When the photocatalyst absorbs sufficient light energy, electron holes are formed in photocatalytic reactions. The mechanism of dye degradation can be illustrated through the following Equations (3)–(10):

Activation:



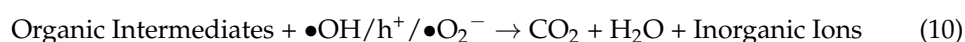
ROS Generation:



Dye Degradation:



Mineralization:



Upon exposure to photon energy on the surface of the NPs, the conduction band will excite electrons from the valence band, resulting in the formation of electron–hole pairs. After that, the photogenerated electron holes convert hydroxide ions (OH[−]) or water molecules (H₂O) into hydroxyl radicals (•OH). Subsequently, the photoexcited electrons will convert oxygen into superoxide radicals (•O₂[−]), which can then be protonated by H⁺ ions in water to generate hydroxyl radicals (HO₂•). These radicals then transform into H₂O₂, which dissociates into the more reactive hydroxyl radical species OH•. The hydroxyl and superoxide radicals are crucial to dye degradation [23]. Previous research has also demonstrated the photocatalytic capability of such NPs. In a recent study, for example, MB dye was degraded in the presence of a ZnFe₂O₄/Ag₂WO₄ photocatalyst, which was made using a straightforward combustion process and a solution mixing approach. By adjusting the catalyst dosage and pH of the media used for the degradation process, the photocatalytic activity of the composite material was determined. The results indicated that 72.7% of the MB dye was degraded by the composite material [24]. In a similar study, Kumar S. et al. synthesized zinc ferrite NPs using the sol–gel auto-combustion (SG) technology in order to use photocatalysis and adsorption to remove the crystal violet dye present in wastewater from the textile sector. The nanomaterial had an 83% degradation efficiency, indicating that it is very advantageous for use in the environment [25]. A different study by Duraisamy K. et al. described an environmentally benign way to create ZnFe₂O₄ NPs using a hydrothermal process with *Nyctanthes arbortristis* leaf extract. This nanomaterial's photocatalytic effectiveness was evaluated using MB dye, and the findings were encouraging. The results indicated that the photocatalyst showed degradation of more than 80% [26]. Some examples of nanocomposites exhibiting dye degradation are discussed in Table 1.

Table 1. Some examples of nanomaterials exhibiting dye degradation.

S.No.	Nanomaterial	Dye	Time (Minutes)	Source	Degradation	Reference
1	ZrO ₂ NPs	Methylene blue	80	Sunlight	80%	[27]
2	CeVO ₄ NPs	Congo red	80	Sunlight	90%	[28]
3	V ₂ O ₅ NPs	Methyl violet	100	Visible light	85%	[29]
4	BiFeO ₃ NPs	Methylene blue	120	Visible light	90%	[30]
5	BiFeO ₃ NPs	Rhodamine B	90	UV light	80%	[31]
6	NiFe ₂ O ₄ NPs	Crystal violet	90	UV light	75.5%	[32]
7	SnO ₂ /NGO nanocomposite	Methyl orange	150	Sunlight	89.4%	[33]
8	LaFe ₂ O ₃ /Sb ₂ O ₃ nanocomposite	Malachite green	80	Visible light	86%	[34]
9	ZnO and α -Fe ₂ O ₃ NPs	Malachite green	90	Visible light	79% and 68%	[35]
10	ZnO–Thiosemicarbazone Nanoconjugates	Malachite green	90	Visible light	90%	[36]
11	CuO–thiosemicarbazone nanoconjugates	Malachite green	90	Visible light	84%	[37]
12	B-CuO/rGO nanocomposite	Methylene blue	90	Visible light	90%	[38]

5. Conclusions

ZnFe₂O₄ NPs were successfully synthesized via a co-precipitation method and showed significant photocatalytic activity for the breakdown of Fast Green FCF and Orange II in aqueous media. The photocatalyst showed a significant degradation capability of about 60% in the case of Fast Green FCF and 73% in the case of Orange II in 90 min, proving its versatility for wastewater remediation. Overall, ZnFe₂O₄ offers a promising strategy for the treatment of dyes in water, supporting the advancement of sustainable wastewater treatment technologies aligned with global environmental goals.

Author Contributions: Conceptualization, T.K.; methodology, N.F. and T.K.; data curation, N.F. and S.D.; writing—original draft preparation, writing—review and editing, N.F., E.V. and A.P.; supervision, T.K. All authors have read and agreed to the published version of the manuscript.

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Conflicts of Interest: The authors declare no conflicts of interest.

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