

Structure, Optical, Magnetic and Photocatalytic Properties of Cu doped ZnFe₂O₄ Nanoparticles

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Copper doped zinc ferrite (Cu-ZnFe₂O₄) nanocrystallites were synthesised via the sol-gel process. X-ray diffraction (XRD), Field Emission Scanning Electron Microscopy (FE-SEM), UV-visible spectroscopy, and Vibrating Sample Magnetometry (VSM) were utilised to analyse the structural behaviour and magnetic properties of the sample. X-ray diffraction studies revealed the spinel FCC structure of the sample. The scanning electron microscopy micrographs indicated the formation of crystallites with dense, compact and platelet structure. The absorption spectrum and the Tauc plot revealed the bandgap to be 2.0 eV. The sample's magnetic property was analyzed using a vibrating sample magnetometer, confirming its superparamagnetic behaviour at room temperature. The photocatalytic degradation of the sample was assessed against Methylene Blue (MB) dye in visible light irradiation, demonstrating an efficiency of 83%. The elemental composition of the sample and its oxidation state were determined using X-ray photoelectron spectroscopy (XPS), which confirmed the synthesis of Cu-doped ZnFe₂O₄.

Keywords: Cu doped zinc ferrite, nanostructure, magnetic property, XPS, photocatalytic activity, nanoparticles.

1. INTRODUCTION

Ferrites are ceramic-like materials with unique magnetic properties that are used in many electronic devices. Spinel-structured ferrites are the important materials employed in contemporary engineering, both in scientific and practical applications [1]. Ferrites have high magnetic permeability, high electrical resistance, high coercivity and so on. Magnetic ceramics are classified as hard (hexaferrites) and soft (spinel and garnets) magnetic materials [2, 3]. In recent decades, spinel soft ferrites have garnered significant attention. Spinel soft ferrites have general chemical formula AB₂O₄, which crystallize in cubic structure with thermal stability, making them suitable for various applications. These materials are utilized in microelectronics, magnetic memory, gas sensors, water purifiers, catalysis, biomedical, antimicrobial and magnetic resonance imaging applications [4]. Among the ferrites, zinc ferrite (ZnFe₂O₄) is an outstanding material, suitable for several applications [5]. ZnFe₂O₄ possesses significant applications in several domains, including sensors, energy, photocatalysis, spintronics and information storage. The performance of this material is critically dependent on its inherent features. The incorporation of different metal ions, including magnetic and nonmagnetic ions into the spinel structure alters the physical and chemical properties [6, 7]. The distribution of cations is contingent upon the ionic radii of cation, synthesis method, preparation parameters, doping, sintering temperature, and substitution process. These results in unique electric and magnetic characteristics for

specific applications. The doping of copper (Cu) in zinc ferrites leads to structural modification and change in properties, optimizing their performance for certain applications. Moreover, it is interesting to note that Cu-doped ZnFe₂O₄ nanoparticles offer promising properties. Their performance is highly dependent on the precise control of synthesis parameters and doping concentrations. Ongoing research continues to optimize these factors to fully harness their potential in various applications [8, 9]. Cu-doped ZnFe₂O₄ nanoparticles belong to a category of spinel ferrite nanoparticles, which attracted considerable interest due to their improved catalytic properties and prospective applications in magnetic data storage, wastewater treatment, and biomedicine [10–13].

Prithviraj Swamy et al. prepared nano crystalline zinc ferrite using the low-temperature combustion method and their XRD results showed single-phase ferrite NPs with a crystallite size of 40 nm [4]. Komal Yadav prepared the Cu doped zinc ferrites using green synthesis route. XRD studies of the samples showed crystalline nature with a crystallite size of 9 nm [14]. Nadhiya et al synthesized Cu_xZn_(1-x)Fe₂O₄ NPs using sol-gel process and studied the structure and magnetic properties [15]. Vani et al. synthesized the copper doped zinc ferrite NPs and studied the structure and optical properties [16]. Bera et al. prepared the zinc ferrite and the XRD and XPS were used to study the ferrite NPs [17]. The synthesis method and preparation conditions significantly influence the properties of ferrite NPs for their intended uses in the present context. Numerous strategies have been documented for the preparation of nanomaterials, including precipitation, high-energy ball milling, hydrothermal methods, combustion routes, solvothermal methods, microwave-assisted synthesis, thermal plasma, micro-emulsion techniques and sol-gel processes [18, 19]. In the

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present work, Cu doped zinc ferrite nanoparticles have been synthesized using sol-gel technique due to its simplicity, cleanliness, low cost and eco-friendliness. This study focuses on the synthesis and characterization of Cu doped zinc ferrite NPs for magnetic and photocatalytic applications.

2. EXPERIMENTAL DETAILS

The analytical reagent (AR) grade compounds were utilized in this study without additional processing. The experiment employed deionized (DI) water consistently. Iron nitrate nonahydrate ($\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$) and zinc acetate dihydrate ($\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2(\text{H}_2\text{O})$) were individually dissolved in deionised water. Both solutions were mixed and agitated for 10 min. Copper nitrate was subsequently dissolved in deionized water and incorporated into the preceding solution for a doping of 10 %. Subsequently, CTAB was incorporated as a surfactant. The citric acid served as the gelling agent. The solution was maintained at around 70–80 °C and calcined at 600 °C for 2 h.

X-ray diffraction studies were carried out for the sample Cu doped ZnFe_2O_4 using powder diffractometer (BRUKER D8) with $\text{CuK}\alpha$ radiation in the range of 20–80°. The surface morphology and composition were analyzed using FESEM (CARL ZEISS). The sample's optical characteristics were examined using UV-visible spectrophotometer (Shimadzu) in the 200–1000 nm wavelength range. The sample's magnetic characteristics were examined with a vibrating sample magnetometer (LAKESHORE, Model 7407). A 10 ppm MB dye solution was used to assess the sample's degradation performance. The solution was kept under dark condition for 30 min to attain equilibrium and the visible light source (550 nm and 125 W) was used to find the degradation of MB dye solution for 120 min duration. 2 ml of the solution was removed for every thirty minutes while it was exposed to visible light (550 nm). The UV-Vis spectrophotometer was employed to examine the absorbance the sample. The XPS spectra were acquired with a Versa Probe II (ULVAC-PHI, Chigasaki, Japan).

3. RESULTS AND DISCUSSION

3.1. Microstructure analysis

The XRD pattern (Fig.1) shows prominent intensity peaks at angles 18.3°, 30.0°, 35.35°, 36.3°, 42.9°, 47.6°, 53.3°, and 56.7°, which correspond to (111), (220), (311), (222), (400), (331), (422), and (333) reflecting planes, respectively, indicating the single phase spinel fcc structure of ZnFe_2O_4 , (JCPDS No. 22-1012) [20,21]. In addition, there are peaks at angles 31.8°, and 38.8° correspond to (-110) and (111) planes, indicating the monoclinic structure of CuO. The crystallite size was calculated using the Scherrer formula from the prominent peak (311):

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

where K is the Scherrer constant (0.9); λ is the wavelength of X-ray; β is the width at half maximum (FWHM) in

radian; θ is the diffraction angle. The crystallite size was found to be 23 nm. Arthur Charles et al. synthesized zinc copper ferrite nanostructures using co-precipitation. XRD studies confirmed the cubic spinel structure of the samples and the crystallite size was found to be 32 nm [22]. Komal et al. [14] prepared copper doped zinc ferrite nanoparticles using green synthesis. The prepared samples showed a crystalline nature with a crystallite size of 9–21 nm range. Shwetambaram et al synthesized Cu doped zinc ferrite ($\text{Zn}_{(1-x)}\text{Cu}_x\text{Fe}_2\text{O}_4$) NPs by the hydrothermal method, and the XRD studies confirmed single phase spinel cubic structure [23].

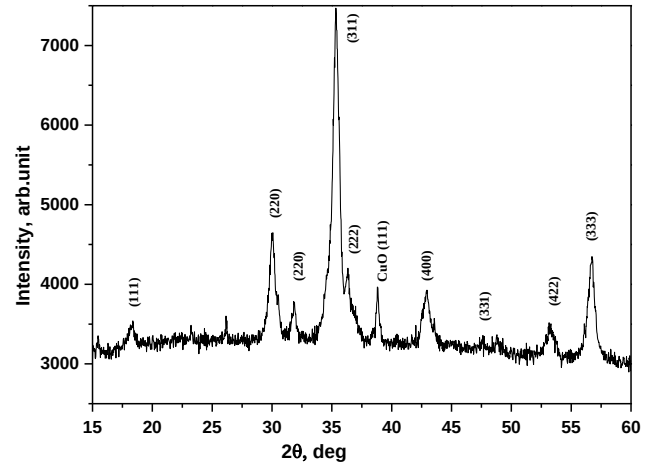


Fig. 1. XRD pattern of Cu doped ZnFe_2O_4 nanoparticles

The surface morphology of the NPs was analyzed using FE-SEM. Fig. 2 shows the formation of crystallites with irregular shapes and sizes. It shows the dense structure, smooth morphology with agglomerations. Each grain is constituted by the aggregation of many nanocrystallites. The EDX analysis verified the presence of Zn, Cu, Fe, and O elements in the sample, with no further contaminants detected. This signifies the effective incorporation of Cu into the ZnFe_2O_4 matrix. The samples exhibit uneven forms and sizes, with grain cohesion resulting from magnetic attraction [22, 24].

3.2. Optical properties - UV-visible spectroscopy

The absorption spectrum of the Cu-doped ZnFe_2O_4 nanoparticles is shown in Fig. 3. The bandgap (E_g) was calculated using absorption phenomena associated with the valence-conduction band transition. A noticeable absorption was observed at about 800 nm, signifying the band-to-band transition. The bandgap (E_g) is determined by the Tauc plot, a plot of $(\alpha h\nu)^2$ versus $h\nu$ at $\alpha=0$. (Fig. 3). Eq. 2 is employed to determine the bandgap of the sample.

$$(\alpha h\nu)^2 = A(h\nu - E_g), \quad (2)$$

where α is the absorption coefficient, ν is the frequency of radiation. The bandgap of the sample was found to be 2.0 eV. Vani et al., determined the bandgap of pure ZnFe_2O_4 nanoparticles and copper doped nanoparticles. The bandgap was observed to be increasing for higher Cu content in the zinc ferrite system, increasing from 1.825 to 2.776 eV [16].

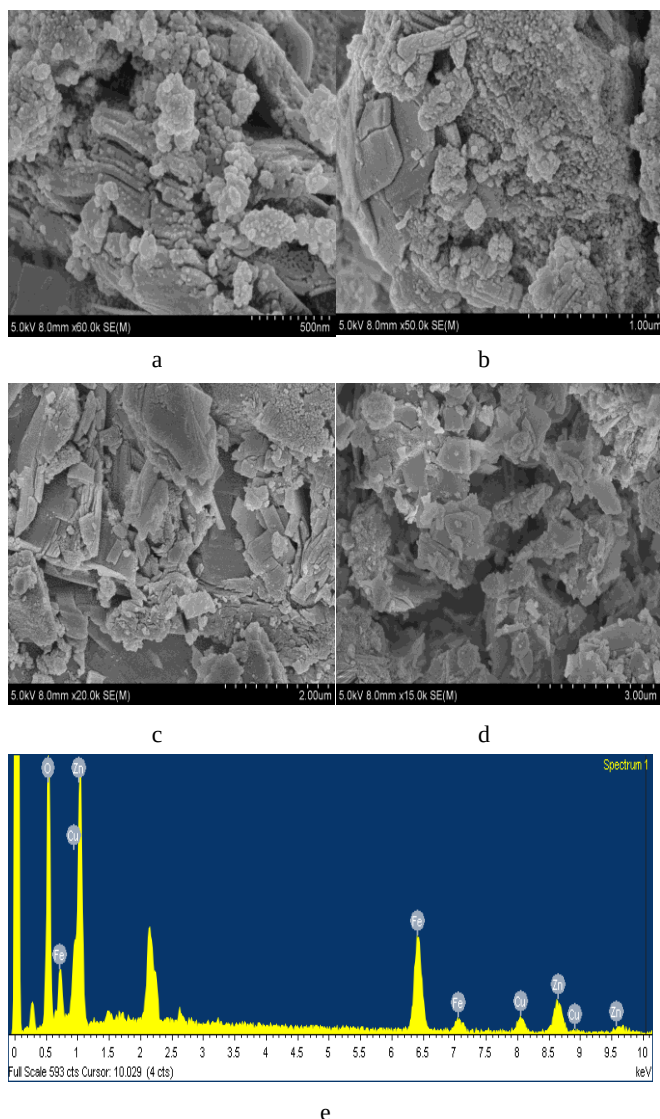


Fig. 2. a–60 KX; b–50 KX; c–20 KX; d–15 KX; e–EDX analysis of Cu doped ZnFe_2O_4 nanoparticles

Ramamurthi et al studied the optical property of the NPs and the bandgap was found to be ~ 3.05 eV and ~ 3.51 eV for ZnFe_2O_4 NPs prepared using polyethylene glycol (PEG) and cetyltrimethylammonium bromide (CTAB) capping agents, respectively [24]. The present result is in agreement with the earlier reported values.

3.3. Magnetic properties – vibrating sample magnetometer (VSM)

The magnetization behaviour of the Cu-doped ZnFe_2O_4 compound was assessed in relation to an applied magnetic field ranging from 0 to 1.5 T. The obtained magnetization curve is shown in Fig. 4, which provides crucial insights into the material's magnetic characteristics. The coercivity (H_c) and retentivity (M_r) values obtained from the magnetization loops, are 19.40 Oe and 0.125×10^{-6} emu, respectively. The M-H curve reveals that the compound possesses a minimal retentivity of 0.125×10^{-6} emu, signifying its feeble remanent magnetization following the removal of the external magnetic field. The dependency of coercivity on crystallite size is well-documented, with smaller crystallite sizes

often leading to a transition from multidomain to single domain regimes or superparamagnetic states [25].

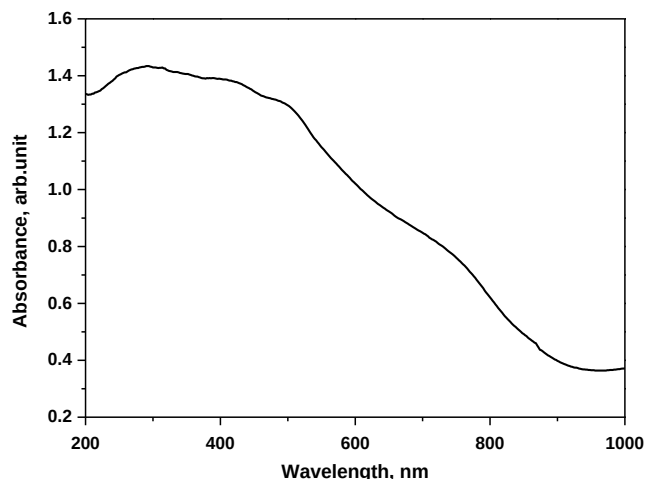
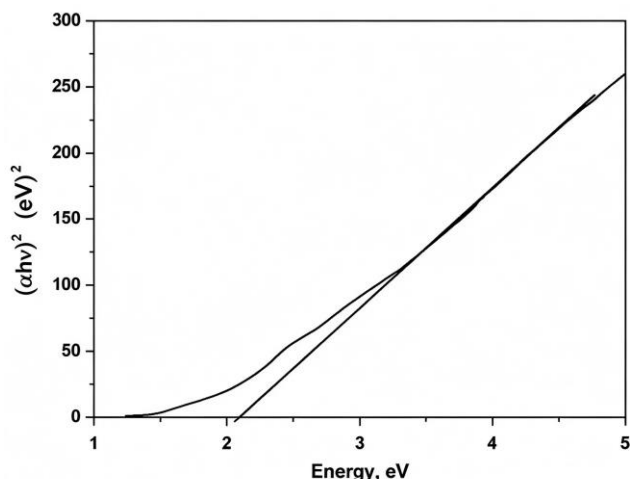


Fig. 3. UV–visible spectroscopy analysis of Cu doped ZnFe_2O_4 nanoparticles: a – absorption spectrum; b – Tauc plot

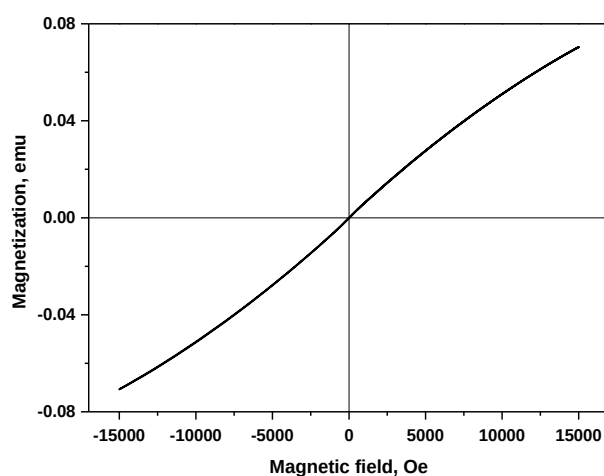


Fig. 4. Magnetic hysteresis loop of Cu doped ZnFe_2O_4 nanoparticles

Furthermore, the M–H curve reveals a non-saturation magnetization nature even at the maximum applied

The reduced coercivity and lack of magnetic saturation further indicate the material's transition from a superparamagnetic to paramagnetic state. This transition arises due to the reduced magnetic coupling and spin canting effects caused by the presence of Cu^{2+} ions, which have a lower magnetic moment compared to Fe^{3+} . The reduced magnetic moment of Cu^{2+} (1 μB) compared to Fe^{3+} (5 μB) significantly impacts the net magnetization of the compound. Hence, the magnetization studies demonstrate that the Cu doped ZnFe_2O_4 compound undergoes a superparamagnetic-to-paramagnetic transition due to the doping of non-magnetic Cu^{2+} ions, which disrupts the intrinsic ferrimagnetic ordering of ZnFe_2O_4 compound. This transition and the accompanying magnetic properties can be attributed to changes in crystallite size, spin interactions, and cation distribution within the spinel structure [27]. Arthur Charles et al. synthesized zinc copper ferrite NPs by co-precipitation process and studied the magnetic properties. The results clearly indicated the superparamagnetic behaviour of the sample [20]. zinc ferrite quantum dot (QD) is produced using microwave assisted hydrothermal synthesis and observed dual magnetic behaviour (superparamagnetism/perfect paramagnetism) [28].

The photodegradation of MB dye of copper doped zinc ferrite NPs is assessed. The photocatalytic efficiency of the nanoparticles is evaluated over a period of time against MB dye. The characteristic peak about 664 nm diminishes progressively with prolonged exposure from 0 to 2 h. This signifies that the MB dye has been nearly decomposed after 2 h. The performance of zinc copper ferrite has been recorded as 83 % dye degradation due to its capacity to absorb visible light and diminish the electron-hole recombination rate [29–31]. Arthur Charles et al. investigated the photocatalytic efficacy of MB dye using visible light, revealing a 65 % efficiency with zinc copper ferrite nanoparticles [18]. Wahid et al. synthesized Cu doped ZnFe_2O_4 using a hydrothermal method for MB degradation under visible light radiation and found the degradation efficiency of 97.06 % for $\text{Cu}_{0.75}\text{Zn}_{0.25}\text{Fe}_2\text{O}_4$ [29].

XPS spectrum is shown in Fig. 5, which illustrates the presence of Zn, Fe, Cu, and O elements in the sample. The Zn2p spectrum display peaks at 1022 eV and 1045 eV, denoted Zn2p_{3/2} and Zn2p_{1/2}, respectively (Fig. 6 a). The

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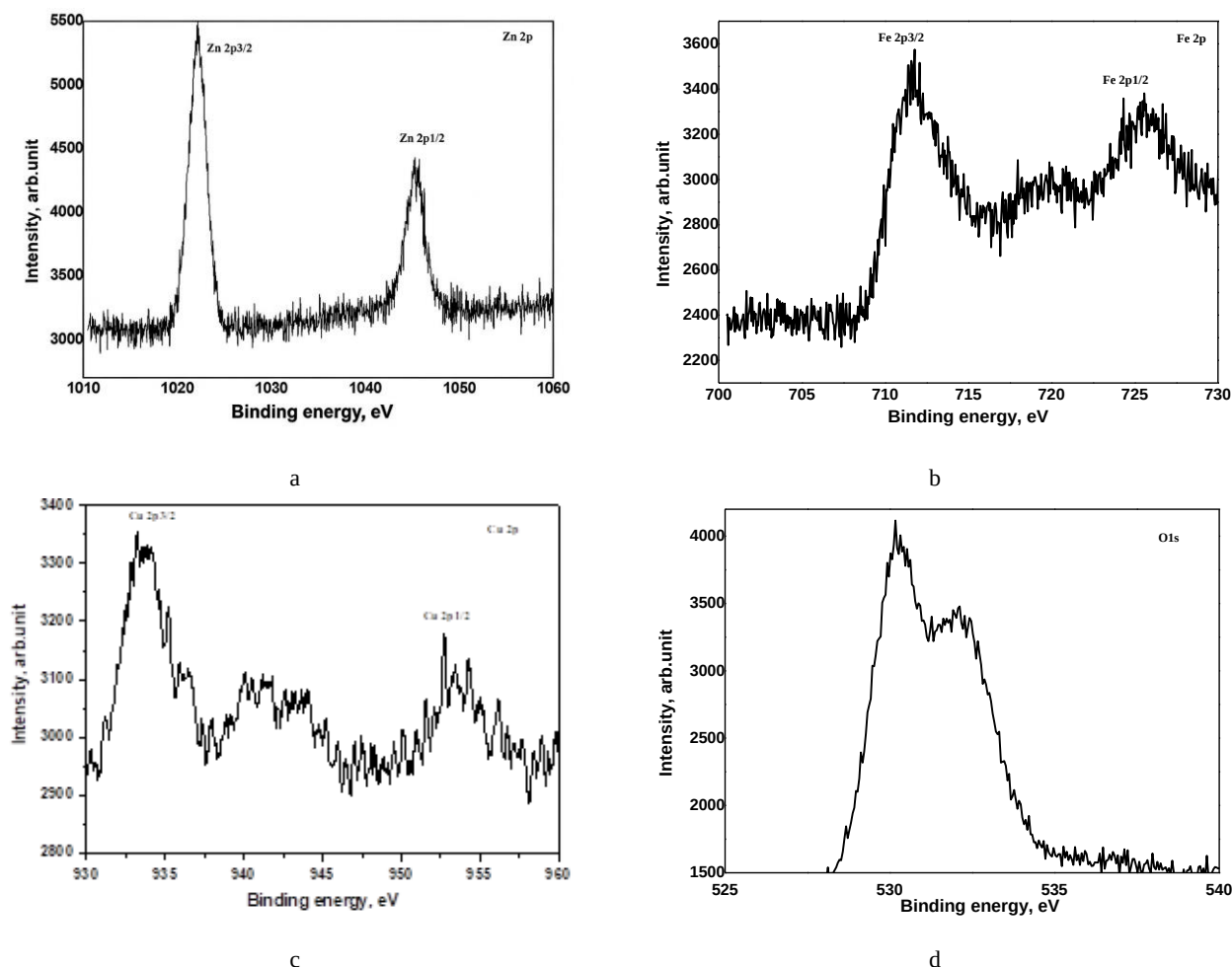


Fig. 6. XPS analysis for Cu doped ZnFe_2O_4 nanoparticles: a– Zn 2p; b– Fe 2p; c– Cu 2p; d– O 1s

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