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Facile synthesis, characterization and magnetic properties of $ZnFe_2O_4$ nanoparticles

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Abstract
Zinc ferrite ($ZnFe_2O_4$) nanoparticles were successfully synthesized via a hydrothermal technique using stoichiometric ratios of zinc nitrate and ferric nitrate as metal precursors, and sodium hydroxide as a precipitating agent. The structural, morphological, and magnetic properties of the synthesized nanoparticles were studied using X-ray diffraction (XRD), scanning electron microscopy (SEM), and vibrating sample magnetometer (VSM). XRD analysis confirmed the formation of a single-phase cubic spinel structure of $ZnFe_2O_4$ with an average crystallite size of ~7.48 nm. Morphological analysis revealed nearly spherical and uniformly distributed, forming agglomerated clusters of nanoparticles with uniform distribution. Magnetic studies demonstrated a superparamagnetic behavior of $ZnFe_2O_4$ nanoparticles.

Keywords: $ZnFe_2O_4$ nanoparticles, Zinc ferrite, Hydrothermal synthesis, X-ray diffraction (XRD), Scanning electron microscopy (SEM)

Introduction

Spinel ferrite is a class of ferrite with a general formula AB_2O_4 having a specific crystalline crystal structure known as the spinel structure. Zinc ferrite has gained considerable attention because of its soft magnetic behavior, high electrical resistivity, chemical stability, and low eddy current losses. It possesses a normal spinel structure, where Zn^{2+} ions preferentially occupy tetrahedral (A) sites and Fe^{3+} ions occupy octahedral (B) sites. [1] These properties make $ZnFe_2O_4$ an excellent material for applications in magnetic storage devices [2], gas sensors [3], photocatalysis [4], catalysis [5], biomedical and environmental applications [6]. The physical and magnetic properties of ferrite nanoparticles are strongly influenced by the synthesis method, particle size, cation distribution, and degree of crystallinity [7]. Therefore, developing an efficient and controllable synthesis route is crucial to tailor these parameters. Various synthesis methods, such as sol–gel [8], co-precipitation [9], microemulsions [10], and solid-state reactions [11], have been explored for $ZnFe_2O_4$ preparation. However, these conventional techniques often require high calcination temperatures, long processing times, or result in particle agglomeration and impurity formation. The hydrothermal method has emerged as a superior approach for synthesizing ferrite nanoparticles because it enables controlled nucleation and crystal growth under moderate temperature and pressure, resulting in highly crystalline, uniform, and phase-pure nanoparticles. This method can be more environmentally friendly as it typically uses water as a solvent, reducing the need for harmful organic solvents that are often used in other chemical reactions.

In this study, $ZnFe_2O_4$ nanoparticles were synthesized using a hydrothermal process employing zinc nitrate and ferric nitrate as precursors and characterized using XRD, SEM, and VSM to evaluate their structural, morphological, and magnetic properties.

Chemicals

Zinc nitrate hexahydrate [Zn $(NO_3)_2.6H_2O$] (98%), ferric nitrate nonahydrate [Fe $(NO_3)_3.9H_2O$] (98%), while sodium hydroxide (NaOH) were obtained from Loba Chemie Pt. Ltd. All reagents were of analytical grade and used without additional purification. The Deionized Water was used in the synthesis as a solvent.

Synthesis

A facial hydrothermal method used for synthesis was carried out by dissolving stoichiometric amounts of Zn $(NO_3)_2 \cdot 6H_2O$ and Fe $(NO_3)_3 \cdot 9H_2O$ with molar ratio 1:2 in 40mL deionized water under magnetic stirring to obtain a homogeneous solution at room temperature. Subsequently, NaOH solution (2 M) was added dropwise until the pH reached ~10. The resulting solution was transferred into a 50mL Teflon-lined stainless-steel autoclave and autoclave was tightly sealed and the reaction temperature maintained at 160˚C for 10h in a hot air oven, the autoclave was cooled to room temperature naturally. The precipitate was washed several times with deionized water and ethanol respectively, then dried at 80 °C for 6 h in hot air oven. The obtained powder was calcined at 350 °C for 2h to enhance crystallinity [12].

Characterization

The structural properties of ZnFe2O4 ferrite nanoparticles were examined by X-ray powder diffraction using an Ultima IV diffractometer (Rigaku Corporation, Japan) with monochromatic Cu Kα radiation with X-ray wavelength 1.5406 Å at 40kV and 40mA. The SEM used to examine surface morphology were obtained on instruments Carl Zeiss (Supra 55, Germany). The magnetic properties measurement of the synthesized nanostructure was carried out with a vibrating sample magnetometer (Micros Sense EZ-9, Milano, Italy) at room temperature with a maximum magnetic field of 15kOe.

Result and discussion

Structural Analysis

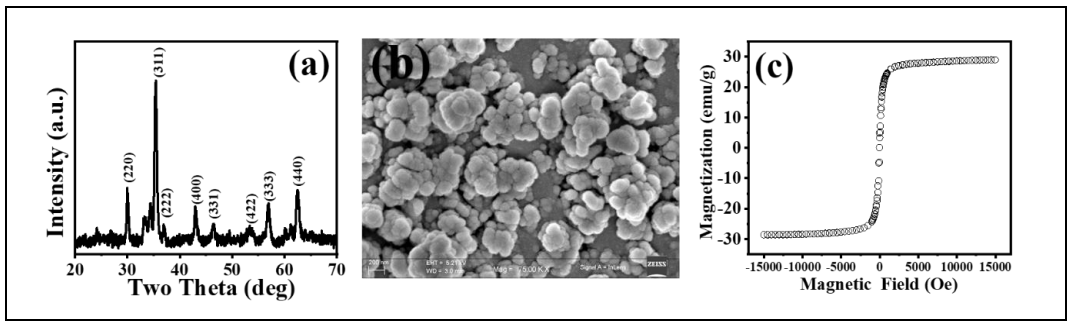

Fig 1: a) XRD pattern) SEM C) VSM of $ZnFe_2O_4$ nanoparticles

The X-ray diffraction pattern of $ZnFe_2O_4$ of hydrothermally synthesized nanoparticles with a view to investigating crystal structure is shown in Figure 1a, The reflections observed correspond to (220), (311), (400), (422), (333), (440), and (533) planes evidencing the formation of an FCC cubic spinel structure with the space group of Fd3m $ZnFe_2O_4$ nanoparticles and it is well-matched with the standard diffraction data JCPDS No. 82–1049. The average crystallite size of $ZnFe_2O_4$ is estimated by using the Scherrer's equation and is found to be 7.48 nm. [13]

Figure 1b shows the SEM micrograph of $ZnFe_2O_4$ nanoparticles synthesized via the hydrothermal method. The image reveals that the particles are nearly spherical and uniformly distributed, forming agglomerated clusters due to magnetic dipole–dipole attractions and surface tension effects among fine nanoparticles. [14] The hydrothermal route facilitated controlled nucleation and homogeneous growth, leading to smooth and well-defined grain boundaries. [15] No evidence of secondary or impurity phases is observed, confirming the phase purity of the synthesized ferrite. The observed agglomeration is typical for magnetic ferrites and can be attributed to the intrinsic magnetic interactions between neighboring nanoparticles. [16] The fine particle size and uniform morphology are advantageous for improving magnetic and catalytic performance owing to the enhanced surface-to-volume ratio. Thus, the SEM analysis validates that the hydrothermal synthesis method provides an efficient pathway for producing highly crystalline, nanosized $ZnFe_2O_4$ particles with uniform morphology and dense packing suitable for technological applications. [17]

Magnetic Properties

The magnetic behavior of hydrothermally synthesized $ZnFe_2O_4$ nanoparticles was analyzed using a vibrating sample magnetometer (VSM) at room temperature, as shown in Figure X. The obtained M–H hysteresis loop exhibits a narrow loop with low coercivity (Hc=40.43 Oe) and remanent magnetization (Mr=4.60 emu/gm), indicating a superparamagnetic in nature. The measured saturation magnetization (Ms=28.44 emu/gm) value is significantly lower than that of bulk $ZnFe_2O_4$, which can be attributed to surface spin disorder, finite-size effects, and cation redistribution between the tetrahedral (A) and octahedral (B) sites in the spinel lattice. In nanosized ferrites, partial inversion occurs where some Zn^{2+} ions migrate to B sites and Fe^{3+} ions occupy A sites, leading to uncompensated magnetic moments and hence a weak ferromagnetic response. The small particle size and high surface-to-volume ratio enhance surface spin canting, resulting in reduced Ms. The negligible coercivity further confirms the single-domain nature of the nanoparticles and the absence of magnetic anisotropy barriers, a typical feature of hydrothermally synthesized ferrites. Overall, the magnetic analysis confirms that $ZnFe_2O_4$ nanoparticles exhibit superparamagnetic characteristics, making them suitable for potential applications in magnetic separation, biomedical drug delivery, and photocatalytic systems where controllable magnetic response and low remanence are desired. [18]

Conclusion

Zinc ferrite ($ZnFe_2O_4$) nanoparticles were successfully synthesized using a simple hydrothermal method. The XRD analyses confirmed the formation of a pure spinel phase. Morphological studies revealed uniform nanosized particles, while magnetic analysis demonstrated superparamagnetic behavior. The results suggest that hydrothermally synthesized $ZnFe_2O_4$ nanoparticles possess desirable structural and magnetic properties for potential applications in photocatalysis, magnetic separation, and biomedical fields.

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