Synthesis and investigation of Structural, Morphological and Magnetic properties of Ni and Zn Spinel ferrites

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Abstract:- This research investigates the structural, morphological, and magnetic properties of nickel (Ni) and zinc (Zn) spinel ferrites synthesized by a sol-gel auto-combustion method. The structural properties were analyzed using X-ray diffraction (XRD), confirming the formation of single-phase spinel structure with cubic symmetry and calculating the lattice parameters. Scanning electron microscopy (SEM) was employed to study the morphological characteristics, revealing uniformly distributed grain sizes and surface morphology. Magnetic properties were examined using a vibrating sample magnetometer (VSM), which provided insights into the saturation magnetization, coercivity, and remanence. The structural, morphological, and magnetic properties were systematically explored, demonstrating that Ni & Zn substitution significantly alters the magnetic behavior, optimizing the materials for potential applications in high-frequency devices, magnetic storage media, and biomedical fields.

Keywords:- Spinel ferrites, SEM, VSM, XRD, Sol-gel method

1. Introduction

Spinel ferrites have garnered significant attention due to their versatile structural, magnetic, and electrical properties, which make them suitable for a wide range of applications including magnetic storage, microwave devices, catalysis, and biomedical technologies. Among the various types of spinel ferrites, nickel (Ni) and zinc (Zn) spinel ferrites stand out due to their unique magnetic properties, high electrical resistivity, and chemical stability. These materials are typically represented by the formula MFe_2O_4, where M represents a divalent metal ion, such as Ni or Zn [1, 2]. The sol-gel auto-combustion method is a widely used technique for synthesizing spinel ferrites due to its simplicity, cost-effectiveness, and ability to produce nanoparticles with controlled size and morphology. This method involves the transition from a sol to a gel, followed by a combustion process that yields the desired ferrite nanoparticles. The resulting materials often exhibit high purity and well-defined structural characteristics. In this study, we aim to investigate the structural, morphological, and magnetic properties of Ni and Zn spinel ferrites synthesized using the sol-gel auto-combustion method. By systematically varying the Zn content in the Ni-Zn ferrite matrix, we seek to understand how the structural and morphological attributes influence the magnetic properties of these materials [3]. X-ray diffraction (XRD) will be used to confirm the spinel phase and determine the lattice parameters. Scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDS) will be employed to examine the surface morphology and elemental composition. The magnetic properties will be characterized using a vibrating sample magnetometer (VSM) to evaluate the saturation magnetization, coercivity, and remanence [4, 5]. The findings of this research are expected to provide valuable insights into the relationship between the structural, morphological, and magnetic properties of Ni-Zn spinel ferrites. Understanding these relationships will aid in the design and development of advanced materials for technological applications, such as magnetic storage media, high-frequency devices, and biomedical applications.

2. Experimental Procedure

The Spinel ferrites $NiFe_2O_4$ & $ZnFe_2O_4$ was synthesized by sol-gel auto combustion route. The analytical grade $Fe(NO_3)_3·6H_2O$, $Ni(NO_3)_2·6H_2O$, $Zn(NO_3)_2·6H_2O$ and citric acid ($C_6H_8O_7·H_2O$) were used as raw materials. The appropriate amount of nitrates and citric acid is first dissolved into de-ionized water to form a mixed solution with molar ratio of nitrates to citric acid 1:1. The pH value of solution was adjusted to about 7 by adding ammonia. Then, the mixed solution was poured into a bicker and heated at 85 °C under constant stirring to transform into a dried gel. Being ignited, the dried gel was burnt in a self-propagating combustion way to form loose powder. The solutions were evaporated by continuous heating at 100 °C with agitation until the formation of viscous gel. The samples were dried, and the dried powders were annealed in air furnace at 800 °C for 7 h., and then slowly cooled to the room temperature. Finally, the annealed powder was grounded using the mortar and pestle [6, 7].

The equation used for calculating the stoichiometric values of the metal nitrates is as follows:

$$Ni(NO_3)_2·6H_2O + 2Fe(NO_3)_3·6H_2O \longrightarrow NiFe_2O_4 + 4N_2O_5 + 12\ H_2O + \text{volatile}$$

$$Zn(NO_3)_2·6H_2O + 2Fe(NO_3)_3·6H_2O \longrightarrow ZnFe_2O_4 + 4N_2O_5 + 12\ H_2O + \text{volatile}$$

The microstructure and the chemical composition of the samples were investigated by X-ray diffraction using Cu–Kα (λ = 1.5406 Å), scanning electron microscopy (SEM). The values of the coercive fields, saturation and remanent magnetizations were obtained from the hysteresis loops obtained from a vibrating sample magnetometer (VSM).

3. Result and Discussion

i) Structural Study

The XRD pattern of the both the sample under investigation is shown in fig. 1. and fig. 2. In the XRD of $NiFe_2O_4$ higher intensity peaks were indexed corresponding to (220), (311), (400), (422), (511) and (440) planes. Similarly in the XRD of $ZnFe_2O_4$ higher intensity peaks were indexed corresponding to (220), (311), (222), (400), (422), (511) and (440) planes. It is observed that (222) absent in XRD of $NiFe_2O_4$. All XRD patterns show the single-phase structure of the Ni and Zn spinel nanoferrites. Table 1 show the different parameter calculated from the XRD patterns of $NiFe_2O_4$ & $ZnFe_2O_4$ nanoferrites. The broadness of the maximum intensity peak shows the nano-sized ferrites [4]. These measured planes for the $NiFe_2O_4$ and $ZnFe_2O_4$ sample are in very good agreement with the reported values of JCPDS card No. 44-1485 and JCPDS card No. 89-4926 respectively. The diffraction lines provide clear evidence for the formation of cubic phase of pure inverse spinel structure of nickel ferrite with Fd3m space group [8,9].

The crystallite size was calculated using the Debye's Scherrer formula

$$D = \frac{0.9\,\lambda}{\beta\cos\theta} \qquad (1)$$

Where D is the particle size, β is the full-width at half-maximum; λ is the wavelength of x-ray (1.5406Å); and θ the angle of diffraction. The particle size for this sample obtained is 18.15 nm and 19.39nm.

The x- ray density of the samples is calculated by relation

$$\rho_x = \frac{8M}{Na3} \qquad (2)$$

Where M is the molecular weight of the sample, N the Avogadro's number and a^3 the volume of the cubic unit cell [6, 10].

Ferrites	Lattice constant(Å)	Crystallite size d (nm)	X-ray density (gm/cm^3)	Volume
$NiFe_2O_4$	8.432	18.15	5.293	599.503 (Å)^3
$ZnFe_2O_4$	8. 440	19.39	5.596	601.211 (Å)^3

Table 1. Variation of structural properties of $NiFe_2O_4$ & $ZnFe_2O_4$

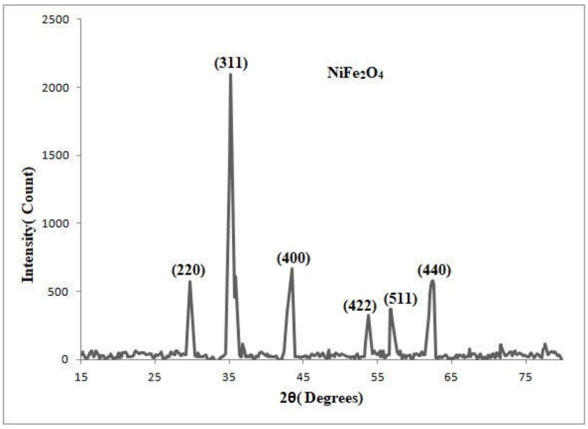

Fig. 1. XRD of $NiFe_2O_4$

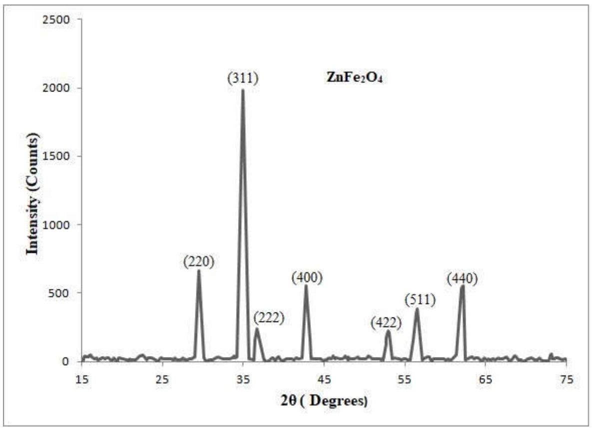

Fig. 2. XRD of $ZnFe_2O_4$

ii) Morphological Study

The microstructure of the $NiFe_2O_4$ & $ZnFe_2O_4$ ferrite samples is analyzed using SEM micrographs as shown in Fig. 3 and Fig. 4. The micrographs show the agglomerated grainy structure [11,12]. Nanosized of the particle is confirmed from SEM images. The SEM image revealed that the grains have an almost regular shape and homogeneous distribution.

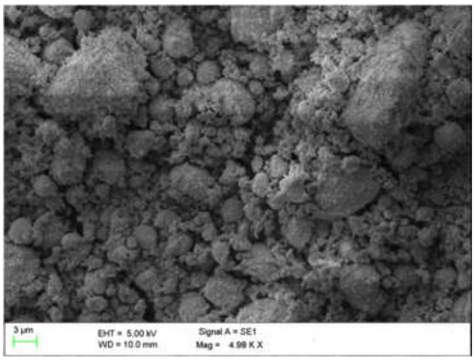

Fig. 3. SEM Image of $NiFe_2O_4$

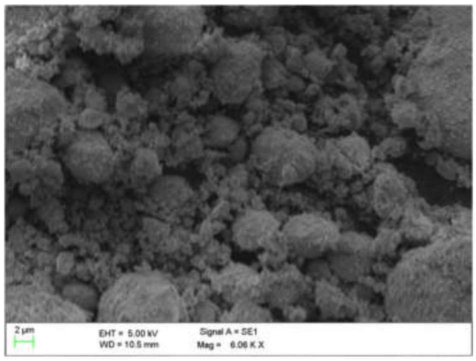

Fig. 4. SEM Image of $ZnFe_2O_4$

iii) Magnetic study

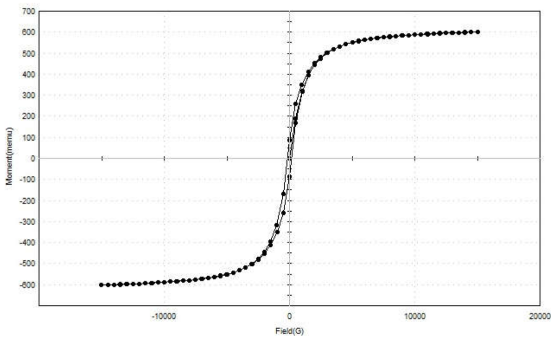

Fig. 5. Saturation Magnetization of $NiFe_2O_4$

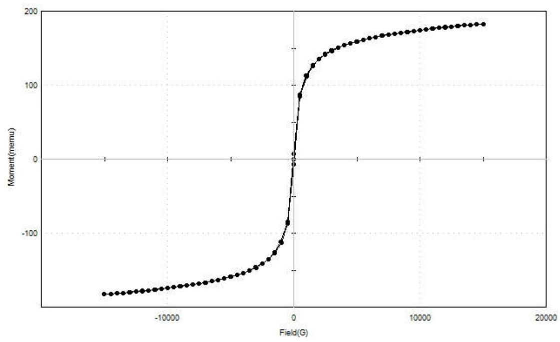

Fig. 6. Saturation Magnetization of $ZnFe_2O_4$

The Room temperature hysteresis loops of $NiFe_2O_4$ & $ZnFe_2O_4$ nanoferrites are recorded under the maximum applied field of 15000 Gauss. The magnetic loops as shown in the Fig. 4. Table.2 depicts the magnetic parameters which are calculated from M-H loops [13]. The saturation magnetization and magnetic remanence of $NiFe_2O_4$ is 60.83 & 17.39 & decreases in saturation magnetization and magnetic remanence is observed for $ZnFe_2O_4$ also the coercivity decreased accordingly [14]. It is noticed that the magnetic parameters are influenced by the extrinsic factors such as porosity, homogeneity, morphology, density and distribution of cations at lattice [15]. Table 2 shows the magnetic parameters of the sample.

Table 2 Magnetic properties of $NiFe_2O_4$ and $ZnFe_2O_4$ nanoparticles

Ferrites	Ms (emu/g)	Mr (emu/g)	Hc (Oe)
$NiFe_2O_4$	60.83	17.39	111.98
$ZnFe_2O_4$	51.75	6.397	159.16

4. Conclusion

In this study, we investigated the structural, morphological, and magnetic properties of Ni and Zn spinel ferrites synthesized using the sol-gel auto-combustion method. The X-ray diffraction (XRD) analysis confirmed the formation of a single-phase spinel structure with cubic symmetry for all synthesized samples, with the lattice parameters varying systematically with the substitution of Zn. Scanning electron microscopy (SEM) revealed uniform grain distribution and consistent surface morphology, while energy-dispersive X-ray spectroscopy (EDS) confirmed the elemental composition and purity of the ferrites. The magnetic properties, characterized using a vibrating sample magnetometer (VSM), demonstrated that the substitution of Zn significantly influences the magnetic behavior of Ni-Zn ferrites. Overall, our results underscore the importance of Zn substitution in tailoring the properties of Ni ferrites. The ability to fine-tune the magnetic properties by adjusting the Zn content makes these materials promising candidates for high-frequency devices, magnetic storage media, and biomedical applications. Further research could explore the optimization of synthesis parameters and the potential integration of these ferrites into practical devices, expanding their applicability in advanced technological fields.

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