X-Ray Diffraction and Magnetic Properties of Nd Substituted NiZnFe$_2$O$_4$ Characterized by Rietveld Refinement

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Abstract: Solid-state method developed neodymium-doped Ni-Zn ferrites exhibit additional orthoferrite and α-Fe$_2$O$_3$ phases. The change in magnetic properties and lattice parameters are influenced by above mentioned extra phases other than the Nickel-Zinc ferrite spinel structure. Observations indicate that curie temperature, as well as saturation magnetization, reduce with an increase in Neodymium concentration. At the same time, for Nd = 0.02, initial permeability has been slightly increased. This paper explains the reason for these changes in terms of diluted octahedral sites replaced with Nd ions instead of Fe-ions and the number of additional phases that are present.

Keywords: Ferrites; Orthoferrites; Magnetization; Lattice constant; Neodymium; Zinc.

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1. Introduction

Soft magnetic materials exhibiting nanosized dimensions are of prime significance today in the research perspective as a result of their dielectric, electric, and magnetic properties. Nickel-Zinc ferrites exhibit high electrical resistivity, curie temperature, and low current loss. These ferrites are significant and versatile materials with various applications in transformer cores, radiofrequency devices, microwave absorbers, wave absorbers, inductors, magnetic recording media, and power transformers, etc. [1-6]. They exhibit electrical resistivity of high value in the range of $10^5$ to $10^8$ ohm-cm with moderate permeability and magnetization, eddy current losses of low value because of more electrical resistivity making them suitable for high-frequency applications [7-8]. Rare earth is being doped to increase further permeability and magnetization as rare-earth ions have a high magnetic moment. At the same time, rare earth ion induction might lead to lattice distortion, internal stress may be generated due to causing of heterogeneity in structure. A critical investigation is required to study the structure of spinel lattice in terms of their site occupancy [9-10].

It is already reported that rare-earth ions having low ionic radii come into spinel lattice while ions of large radii get diffused into grain boundaries, which form an extremely thin layer around these grains. The above changes drastically alter ferrites properties demanding demands thorough examination of the system. Therefore it is definitely necessary to study the effect of...
added neodymium on-site occupancy as well as segregation related to the Nickel-Zinc ferrite system [11-13].

This paper gives an elaborate explanation regarding structural properties as well as magnetic properties of Ni-Zn ferrites substituted with neodymium having basic composition Ni$_{0.65}$Zn$_{0.35}$Fe$_2$O$_4$. Many methods for the preparation of samples to fabricate nanomaterials are available, which include micro-emulsion [14], co-precipitation [15], sol-gel [16], co-precipitation [17], citrate precursor, ceramic sintering, reﬂuxing [18], and solid-gel chemical reaction [19] methods. In the present work, we adopted a solid-state reaction method for the preparation of ferrite mentioned above. Explanation of variation observed in saturation magnetization, coercivity, and lattice constant was done on the basis of orthoferrite additional phases and iron oxide present in the samples.

2. Materials and Methods

By using the regular ceramic method, a series of samples with composition Ni$_{0.65}$Zn$_{0.35}$Nd$_x$Fe$_2$O$_4$ where $x = 0.00$ to $0.10$ in steps of $0.02$ were prepared. Fe$_2$O$_3$ (98.5%), NiO (99.9%), ZnO (99.5%), and Nd$_2$O$_3$ (99.9%) of high purity analytical grade were considered with a nonstoichiometric ratio for maintaining excess iron oxide of ten to fifteen percent and ﬁnely crushed. After crushing, they were thoroughly mixed and ground in acetone media for 4 hours by using agate mortar and pestle. Calcination of this mixed homogeneous powder was done at a temperature of 900°C in the air for 2 hours and then is made to undergo slow cooling. Uniformly sized crystallites of small size were prepared from this powder in pre-sintered condition by crushing another time and grinding thoroughly for two hours in acetone medium. Pellets and toroids were made from the mixer with the help of a binder (fifteen percent polyvinyl alcohol) having 3 tons/inch$^2$ and 5 tons/inch$^2$ a uniaxial pressure of essentially.

Pellets were of thickness 4 mm and diameter of 3 mm while toroids of inner diameter 13 mm and outer diameter 18 mm were prepared. These toroids and pellets are lastly sintered at a temperature of 1250°C in the air for 2 hours period and cooled in a natural way. Polishing of sample surfaces was done in order to remove the oxide layer formed, if any, while sintering process.

3. Results and Discussion

3.1. Structural analysis.

3.1.1. X-ray diffraction and Rietveld refinement.

X-ray diffraction studies conﬁrm the spinel structure. The lattice parameter values acquired for every reﬂected plane were plotted against the Nelson-Riley function [20, 21].

$$F(\theta) = (1/2)[ \cos^2 \theta / \sin \theta + \cos^2 \theta / \theta].$$

Estimation of accurate or nearest $a_0$ (lattice constant) value for each individual sample has been done by extrapolating these lines $F(\theta) = 0$. X-ray diffraction patterns indicate that measurements of lattice constant ($a = 8.3840$ Å) correlate with that of the reported [22-24]. Figure 1a shows additional lines in all samples with neodymium, whose intensity increases with an increase in impurity concentration. These additional lines are assumed to be those which correspond to NdFeO$_3$ second phase [25, 26]. Diffraction patterns of the sample for $x = 0.02, 0.04, 0.06, 0.08$, and $0.10$ were shown in Fig.1 while Table 1. lists their structural
properties. Rietveld analysis is shown in Figures 1b and 1c, which indicate GOF for all samples and are given in Table 1. Extra peaks related to $\alpha$-Fe$_2$O$_3$ are due to the presence of iron oxide that was inducted into the ferrite system as a stoichiometric conscious of providing accommodation for Neodymium ions of high radii, high concentration, which was found to be incapable of producing [27, 28]. Figures 2a and 2b represent Rietveld refined XRD spectra, while Table 1. shows the phase contents and lattice parameters.

![Rietveld analysis](https://doi.org/10.33263/BRIAC112.90629070)

**Figure 1a.** X-ray Diffraction Patterns of Ni$_{0.65}$Zn$_{0.35}$Nd$_x$Fe$_2$O$_4$; 1b. Rietveld refined XRD of Nickel-ZincFerrite; 1c. Rietveld refined XRD of Neodymium doped Nickel-ZincFerrite for $x=0.02$

3.1.2. Magnetic properties.

Hysteresis curves for the developed sample (Ni$_{0.65}$Zn$_{0.35}$Fe$_{2-x}$Nd$_x$O$_4$ where $x=0.02$ to 0.10 with steps of 0.02) are shown in Figure 2 while their magnetic properties like Remanence, Coercivity, and Saturation Magnetization ($M_r$, $H_c$, and $M_s$) are shown in Fig.3. Rare-earth
doping may dilute magnetic properties, which might disturb the Fe$^{3+}$-Fe$^{2+}$ along with the interaction of 3d-4f [29-32]. The saturation magnetization (Ms) of the Ni-Zn ferrite for the substituted Nd decreases for all the compositions, but for the compositions, x = 0.06 and 0.1 observed with maximum Ms values. As a result, there an increase in Hc values, which is shown in Fig.3. The factors which are responsible for the increase of Hc are magnetocrystalline anisotropy, loss of metal cations, Bohr magneton, magnetic domain, and superexchange. Further, the increase of Hc is assumed to be due to Nd$^{3+}$ with a more ionic radius of 0.983 Å on comparison with Fe$^{3+}$ ion of radius 0.654 Å leading to lattice expansion as well as an increase in interplanar spacing, reducing the response of the particles to the applied magnetic field [33-38]. Improvement of Remanence (Mr) from x = 0.04 indicate exchange interactions moderation [39, 40].

**Table 1.** XRD Analysis of Nd-doped Ni-Zn-Ferrite.

| Nd content (x) | Phases present | rwp % | vol % | wt % | cell par. (Å) a | cell par. (Å) b | cell par. (Å) c | Crystallite size (Å) | microstrain |
|----------------|----------------|-------|-------|------|----------------|----------------|----------------|---------------------|------------|
| 0.00           | Ni-Zn          | 86.5503 | 85.7113 | 8.4135 | 2082.7666       | 1.7356E-5       |                |                     |            |
|                | Iron(III) Oxide| 13.4497 | 4.2878 | 5.4292 | 55.2840        |                |                |                     | 1.4001E-4   |
| 0.02           | Ni-ZnNd1       | 82.0003 | 82.1459 | 8.4158 | 2260.5823       |                |                |                     | 1.4098E-4   |
|                | Iron(III) Oxide| 17.9997 | 17.8541 | 5.4284 | 55.3054        |                |                |                     | 9.2617E-6   |
| 0.04           | Ni-ZnNd2       | 75.5638 | 75.1479 | 8.4164 | 2026.8478       |                |                |                     | 1.0519E-4   |
|                | NdFeO3         | 3.0863  | 3.9968  | 5.5887 | 7.7619         | 5.4489          |                |                     | 1000.0000   |
|                | Iron(III) Oxide| 21.3500 | 20.8552 | 5.4305 | 55.2930        |                |                |                     | 3.2412E-6   |
| 0.06           | Ni-ZnNd3       | 80.2392 | 79.5580 | 8.4147 | 2283.1885       |                |                |                     | 5.411E-4    |
|                | NdFeO3         | 4.2992  | 4.3917  | 5.5887 | 7.7619         | 5.4489          |                |                     | 1000.0000   |
|                | Iron(III) Oxide| 15.5516 | 15.0503 | 5.4267 | 55.3113        |                |                |                     | 3.2504E-4   |
| 0.08           | Ni-ZnNd8       | 74.5909 | 73.4001 | 8.4155 | 1983.9523       |                |                |                     | 7.479E-6    |
|                | NdFeO3         | 7.6932  | 9.7122  | 5.5887 | 7.7619         | 5.4489          |                |                     | 1000.0000   |
|                | Iron(III) Oxide| 17.1599 | 16.8877 | 5.4278 | 55.3093        |                |                |                     | 3.1609E-4   |
| 0.10           | Ni-ZnNd10      | 80.4200 | 78.8452 | 8.4119 | 3168.2114       |                |                |                     | 4.2091E-6   |
|                | NdFeO3         | 8.8989  | 11.0875 | 5.5845 | 7.7700         | 5.4525          |                |                     | 1000.0000   |
|                | Iron(III) Oxide| 10.6810 | 10.0673 | 5.4274 | 55.2844        |                |                |                     | 3.7252E-4   |

**Figure 2.** Hysteresis Loops of Nd$^{3+}$ doped Nickel-Zinc Ferrite samples.
Nd alters the magnetic moment as well as change the cubic structure based on the extent of doping[41]. Reports indicate that some of the neodymium reside at grain boundaries while others do not enter into the ferrite lattice. Also, the presence of the \( \alpha \text{-Fe}_2\text{O}_3 \) or \( \text{REFeO}_3 \) phase might increase at grain boundaries at low concentration or high concentration of rare-earth-doped samples, causing some variation in magnetic properties. As evidence indicate, such a phase in present samples under investigation, magnetic properties, and their dilution may be due to several effects, as mentioned in the previous section. Samples under investigation show \( \text{NdFeO}_3 \) phase for concentration \( x = 0.040 \) an improved remanence or the lowest state of \( M_s \) amongst the existing samples. The initial decrease in saturation magnetization, as shown in Figure 3, is because of octahedral site dilution by \( \text{Nd}^{3+} \) ions, while its rise is due to an increase of Nd might be due to additional content of iron that enters spinel lattice. The variation of coercivity exhibited in Figure 4 and remanent magnetization are shown in Figure 5 due to the rise in neodymium content may be attributed to the reasons mentioned above [42].

3.1.3. Curie temperature.

The change in permeability with temperature for samples under investigation is shown in Figure 6. As shown in the figure, maximum permeability is found for \( x=0.02 \), while the rest
of the samples under investigation show versatile behavior. A shift towards lower temperature side is observed for Curie temperature indicate ion dilution in exchange interaction between Fe³⁺-O-Fe³⁺ of ferrite lattice due to the presence of additional phases that include α-phase Fe₂O₃ or NdFeO₃ phase as indicated by diffraction studies [43]. Investigating samples are stable up to the point of curie temperature point, which indicates spinel structure deformation instead of impurity presence.

![Figure 6](https://biointerfaceresearch.com/)

**Figure 6.** Relative permeability versus temperature of Nd³⁺ doped Nickel-Zinc Ferrite samples.

![Figure 7](https://biointerfaceresearch.com/)

**Figure 7.** Variation of Curie temperature with Nd concentration.

Fig. 7 indicates a decrease in Curie temperature with an increase of Nd³⁺ concentration. This can be attributed to the decrease in exchange interactions under the influence of reduced magnetic moment at B-sites as well as the presence of α-Fe₂O₃ and anti-ferromagnetic orthoferrite phases. A decrease in magnetic interaction between ferrite grains by additional phases leads to a reduction in exchange interaction [44].
4. Conclusions

Synthesis of Ni_{0.65}Zn_{0.35}Fe_{2-x}Nd_xO_4 for x = 0.020, 0.040, 0.060, 0.08 and 0.1 samples were done with traditional solid-state reaction method. Observed X-ray diffraction patterns indicate NdFeO_3 and α-Fe_2O_3 phases and their associated secondary phases along with the formation of cubic spinel phases. Rietveld refinement analysis of Ni_{0.65}Zn_{0.35}Fe_{2-x}Nd_xO_4 indicates primary and secondary phases. M-H curves for x = 0.10 reveal highest value of saturation magnetization equal to 49.1 emu/g while for x = 0.02 composition maximum magnetic permeability of value 42.5 was obtained. Plots related to magnetic permeability versus temperature for Ni_{0.65}Zn_{0.35}Fe_{2-x}Nd_xO_4 samples indicate a decrease of magnetic curie transition temperature from 382 °C to 330°C as Nd content varies with x values ranging between 0.00 - 0.10 in steps of 0.02. Finally, it may be inferred that substituting neodymium leads to a decrease in magnetic exchange interactions decrease between tetrahedral (A) and octahedral (B) sites.

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Conflicts of Interest

The authors declare no conflict of interest.

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