Studies on Synthesis and Structural Properties of Nickel Ferrite before and after Gamma Irradiation

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Abstract. In the present work nanocrystalline NiFe₂O₄ samples were prepared by the sol-gel auto combustion technique. The synthesis was carried out by taking citric acid as fuel with metal nitrate to fuel ratio as 1:3. The obtained powder was annealed at 550°C for 4 h and then used for structural and magnetic investigations. Prepared nickel ferrite samples have been irradiated by gamma-ray (⁶⁰Co) to examine the changes that occurred in structural properties. Structural properties of nickel ferrite nanoparticles before and after gamma irradiation were carried out by X-ray diffraction (XRD) technique. From the XRD pattern, it was observed that all the Braggs planes reveal cubic spinel structure before and after gamma irradiation. A close examination of the XRD pattern revealed the crystallite size of 21 nm and 19 nm for nickel ferrite samples before and after gamma irradiation respectively. The obtained results help in providing interesting and useful study for various applications of nickel ferrites.

1. Introduction
Now-a-days magnetic materials are grabbing the attention of researchers and scientists because of their novel physicochemical properties [1]. Among magnetic materials, ferrites are the most attention-grabbing materials because of their magnetic and insulator properties [2, 3]. Among the various ferrites, nickel ferrites are vitally attributable to their astounding properties, such as high magnetic permeability, lower eddy current losses, and high resistivity making them a potential material for high-frequency applications [4-6].
The development of nanoscience and nanotechnology leads us to deal with and fabricating the material at nanoscale for a particular application. The unique characteristics of nanostructured materials are imported due to their changed electronic structure, close to that of an isolated atom or molecule [3]. Modifications and improvements of these materials are important to adjust the performance and efficiency of the different devices that use them. In recent times, in order to study the effect of irradiation on the properties of ferrite materials, fast heavy ions, laser beams, and gamma rays have been used [7]. Irradiation can be an effective tool to enhance crystal defects and adjust the properties of ferrite (soft and hard magnetic) in a controlled manner. A lot of scientific focus is thus given to the gamma-irradiation caused by the formation and alteration of defects leading to tunable structural and magnetic properties of ferrites. Radiation energy such as gamma rays interacts with materials (atomic electrons and atomic nuclei) [8, 9]. These interactions result in the scattering of particles, the excitation of electrons and vibrations (thermal), and the ionization of atoms, which usually cause interference in the material structure. This in turn modifies the material's electrical and magnetic properties [10, 11]. These improvements can be due to the breakage of ferrimagnetic ordering, surface-state pinning, and cation inversion, etc. Such modifications are quantitatively functions of the dose intensity of irradiation, dose duration, dose absorbed by the materials and quality of the target materials, etc [12, 13].

In the present study, we have carried out the synthesis of nickel ferrite by the sol-gel auto combustion method and irradiated with gamma-ray to understand the effect of gamma radiation on the structural properties of nickel ferrite.

2 Experimental

2.1 Materials

Synthesis of nickel ferrite was carried out by using chemicals such as ferric nitrate (Fe(NO\textsubscript{3})\textsubscript{3}⋅9H\textsubscript{2}O), nickel nitrate (Ni(NO\textsubscript{3})\textsubscript{2}⋅6H\textsubscript{2}O), citric acid (C\textsubscript{6}H\textsubscript{8}O\textsubscript{7}), ammonia (NH\textsubscript{3}), and distilled water. All the chemicals were used without further purification.

2.2 Preparation of nickel ferrite

The nickel ferrite nanopowder was prepared by using a cost-effective safe sol-gel auto combustion technique. To obtain better combustion citric acid was used as a chelating agent. The detailed procedure of sol-gel auto combustion is explained in our earlier reports [14, 15]. The prepared fluffy powder was sintered at temperature 550 °C for 4 h using a muffle furnace to get a better crystalline nature and purity.

2.3 Characterization

The synthesized nickel ferrite samples were characterized by X-ray diffraction (XRD) technology to identify the crystalline phase. The Bruker D-8 X-ray diffractometer has a (2θ) angle range of 20-80°.

Results and Discussions

2.4 X-ray Diffraction

The X-ray diffraction pattern of nickel ferrite synthesized by the sol-gel auto-combustion method recorded at room temperature in 2θ range from 20-80° showed in figure 1. The values of the lattice parameter of the prepared sample calculated by using the following relation [16, 17].

$$a = \frac{d_{hkl}}{\sqrt{h^2 + k^2 + l^2}}$$

Where d is the interplanar spacing of two planes, ‘a’ is the lattice constant, and (hkl) is the miller indices. It revealed that the lattice parameter decreases after irradiation and caused increase in X-ray density.

$$d_B = \frac{m}{V}$$

Where, m is the mass and V is the volume (πr\textsuperscript{2}h) of pallets. The obtained XRD patterns revealed the formation of the cubic spinel structure with Fd-3m space group. There is no impurity peak observed in the XRD pattern. The crystallite size of nickel ferrite before and after irradiation was found to be 21 nm and 19 nm respectively, which is calculated by using Debye-Scherrer’s formula [18],
Where, \( D = \frac{k\lambda}{\beta \cos \theta} \)

Where, \( k \) is the constant having value 0.89, \( \lambda \) is the X-ray light source wavelength (1.540 Å), \( \beta \) is full width at half maximum (FWHM) and \( \theta \) is the glancing angle. The peak positions of the irradiated sample are shifted to the lower angle (2θ). The slight change of the reflective peaks in the irradiated samples is due to some induced disorder (compressive strain) in the crystal structure resulting from ion migration into interstitial positions. X-ray density (\( dx \)) of nickel ferrite was calculated by using the relation [19, 20],

\[
d_{x} = \frac{8M}{N_{A}a^{3}}
\]

Where \( dx \) is the X-ray density, \( M \) is the molecular weight of the composition, \( N_{A} \) is the Avogadro’s number and ‘\( a \)’ is the lattice constant. Calculated values of lattice constant, unit cell volume, average crystallite size, X-ray density, bulk density, the porosity of before and after irradiation of prepared nickel ferrite is tabulated in table 1.

**Figure 1.** X-ray diffraction pattern of NiFe\(_{2}\)O\(_{4}\) nanoparticles before and after radiation.

**Table 1-** Values of ‘Lattice constant (\( a \))’, ‘Unit cell volume (\( V \))’, ‘Average crystallite size (\( D \))’, ‘X-ray density (\( dx \))’, ‘Bulk density (\( d_{B} \))’, ‘Porosity (\( P \))’ of nickel ferrites nanoparticles before and after gamma radiation

| NiFe\(_{2}\)O\(_{4}\) | \( a \) (Å) | FWHM (θ) | \( V \) | \( D \) (nm) | \( dx \) | \( d_{B} \) | Porosity % |
|-----------------|----------|----------|------|---------|------|-------|---------|
| **Before radiation** | 8.336   | 0.3418 | 579.3 | 21.75  | 5.374 | 3.638 | 33.00   |
| **After radiation**  | 8.329   | 0.3021 | 577.8 | 19.69  | 5.389 | 3.617 | 33.14   |
4. Conclusions
Nanostructured nickel ferrite sample was successfully prepared by the sol-gel auto-combustion method. The prepared ferrite sample was irradiated by $^{60}$Co gamma-ray source. The XRD patterns confirmed the formation of cubic spinel ferrite with the Fd3m space group. The lattice parameter and crystallite size decreases after gamma irradiation.

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