Structural and Magnetic Properties of Nickel Ferrite Nanoparticles Prepared by Solution Combustion Method

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Abstract. Nickel ferrite nanoparticles with chemical formula NiFe₂O₄ were prepared by standard sol-gel auto ignition technique. Citric acid was used as chelating agent and mixed with solution of nickel nitrate and ferric nitrate in 3:1 proportion to balance the oxidizer to reducer ratio. The obtained particles were sintered at 550°C for 4h to remove the water content, impurity and to obtain better crystallinity. The phase purity and structural formation was confirmed through the analysis of X-ray diffraction technique. The results of X-ray diffraction analysis show the single phase cubic spinel nanocrystalline structure. The particles size calculated from FWHM of the strongest peak (311) is 22 nm in the nanometer dimension. The lattice constant, X-ray density and other structural parameters calculated from XRD data are in good agreement with the literature values. The magnetic measurements were taken at room temperature using hysteresis loop technique. Using M-H plot, the different magnetic parameters such as saturation magnetization, coercivity, remanence magnetization and magneton number were deduced.

1. Introduction
In the recent years, research into nanoscience and nanotechnologies has grown tremendously in a large panel of scientific and technological fields. The importance of nanotechnology and nanoscience has been increased due to their drastic effects in improving the properties of the materials and newer applications [1, 2]. It is the establish fact that, the materials when brought to nano dimension from micro dimensions exhibit important, unusual and superior properties. Due to advancement of the measuring techniques viz. atomic force microscopy, scanning electron microscopy etc. it has become possible and convenient to understand the structure and properties of nanomaterials [3].

Magnetic nanoparticles of ferrite have become prime importance in the field of medical science, environment, magnetic recording media, sensors, catalyst due o their very good magnetic properties
The magnetic nanoparticles exhibit superparamagnetic behavior and quantum effects which are useful in many applications. Spinel ferrites on account of their excellent magnetic and electrical properties are very much useful in various technological devices such as switching circuits, high density magnetic storage, microwave-based instruments, magnetic fluids, gas sensors, catalyst, water purification etc [7-9]. Generally, a spinel ferrite is described as (MFe$_2$O$_4$) where M is a divalent metal ion such as Co, Ni, Zn, Mg, Cu Mn etc. Spinell ferrite possesses two interstitial sites namely tetrahedral (A) and octahedral [B] site. The cations of different valence and nature can accommodate in these sites bringing wide variations in the structural and magnetic properties. On the basis of distribution of cations at tetrahedral A and octahedral B sites, the spinel ferrites are classified as normal ferrite, inverse ferrite and random ferrites [10].

It is known that the structural, electrical and magnetic properties of ferrites are highly sensitive to preparative conditions, amount and type of compositions, magnetic interactions and distribution of cations at tetrahedral (A) and octahedral [B] sites. The cation distribution at tetrahedral and octahedral sites is sensitive to ionic radii of Fe/Co and M ions, the type of bonding and the preparation method. Among the spinel ferrites, nickel ferrite is a soft magnetic material with moderate coercivity and saturation magnetization. It possesses inverse spinel structure [11]. In the literature, nickel ferrite with different substitution of divalent cations has been studied by number of researchers [12-14]. Although numbers of reports are available on the magnetic, electric and dielectric properties of nickel ferrites, work related to systematic study of structural and magnetic properties of nickel ferrites prepared by sol-gel auto combustion method is still lacking. It is important to note that the sol-gel auto-combustion method have definite advantages over other wet chemical methods. The method is simple, cost effective and produces homogeneous fine particles of nanometer dimensions. Therefore, in the present work, we have attempted to investigate the structural and magnetic properties of nickel ferrite prepared by sol-gel auto combustion method.

2. Experimental

Nanostructured nickel ferrite nanoparticles were prepared by the sol-gel auto-combustion method using citric acid as a fuel and complexing agent. The analytical reagents of ferric nitrate [Fe(NO$_3$)$_3$·9H$_2$O], nickel nitrate [Ni(NO$_3$)$_2$·6H$_2$O] and Citric acid (C$_6$H$_8$O$_7$) were used as a raw materials for synthesis. Depending upon the composition, the appropriate amount of metal nitrates and citric acid were dissolved in deionized water to obtain a mixed solution. During the mixing process, the solution was continuously stirred for 30 min at 100 °C using a magnetic agitator. The mixed solution was kept stirred at 100 °C for 3 h to transform into gel. The dried gel was burnt in an auto combustion manner to form a loose powder. The prepared powder was sintered at the temperature 550°C for 4 h for better crystallinity and purity. Structural and magnetic properties of prepared nanoparticles were evaluated by different characterization techniques. The powder X-ray diffraction pattern of the sample was recorded out using Bruker D8 Advance X-ray diffractometer with Cu-Kα radiation (λ = 0.15406 nm) to identify the phase and structure prepared samples. The pulsed field hysteresis loop tracer was used to obtain magnetic properties of prepared samples.

3. Results and discussion

3.1. Structural properties

X-ray diffraction (XRD) pattern prepared nickel ferrite nanoparticles is shown in Fig 1. The XRD patterns reveal the (220), (311), (222), (400), (422), (511), (440), (620) and (533) reflections belonging to cubic spinel structure. The analysis of XRD pattern proves that the samples possess single phase cubic spinel structure. From the XRD results, various structural parameters were calculated using standard relation as follows:

Using the values of Bragg’s angle 2θ and interplanar spacing d, the values of lattice constant of nickel ferrite nanoparticles was calculated using the following relation [15],

\[ a = d \sqrt{h^2 + k^2 + l^2} \text{ Å} \] ...1
The crystallite size (t) of the nickel ferrite nanoparticles was calculated from the most intense peak (311) present in the XRD patterns using the well known Scherrer’s formula [16],

\[ t = \frac{0.9\lambda}{\beta \cos \theta} \text{ nm} \quad \ldots 2 \]

The dislocation density (\( \delta \)) of nickel ferrite nanoparticles was calculated using the standard relation given by,

\[ \delta = \frac{1}{t^2} \text{ line/m}^2 \quad \ldots 3 \]

The X-ray density of nickel ferrite nanoparticles was calculated using the standard relation given by equation,

\[ d_X = \frac{Z \times M}{V \times N_A} \text{ gm/cm}^3 \quad \ldots 4 \]

The bulk density of the present samples was determined using Archimedes principle [17]. Toluene was used as a liquid medium to measure the bulk density. The percentage porosity of the nickel ferrite nanoparticles was calculated from the following relation;

\[ P = 1 - \frac{d_B}{d_X} \% \quad \ldots 5 \]

All the values of above parameters are tabulated in table 1.

![Fig. 1 X-ray diffraction pattern of NiFe\textsubscript{2}O\textsubscript{4} nanoparticles](image)

**Table 1** Values of different structural parameters of NiFe\textsubscript{2}O\textsubscript{4} nanoparticles

| Parameters         | NiFe\textsubscript{2}O\textsubscript{4} |
|--------------------|-----------------------------------------|
| Molecular weight   | 234.38                                  |
| \( a \) (Å)        | 8.341                                   |
| \( t \) (nm)       | 22                                      |
| \( V \) (Å\textsuperscript{3}) | 580.34                                  |
| \( d_X \) (gm/cm\textsuperscript{3}) | 5.365                                   |
| \( d_B \) (gm/cm\textsuperscript{3}) | 2.686                                   |
| \( P \) (%)        | 0.49                                    |
| \( \delta \) (lines/m\textsuperscript{2}) \times 10\textsuperscript{14} | 6.30                                    |
| \( \varepsilon \) (%) | 0.087                                   |
3.2. Magnetic properties

The magnetic behaviour of the nanoparticles is of more interest to the scientist and technologist. In the present study, the magnetic behaviour of NiFe₂O₄ nanoparticles was studied through pulsed field hysteresis loop technique. The magnetization versus applied field plots i.e. M-H plots for the present sample under investigation were recorded at room temperature and shown in Figure 2. The M-H plot exhibits typical hysteresis curve indicating the ferromagnetic behaviour of the present samples. This M-H plot is useful to know the saturation magnetization, coercivity and remanence magnetization of the present samples. The values of these magnetic parameters are given in Table 2.

![Fig. 2 M-H plot of NiFe₂O₄ nanoparticles](image)

Table 2 Values of saturation magnetization ($M_s$), remanence magnetization ($M_r$), coercivity ($H_C$), remanence ratio ($M_r/M_s$) and magneton number ($n_B$) for NiFe₂O₄ nanoparticles

| Parameters | $M_s$ (emu/gm) | $M_r$ (emu/gm) | $H_C$ (Oe) | $M_r/M_s$ | $n_B$ obs. ($\mu_B$) |
|------------|----------------|----------------|-------------|------------|---------------------|
| NiFe₂O₄   | 53.16          | 12.96          | 1.97        | 0.037      | 2.234               |

4. Conclusion

The experimental results on structural and magnetic properties have leaded us to draw the following conclusions. The spinel structured nickel ferrite nanoparticles were successfully synthesized by sol-gel auto combustion method assisted by citric acid. Pure phase formation of nickel ferrite with no impurities was observed from X-ray diffraction pattern. The average crystallite size of the prepared sample was found to be 22 nm. The values of saturation magnetization ($M_s$) and remanent magnetization ($M_r$) were found to be 53.16 emu/gm, and 12.96 emu/gm respectively.

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