X-ray Diffraction, Infrared and Magnetic Studies of NiFe$_2$O$_4$ Nanoparticles

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Abstract: Spinel ferrite nanoparticles plays important role in many applications due to their excellent structural, magnetic and electrical properties. Among the spinel ferrite nickel ferrite is a prominent candidate for various applications and is the subject of interest to many researchers. Therefore, attempt is made to synthesize NiFe$_2$O$_4$ nanoparticles and investigate its structural, infrared and magnetic properties for high frequency applications. NiFe$_2$O$_4$ nanoparticles were synthesized by sol-gel auto-ignition technique. Efforts are made to obtain nanopowder of high quality by taking extra care and some modification in synthesis procedure. In order to examine the phase purity and nanocrystalline nature, prepared nanopowder was subjected to X-ray diffraction studies. A close examination of XRD pattern revealed the presence of the reflections belonging only to cubic spinel structure. No extra peak was seen in the XRD pattern. The crystallite size was estimated using Debye-Scherrer’s formula and found to be 22nm. FT-IR spectrum of NiFe$_2$O$_4$ shows the two prominent absorption bands near the frequency range 400 cm$^{-1}$ and 600 cm$^{-1}$. The important magnetic property was studied by M–H plot which was recorded using pulse field hysteresis loop tracer at room temperature. Using the M–H plots the values of saturation magnetization, coercivity, and remanence magnetization are recorded as 37.61 emu/g, 177 Oe, and 11.71 emu/g respectively. The obtained data for structural and magnetic properties will be useful for high frequency applications.

1. Introduction

Nanocrystalline magnetic materials play a very important role in many technological applications due to their unusual and interesting properties such as nanoscale dimensions, better reactivity at atomic scale, higher surface to volume ratio, greater homogeneity, high purity, better yield, separable from solution mixture, less toxic, superparamagnetic, act as catalyst, etc [1-4]. The use of nanoscience and
nanotechnology in the development of many areas viz medicine, industry, agriculture, environment, etc. has been tremendously increased. Many researchers have focused their research on synthesis and characterization of various nanomaterials specially, the magnetic nanomaterials. In view of this, magnetic nanomaterials like spinel ferrite has gained a lot of importance amongst scientist and technologists as they exhibit superior, excellent magnetic and electrical properties compared to their bulk counterparts. Spinel ferrite chemically recognized by the formula $\text{AB}_2\text{O}_4$, where A is divalent metal ion like Cu, Zn, Co, Mg, Mn, Ni etc and B is iron, are important class of magnetic materials in the family of ferrite. They exhibit high electrical resistivity, high saturation magnetization, high Curie temperature, low eddy current and dielectric loss which can be modified so as to suit the required application. Nickel ferrite (NiFe$_2$O$_4$) is one such magnetic material in the group of spinel ferrite. It has cubic spinel structure with space group Fd-3m$^h$. On the basis of crystal structure, it belongs to cubic inverse structure i.e. Ni$^{2+}$ ions distributed to octahedral [B] sites while Fe$^{3+}$ ions equally distributed over both tetrahedral (A) and octahedral [B] sites. However, distribution of Ni$^{2+}$ ions and Fe$^{3+}$ ions depends on method of preparation, role of synthesis parameters, the nature of dopant. There are various synthesis methods available for the preparation of spinel ferrite in nano-crystalline form. Wet chemical methods include, sol-gel, sol-gel auto combustion, co-precipitation, hydrothermal, etc are promising as they yield ultrafine powder of nano-scale dimension, more chemically stable, highly homogeneous nanopowder. Usually the sol-gel auto combustion method is used by many researchers for the reason that the method is simple, less expensive, requires lower temperature, and requires less equipment and raw materials. This method can be modified by choosing a proper fuel, taking extra care in the formation of sol-gel and combustion process, stirring temperature, stirring speed, stirring time, etc. By modifying the sol-gel auto ignition method we may obtain high quality of nanopowder. Nickel ferrite has been studied for the electrical and magnetic properties by many researchers. Though, nickel ferrite was studied by many researchers [10] still interest in it has motivated us due to its excellent electrical, magnetic and other properties which can be varied by many ways. With this background in the present report results on the synthesis, structure, infrared and magnetic properties investigated for high frequency applications.

2. Experimental

Nickel nitrate (Ni(NO$_3$)$_2$·6H$_2$O), ferric nitrate (Fe(NO$_3$)$_3$·9H$_2$O) as oxidants and glycine as a reducing agent were used to synthesize NiFe$_2$O$_4$ nanoparticles. All the reagents were of analytical grade (AR) and used as received without further purification. The metal nitrates to fuel (glycine) ratio was chosen as 1:4.4. The detailed procedure followed here was as explained in our earlier articles [5, 6]. The as-prepared loose powder was ground followed by annealing at 800°C for 5 h in muffle furnace. The annealed powder was used for further characterizations.

The X-ray diffractometer PanAnalytical Xpert-Pro with Cu-ka X-ray radiation ($\lambda=1.5409\text{Å}$) was employed to study the phase purity and structure of the prepared nickel ferrite nanoparticles. FT-IR spectrometer FTIR-6100 type-A model used to know the characteristic features of ferrite skeleton. Moreover, magnetic measurements were carried out by pulse field hysteresis loop tracer at room temperature by applying magnetic field $\pm5\text{kOe}$.

3. Results and discussion

3.1 X-ray diffraction

X-ray diffraction is a unique technique to understand the phase purity and structure of the material. In the present study X-ray diffraction of pure nickel ferrite nanoparticles was recorded in the 20 range of 20$^0$ to 80$^0$ at room temperature. Figure 1 shows XRD pattern of nickel ferrite nanoparticles in which reflections (220), (311), (222), (400), (422), (511), (440) occurred which are allowed reflection of cubic spinel structure. All the reflections were indexed using Bragg’s law. These reflections are intense
and show slight broadening which may be due to nanocrystalline nature of the prepared nickel ferrite. All the reflections present in the XRD pattern belongs to fcc type cubic spinel structure. No extra peak other than expected reflections occurred in the XRD pattern. The analysis of XRD pattern proved that the prepared nickel ferrite possesses single phase cubic spinel structure. XRD pattern matches well with JCPDS card 74-2081. The XRD data was used to obtain various structural parameters. The crystallite size (t) was calculated using Scherrer’s formula given by

\[ t = \frac{0.9\lambda}{\beta \cos \theta} \]  

Where, \( \lambda \) is wavelength, \( \beta \) is FWHM and \( \theta \) is diffraction angle. The lattice constant (a) was determined by using the standard relation given by

\[ a = \frac{d}{\sqrt{N}} \] 

Where, ‘a’ is lattice parameter is inter-planer distance and ‘N’ is Miller indices. The other structural parameters like X-ray density (dx) and unit cell volume (V) were also determined by using standard relations. The values of all the structural parameters are listed in table 1. It can be observed from the values of the structural parameters that they match well with the reported values [7, 8].

![Fig. 1: XRD pattern of NiFe2O4 nanoparticles](image)

Table 1: Structural parameters of NiFe2O4 nanoparticles

| Composition | Lattice constant (Å) | Volume (Å³) | X-ray density (g/cm³) | Crystallite size (nm) |
|-------------|---------------------|-------------|-----------------------|----------------------|
| NiFe2O4     | 8.334               | 578.8       | 5.37                  | 22                   |

3.2 FT-IR

The prepared nickel ferrite was also characterized by FT-IR spectroscopy technique. Figure 2 represents FT-IR spectra of nickel ferrite which shows one absorption band near 600 cm⁻¹. Usually, IR spectra of spinel ferrite show two absorption bands, one near 400 cm⁻¹ and another at 600 cm⁻¹. In the present study, we observe band near 579 cm⁻¹ belongs to intrinsic vibrations of octahedral and another
band at 398 cm\(^{-1}\) tetrahedral vibrations. FT-IR results proved the characteristic features of ferrite. Our results on FT-IR analysis are in good agreement with those reported in the literature [9].

![FT-IR spectra of NiFe\(_2\)O\(_4\) nanoparticles](image)

**Fig. 2:** FT-IR spectra of NiFe\(_2\)O\(_4\) nanoparticles

### 3.3 Magnetization measurements

The magnetic properties of spinel ferrite are of prime importance for the scientist and technologist. In the present study, the magnetic properties of nickel ferrite are studied by means of pulse field hysteresis technique. Figure 3 represents typical magnetization versus applied field plot recorded at room temperature. The M-H plot exhibits a typical ferrimagnetic nature of hysteresis curve. Using this M-H plot, values of saturation magnetization, remanence magnetization are obtained and presented in table 2. The saturation magnetization of the present nickel ferrite sample resembles with one reported value [10]. However, coercivity of the prepared sample is very low which indicate that superb paramagnetic behavior of present sample. Using the values of saturation magnetization and molecular weight magneton number was calculated and its value is also given in table 2. The obtained results on magnetization are very much useful for the application in high frequency devices.

![M-H hysteresis loop of NiFe\(_2\)O\(_4\) nanoparticles](image)

**Fig. 3:** M-H hysteresis loop of NiFe\(_2\)O\(_4\) nanoparticles
Table 2: The magnetic parameters as saturation magnetization (Ms), remanence magnetization (Mr), coercivity (Hc), remanence ratio (R) and magneton number ($\eta_B$) 

| Composition | Ms (emu/gm) | Mr (emu/gm) | Hc (Oe) | R= Mr/Ms | $\eta_B$ (µB) |
|-------------|-------------|-------------|---------|----------|---------------|
| NiFe$_2$O$_4$ | 37.61 | 11.71 | 177 | 0.31 | 1.57 |

4. Conclusion
The experimental results on structural, infrared and magnetization studies lead to the following conclusions i) sol-gel auto combustion method was successfully employed for the preparation of nickel ferrite nanoparticles. Careful preparation and slight modification in the synthesis procedure leads to evolution of better structure and thereby enhanced structural and magnetic properties ii) The nickel ferrite exhibit single phase cubic spinel structure which confirmed from obtained crystallite size which is 22 nm. iii) The magnetic properties obtained in the present study are useful for high frequency applications of nickel ferrite nanoparticles.

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