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Synthesis and characterization of nickel ferrite nanoparticles by sol - gel auto combustion method

R.Kesavamoorthi, A.N.Vigneshwaran, Vijayalakshmi Sanyal and C.Ramachandra Raja*
Department of Physics, Government Arts College (Autonomous), Kumbakonam - 612 001.
***Corresponding author: E-Mail: crraja_phy@yahoo.com, crrajaphy@gmail.com**

ABSTRACT

Spinel structure Nickel Ferrite ($NiFe_2O_4$) nanoparticles have been successfully synthesized by sol-gel auto combustion method. The synthesized sample was annealed at 600°C for 2 hrs and characterized by X-ray diffraction (XRD) analysis, Scanning Electron Microscopy (SEM) analysis, Energy Dispersive X-ray spectrum (EDAX) analysis and FT-IR Spectroscopy analysis. The XRD pattern confirmed the formation of cubic spinel structure of nickel ferrite. The lattice parameters were calculated as 8.322 Å and 8.327 Å for prepared and annealed samples respectively. The average crystallite size was found from XRD broadening peak of maximum intensity (311) plane using Debye Scherer's formula and the average crystallite sizes of $NiFe_2O_4$ was found to be 21 and 60 nm for prepared and annealed samples respectively. The SEM analysis revealed that the particles are spherical shaped. The EDAX spectrum confirmed homogeneous mixing of Ni, Fe, and O atoms. The presence of two main metal oxide bands in FT-IR spectrum around 422 cm^{-1} and 585 cm^{-1} confirmed the octahedral and tetrahedral sites of metal oxide bands respectively.

KEY WORDS: Nickel Ferrite, XRD, SEM, EDAX, FT-IR Spectroscopy.

1. INTRODUCTION

Nano crystalline spinel ferrites with general formula MFe_2O_4 (M = Ni, Co, Cu, Zn, Mn, etc.) have face centered-cubic structure. These materials have a cubical spinel structure where oxygen forms a face centered cubic close packing and M^{2+} occupy either tetrahedral or octahedral interstitial sites. Ferrites with uniform particle size and narrow size distribution are desirable for variety of applications like gas sensing applications, high density data storage, ferro-fluid technology, magneto caloric refrigeration, magnetic guided drug delivery and electronic devices for high frequency applications. Nickel ferrite ($NiFe_2O_4$) is one of the most important spinel ferrite which finds applications in the fabrication of soft magnets and low loss materials at high frequencies. It is known that the fascinating electrical and magnetic properties of ferrites depend upon the nature of the ions, charges and their distribution among tetrahedral (A) and octahedral (B) sites. Nickel ferrite is a typical soft ferromagnetic material crystallizing in a completely inverse and cubic spinel structure with all nickel ions located in the B-sites and ferric ions occupying both A-sites and B-sites. The compound, thus can be represented by the formula (Fe^{3+}) A $[Ni^{2+}Fe^{3+}]$ B O_4^{2-}. The nano crystalline ferrite material is being synthesized by various techniques such as Co-precipitation method, hydrothermal technique, Ball milling technique, Micro emulsion process, Sol-gel auto combustion method and so on. In the present work, we report the characterization of single phase cubic spinel ferrite using a sol-gel auto combustion method which is most suitable for the preparation in good stoichiometric control with narrow particle size distribution due to molecular level mixing, short processing time, lower temperatures and low cost.

2. MATERIALS AND METHODS

2.1. Synthesis of nickel ferrite Nanoparticles: The nickel ferrite nanoparticles were synthesized at room temperature by sol-gel auto combustion method. The starting materials, nickel nitrate hexahydrate [Ni (NO_3)].$6H_2O$, Iron nitrate nonahydrate [Fe $(NO_3)_2$].$9H_2O$ and citric acid ($C_6H_8O_7.H_2O$) were purchased from Merck company. The starting material of metal nitrates and citric acid are taken in the molar ratio 1:2. All metal nitrates and citric acid were dissolved in stiochiometric amount of de-ionized water and stirred continuously using magnetic stirrer attached with hot plate. During constant stirring, ammonia (NH_3) solution was added drop wise to reach 7. The mixed nitrate solution was heated at 60°C with constant stirring for 6 hrs. Then the solution was kept in the hot air-oven at the temperature of 120°C for 12 hrs. During the process the mixed nitrate solution gets transform sol into gel, and the gel gets completely dried in 12 hrs. The dried sample was ground using mortar and pestle, and then annealed in muffle furnace at 600°C for 2 hrs and cooled slowly to room temperature.

2.2. Characterization studies: The synthesized samples were characterized for crystal phase identification by Powder X-ray diffraction (XRD) spectroscopy performed with XPERT-PRO PAN Analytical X-ray diffractometer and it is operated at 45kV and 30 mA (X-ray source: Cu Kα, wavelength 1.54439Å). The samples were scanned in continuous normal scan mode from 10 to 80°. The XRD results were compared with the Joint Committee Powder Diffraction Standards (JCPDS Card No.10-0325) data for the phase identification. The lattice parameters (a) of as-prepared and annealed nickel ferrite samples were calculated by considering cubic crystal structure, from the plane (311) main peak of spinel structure using Braggs Equation is $a=d\sqrt{h2+k2+l2}$. Where, "d" is inter planar distance, "h, k and l" are the miller indices and "a" is the lattice parameter. The average crystallite size of synthesized sample were calculated from maximum intensity peaks of (311) plane using Debey Scherer's formula $D = k\lambda/ \beta \cos \theta$, where

D is the mean crystalline size (nm), k is the Scherer's constant (0.89), λ is the wavelength of X-ray beam used (λ = 1.54439 Å), β is the full width at half maximum (FWHM) diffraction and θ is the Bragg's angle. The morphology was determined by scanning electron microscopy (SEM) using JEOL/JSM-5610 NE Instrument model, and also the chemical composition of samples was estimated by EDAX spectroscopy technique. Fourier transform infrared (FT-IR) spectroscopy are recorded using KBr pellets on a BRUKER® Tensor 27 FT-IR spectrometer, in the range of 4000 - 400 cm^{-1}.

3. RESULTS AND DISCUSSION

3.1. XRD Analysis of Nickel Ferrite nanoparticles: The powder XRD pattern of as prepared and annealed sample are shown in figure 1(a) and 1(b) respectively. Figure 1 (a) shows that as prepared sample with broad peaks are observed to spinel phase of nickel ferrite and small amount of α-Fe_2O_3 (hematite) phase also observed at low intensity. The XRD pattern of annealed sample exhibits typical reflections of (220), (311), (222), (400), (511) and (440) planes that are indications of the presence of cubic spinel structure. These diffraction lines provide clear evidence on the formation of nickel ferrite. The entire diffraction peaks match well with the reported values and (JCPDS file No: 10-0325). No secondary phase was detected in XRD pattern of annealed sample, ensuring the phase purity of the final product. Annealed sample have more intense peaks than as prepared sample. It indicates more crystalline form of nickel ferrite. The effect of increasing temperature on improvement of the crystalline properties of nickel ferrite and also the conversion of some nickel and iron oxides to produce nickel ferrite crystallites. It is clear that the crystallization of nickel ferrite completed at increasing temperature. The lattice parameter was calculated as a = 8.322 Å and 8.327 Å and the average crystallite sizes are found to be 21 to 60 nm from the maximum intensity peak (311) using Debye Scherer formula. The lattice parameters and average crystallite sizes are shown in table 1.

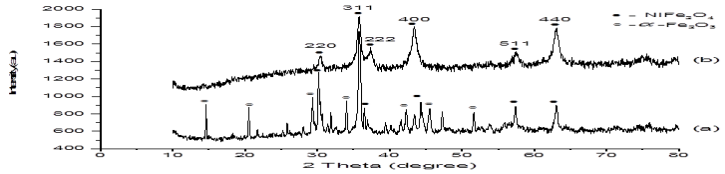

Figure.1.XRD pattern of $NiFe_2O_4$ (a) as prepared sample, (b) annealed at 600°C

Table 1: Structural parameters of Ni ferrite sample

Sample	2 Theta (deg)	d-spacing (Å)	Crystallite size (nm)	Lattice constant (Å)
As prepared	35.76	2.510	21	8.322
Annealed	35.78	2.509	60	8.327

3.2. Scanning Electron Microscopy (SEM) Analysis: The Scanning Electron Microscopy images of as prepared and annealed samples are shown in figure.2 (a) and (b) respectively. These figures reveal the remarkable changes in the microstructures. The micrographs of as prepared sample, particles are combined to form clusters at room temperature. Figure.2 (b) shows SEM images of annealed sample are separately distributed. The powder has an aggregation of particles are found less than 100 nm. The particles were observed as uniform grains with spherical shaped confirming the crystalline structure of nickel ferrite which is also detected by the XRD profile. Similar results were reported by Sukhvir Singh (2011) and Sharma (2006). The temperature of annealing must be well controlled for an optimal particle size distribution and to obtain the regular structure. The observed results shows that distribution of the particles sizes are varied at room temperature and uniform for annealed samples.

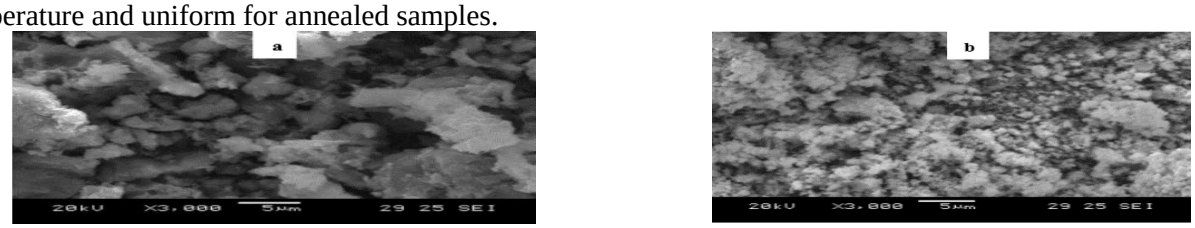

Figure.2.SEM image of $NiFe_2O_4$ (a) as prepared sample, (b) Annealed at 600°C

3.3. Energy Dispersive X-ray Spectrum (EDAX) analysis: The purity and chemical composition of synthesized samples was checked by EDAX spectrum. The stiochiometry compositions of EDAX spectrum are shown in figure 3. The EDAX pattern confirms homogeneous mixing of Ni, Fe, and O atoms in synthesized sample. It is clear that there is no chemical or any loss of ingredients after final stage of annealing. It reveals that the compositional stiochiometry of the nickel ferrite were unaffected. The molar proportions of the elements are in good agreement with that of expected values. The observed composition is almost equal to that of the samples produced by stiochiometric calculations while taking oxygen as balanced. It is quite clear that the experimentally observed percentages of elements are in a good agreement with those theoretically calculated.

No impurity peaks were observed except for extra gold (Au) peak. The gold peak appears due to the thin coating on the sample surface to make it conducting, which is required to record the SEM picture. The EDAX spectrum reveals that there is no contamination in the samples. Similar results were reported by Huo and Wei (2009).

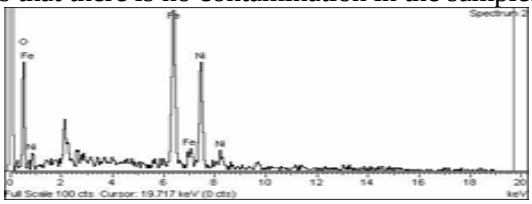

Fig.3.EDAX spectrum $NiFe_2O_4$ sample

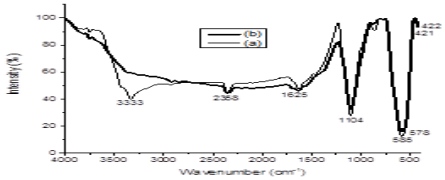

Fig.4.FT-IR spectrum of (a) as prepared and (b) annealed sample

3.4. Fourier Transform Infrared (FT-IR) Spectroscopy Analysis: The FT-IR spectroscopy provides valuable information regarding the nature of the functional groups present in the nickel ferrite samples and shown in figure 4 (a) and (b) of as prepared and annealed samples respectively. The spectrum show prominent bands near 3333 and 1625 cm^{-1}, which are attributed to the stretching modes and H-O-H bending vibrations of the free or absorbed water. The band near 1104 cm^{-1} is due to the antisymmetric NO-stretching vibrations arising from the nitrate group which is present as residue in the samples. This band is very weak in the spectra of sol–gel auto combustion derived sample, indicating the purity of nickel ferrite nanoparticles synthesized by this method. From the spectrum two main broad metal-oxygen bands are seen in the infrared spectrum of all spinels, especially ferrites. The higher one generally observed in the range 585-578 cm^{-1}, is caused by the stretching vibrations of the tetrahedral metal-oxygen bond. The lowest band usually observed in the range 422-421 cm^{-1}, is caused by the metal-oxygen vibrations in the octahedral sites. The vibrational frequencies of IR bands and of samples prepared by sol-gel auto combustion method, which are in perfect agreement with reported values Montemayor (2007) and Priyadharsini (2009).

4. CONCLUSION

The nickel ferrite nanoparticles have been successfully synthesized at room temperature by sol-gel auto combustion method and synthesized samples were characterized. The powder XRD pattern shows the structure of $NiFe_2O_4$ have been cubic spinel structure. The lattice parameters were calculated as 8.322 Å and 8.327 Å for prepared and annealed samples respectively. The average crystallite sizes was found to be 21 and 60 nm from the XRD broadening peak using Debye Scherer's formula. The SEM micrographs revealed that the particles are spherical shaped. The EDAX spectrum confirmed homogeneous mixing of Ni, Fe, and O atoms. The presence of two main metal oxide bands in FT-IR spectrum around 585 cm^{-1} and 422 cm^{-1} confirmed the tetrahedral and octahedral sites of metal oxide bands respectively.

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