

ChemTech

International Journal of ChemTech Research

CODEN (USA): IJCRGG ISSN: 0974-4290 Vol.8, No.5, pp 113-116, **2015**

National conference on Nanomaterials for Environmental [NCNER-2015] 19th & 20th of March 2015

Synthesis and Characterization of Nickel ferrites nanoparticles

R.Suresh¹*, P. Moganavally¹, M.Deepa²

¹Research and Development Centre, Bharathiyar University, Coimbatore, India ²Department of Chemistry, Muthurangam Govt. Arts College, Vellore, India

Abstract: Ultrafine powders of nickel ferrites were synthesized at low temperature through the chemical co- precipitation method. The synthesized powders were characterized using X-ray Diffraction (XRD) for crystallite size, X-ray density and lattice parameter calculation. It reveals the presence of cubic spinel structure of ferrites with crystallite size of approximately 23nm. Scanning Electron micrograph (SEM) shows uniform distribution of ferrite particles with some agglomeration and Energy Dispersive X-ray analysis (EDAX) was used to determine the compositional mass ratio. Magnetic measurements of the samples were carried out by means of vibrating sample magnetometer (VSM) and these studies reveal that the formed nickel ferrite exhibits ferromagnetic behavior.

Keywords: Ferrites, Co-precipitation, Nanoparticles, Lattice parameter, Magnetic properties.

Introduction

Ferrites have gained importance because they possess the combined properties of magnetic materials and insulators. The soft ferrites with their tailerable properties have some stringent requirements^{1, 2}. The soft ferrites have been adopted for excellent applications in microelectronics such as wave absorber, magnetic core memory devices, etc. Fine grained powder ferrites have many microwave applications (Multi layer chip inductor) because of its sharp square hysteresis loop behavior, high stability compared to conventional methods. The different compositions of the ferrites have been synthesized with different substitutions for tuning their electrical, magnetic properties and super magnetism features³⁻⁶. The magnetic properties of ferrites are attracting a great deal of attention because of their versatile properties and applications in electronics, magnetic storage devices, and ferrite wave absorber and humidity sensors^{7,8}.

Ultra- fine powder of ferrites can be prepared through various wet chemical methods like coprecipitation, sol-gel method, combustion method, hydrothermal method, gel-assistant hydrothermal route, thermolysis, micro emulsion method⁹⁻¹². We synthesized the nickel ferrite nanoparticles by co-precipitation method through low process temperature. This method has the advantages of simple preparation, cost-effective and gentle chemistry route resulting in ultra fine and homogenous powder. Many researchers synthesized the nickel ferrite nanoparticles by different methods and at high temperatures only. But we synthesized the nickel ferrite nanoparticles at low temperature only and it exhibits amorphous nature.

Experimental

All the reagents are of analytical grade and are used as received without further purification. Ferric chloride [FeCl₃.6H₂O], Nickel chloride [NiCl₂.6H₂O] and sodium hydroxide [NaOH].

The Nickel ferrite nanoparticles were prepared by co-precipitation method. An appropriate amount of metal chlorides were dissolved to get an aqueous solution. Then add 8M sodium hydroxide and the formed precipitates are heated to 80 °C and also maintain the pH around 11.5 for 40 minutes. The formed precipitate washed with warm water and followed by acetone for many times. Then precipitate is collected, dried and kept in oven for 3 hours at 100 °C to get nickel ferrite nanoparticles.

The average crystallite size of the ferrite nanoparticles were analyzed using powder X-ray diffractometer (XRD) using CuK α (λ =1.5418Å) radiation. The scanning was done in the 2 θ range from 20 ° to 80 °. The morphology, structure and elemental composition of the sample were characterized by Scanning electron microscopy (SEM) and Energy dispersive X-ray spectra (EDS). Room temperature magnetic measurements were carried out using vibrating sample magnetometer (VSM) and various parameters were evaluated.

Results and Discussion

The precipitated fine particles were characterized by XRD as shown in Fig.1. The structure and their crystallite size were evaluated. The crystallite size of the nano-crystalline samples was measured using Debye-Scherer formula¹³,

 $D_{XRD} = 0.98\lambda/\beta \cos \theta$

Where λ is the wavelength of X-ray used in Å, β is the full width at half-maximum (FWHM) in radians in the 2 θ scale, θ is the Bragg angle, D_{XRD} is the crystallite size in nm.



Fig.1. XRD pattern of the NiFe₂O₄ nanoparticles

The lattice constant (a) was computed using the'd' value and with their respective (h k l) parameters. Analysis of the diffraction pattern confirms the formation of cubic spinel structure. The peaks indexed to (220), (311), (400), (422), (511) and (440) planes of a cubic unit cell, correspond to cubic spinel structure. The lattice constant calculated using the equation given below and tabulated in Table 1

 $a=d(h^2+k^2+l^2)^{1/2}$

Table 1 XRD parameters of NiFe₂O₄ Nanoparticles

Chemical formulae	NiFe ₂ O ₄
d-spacing	2.50742
Lattice constant 'a' in Å	8.3162
Average particle size 'D' in nm	23.1055
Molecular Mass	235
X-ray Density $(x10^3 \text{ kg/m}^3)$	5.427

The X-ray density was calculated using the molecular weight and the lattice constant. The XRD density was calculated by formula ¹⁴

XRD density = $8M/Na^3$

Where M is molecular weight of the sample and N is the Avogadro number and 'a' is the lattice parameter.

Fig.2 shows scanning electron micrograph of the sample nickel ferrite nanoparticles. The uniform nature of the ferrite particle is shown with some agglomeration, since it is prepared by low temperature coprecipitation technique. The EDX (Energy dispersive X-ray spectroscopy analysis) was done in order to determine the chemical composition and the results of EDX analysis evaluates the atomic weight percentage of various cations in the investigated samples are found to be approximately correct, which corresponds to a composition ratio and these ratios are expected by the preparation method.



Fig.2 SEM and EDX images of NiFe₂O₄ NPs

Magnetic properties of NiFe₂O₄ were investigated by Vibrating Sample Magnetometer (VSM) at room temperature and it exhibits the ferromagnetic behavior. The various parameters like Saturation Magnetization (Ms), Coercivity (Hc) were tabulated in Table-2 and Magnetic hysteresis loop is shown in Fig.3.



Fig. 3 Magnetic hysteresis loop of Nickel ferrite Nanoparticles

Chemical Formulae	Coercivity H _c (Gauss)	Magnetization M _s (emu/gm)
NiFe ₂ O ₄	53.189	12.125

Table 2 Magnetic Parameters of NiFe₂O₄ NPs

Conclusion

 $NiFe_2O_4$ ferrites have been successfully synthesized by co-precipitation method at low temperature and successful formation of cubic spinel structure confirmed by X-ray diffraction analysis. The lattice parameter and x-ray density were found using the XRD parameters. SEM micrographs and EDX analysis confirms the chemical compositions, which support our observations on the structure of the ferrite. The Magnetic hysteresis loop of nickel ferrites confirms the formation of ferromagnetic behavior. Since this method is so easy to synthesize and highly cost effective, it has found many applications.

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