Synthesis and Characterization of Nano Crystalline Nickel Ferrite

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Abstract— $NiFe_2O_4$ nanoparticles have been synthesized by microwave route. The sample was characterized by Fourier Transform infrared (FTIR) spectroscopy; FTIR spectra show the stretching and bending vibrations of Ni–O and Fe–O bonds in nickel ferrite. Structures and morphology of sample was studied by X-Ray Diffraction (XRD) and Transmission Electron Microscopy (TEM) analyses confirmed the formation of single-phase nickel ferrite nanoparticles of average size 31.42 nm and 25.39 nm respectively. The magnetic properties of the sample were measured at room temperature using Vibrating Sample Magnetometer (VSM) in the field range ±15000G. Hysteresis loop obtained room temperature for $NiFe_2O_4$ nanoparticles indicates that the nanoparticles are ferromagnetic in nature.

Key words: Nickel Ferrite, Synthesis, X-Ray Diffraction, TEM, Magnetic Property, Saturation Magnetization

I. INTRODUCTION

During the last three decades, nanosized materials have been extensively studied by many researchers worldwide because of their unique physical properties, such as electrical conductivity, refractive index and magnetic properties, and superior mechanical properties such as hardness of nanomaterials and chemical properties compared with their counterpart bulk materials [1]. As the research community is gaining the knowledge to understand these unique behaviors and various applications of nanostructured materials, a new field of technology has been emerged known as 'nanotechnology' [2, 3].

Among various nanomaterials, mainly spinel ferrite (MFe_2O_4, M = Ni, Co, Mn, Zn, etc.) nanoparticles have become immensely popular magnetic materials for a wide variety of applications such as electronic ignition systems, generators, vending machines, medical implants, site-specific drug delivery, wrist watches, inductor core, transformer circuits, magnetic sensors and recording equipment, telecommunications, magnetic fluids, magnetic resonance imaging, photo magnetic materials, microwave absorbers and other high-frequency applications [4-13]. A distinct merit of ferrites over other magnetic materials such as iron and metallic alloys is their high electrical resistivity due to which ferrites perform much better at high frequencies. High electrical resistivity prevents induction of eddy currents and the resultant loss of energy. High permeability and temperature stability are other advantages. In addition, ferrites are most economic than most of other magnetic materials and their magnetic and mechanical properties can be tuned as per the requirement of application [14].

II. EXPERIMENTAL

A. Materials and Methods

All the chemicals were of AR grade and were used as received. Double-distilled water was used for preparation of the required solutions.

B. Synthesis of Nickel Ferrite ($NiFe_2O_4$) Nanoparticles (Microwave Route)

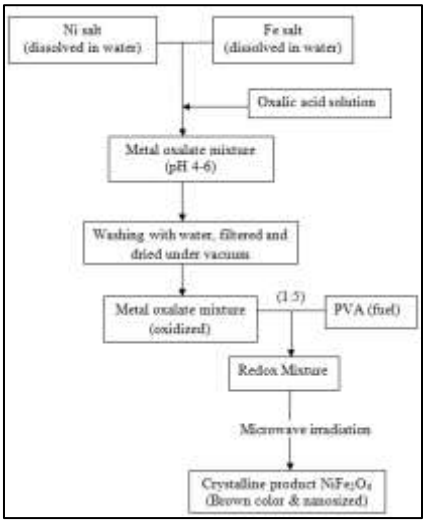

Fig. 1: Flow chart: Synthesis of $NiFe_2O_4$ Nanoparticles

$NiFe_2O_4$ nanoparticles are prepared by, known quantity of nickel salt and iron salt were dissolved in minimum amount of water and similarly oxalic acid was dissolved in water in a separate container. These two solutions were mixed well to form a metal oxalate a precipitate. The precipitate was filtered through sintered glass and washed with double distilled water. Finally, washed with dry acetone and dried under vacuum.

Metal oxalate mixture is grounded well with polyvinyl alcohol in 1:5 in a pestle and mortar. The reaction was transferred into crucible and initially it was burnt in an electrical oven for complete combustion of the fumes. The resultant residue was transferred into microwave oven for complete combustion process. The reaction was completed within 10 minutes at high power level to form nanosized $NiFe_2O_4$ particles. On cooling to room temperature treating with acetone separates carbon impurity. The various steps involved in chemical synthesis of nanosized $NiFe_2O_4$ particles is shown in flow chart (figure 1).

III. CHARACTERIZATION TECHNIQUES

The IR spectra of all the samples are recorded on Perkin Elmer (model 783) IR spectrometer in KBr medium at room temperature. For recording IR spectra, powders are mixed with KBr in the ratio 1:25 by weight to ensure uniform dispersion in KBr pellets. The mixed powders are pressed in a cylindrical die to obtain clean discs of approximately 1 mm thickness. The X-ray diffraction patterns of the samples in this present study are obtained on Bruker AXS D8 Advance, X-ray diffractometer using CuK_α radiation (λ = 1.5406 Å). The diffractograms were recorded in terms of 2θ in the range $20^0 – 120^0$ with a scanning rate of 2^0 per minute. The powder morphology of $NiFe_2O_4$ nanoparticles sintered in the form of pellets (to measure grain size) is investigated using PHILIPS CM200 Transmission Electron Microscope (TEM). The magnetization measurements of the samples were carried out using measured using vibrating sample magnetometer (LakeShore, Model-7410) at room temperature with a maximum field of -15000 G to +15000 G.

IV. RESULTS AND DISCUSSION

A. FTIR- Analysis

The IR spectrum of pure $NiFe_2O_4$ nanoparticles is shown in fig. 2. The important peaks observed in $NiFe_2O_4$ are 3416, 1440, 1113, 553 and 461 cm^{-1}. From the figure, the characteristic absorption peaks are found to be at 553 cm^{-1} corresponds to intrinsic stretching vibration of the metal at the tetrahedral site, 461 cm^{-1} corresponds to intrinsic stretching vibration at the octahedral site [15]. The peak at 3416 and 1440 cm^{-1} corresponds to water of hydration [16].

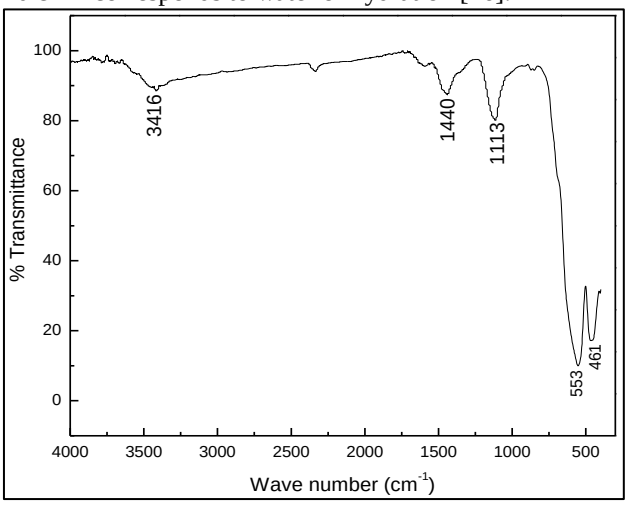

Fig. 2: FTIR spectra of NiFe2O4 nanoparticles

B. XRD Analysis

Figure 3 shows the XRD pattern of $NiFe_2O_4$ nanoparticles. The prominent peaks at 2θ= 30.84º, 35.48º, 43.16º, 53.95º, 57.24º, 62.74º, 75.31º and 79.43º corresponds to (2 2 0), (3 1 1), (4 0 0), (4 2 2), (5 1 1), (4 4 0), (6 2 2) and (4 4 4) planes, matching with the JCPDS pattern of $NiFe_2O_4$ nanoparticles (JCPDS-10-325). The crystalline size of the nanoparticles can be estimated by XRD using Scherrer formula [17]

$$D = \frac{k\,\lambda}{\beta_{1/2}\cos\theta}$$

Where, D is the crystal size, $\beta_{1/2}$ is the FWHM of stronger peak, λ is the wave length of the X-ray, θ is the peak position and k is the constant. The average crystallite size calculated from peak width is about 31.42 nm, which is in accordance with the TEM results discussed in next section.

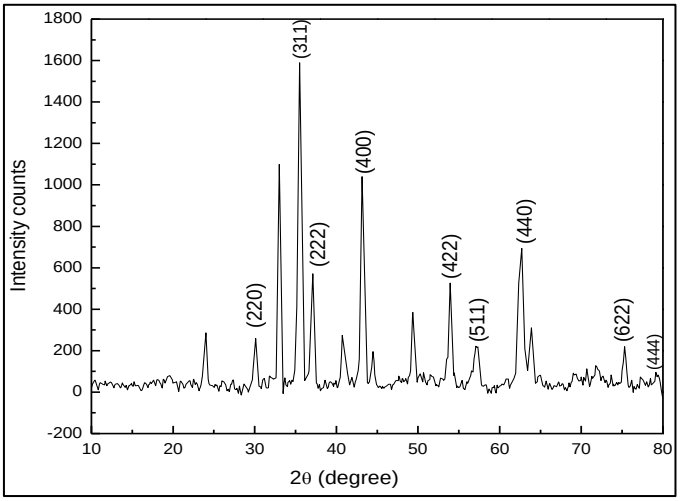

Fig. 3: XRD Pattern of $NiFe_2O_4$ Nanoparticles

C. TEM Analysis

The morphological characteristics of the obtained $NiFe_2O_4$ were investigated by TEM analysis. TEM was employed to visualize the size and shape and to confirm the nanocrystalline nature of the as synthesized nickel ferrite. Figure 4 shows the typical bright field TEM image of the as synthesized nickel ferrite nanoparticles. The TEM image clearly shows dense assembly of uniformly sized ferrite nanoparticles. The whole surface of the grid was covered with ferrite nanoparticles as shown in this image. It can be seen from the TEM image that the distribution of nickel ferrite nanoaprticles are polydispersed. According to figure 4, it is seen that $NiFe_2O_4$ nanoparticles appear uniform sphere like shape and highly crystalline spinel structure with average particle size about ~ 25.39 nm.

The selected area electron diffraction (SAED) patterns of the $NiFe_2O_4$ nanoparticles are shown in figure 5. The electron diffraction consists of concentric rings with spots over the rings. This feature indicates crystalline nature of the $NiFe_2O_4$ nanoparticles [18, 19].

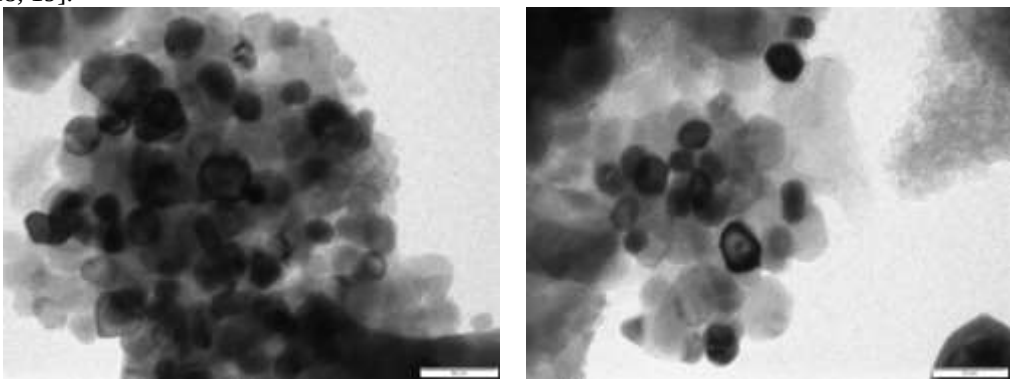

Fig. 4: TEM images of $NiFe_2O_4$ nanoparticles

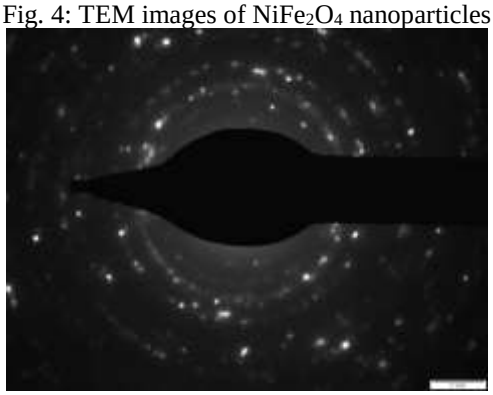

Fig. 5: SAED patterns of NiFe2O4 nanoparticles

D. Magnetic Properties

Figure 6 shows a typical hysteresis loop obtained room temperature for $NiFe_2O_4$ nanoparticles. Saturation magnetization (M_S) for $NiFe_2O_4$ nanoparticles was found to be 20.25 emu/g with coercive field (H_C) 301.77 G and the remnant magnetization (M_R) 4.76 emu/g (Table 1) indicating that $NiFe_2O_4$ nanoparticles are ferromagnetic in nature.

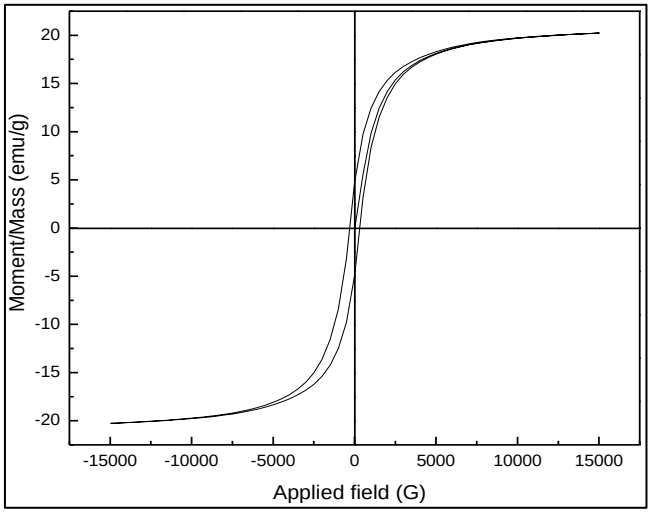

Fig. 6: Magnetization curve for $NiFe_2O_4$ nanoparticles

Samples	M_S (emu/g)	M_R (emu/g)	H_C (G)	SQR
$NiFe_2O_4$ nanoparticles	20.25	4.76	301.77	0.23

Table 1: Magnetic parameters of $NiFe_2O_4$ nanoparticles

V. CONCLUSIONS

Nanocrystalline $NiFe_2O_4$ has been synthesized by microwave route. The FT-IR spectrum confirmed the presence of metal oxides. XRD analysis validated the structure of $NiFe_2O_4$ nanoparticles with the crystallite size of 31.42 nm supported by the TEM images. The results of VSM indicate that the saturation magnetization of $NiFe_2O_4$ shows a maximum of 20.25 emu/g at room temperature.

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