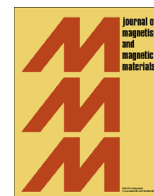




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Structural, electrical and magnetic characteristics of nickel substituted cobalt ferrite nano particles, synthesized by self combustion method



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ABSTRACT

Nickel-substituted cobalt ferrite nano-particles are synthesized using a self-combustion method. Aqueous metal nitrates and citric acid form the precursors. No external oxidizing agents are used to change the pH of the precursors; this resulted in a more environment friendly synthesis. Structural, magnetic and electrical characteristics of the nano ferrites are verified using X-ray diffractometer (XRD), VSM and impedance analyzer respectively. Phase formation, particle size, lattice parameter, X-ray density, saturation magnetization, coercivity, dielectric constant and electrical activation energy as function of nickel substitution in cobalt ferrite are studied. It is shown here that the magnetic and electrical properties can be tuned by varying the nickel concentration.

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1. Introduction

Nano-ferrites have drawn wide spread attention in the past few decades due to their technological importance. Applications of ferrites are in as diverse fields as catalysts [1], micro-wave devices [2], high frequency transformer core, antenna material and magnetically-guided drug delivery devices [3–8]. Preparation methods [9,10], doping and substitutions play a crucial role in controlling the properties of ferrites. Among these magnetic materials, spinel-type ferrite nanoparticles, MFe_2O_4 ($M=Mn, Co, Ni, Zn, Mg, Fe$, etc.), have gained great interest because of their good electromagnetic performance and wide range of applications in diverse fields. Soft magnetic properties, high electrical resistance, low eddy-current loss, high permeability at high frequencies make nickel ferrite technologically important material. Nickel-based mixed ferrites are also investigated extensively [11,12]. Cobalt ferrite with mixed spinel structure has high coercivity and lower resistivity. Bulk nickel ferrite has inverse spinel structure. Nickel substitution into cobalt ferrites allows us to tune the magnetic and electrical properties of ferrites as per the desired application. Ni–Co–Fe alloys are reported to be magnetostrictive and used as magneto resistance sensors. These can also be used for thin film magnetic heads for high-density recording [34]. The spinel structure is face-centered cubic with each unit cell

containing $32 O^{2-}$, $8 Ni^{2+}/Co^{2+}$ and $16 Fe^{3+}$ ions. The lattice of oxygen ions contains 64 tetrahedral and 32 octahedral vacancy sites. 24 cations are distributed into these sites. The eight Ni^{2+}/Co^{2+} and eight Fe^{3+} cations occupy half of the octahedral sites and the other eight Fe^{3+} ions occupy eight of the 64 tetrahedral sites [13,14]. This forms the inverse spinel structure. In the mixed spinel structures the number of Fe^{3+} ions at octahedral sites increase, while that of the Fe^{3+} ions at tetrahedral sites decrease because some divalent metal ions (Ni^{2+}/Co^{2+}) enter into tetrahedral sites.

In this study we report an environmentally benign synthesis for $Co_{1-x}Ni_xFe_2O_4$ nano particles. The ferrite powders are prepared by the sol–gel self-combustion synthesis method. This method is simple and results in stoichiometrically good compounds. There are several reports on use of this method of preparation with precursor solution changed to pH values 7, 9, 10 and 11 [34]. Here we are reporting the self-combustion method without altering the pH value of the precursor solution. Structural, electrical and magnetic characterization studies are done on the prepared ferrite nano-particles. This method of preparation is more environment friendly as it produces lesser amount of pollutant gases during the synthesis.

2. Experimental

Nickel-substituted cobalt Ferrite ($Co_{1-x}Ni_xFe_2O_4/x=0, 0.25, 0.5, 0.75$ and 1.0) nano-particles are prepared by sol–gel auto-combustion method. All of the chemicals used are of analytical

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grade and used without further purification. The aqueous solutions of $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ and $\text{Co}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ are taken in stoichiometric ratio, form the precursor material. Citric acid in 2:1 M ratio to the metal ions is used as chelating agent and fuel for self-combustion. For preparing the sample powders, aqueous ferric nitrate solution taken in a beaker is placed on a hot plate with magnetic stirrer. The solution temperature is maintained at 80 °C and stirred for about half an hour. Nickel nitrate solution, cobalt nitrate solution and citric acid solution are added one after the other, with a gap of half an hour to the ferric nitrate solution. The complex solution of metal nitrate and citric acid is stirred further for evaporation of water molecules and gelation of the solution. The viscous gel is transferred to a porcelain dish. The temperature of the dish is maintained at about 200 °C for half an hour in the open air. The gel starts combusting and the combustion continues for half an hour. After combustion a puffy solid remains. Before using for characterization, the puffy solid is grinded to powder and sintered at 600 °C for 2 h with a heating rate of 10 °C per minute.

The structural characterization is done with X-ray diffractometer (XRD) on the sintered powders. XRD is done using Phillips X'Pert diffractometer for the diffraction angle (2θ) ranging from 10° to 80° with a step size of 0.02°. A continuous scan with step time of 0.15 s is performed. The measurements are taken at 25 °C with Cu as anode material and the K_α radiation of wave length 1.5406 Å as the incident/diffracting wave. Particle size is calculated using Debye–Scherer formula. Lattice parameter, X-ray density is determined for these powders. These powders are pelletized with PVA binder and 4 t pressure. The pellets are heat treated for 2 h at 350 °C and further at 600 °C for 1 h for the removal of water and carbon compounds. Heating rate is maintained at 10 °C per minute. Samples are allowed to cool to room temperature within the furnace. Magnetic characterization is carried out on the solid samples with Lake Shore Cryotronics VSM (Vibrating Sample Magnetometer). Magnetic field is swept in the range from –5000 kOe to 5000 kOe at room temperature. Temperature dependent DC conductivity is measured on the pelletized samples with a Model 196, Keithley digital multimeter. The samples are silver coated for a good contact and heat treated before the conductivity studies. AC conductivity measurements of the ferrite samples are performed in the frequency range from 100 Hz to 20 MHz at room temperature using Wayne Kerr 6500B series, precision impedance analyzer. The instrument is calibrated for wire capacitance compensation before usage. The sample testing is done for an applied voltage of 1.00 V signals without any bias voltage.

3. Results and discussion

3.1. Structural characterization

Spinel structure of annealed powders of nickel-substituted cobalt ferrite ($\text{Co}_{1-x}\text{Ni}_x\text{Fe}_2\text{O}_4$ / $x=0, 0.25, 0.5, 0.75, 1.0$) nanoparticles prepared by sol–gel auto-combustion has been confirmed by the XRD. Fig. 1 shows the XRD of ($\text{Co}_{1-x}\text{Ni}_x\text{Fe}_2\text{O}_4$ / $x=0, 0.25, 0.5, 0.75, 1.0$). Nickel-rich ferrite nano powders show extra peaks of Fe_2O_3 . These extra peaks may be because of the combustion taking place in an oxygen-rich environment and small percentage of the sample is not reduced from Fe_2O_3 to Fe_3O_4 . Nickel is less oxidizing than cobalt and so this is found to be common, in particular for nickel-rich samples prepared at low sintering temperatures. Similar reports are found in the literature [15,16]. All the diffraction peaks of ferrite samples are indexed on the basis of the face centered cubic unit cell (JCPDS no. 00-022-1086 for CoFe_2O_4 and JCPDS no.00-044-1485 for NiFe_2O_4). All the samples are found to have Fd-3 m space group symmetry. The cubic lattice parameter

' a ' is calculated using the relation

$$a = d\sqrt{h^2 + k^2 + l^2} \quad (1)$$

Where, d is the inter planar distance and (hkl) are Miller indices. The correction to the lattice parameter is done employing the Nelson–Riley scheme. The Nelson–Riley function $\text{NR} = 1/2(\cos 2\theta / \sin \theta + \cos 2\theta / \theta)$ is plotted against the lattice constants, calculated from single reflection peaks, and is extrapolated to $\theta = 90^\circ$.

The X-ray density (the theoretical density) ρ_x was calculated using the relation

$$\rho_x = \frac{8M}{N_A a^3} \quad (2)$$

where M is the molar mass of the ferrite, ' a ' is the lattice parameter, and N_A is Avogadro's number.

The particle size ' D ' was determined using Debye–Scherer formula [17].

$$D = \frac{0.9\lambda}{\beta \cos \theta} \quad (3)$$

where λ is the wavelength of the Cu K_α (1.5406 Å), β is the full-width at half-maximum (FWHM) of the (3 1 1) peak in radians, and θ is the Bragg's angle.

The tabulated particle size, as in Table 1, indicates that the variation in the chemical content of the sample does not affect the particle size of the formed sample. The particle sizes of the samples fall in a very small range from 38 nm to 50 nm. The nanoferrites prepared at considerably low temperatures of about 200 °C and sintered at 600 °C for just 2 h are showing good crystallinity and agreeing well with JCPDS data. As the ionic radius of Ni^{2+} is smaller (0.74 Å) than that of Co^{2+} (0.78 Å), we see that the (3 1 1) peak position is continuously shifting towards lower angles suggesting the reduction in the ' d ' spacing. The lattice parameter

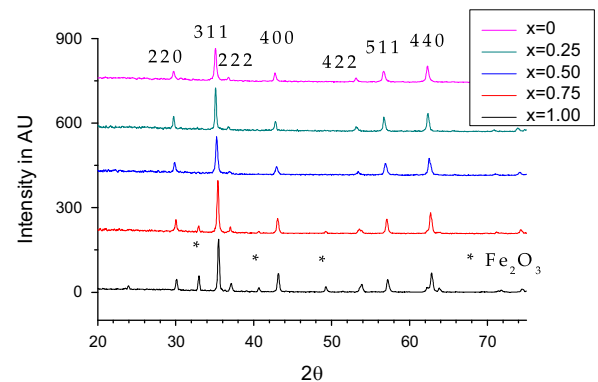


Fig. 1. XRD of $\text{Co}_{1-x}\text{Ni}_x\text{Fe}_2\text{O}_4$.

Table 1
Physical properties of cobalt and nickel-substituted cobalt ferrites.

Sample	Particle size (nm)	Lattice parameter (Å)	2θ of (3 1 1) peak	X-ray density (Kg/M^3)
CoFe_2O_4	35.24	8.416	35.5	5229.53
$\text{Co}_{0.75}\text{Ni}_{0.25}\text{Fe}_2\text{O}_4$	49.34	8.407	35.41	5244.12
$\text{Co}_{0.5}\text{Ni}_{0.5}\text{Fe}_2\text{O}_4$	38.02	8.380	35.26	5286.99
$\text{Co}_{0.25}\text{Ni}_{0.75}\text{Fe}_2\text{O}_4$	49.52	8.374	35.12	5303.41
NiFe_2O_4	38.12	8.347	35.01	5354.19

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