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Synthesis together with Structural & Magnetic Studies of Nickel Substituted Cobalt Ferrite Nanomaterials ($Ni_{0.07}Co_{0.93}Fe_2O_4$) Via Citrate Precursor Method

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Nickel substituted cobalt ferrite Magnetic nanoparticles of having formula $Ni_{0.07}Co_{0.93}Fe_2O_4$ have been synthesized by citrate precursor method using Ferric nitrate, Cobalt nitrate, Nickel nitrate and Citric acid as starting materials. The nanoparticles were prepared by annealing a citrate precursor at two different temperatures 650°C and 700°C for an hour. The samples were characterized using X-ray diffraction (XRD) and Vibrating sample magnetometer (VSM). Using Scherrer formula, the crystallite size was found to be 69 nm and 87 nm and lattice constants as 8.360Å and 8.333 Å respectively. Magnetic parameters were measured using Vibrating Sample Magnetometer (VSM). The maximum magnetization, retentivity was observed equal to 61.80 emu/g, 30.89 emu/g and coercivity as 808 Oe.

Key words : *Ferrite, Nanoparticles, Magnetic properties, Citrate Precursor method.*

Introduction : Ferrites being the mixed oxides with iron is the main component having the general formula (MO) (Fe_2O_3), where M stands for some divalent metal like Zinc, Cobalt, Nickel, etc. Ferrites have been receiving growing attention because of their various commercial and technological applications (Sugimoto Mitsuo., 1999, Mazaleyrat F. et al., 2000). The spinel structure of ferrites was first proposed in 1909 by Hilpert and has been investigated since then in quite detail (Hilpert S., 1909, Hilpert S. and Wille A., 1932, Hilpert S. and Lindner A., 1933, Hilpert S. and Schweinhagen R.,1935). Ferrites were prepared in nanocrystalline state for the first time in late eighties (Yoshizawa Y. et. al., 1988). In the nanocrystalline phase, ferrites have exhibited properties that are notably different from their bulk phase properties and are strongly dependent on the conditions and method of preparation (Fresh D.L., 1956, Rath Chandan et. al., 2000). This dependence has not been adequately investigated and it still lacks standardized procedures for obtaining ferrites with desired properties. Thus it appears relevant to study and understand the behaviour of ferrite samples prepared under varied conditions. There are several routes for preparation of nanocrystalline samples. One technique that has been widely used is by powdering. Ball Mills are normally employed for this. Fine powders have also been obtained using chemical precipitation and annealing. Electrolytic techniques have also been used. Several other methods exist besides these. A review of the different preparation techniques has been given by M. Pal and D. Chakravorty (Pal M. and Chakravorty D., 2003). Each of these techniques has its own advantage and disadvantage. The chemical route has a number of attractive features like simplicity and low cost of preparation so that it becomes an attractive alternative for preparation of nanocrystalline phase ferrite. **We have used chemical based Citrate Precursor method to synthesize Ni substituted Cobalt ferrite Nanoparticles**. Polycrystalline ferrite materials have been attractive for microwave applications, radio frequency circuits, transformer cores, rod antennas, read/ write heads for high speed digital tapes, sensors due to their high resistivity, low magnetic coercivity, low eddy current losses, high curie temperature and chemical stability etc. (Sugimoto Mitsuo., 1999, Mazaleyrat F. et al., 2001, Hilpert S. and Lindner A.,1933).

Materials and Methods: Experimental Procedure

Magnetic nanoparticles of Nickel substituted Cobalt ferrite having formula $Ni_{0.07}Co_{0.93}$ Fe_2O_4, have been synthesized using the Citrate precursor method. Ferric nitrate, nitrate of divalent metal (Ni and Co, AR grade chemical) were taken in Stoichiometric proportions as starting materials. Aqueous solutions of these salts were prepared separately by dissolving the salt in minimum amount of deionized water while stirring constantly. The solutions were then mixed together. Aqueous solution of citric acid was prepared in adequate quantity by weight and was added to the prepared salt solutions. The mixture was heated at temperature about 60°C to 80°C for two hours with continuous stirring. This solution was allowed to cool at room temperature and finally it was dried at 60°C–65°C temperature in an oven until it formed a brown color fluffy mass. The gels were annealed at temperature 650°C and 700°C for one hour in a muffle furnace. By this process, the precursor thermally decomposed to give ferrite powder that were later proved to be nanometer size particles.

Results and Discussion :

The XRD patterns were recorded using a diffrectometer (model D/max-IIB, Rigaku, Tokyo, Japan) and Magnetic measurement were carried out at room temperature using vibrating sample magnetometer (VSM, Lakeshore 7404).

The X-ray diffraction pattern of synthesized samples are shown in figure 1 and 2, hysteresis curves are shown in figure 3 and figure 4. As the annealing temperature increases, particle size increases and lattice constant decreases. Crystalline size calculated using Scherrer's formula (Culity B.D., 1978) and was found to be 69 nm and 87 nm annealed at 650°C and 700°C.The phase of these materials are well defined and belongs to nickel cobalt ferrite with spinel structure compared to JCPDS data file (JCPDS diffraction file, File No.22-1086). Crystalline property Increases as the annealing temperature increases.

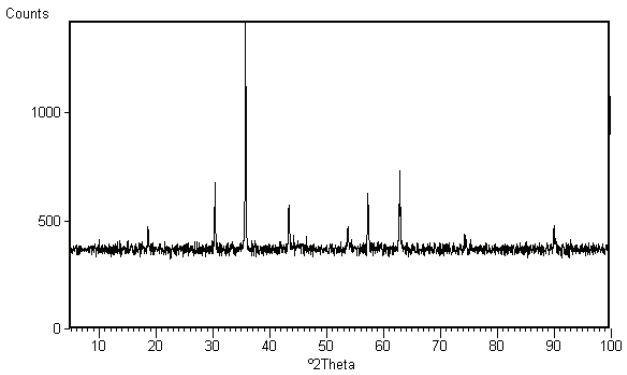

Fig. 1. : X-ray diffraction pattern of $Ni_{0.07}$ $Co_{0.93}$ Fe_2O_4 annealed at 650°C

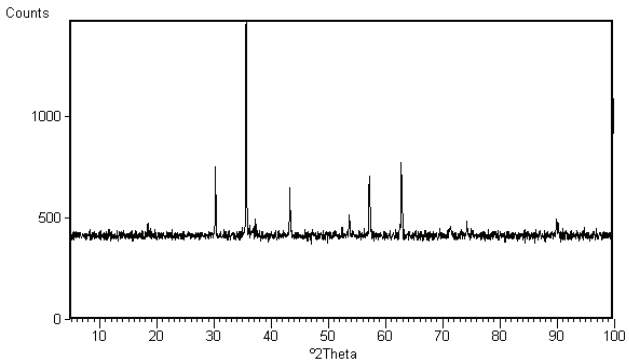

Fig. 2 - X-ray diffraction pattern of ferrite $Ni_{0.07}$ $Co_{0.93}$ Fe_2O_4 nanoparticle annealed at 700°C

Table 1: Structural data observed from X-ray diffraction

Sample	Annealing Temp.	Average Particle size	Lattice Constant
$Ni_{0.07}$ $Co_{0.93}Fe_2o_4$	650°C	69nm	8.360 Å
	700°C	87nm	8.333 Å

With increase in annealing temperature from 650°C to 700°C. Value of magnetic parameter such as Coercivity increases from 624Oe to 808Oe, retentivity increases from 27.71 emu/g to 30.89 emu/g and magnetization increases from 56.10 emu/g to 61.80 emu/g (Table 2). Larger particle size have larger magnetic parameters. Thus magnetic properties depend on particle size as well as annealing temperature (Verma A. et. al., 1999). The values of magnetization 61.80 emu/g is the highest value at this annealing temperature as our knowledge. This might be a feature of citrate precursor method (Singh et. al., 2010). The bulk value of magnetization for Ni-CoFerrite is 50 emu/gram.

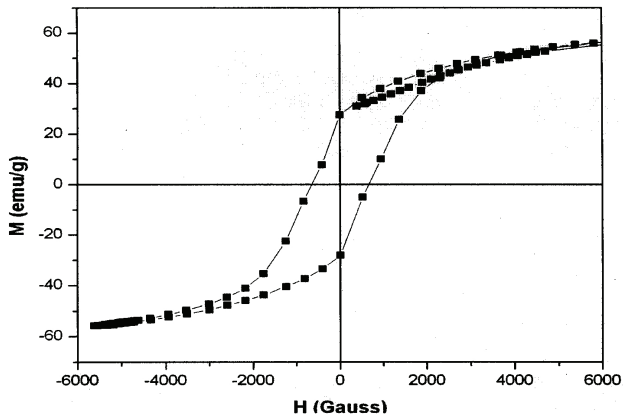

Fig 3 : Hysteresis curve for $Ni_{0.07}Co_{0.93}Fe_2O_4$ annealed at 650°C

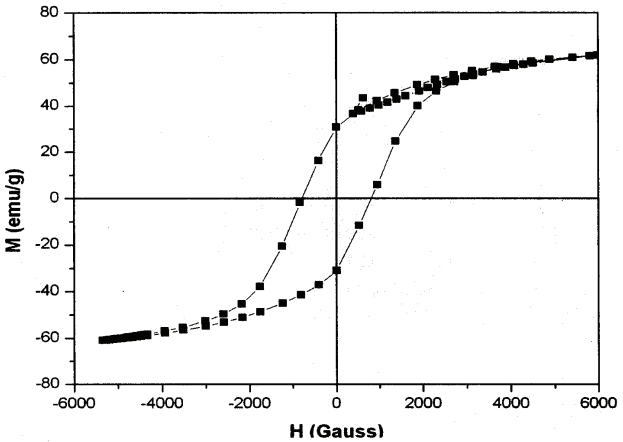

Fig 4 : Hysteresis curve for $Ni_{0.07}Co_{0.93}Fe_2O_4$ annealed at 700°C

Table 2: Values of Magnetic Parameters obtained from VSM

Annealing temperature	Coercivity Hc	Retentivity Mr	Magnetization
650°c	624 Oe	27.71 emu/g	56.10 emu/g
700°c	808 Oe	30.89 emu/g	61.80 emu/g

Thus with annealing temperature all the Magnetic parameters increases as well as particle size was also found to increases. Hysteresis loop area also found to increase. Nickel and Cobalt both have site preference in octahedral site. The magnetic properties have seen to alter with change cations distributions (Albuquerque A.S. et. a., 2001).

Conclusion :

In this work, $Ni_{0.07}Co_{0.93}Fe_2O_4$ ferrite nanomaterials synthesized using Citrate Precursor method. The cryatalline size was observed 69nm and 87nm at the annealing Temperatures 650°C and 700°C. The lattice constant was found to decrease from 8.360 Åto 8.333 Å. The maximum magnetization was found 56 emu/g and 61.80 emu/g at annealing Temperature 650°C and 700°C respectively. Coercivity, retentivity and magnetization and particle size were found to increase with the increase of annealing temperature while lattice constant was found to decrease. The magnetization 61.80 emu/g observed at 700°C is greater than bulk value.

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