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Synthesis, Structural and Magnetic Properties of Nickel Substituted Cobalt Ferrite Nanoparticle ($Ni_{0.03}Co_{0.97}Fe_2O_4$) Via Citrate Precursor Method

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Nickel substituted Cobalt ferrite Magnetic nanoparticles of having formula $Ni_{0.03}Co_{0.97}Fe_2O_4$, have been synthesized by citrate precursor method using Ferric nitrate, Cobalt 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 . The samples were characterized using X-ray diffraction (XRD) and Vibrating sample magnetometer (VSM). Using Scherrer formula, the crystallite size was found to be 95 nm and 69 nm and interplanner distance d equal to 2.526 Å and 2.519 Å respectively. Magnetic parameters were measured using Vibrating Sample Magnetometer (VSM). The maximum magnetization, retentivity and coercivity was observed 68.20 emu/g, 32.89 emu/g and 833 Oe respectively.

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

Introduction : Ferrites are mixed oxides with iron as 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 there is still lack of 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 synthesized 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 $Ni_{0.03}Co_{0.97}Fe_2O_4$, have been synthesized using the Citrate precursor method. Ferric nitrate, nitrate of divalent metal (Ni and Co, AR grade chemicals) 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 & 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 show in figures 1 and 2 and the hysteresis curves are shown in figure 3 and figure 4.

The average Particle size calculated using Scherrer equation (Culity B.D., 1978), decreases from 95 nm to 69nm and interplaner distance also decreases as annealing temperature increases from 650°C to 700°C. The phase of the material are well defined and belongs to Ni-Co ferrite with spinel structure compared to JCPDS data file (File No.22-1086).

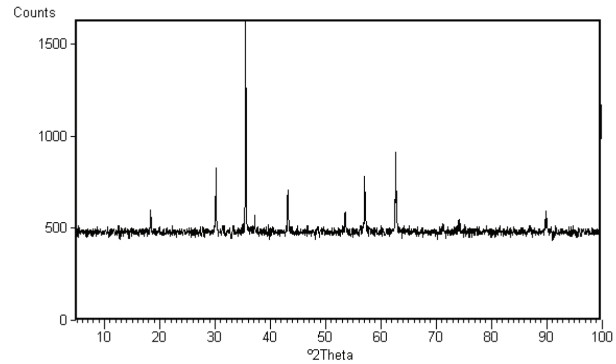

Fig.1 - XRD Pattern of $Ni_{0.03}$ $Co_{0.97}$ Fe_2O_4 annealed at 650°C

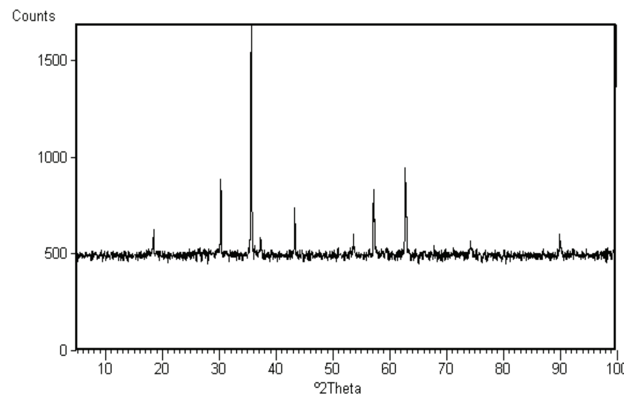

Fig. 2–XRD Pattern of $Ni_{0.03}$ $Co_{0.97}$ Fe_2O_4 annealed at 700°C

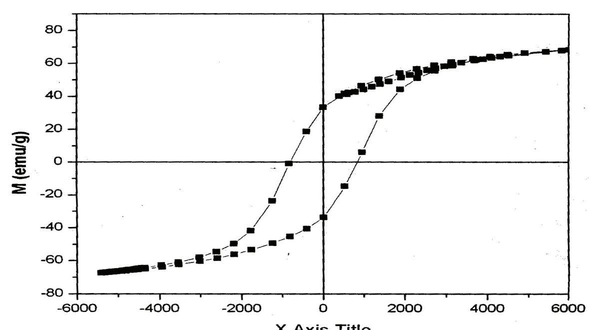

Fig: 3 - Magnetization curve for $Ni_{0.03}$ $Co_{0.97}$ Fe_2O_4 annealed at 650°C

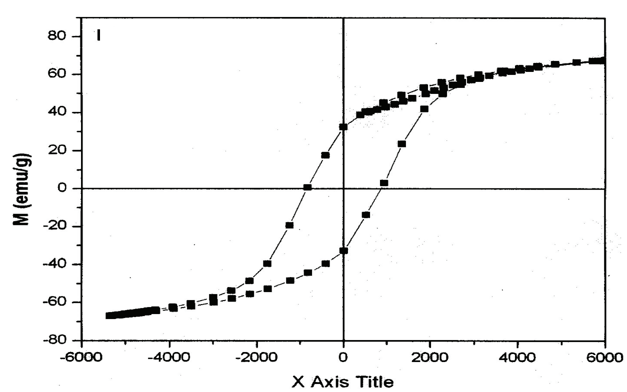

Fig: 4 - Magnetization curve for $Ni_{0.03}$ $Co_{0.97}$ Fe_2O_4 annealed at 700°C

Table 1 : Observed data from XRD and VSM

Sample	Annealing Tempe-rature	Coerci-vity (Oersted) (Oe)	Reten-tivity (emu/g)	Magn-etisa-tion (emu/g)	Inter-planner Distance (Å)	Parti-cle Size (nm)
$Ni_{0.03}Co_{0.97}Fe_2O_4$	650°C	808	33.33	68.20	2.526 Å	95
	700°C	833	32.88	67.75	2.519 Å	69

Ni has site preference to go octahedral sites. Particle size decreases with annealing temperature because, migration of divalent metals are randomly distributed over tetrahedral end octahedral sites. Particle size decrease with the increase in annealing temperature may be due to spin canting phenomenon induced at the surface by thermal gradient (Mohamed M.R. et. al., 2010). This phenomenon is more significant for the nano particles due to their large surface to volume ratio. Another possible reason for the dimination in magnetization and increase in coercivity might be due to the one hour of annealing temp.

When annealing temperature is increased from 650°C to 700°C, coercivity was found to increased from 808 Oe to 833 Oe while Retentivity decreases from 33.33 to 32.88, magnetization decreases from 68.20 to 67.75 emu/g. The magnetization 68.20 emu/g, greater than bulk size of Ni-Co ferrite. This may be due to migration of Co^{2+} ions from octahedral site to tetrahedral site. Because Co^{2+} ions have site preference in both sites, depend on heat treatment.

Conclusion :

In this work the method of preparation was used Citrate Precursor method to synthesize nanoparticles. The cryatalline size was observed 95 nm and 69 nm at annealing temperatures 650˚C and 700˚C respectively. The interplanner distance d was found to decrease (2.526 Å to 2.519 Å) as well as particles size(95nm to 69nm) with the increase in annealing temperature. The maximum magnetization was found to be 68.20 emu/g which is greater than bulk value.

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