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Magnetic properties of Zinc and Nickel substituted Cobalt Ferrite Nanoparticles synthesized using Citrate precursor method, annealed at 450°C.

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Zinc and Nickel substituted Cobalt ferrite Magnetic nanoparticles of having formula $M_{0.01}Co_{1.99}Fe_2O_4$, (where, M= Zn, Ni) 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 temperatures 450°C only 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 29nm, 58 nm and 87 nm respectively. Magnetic parameters were measured using Vibrating Sample Magnetometer (VSM). The maximum magnetization was observed 43 emu/g and coercivity 1325 G.

***Key words**: Cobalt Ferrite, Nanoparticles, Magnetic Properties, Citrate precursor method.*

Introduction :

Cobalt ferrite nanoparicles find applications in several areas including Magneto-optic devices, high density recording media, ferro-fluids and Medical diagnostics (Mohamed M.R et. al., 2010). The magnetic properties of fine particles is of considerable interest both from scientific and practical point of view. This is due to both size effects and surface effects (Papaefthymiou C.et al., 2009, Leslie-Pelecky et al. 1996, Singh et al. 2010). The major size effects involve the reduction of domain boundaries, that leads to single domain particles and a thermal randomization of the total spin system, commonly called superparamagnetism. This superparamagnetic behaviour originating from surface effects include canted spin structures and magnetic dead layers at surfaces. In small particles, saturation magnetization (M_s), Magnetocrystalline anisotropy (K), Coercivity (H_c), Retentivity (M_r) values are found to differ from bulk properties*, (Leslie-Pelecky et al. 1996)*. In Spinel structure of ferrite Nickel has tendency to occupy octahedral sites and Zinc to occupy tetrahedral sites. Thus magnetic properties of ferrites are found to changed.

Materials and Methods: Experimental Procedure

Magnetic nanoparticles of Zinc and Nickel substituted cobalt ferrite having formula $M_{0.01}Co_{1.99}Fe_2O_4$, (where, M= Zn, Ni) have been synthesized using the Citrate precursor method. Ferric nitrate, nitrate of divalent metal (Zn and Ni, purity- 99%) 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 90°C-95°C temperature in an oven until it formed a brown color fluffy mass. The gels were annealed at temperature 450°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 :

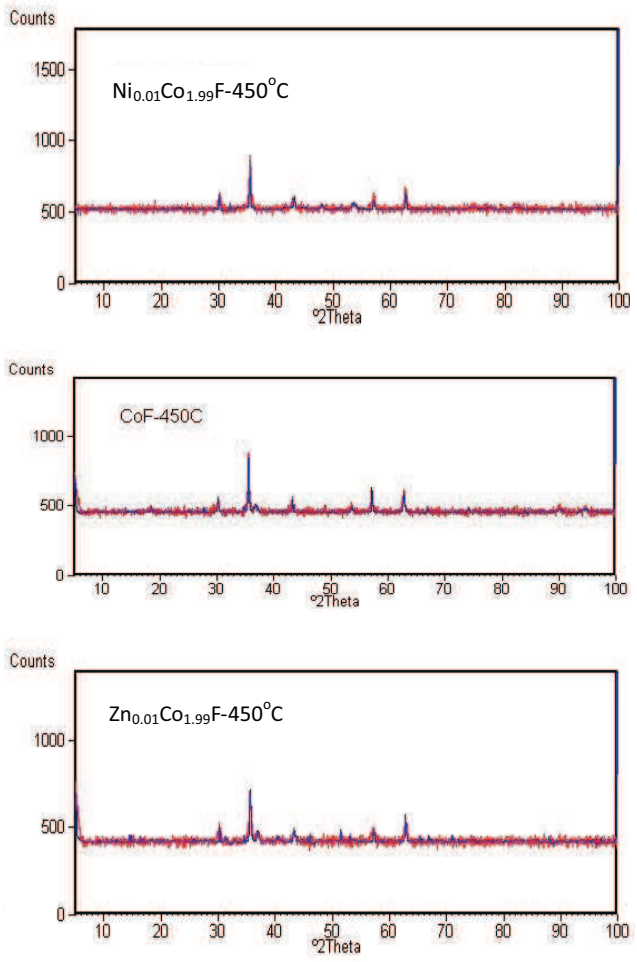

Fig.: 1- X-ray diffraction pattern of $CoFe_2O_4$ (CoF) $Ni_{0.01}Co_{1.99}Fe_2O_4$ and $Zn_{0.01}Co_{1.99}Fe_2O_4$, annealed at 450°C

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 XRD pattern were recorded using a diffractometer in the 2θ range, 10°-90° using Cu K_α (Wavelength = 1.5405Å) radiation. XRD studies identify that these nanoparticles samples are of spinel phase (JCPDS diffraction file, Card No.22-1086.) The X- diffraction pattern of these samples are shown in figure 1. Zn and Ni substituted ferrite have intensity peaks are smaller compared to Cobalt substituted ferrite(fig1).

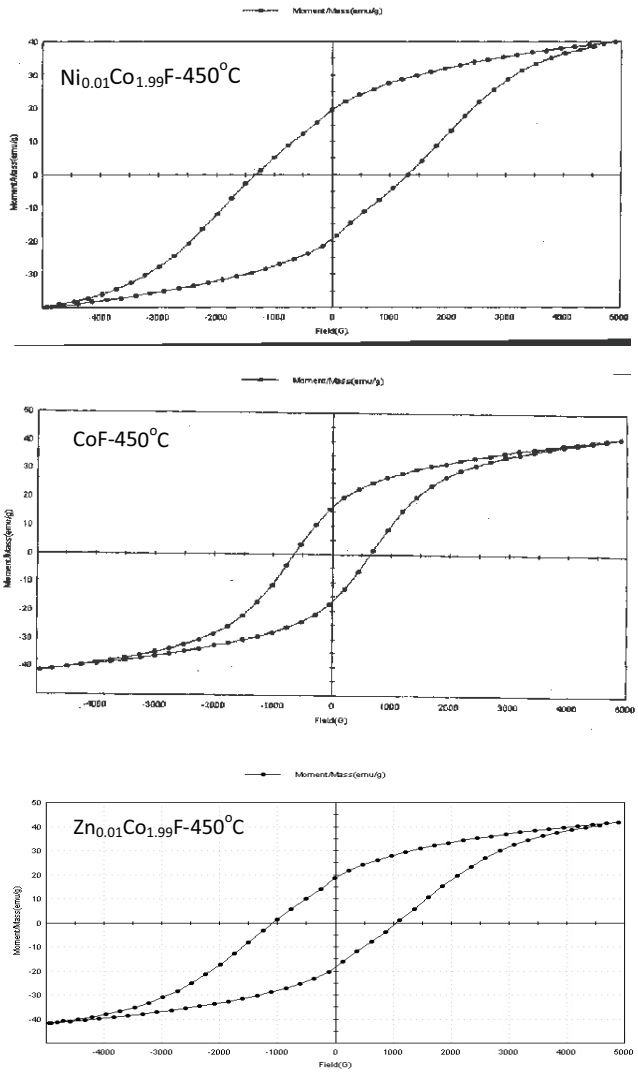

Fig. : 2 - Magnetization curve for $CoFe_2O_4$ (CoF) $Ni_{0.01}Co_{1.99}Fe_2O_4$ and $Zn_{0.01}Co_{1.99}Fe_2O_4$

The XRD pattern of the ferrite samples show well defined and sharp peaks. Using Scherrer formula (Culity B.D., 1978), $D= 0.9\lambda/ \beta COS\theta$, the crystallite size was found to be in the range 34 nm, 24 nm and 43 nm. Here D is the mean diameter, β is FWHM (Broadening of the intensity in radian), λ is the wavelength of X-ray radiation and θ is the Bragg's diffraction angle. It is also observed that the intensity of the Bragg's reflection peaks goes on decreasing with the substitution of Ni^{2+} and Zn^{2+} ions, indicating that these ions obstructs the crystal growth formation and particle sizes were found to change (Table 1).

The ferrite samples were magnetically characterized using VSM. The magnetic parameters obtained from VSM measurements of these samples of Cobalt ferrite

particles are tabulated in Table1 and their magnetic hysteresis curves are shown in figure 2. The net magnetic moment in the ferrimagnetic materials depends on the number of magnetic ions occupying in the tetrahedral (A) and octahedral sites (B).

Table 1: Observed data from XRD and VSM

Sample Name	Particle Size (nm)	Purity of Phase	Co-ercivity (G)	Reten-tivity (emu/g)	Magne-tizatiom (emu/g)
$CoFe_2O_4$	34	Good	650.26	16.75	41.38
$Zn_{0.01}Co_{1.99}Fe_2O_4$	24	Good	1074.81	18.77	40.05
$Ni_{0.01}Co_{1.99}Fe_2O_4$	43	Good	1323.62	19.53	41.74

The two important things for ferrite materials are (1) Ferrimagnetic behavior of cobalt ferrite depending upon heat treatment and (2) 450^0C is the standard temperature for complete cryatallization of Ferrite (Panda R.N. et al., 2003, Singh et al., 2010, Calvin S. et. al, 2008, Sangmanee et. al, 2009). In the present work, as the composition of Zn and Ni is substituted by 0.01 mole, both retentivity and coercivity are found to increase (Table 1) whereas change of magnetization is very small. This is due to different partile sizes, ions site preference and partly due to temperature susceptability property of cobalt ferrite (Iyer Rajesh et. al, 2009).

Conclusion :

A single annealing Temperature of 450^0C for the synthesis of these ferrite materials has been used. Particles sizes were found to be different i.e. 34nm, 24 nm and 43 nm. Substitution of 0.01 mole of Zn and Ni increased the Coercivity and retentivity in cobalt ferrite but magnetization is almost unchanged. Magnetic properties were found to depend on particles size and tetrahedral and octahedral site preferences of Zn and Ni. Substitution of Zn^{2+} and Ni^{2+} ions obstract the cystal growth formation and particle size of $CoFe_2O_4$ nanoparticles .

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