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Synthesis, Characterization and Magnetic properties of Nanoparticles of Cobalt Doped Ferrite

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Abstract**—** *Ferrites are ceramic like material having magnetic properties which are being utilized for several applications. Cobalt ferrites are hard magnetic material with high coercivity. In our study Crystalline, Magnetic nanoparticles of Cobalt ferrite $Co_{0.8}Fe_{2.2}O_4$ were synthesized by Sol Gel Method using ferric chloride and cobalt nitrate with NaOH as a reactant. Structural characteristics of samples were determined by X-Ray diffraction, FESEM and TEM. Particle size found 14.26nm by using Debye Scherrer method. Scanning electron microscopic (SEM) studies revealed nano-crystalline nature of the sample. AFM showed surface roughness. Magnetic properties were investigated using VSM (vibrating sample magnetometer). Various magnetic parameters such as saturation magnetization (Ms) and remanence (Mr) and coercivity (Hc) are obtained from the hysteresis loops. The calculated value of saturation magnetization in our study for Cobalt ferrite was found lower than the value reported for the bulk. The coercivity was found very high which indicate that the nanoparticles exhibit ferromagnetic behavior.*

Keywords**— Cobalt ferrites, Hysteresis loop, Nanoparticles, VSM, XRD.**

I. INTRODUCTION

Nanotechnology is the understanding and control of matter at dimensions of roughly 1 to 100 nanometers, where unique phenomena enable novel applications. Encompassing nanoscale science, engineering and technology, nanotechnology involves imaging, measuring, modeling and manipulating matter at this length scale. Now a days metal-oxide nanoparticles due to their unusual optical, magnetic and electronic properties, which are quite different from the bulk, being a subject of interest. Cobalt ferrites ($CoFe_2O_4$) are hard magnetic material having high coercivity and moderate magnetization [1, 2]. Above mentioned properties and their high chemical and physical stability, make cobalt ferrite nanoparticles suitable for various purpose like magnetic recording device as audio and videotape and digital recording disks with high-density etc. [3, 4]. The nanoparticles which are used for recording media, their magnetic characters crucially depend on the shape, size and purity of them [5]. A number of research reports are available concerning the preparation techniques thermal decomposition [6] ceramic [7], sol-gel [8], combustion methods [9], hydrothermal [10] and co-precipitation [11] etc. Among the various methods we used sol gel method [8]. Sol gel method is quite easy and efficient method. The presented work is about cobalt ferrite nanoparticles synthesis, characterization and their magnetic properties. Characterization of cobalt nanoparticles was done by *XRD, FESEM, TEM* and *AFM*. Particle size using *XRD* characterization was calculated by debye Scherrer method [12]. Magnetic characterization was done by VSM.

II. METHODOLOGY

2.1 Synthesis: Magnetic nanoparticles were prepared by the sol-gel method. We used $FeCl_3.6H_2O$, $FeSO_4.7H_2O$, $Co(NO_3)_2.6H_2O$ and *NaOH* as reactant to make cobalt doped ferrites. We made four types of cobalt doped ferrites using reactants in different concentrations. We made solutions of stoichiometric amount of $FeCl_3.6H_2O$, $Co(NO_3)_2.6H_2O$ and $FeSO_4.7H_2O$ in 100ml distilled water and a solution by using 6.4g *NaOH* dissolved in 200 ml distilled water.

All solutions were stirred constantly using magnetic stirrer for 20minute separately. Then took 100ml solution of *NaOH* into a beaker of 500 ml capacity and add cobalt

nitrate, ferric chloride and ferrous sulphate solution into it with stirrer on. *pH* of this mixed solution was between 8-9 then *NaOH* was added drop wise in line to achieve *pH* between 11- 12 under continuous stirring for 15minute. Then shifted this mixed solution on hot plate and raised its temperature till 80^0C. After 10minute, oleic acid 5ml was added in mixed solution. Then kept Mixture at 80^0C for 20minute. Now switched off hot plate for one hour while Stirrer was in running (on) state. Hot plate was switched on after 1 hour and its temperature was raised till 90^0C. Switched off hot plate again and let the solution to reach at room temperature meanwhile stirrer was in running (on) state. Then added 6 to 7 drops of HNO_3 into it, precipitate and dirty water got separated. We removed dirty water and washed precipitate using distilled water and kept precipitate in distilled water for overnight. Next day we washed precipitate first with boiled water for 5-6 time then with acetone for 5-6 time. To make sample dry we kept precipitate on filter paper for some time then in petri dish in sunlight and sample in powder form prepared.

2.2 Characterization: Various techniques were used for the characterization of nanomaterial properties. A complex analytical system was needed which should be capable to determine the composition and other properties of the substances. We used Transmission Electron Microscopy (TEM), X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM), atomic force microscopy (AFM) to study Structural morphology**.** These methods aimed at determining the crystal structure, chemical analysis, Phase identification and crystal or grain size. We used vibrating sample magnetometer (VSM) to study magnetic properties.

III. RESULT AND DISCUSSION

3.1 X-Ray diffraction analysis: Composition, phase structure and morphology were characterized by X-ray diffraction (Cu target, Wavelength 1.54184 Å). *XRD* patterns of different cobalt doped ferrites are shown in ***Figure* (3.1).** In these patterns one peak (h k l) value (3 1 1) was presented intensively. Crystalline size of every sample was calculated by debye scherrer formula [12] –

$$t=k\lambda/\beta cos\theta$$

Where k is shape factor. The value of k is 0.9. λ is the wavelength of *X* Ray used in analysis, θ represent Bragg's angle and β represent full width at half maximum (*FWHM*) (radian). Particle size, D spacing and lattice constant of cobalt ferrite are shown in below table-

$Co_x Fe_{1-x} Fe_2 O_4$	x =0.8
Particle size	14.26nm
D spacing	2.50$\AA$
Lattice constant	8.31 $\AA$

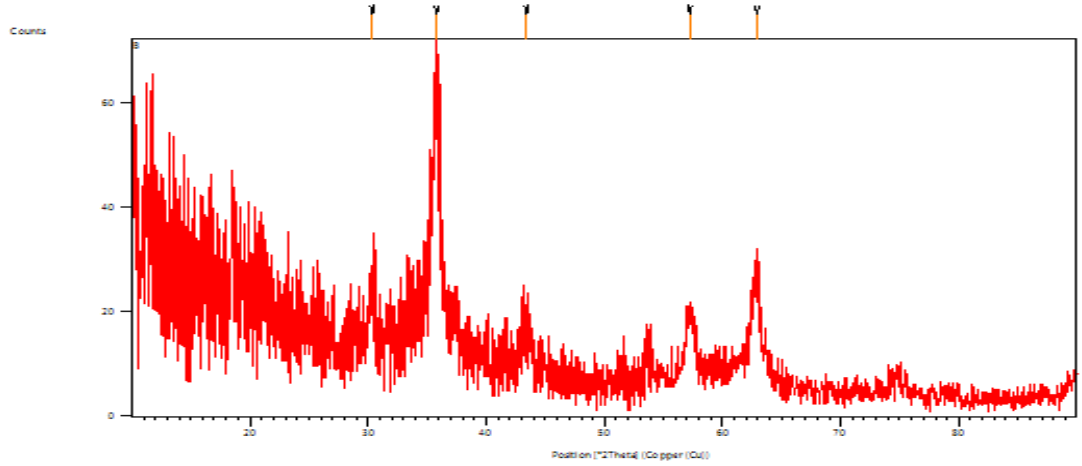


Fig. 3.1: *XRD* pattern of $Co_{0.8}$ $Fe_{2.2}$ O_4

3.2 SEM analysis: *FE-SEM* analysis of one cobalt nano crystalline ferrite was done. Cobalt ferrite's *SEM* images are shown in Figure (3.2). The particle size of this sample was not uniform and was found a little bit large from what we analyzed by *XRD*. Particle size was found approximate 40 nm by *FESEM* analysis. Similar images were also found by A. flores et al. [13].

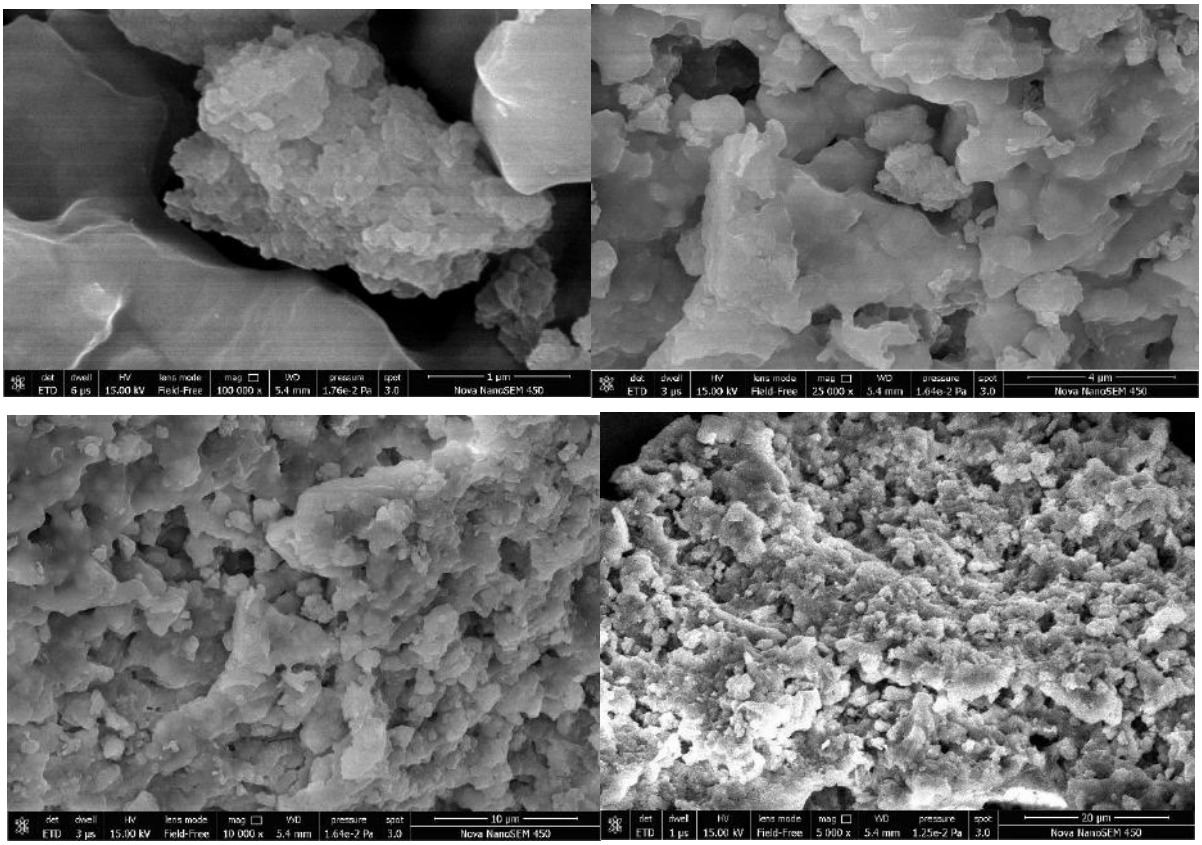
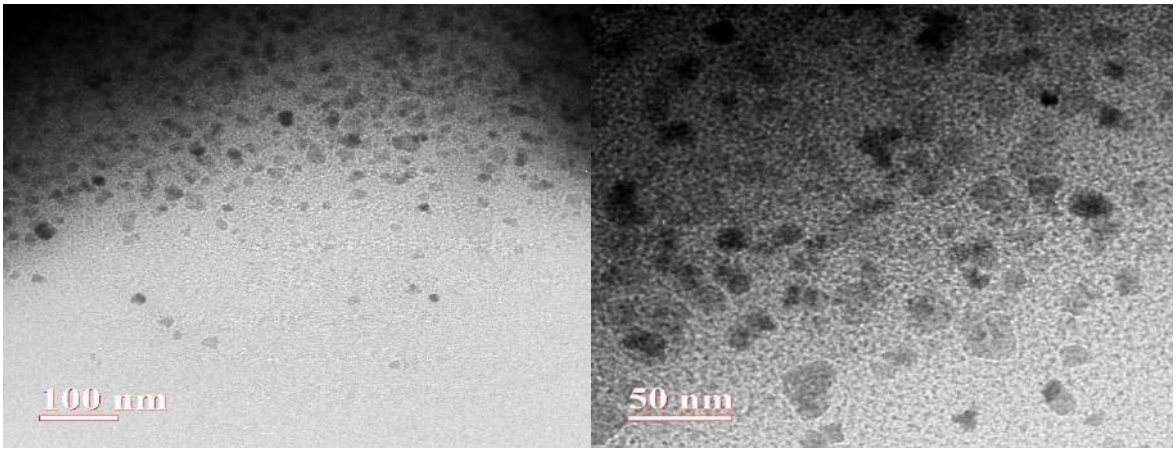


Fig. 3.2: FESEM images of $Co_{0.8}Fe_{2.2}O_4$

3.3 TEM analysis: Transmission electron microscopy was performed for cobalt ferrite. The images of *TEM* are shown in Figure (3.3). *SEAD* pattern of this ferrite is also shown in last image. Plane (3 1 1) shows in this pattern intensively. Other planes are also shown in this pattern.



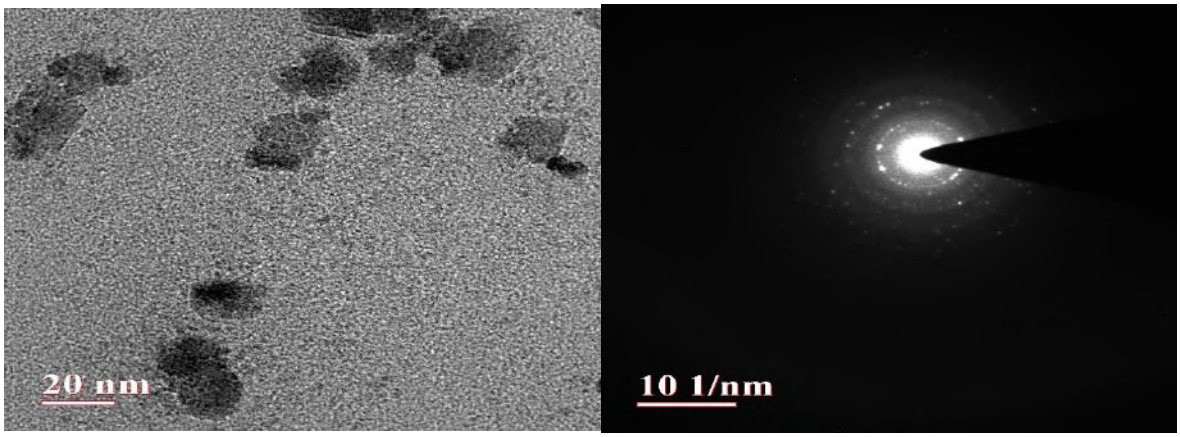


Fig. 3.3: TEM images and SEAD pattern of $Co_{0.8}$ $Fe_{2.2}$ O_4

3.4 AFM analysis: Atomic force microscopy was also done for this cobalt ferrite. AFM shows roughness of the surface. Images of AFM are shown in figure 3.4.

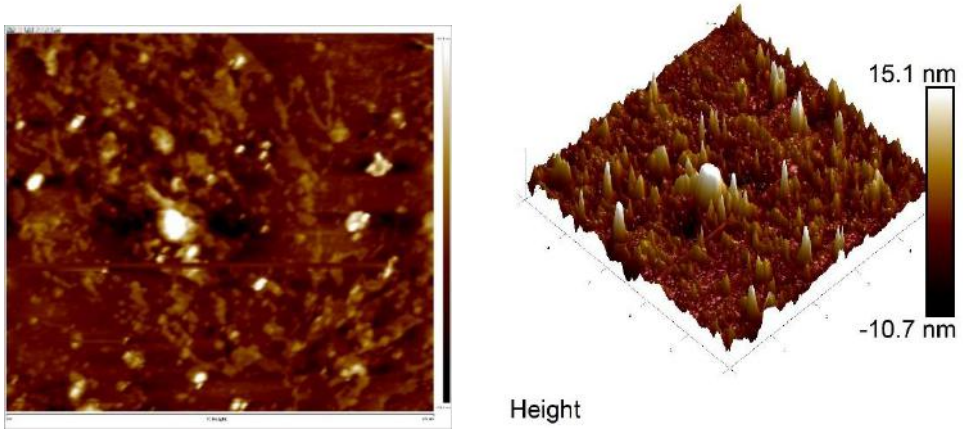


Fig.3.4: AFM images of $Co_{0.8}$ $Fe_{2.2}$ O_4

3.5 VSM Analysis: Vibrating sample magnetometer (VSM) was done for the cobalt sample. Sample mass was 20.6 gm. A hysteresis curve of $Co_{0.8}Fe_{2.2}O_4$ nanoparticles is shown in figure 3.5. The calculated value of saturation magnetization (Ms) for $Co_{0.8}Fe_{2.2}O_4$ nanoparticles is 25.49 emu/g, which was found lower than the value reported for the bulk samples (80 emu/g) [14], one was attributed to the existence of a structural "dead" surface layer, due to the formation of small nanoscale crystallite and residual strains during the sample synthesis [15]. In another report, the value of saturation magnetization (Ms) for $CoFe_2O_4$ nanoparticles was found 30 emu/g, which is very similar to our calculated value [16]. L.D.Tung et al. and L. Ajroudi et al. also found similar lower value of Ms for cobalt ferrites nanoparticles [17, 18]. The density of magnetization of the nanoparticles decreased with respect to the bulk can be attributed to surface defects and their morphology. The surface defects are the results of finite-size scaling of nano crystallites, which in turn leads to a non-collinearity of magnetic moments on their surface. These effects are more intense in ferromagnetic system, where the super-exchange interaction occurs through the oxygen ion O_2^- [19].

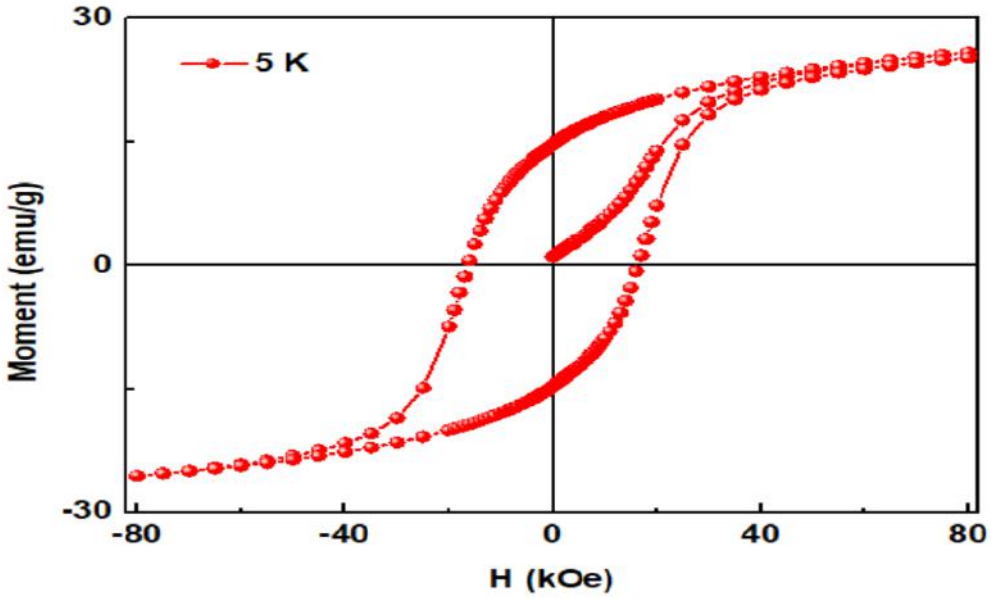


Fig. 3.5: hysteresis curve of $Co_{0.8}$ $Fe_{2.2}$ O_4 at 5K

The hysteresis loop of the as-prepared $Co_{0.8}Fe_{2.2}O_4$ nanoparticles carried out at temperature of 5 K, with applied fields of up to 80 kOe, is presented in Fig. 3.5. An open hysteresis loop with a coercivity field (Hc) of about 16 kOe was observed. Thus the nanoparticles exhibit ferromagnetic behavior with non-zero coercivity. This behavior is characteristic of single domain cobalt ferrite nanoparticles. Remanence (Mr) value calculated for $Co_{0.8}Fe_{2.2}O_4$ nanoparticles was 14.5 emu/gm. The squareness ratio Mr/Ms at 5K is 0.57, thus near the expected value for uniaxial single-domain particles without interaction and with a randomly orientation of the easy magnetic axis [18].

IV. CONCLUSION

In our paper we presented method of preparing cobalt ferrite ($Co_{0.8}$ $Fe_{2.2}$ O_4) nanoparticles by Sol Gel method. Their structural morphology was studied using *XRD*, *FESEM*, *TEM* and AFM. Debye Scherrer method was used to find out particle size from *X*-Ray diffraction pattern and using this method we got particle size between 14.26nm. After analyzing the images of *FESEM* and *TEM* we found that particle shape was not perfectly spherical but followed symmetrical pattern. *TEM* images showed that the similar particle size. AFM showed surface roughness. During VSM analysis Hysteresis loop followed similar trend. The calculated value of saturation magnetization (Ms) for CoFe2O4 nanoparticles was 25.49 emu/g, which was lower than the value reported for the bulk samples. An open hysteresis loop with a coercivity field (Hc) of about 16 kOe was observed. Thus the nanoparticles exhibit ferromagnetic behavior with non-zero coercivity. The squareness ratio Mr/Ms at 5K was 0.57, thus near the expected value for uniaxial single-domain particles.

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