



## **STUDY OF EFFECT OF SUBSTITUTION OF CADMIUM IN COPPER FERRITE BY WET CHEMICAL CO-PRECIPIATION METHOD**

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**Abstract:** Mixed ferrite system having the general  $Cu_{1-x}Cd_xFe_2O_4$  for  $x=0.0-0.6$  have been prepared by using wet chemical co-precipitation method in air oxidization by taking  $Cu^{2+}$ ,  $Cd^{2+}$  and  $Fe^{3+}$  cations in their weight proportion. The powders were characterized by XRD, and hysteresis techniques. For structural characterization the most intense peak (311) of XRD patterns were considered. The particle size obtained from XRD data was found in the range 25-26nm. The magnetic properties were investigated by using room temperature pulse field hysteresis loop technique. The saturation magnetization ( $M_s$ ) increases with cadmium concentration 'x' up to  $x=0.3$  and thereafter it decreases.

**Keywords:** Structural properties, magnetization, particle size.

### **INTRODUCTION**

Due to variety in technological and industrial applications scientists from physics and chemistry put their efforts for studying the ferrite materials at nanoscale dimension [1-4]. Reduced size of nanostructured materials than the bulk has unique and different properties in particular

copper ferrite. The microstructure and the surface properties of such fine powders have large implication in controlling the parameters required for any particular application which in turn depends on the method adopted for synthesis [5]. Physical methods, such as conventional ceramic method has some limitations for the

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synthesis of ferrite powders at nano structure. But in chemical methods which includes chemical co-precipitation [6], sol-gel [7], oxalate precipitate method [5], hydrothermal [8], citrate method [9], and combustion method [10] overcomes the limitations of physical methods. In the present work we have prepared the samples by using co-precipitation method. The electrical properties of ferrites depend upon chemical composition, methods of preparation and sintering temperature [11, 12]. The electrical conductivity in ferrites can be explained on the basis of Verwey mechanism [13]. The study of dielectric properties of ferrites produces valuable information on the behaviour of electronic charge carriers leading to greater understanding of the mechanism of dielectric polarization. It is reported that incorporation of Cd in  $\text{CuFe}_2\text{O}_4$  necessitates adjustment of both  $\text{Cu}^{2+}$  and  $\text{Fe}^{3+}$  ions [14].

### EXPERIMENTAL:

The spinel ferrite system  $\text{Cu}_{1-x}\text{Cd}_x\text{Fe}_2\text{O}_4$  with variable composition (for  $x = 0.0-0.6$ ) was prepared by air oxidation of an aqueous suspension containing  $\text{Cu}^{2+}$ ,  $\text{Cd}^{3+}$  and  $\text{Fe}^{3+}$  cations in proportion. The starting solution was prepared by mixing 50 ml of aqueous solutions of Cupric Sulphate ( $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ ) of Qualigens (99.5%), Cadmium Sulphate ( $3\text{CdSO}_4 \cdot 8\text{H}_2\text{O}$ ) A.R. grade (99%) and Ferrous Sulphate ( $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ ) of Qualigens (99%) in stoichiometric proportions. A two molar

(2M) solution of NaOH was prepared as a precipitant. It has been suggested that the solubility product constant ( $K_{sp}$ ) of all the constituents always exceeds when the starting solution is added in to the precipitant therefore in order to achieve simultaneous precipitation of all the sulphates the starting solutions (pH=3) was added to solution of NaOH and  $\text{H}_2\text{O}_2$  was also added and a suspension (pH =11) containing dark intermediate precipitation was found then the suspension was heated and kept at a temperature  $60^\circ\text{C}$ , while oxygen gas was bubbled uniformly into the suspension to stirrer it and to promote the oxidation reaction until all the intermediate precipitant changed into the dark brownish precipitate of spinel ferrite. The samples were filtered, washed several times by distilled water and acetone and then dried. The samples were heated at  $150^\circ\text{C}$  for 4 hours to remove the water content present in the sample. The wet samples of Cu-Cd system were annealed at  $950^\circ\text{C}$  for 12 hours. The prepared samples were then characterized by XRD for the determination of structural parameters.

### RESULTS AND DISCUSSION:

#### X-ray diffraction:

The powder X-ray diffraction (Philips, USA) of the prepared sample has been recorded to identify the crystalline phases present in the sample. When monochromatic beam of X-ray is incident on powder sample, it is reflected, the reflected rays

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interfere and interference is constructive only if the path difference between the interfacing rays is in integral multiple of the wavelength of incident X-ray beam. The condition of constructive interference is given by Bragg's law,

$$2d \sin \theta = n\lambda \quad (1)$$

where,  $d$  is the inter-planer spacing,  $\theta$  is glancing angle,  $\lambda$  is wavelength of incident radiation and  $n$  is an integer. The diffraction pattern were recorded in the  $2\theta$  range  $20^\circ$  to  $80^\circ$  with scanning rate of  $2^\circ$  per minute using  $\text{Cu-K}_\alpha$  radiation of wavelength  $1.5418 \text{ \AA}$ . The unit cell dimensions are determined from the  $d$ -spacing of a line by making use of the cubic formula for inter planer spacing.

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

where,  $a$  is the lattice constant,  $d$  is inter-planer spacing and  $(h \ k \ l)$  is the miller indices.

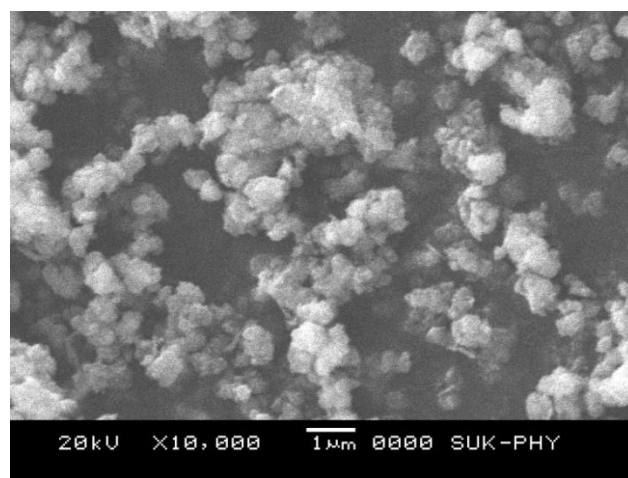
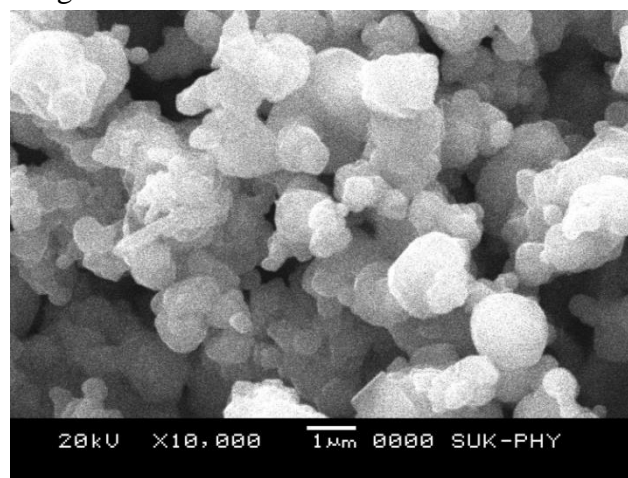
Scherrer [5], shows that the mean diameter  $d$  of the crystalline composing a powder is related to the X- ray diffraction broadening ( $B$ ) by the equation,

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

where,  $\lambda$  is the wavelength of incident radiation,  $B$  is the full width of half maxima, and  $\theta$  is glancing angle for (311) peak.

The average crystallite size of Cu-Cd series was determined by Scherrer formula using the strongest peak of (311) of the XRD pattern. The average sizes of the

particles were found to be 26 nm. The values of particle size obtained from Scherrer formula are given in Table 1. The crystallite size was also determined from SEM (Scanning Electron Micrograph). The SEM images are given in Fig. 1 for typical samples ( $x = 0.0, 0.2, \text{ and } 0.4, 0.6$ ). The particle size obtained from SEM data is of the order of 24nm to 27nm and the values are given in Table 1.



*Fig.1: Scanning electron micrographs for  $\text{Cu}_{1-x}\text{Cd}_x\text{Fe}_2\text{O}_4$  ( $x = 0.0$  And  $x = 0.2$ )*

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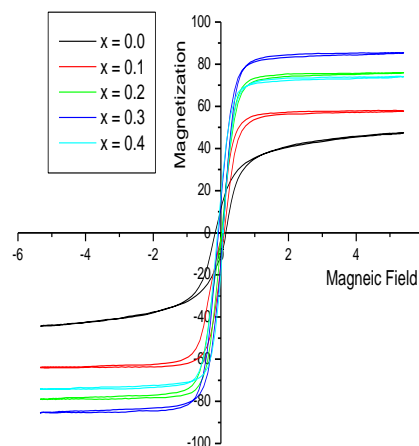
prepared by wet chemical co-precipitation technique.

Comp. 'x'	Lattice constant (Å)	X-ray density 'd <sub>x</sub> ' (gm/cm <sup>3</sup> )	Bulk density 'd' (gm/cm <sup>3</sup> )	Porosity %	Particle size 't' (nm)	
					XRD	SEM
0.0	a=8.41 c= 8.88	5.342	4.390	17.833	26	24
0.1	8.43	5.413	4.293	20.684	25	-----
0.2	8.46	5.463	3.053	44.107	26	27
0.3	8.5	5.492	3.189	41.928	27	-----
0.4	8.53	5.539	3.508	36.659	26	26
0.5	8.57	5.564	4.439	20.225	25	-----

**Table 1:** Lattice constant (a), X-ray density (d<sub>x</sub>), bulk density (d), particle size (t) and average grain size

Magnetic hysteresis loops for all samples of the series Cu<sub>1-x</sub>Cd<sub>x</sub>Fe<sub>2</sub>O<sub>4</sub> were recorded at room temperature. Typical loops for the samples with x = 0.0 -0.6 are given in Fig. 2. Using hysteresis loops, the values of saturation magnetization (M<sub>s</sub>), coercivity (H<sub>C</sub>) and remanence magnetization (M<sub>r</sub>) are obtained. The values of magnetic parameters obtained from hysteresis loop are given in Table 2. The magneton number (n<sub>B</sub>) calculated from observed values of saturation magnetization are listed in Table 2. It can be seen from Table 2 that magneton number n<sub>B</sub> increases up to x = 0.3 and after that it decreases. The increase in saturation magnetization and hence magneton number

may be attributed to the fact that though Cd<sup>2+</sup> (0μ<sub>B</sub>) ions occupy tetrahedral A-site, Cu<sup>2+</sup> ions occupies octahedral B-site thereby increasing the magnetic moment of B-site. The occupancy of Cu<sup>2+</sup> having magnetic moment 1μ<sub>B</sub> ions at octahedral B-site leads to increase in magnetic moment of B-site as compared to A-site magnetic moment. The decrease in magneton number for x = 0.5, x = 0.6 is due to the decrease in A-B super-exchange interaction. The magnetic super-exchange interaction of the ferrite system depends on the cation distribution at tetrahedral (A) site and octahedral [B] site.



**Fig.2:** Hysteresis loops for the samples of the series Cu<sub>1-x</sub>Cd<sub>x</sub>Fe<sub>2</sub>O<sub>4</sub> prepared by wet chemical co-precipitation technique.

**Table 2 :** Magnetization parameters for Cu<sub>1-x</sub>Cd<sub>x</sub>Fe<sub>2</sub>O<sub>4</sub> for (x=0.0-0.5) prepared by wet chemical co-precipitation technique.

Comp. 'x'	Magnetization Parameters			
	Remanent Magnetization Mr (emu/gm)	Saturation Magnetization Ms (emu/gm)	Coercive Field Hc (Oe)	Remnance R=(Mr/Ms)
0.0	14.882	47.554	158.114	0.3129
0.1	1.878	58.116	9.527	0.0323
0.2	4.200	76.007	18.322	0.0553
0.3	1.0815	85.511	33.953	0.0126
0.4	7.917	74.311	23.438	0.1065
0.5	0.831	65.944	18.151	0.0126

### CONCLUSIONS

The samples of the series  $Cu_{1-x}Cd_xFe_2O_4$  were successfully prepared by using wet chemical co-precipitation method. XRD data confirms the cubic spinel structure of the samples. Addition of Cd in copper ferrite decreases the net magnetization and also the coercive force.

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