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# Effect of Cu Substitution on Magnetic Properties of Co<sub>0.6</sub>Ni<sub>0.4</sub>Fe<sub>2</sub>O<sub>4</sub> Nanoferrites

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**Abstract**: This work is focusing on synthesizing the cobalt nanoferrite materials substituted by copper forming  $Co_{0.6}Ni_{0.4-x}Cu_xFe_2O_4$  with x = 0.0, 0.1, and 0.3 using the sol-gel auto-combustion method. The phase analysis of XRD showed the spinel structure with the lattice parameter in the range 8.36-8.39 Å. FESEM image showed the grain size initially decreasing and then increasing with Cu concentration. The FTIR curve's two absorption bands in the specified range of frequency assured the spinel nano ferrite structure. The values of remanent ratios obtained from VSM showed their isotropic nature forming single domain ferrimagnetic particles.

#### Keywords: sol-gel auto-combustion; spinel structure; FTIR; VSM.

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#### 1. Introduction

Two approaches were developed to obtain the high resistance in the core of the transformer, inductors, etc., to reduce loss due to eddy current and make appropriate for high-frequency devices: one is to modify the shape of metallic alloy, and the other was to modify the other to develop magnetic oxides. Nanoferrites are the magnetic oxides or ceramics made up of a large amount of Fe<sub>2</sub>O<sub>3</sub> blended with a small amount of different elements like barium, cobalt, manganese, nickel and zinc [1, 2]. The Fe<sub>2</sub>O<sub>3</sub> type of magnetic oxides has magnetization but electrically insulating. So, they are also called magnetic insulators. The thin tape Ni-Fe alloy was called permalloys among different alloys like Si-Fe, Ni-Fe, Ni-Zn, Mg-Zn, etc. As the next step of improvement, the grain-oriented materials were used in the core and termed superalloys. When mixed with molybdenum, the nickel-iron was termed as molypermalloys used mostly as switching devices due to their square hysteresis loops. The ratio 77%, 16%, 5%, 2% of nickel, iron, copper and molybdenum or chromium, respectively, gives Mu Metal which is used in magnetic shielding.

The ferrimagnetic behavior of nano ferrites are famous for making permanent magnet used in refrigerator, loudspeakers, electric motors etc. due to their high coercivity or difficult of demagnetize and termed as 'hard ferrites' and in high-frequency inductors, transformers, antennas, microwave components etc. due to their low coercivity or easily demagnetize and termed as 'soft ferrites'. While Cobalt nano ferrites are 'semi-hard ferrites'. Many ferrites have spinel structures in the form AB<sub>2</sub>O<sub>4</sub>, A and B are various metal cations (mostly iron). A cation covers 1/8<sup>th</sup> of the tetrahedral and B covers <sup>1</sup>/<sub>2</sub> of the octahedral holes, i.e., A<sup>2+</sup>B<sub>2</sub><sup>3+</sup>O<sub>4</sub><sup>2-</sup>. Ferrites are easy to synthesis and have a simple structure for analysis. Among them, the mid-hard cobalt

ferrites have dragged the concern with their extensive magnetocrystalline nature, wider coercivity, medium magnetic saturation, chemically stability, mechanically strong and high electrical resistivity [3-5]. They can be synthesized by hydrothermal, sol-gel, solid-state reaction method etc. The allocation of the cations in the sample is greatly affected by the synthesis procedure [6, 7]. Numerous works of literature can be found in changing and controlling the properties of ferrites by substituting other metals like Al, Ge, Ni, Cr, Sm-Gd, Ce-Gd etc. But, substituting with highly conducting copper is hardly found. So, we are going to substitute copper in the nickel element system in this study [8, 9].

We have used sol-gel auto-combustion method for synthesis of  $Co_{0.6}Ni_{0.4-x}Cu_xFe_2O_4$  for x = 0.0, 0.1 and 0.3. Studying the magnetic property of ferrite is very important. Among them, the value of saturation magnetization is vital. We expect quite the exotic magnetic nature of our synthesized composition that can be used in advanced devices.

#### 2. Materials and Methods

The cobalt nitrate hexahydrate (Co(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O), nickel nitrate hexahydrate (Ni(NO<sub>3</sub>)<sub>2</sub>· 6H<sub>2</sub>O), copper nitrate monohydrate Cu(NO<sub>3</sub>)<sub>3</sub>· H<sub>2</sub>O, ferric nitrate (Fe(NO<sub>3</sub>)<sub>3</sub>·9H<sub>2</sub>O), and citric acid monohydrate (C<sub>6</sub>H<sub>8</sub>O<sub>7</sub>· H<sub>2</sub>O) analytical grade of 99% purity were taken as starting materials. The total nitrates and citric acid were mixed in 1:1 ratio. Citric acid segregates the metal ions and homogenizes the distribution. The electronegative oxygen atom interacting with electropositive metal ions helps control the precipitation of metal nitrates during water dehydration. As a result, a homogenous nano ferrite is formed even at a relatively low temperature. The addition of ammonia neutralizes the mixed solution to pH-7 and supports forming a gel by increasing the solubility of metal ions. The distilled water is added to get a clear colloidal solution. The resulting solution was heated continuously on the magnetic stirrer around 150°C to allow gel formation. The gel obtained was combusted at normal temperature and pressure to remove excess water, nitrogen and carbon dioxide. It was powdered finely with the help of agate mortar and pestle. The powder was continuously sintered at 1000°C for 3 h. The sintered powder was pressed into pellets using a hydraulic press under 5 tons pressure. Few drops of Poly Vinyl Alcohol were utilized for their shaping.

Rigaku Cu-K $\alpha$  diffractometer was used for finding the X-ray diffraction pattern with the diffracting angle between 20° and 80° in the step of 0.02° to calculate the lattice parameter and crystallite size. XRD data were obtained at room temperature using Cu-K $\alpha$  (1.5406 Å) radiation. A Field-Effect Scanning Electron Microscope (FESEM) image was used to study the morphology of the samples. Fourier transforms infrared (FT-IR) spectra were used to identify the functional groups in all samples. Magnetic properties were studied using VSM at room temperature.

#### 3. Results and Discussion

#### 3.1. X-ray diffraction analysis.

The phase and lattice parameter of  $Co_{0.6}Ni_{0.4-x}Cu_xFe_2O_4$  for x = 0.0, 0.1and 0.3 was identified by X-ray diffraction method. The diffracted beam intensity and diffracting angle in terms of angular width (2 $\theta$ ) in the range 10°-70° are shown in Figure 1. In the Figure, the sharp and narrower peaks show that the sample has a high crystalline nature. Also, the shifting of

peaks towards the lower value of  $2\theta$  with the increase in the concentration of Cu from x = 0.0, 0.1 and 0.3 shows the respective increment of the lattice parameter.

The maximum peak of the X-ray diffraction pattern (311) is used to find the crystallite size using an online calculator. The values of lattice parameter "a" are calculated with the following relation [9]:

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

where d is interplanar spacing for the (hkl) plane. The values of ' $\theta$ ' and 'd' were obtained from the experiment based on Bragg's law. Nelson-Riley extrapolation method was used to determine the exact lattice parameter with error for each sample. This function is given by:

$$F(\theta) = \frac{1}{2} \left[ \left( \frac{\cos^2 \theta}{\sin \theta} \right) + \left( \frac{\cos^2 \theta}{\theta} \right) \right]$$
(2)

The XRD data obtained was with an accuracy of  $\pm 0.002$ Å. The lattice parameters, ionic radii of the sample in tetrahedral and octahedral sites, and cell volume are presented in Table 1.



Figure 1. XRD spectra of Co<sub>0.6</sub>Ni<sub>0.4-x</sub>Cu<sub>x</sub>Fe<sub>2</sub>O<sub>4</sub> nanoferrite samples.

The ionic radius of  $Cu^{2+}$  (0.73 Å) is larger than that of Ni<sup>2+</sup> (0.69 Å) [10, 11]. So, the lattice constant is found to increase from 8.3598 to 8.3906 Å with increasing Cu content. Moreover, the ionic radiuses of the sample on the tetrahedral side are increasing and on the octahedral site are decreasing with Cu concentration. This is due to the transfer of cations from the octahedral site to the tetrahedral site.

The maximum peak from the XRD pattern (311) is considered for calculating Crystallite size with the help of an online CS calculator depending on Scherer's formula. The broadness of diffraction peaks (Figure 1) indicates the smaller crystallite size of the samples. The-broadness of the peak decreases with increasing Cu content and hence showing increasing crystallite size. Scherer's formula is given as [12];

$$D_{311} = \frac{0.9\lambda}{\beta \cos\theta} \tag{3}$$

where, D<sub>311</sub>,  $\lambda$ ,  $\beta$  and  $\theta$  are crystallite size, the wavelength of X-ray (1.5406Å), full width at half maximum (FWHM) of (311) peak and Bragg's angle, respectively.  $\beta = \sqrt{\beta_{exp}^2 - \beta_{inst}^2}$ is the FWHM in radians where  $\beta_{exp}$  is the experimental FWHM and  $\beta_{inst}$  is the instrumentation broadening factor. Its value is 0.06° for silicon powder used as a standard sample. The XRD peak (311) is obtained by Gaussian profile fit that corresponds to the crystallite size of samples under consideration. Similarly, the plot of Lattice constant and Crystallite size against Cu concentration (x) of Co<sub>0.6</sub>Ni<sub>0.4-x</sub>Cu<sub>x</sub>Fe<sub>2</sub>O<sub>4</sub> mixed nanoferrite. The calculated crystallite sizes are in the range of 39.83 to 42.56 nm.

Composition (x)	Lattice constant (Å)	Crystallite size (nm)	Cell volume (Å) <sup>3</sup>
0.0	8.36	39.83	591.39
0.1	8.37	40.25	589.55
0.3	8.38	41.85	589.54

**Table 1.** Lattice constant and crystallite size of Co<sub>0.6</sub>Ni<sub>0.4-x</sub>Cu<sub>x</sub>Fe<sub>2</sub>O<sub>4</sub> nanoferrite samples.

3.2. Field Effect Scanning Electron Microscope (FESEM) studies.

The FESEM gives images of the physical texture and morphology of  $Co_{0.6}Ni_{0.4-x}Cu_xFe_2O_4$  for x = 0.0, 0.1 and 0.3. The images are as shown in Figure 2 (a) to (c). In the figures, the crystal grains are clear and inhomogeneous in shape and size. The decreasing of agglomeration with a concentration of  $Cu^{2+}$  may be due to the addition of non-magnetic dopant in place of magnetic cations and also with the synthesis condition [13, 14]. The pores and voids are created after releasing gases in the synthesis process. Since Cu has a larger ionic radius than Ni, the lattice distortion takes due to size mismatch so that the size decreases first. As the concentration is increased, the bond is set up, and the grain size is increased.



Figure 2. (a-c): FESEM image of Co<sub>0.6</sub>Mg<sub>0.4-x</sub>Cu<sub>x</sub>Fe<sub>2</sub>O<sub>4</sub> nanoferrite samples.

## 3.3. FTIR studies.

The functional groups of Co<sub>0.6</sub>Ni<sub>0.4-x</sub>Cu<sub>x</sub>Fe<sub>2</sub>O<sub>4</sub> for x = 0.0, 0.1 and 0.3 are identified with the help of the FTIR study. The specific feature in a molecule creates infrared absorption due to a change in the electric dipole moment of the molecules. The change in dipole moment is due to the expansion and contraction of its bond. The infrared spectra are shown in Figure 3. The two strong absorption bands are seen on the IR curve. The two bands  $v_1$  with higher frequency and  $v_2$  with lower frequency indicate that metal cation and oxygen bond stretching  $M_{tetra} \leftrightarrow O$  and  $M_{octa} \leftrightarrow O$  with respective force constants  $K_t$  and  $K_o$  at tetrahedral and octahedral sites, respectively. Their values are listed in Table 2.



Figure 3. Wavenumber of Co<sub>0.6</sub>Ni<sub>0.4-x</sub>Cu<sub>x</sub>Fe<sub>2</sub>O<sub>4</sub> nanoferrite sample.

Table 2. Tetrahedral  $(v_1)$  and Octahedral  $(v_2)$  absorption bands of  $Co_{0.6}Ni_{0.4-x}Cu_xFe_2O_4$  samples.

Cu content (x)	v <sub>1</sub> (cm <sup>-1</sup> )	$v_2 ({\rm cm}^{-1})$
0	588.3	402.18
0.1	591.207	397.35
0.3	591.27	399.79

The wavenumber of the tetrahedral absorption band first increases and decreases, while, that of the octahedral absorption band decreases gradually with an increase in the concentration of Cu. The difference in band position of tetrahedral and octahedral is due to the distance between the metal cations and oxygen atoms in two respective sites [15-17]. Their mean distance is found to be 1.89 Å and 1.99Å on their respective sides. This makes the values of wavenumber in the two sites different. The decrease in site radius or bond length increases the fundamental frequency.

## 3.6. Magnetic properties.

The Vibrating Sample magnetometer (VSM) gives the magnetic properties of  $Co_{0.6}Ni_{0.4-x}Cu_xFe_2O_4$  for x = 0.0, 0.1 and 0.3 nanoferrites. Their hysteresis loops under the field of ±10,000 Oe are shown in Figure 4. The S-shaped hysteresis loop indicates that the sample is ferrimagnetic. Different loops are found for different Cu concentrations. The coercivity value of these samples indicates that the nano ferrite is becoming soft with the Cu concentration [18-20]. Higher the retentivity or lower coercivity, the more strongly it gets magnetized and more rapidly loses its magnetism. Next, the higher the remanent magnetization (M<sub>r</sub>), the higher is the coercive force (H<sub>c</sub>) needed to bring it to zero. The value of saturation magnetization increases with increasing the Cu concentration. This may be due to increasing exchange interaction between the ions at tetrahedral and octahedral sites. The particle and magnetic

domain size-reduction decreases the coercivity and hence coercive force [20-23]. The measured values of saturation and remnant magnetization (M<sub>s</sub> and M<sub>r</sub>) and coercive field (H<sub>c</sub>) are listed in Table 3.

The value of the remnant ratio  $(R=M_r/M_s)$  below 0.5 shows the isotropic nature of the materials forming single domain ferrimagnetic particles [24]. Nanoferrites have high Ms and moderate H<sub>c</sub> [25, 26] that are required for magnetic recording media. The coercivity decreases with Cu concentration due to the non-magnetic nature of Cu. The resulting material is becoming soft nano ferrites which we need. The grain size generally does not affect the magnetic saturation (M<sub>s</sub>) and the coercivity (H<sub>c</sub>) of a material. But, M<sub>s</sub> depends on the size, if and only if the number of atoms on the surface is remarkable to the total number of the sample volume. Here, the size of the particle is not affecting the coercive force.



Figure 4. Hysteresis loops of Co<sub>0.6</sub>Ni<sub>0.4-x</sub>Cu<sub>x</sub>Fe<sub>2</sub>O<sub>4</sub> nanoferrite sample at room temperature.

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I al	one 5. $M_s$ , $M_r$ and	$H_c$ of $CO_{0.6}ZII_{0.4-x}C$	$u_x re_2 O_4$ nanoterm	e sample.
tration (x)	M <sub>s</sub> (emu/g)	M <sub>r</sub> (emu/g)	H <sub>c</sub> (Oe)	$R=M_r/I$

Table ? M. Mand II of Co. 7n

Concentration (x)	M <sub>s</sub> (emu/g)	Mr (emu/g)	H <sub>c</sub> (Oe)	$R=M_r / M_s$
0	46.2	18.6	885	0.39
0.1	50.8	13.8	621	0.35
0.3	39.9	14.2	449	0.37

## 4. Conclusions

X-ray diffraction pattern shows the highly crystalline nature The of  $C_{0.6}N_{i_0.4-x}Cu_xFe_2O_4$  (x = 0.0, 0.1, and 0.3) nanoferrite samples. The shifting of peaks towards the lower value of  $2\theta$  Cu concentration indicated the increasing lattice parameter with concentration due to larger ionic radii of the substituent. Crystallite size increased with Cu<sup>2+</sup> concentration and is found in the range of 39.83 nm to 42.56 nm showing the nano-size particles. The decreasing and increasing grain sizes in FESEM images are due to the lattice distortion by size non-uniformity during synthesis. The value of the remnant ratio showed the isotropic nature of the materials forming single domain ferrimagnetic particles.

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## **Conflicts of Interest**

We declare that this article has no conflict of interest.

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