

Synthesis, Characterization and Properties of Ni–Cu–Zn Nano-Composite

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ABSTRACT: This research article describes the magnetic properties of nano-composite composition as $\text{Ni}_{0.75-2x}\text{Cu}_{0.1+x}\text{Zn}_{0.15+x}\text{Fe}_2\text{O}_4$ ($0.01 \leq x \leq 0.05$), synthesized by a simple method using metal nitrates and citrate precursor method. X-ray Diffraction measurements (XRD) justified the formation of single-phase cubic spinel structure. The average crystallite size has been calculated using XRD pattern and the same is confirmed by Scanning Electron Microscope (SEM). The nearly constant lattice parameters obtained with copper substitution is attributed to the small difference in the ionic radius between Ni^{2+} and Cu^{2+} ions. The effect of copper and zinc concentration on the magnetic properties is investigated using Vibrating Sample Magnetometer (VSM). Through the magnetic measurements it can be observed that the entire preparation method increases the magnetic properties of the Nano-composite material. The NiCuZn Nano-composite with increased copper and zinc molar ratio are observed to have better magnetic properties compared to available NiCuZn Nano-composite.

Keywords: Nano-composite; Citrate Precursor Method; magnetic amp-turns; power to weight ratio.

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I. INTRODUCTION

In the present day scenario, Nano-composites are highly commercially important materials with enhanced magnetic properties. NiCuZn Nano-composite ferrite as magnetic core material with high resistivity, permittivity, high Curie temperature and high magnetization have been rapidly developed to operate from a few cycles per second to mega cycles per second. Due to these reasons, NiCuZn ferrites play an essential role in technological applications such as telecommunications, power transformers, electromagnetic interference (EMI) suppressors, SPMS, microwave devices, and read/write heads. Now, NiCuZn ferrites are the dominant materials for MLCI applications due to its relatively low sintering temperature, less loss, and wide applied wave bands.

Typical NiCuZn nano-composite usages of fine powder decrease the sintering temperature of Nano-composites. Fine powders can be prepared through various wet-chemical methods like co-precipitation, hydrothermal synthesis and sol-gel processes. Although the co-precipitation and sol-gel methods are the most popular, they have some disadvantages as most of them are highly pH sensitive and require special attention for complex systems whereas the sol-gel technique requires expensive alkoxide precursor material and stringent process of gel product. Among the

established synthetic methods are critical to finding simple and cost-effective routes to synthesize nano-crystalline NiCuZn Nano-composites by using cheap, nontoxic and environmental friendly citrate precursor method [1-23].

In this paper investigate of magnetic and electrical properties of $\text{Ni}_{0.75-2x}\text{Cu}_{0.1+x}\text{Zn}_{0.15+x}\text{Fe}_2\text{O}_4$ ($0.01 \leq x \leq 0.05$) nano-composite samples are presented. From hysteresis data, the maximum coercivity is obtained for the composition with $x=0.03$. The magnetic properties of the Nano-composite are obtained using VSM at room temperature.

II. EXPERIMENTAL PROCEDURE

This section gives the details of the synthesis of the nano composite $\text{Ni}_{0.75-2x}\text{Cu}_{0.1+x}\text{Zn}_{0.15+x}\text{Fe}_2\text{O}_4$ ($0.01 \leq x \leq 0.05$), in the Nano-composites preparation, aqueous solutions of stoichiometric amounts of Nickel nitrate, Copper nitrate and Zinc nitrate along with ferric citrate are reacted with the citric acid in 1:1 molar ratio. p^{H} of the solution is increased by the addition of ammonia to complete the reaction and Poly Ethylene Glycol (PEG) is added. The solution is evaporated very slowly over a period of eight hours to dryness. Viscosity and color are changed as the solution turned into puffy, porous dry gel. As soon as the solvent removal is completed, dried precursor goes under a self-ignition reaction to

form a very fine powder known as synthesized powder. The synthesized powder thus obtained is calcined in a muffle furnace at 600^oc for two hours to remove the residual carbon and furnace cooled. Then matter is subjected to Ball milling for two hours at speed of 450 rpm [2].

XRD patterns are obtained on Advanced diffractometer at a sweep rate 15° min⁻¹ and time constant 10 s using Cu K α_1 as the radiation source ($\lambda = 1.541841^\circ \text{A}$). SEM examination is performed using a Jeol, Z440 instrument. The magnetic properties are obtained using a VSM at room temperature with an applied magnetic field up to 15 kOe to reach study state.

III. RESULTS AND DISCUSSION

Fig.1 gives the XRD patterns of Ni_{0.75-2x} Cu_{0.1+x} Zn_{0.15+x} Fe₂O₄ (0.01 ≤ x ≤ 0.05) system a single-phase cubic spinel structure. From the XRD plot, it can be observed no undesirable secondary phases are detected. The XRD pattern is studied in detail for the determination of crystallite size by using the classical Scherrer equation [1]:

$$D = \frac{k\lambda}{\beta \cos \theta}$$

where, D is the average crystallite size, k is a constant equal to 0.89, λ is the X-ray wave length (0.1542 nm), θ is the angle of diffraction and β is the full width at half maximum (FWHM) of the peak. The average crystallite sizes of the powders are in the range 32.43– 64.88nm which indicates that the copper and zinc substitution for nickel has effect on the crystal size. The lattice parameter (a) has been calculated from X-ray data using the formula [1, 11, 15 and 20]:

$$\frac{1}{d^2} = \frac{h^2 + k^2 + l^2}{a^2}$$

Magnetic properties of Ni_{0.75-2x} Cu_{1+x} Zn_{1.5+x} Fe₂O₄ (0.01 ≤ x ≤ 0.05) system is carried out using VSM with a field scan up to ±15.0 kOe at room temperature. The hysteresis loops of the samples are shown in Fig. 3. From, the hysteresis data indicated that the maximum coercivity and remanant magnetization is obtained for the Nano-composition with x=0.03.

The saturation magnetization (M_S), remanant magnetization (M_r) and coercivity (H_c) are found to increase gradually with increasing copper and zinc content (Table 1).

The detailed Characterizations and parameter values of nano-composite (NCZ ferrite) given in the Table.1. Here, the average crystal size and curie temperature are observed.

where, d is the lattice spacing and h, k and l are the miller indices of the plane. The theoretical density or the X-ray density (D_x) is calculated according to relation [1, 12 and 14]:

$$D_x = \frac{ZM}{Na^3}$$

where, Z is the number of molecules per unit cell (Z = 8), M is the molecular weight, N is Avogadro's number and a³ is the volume of unit cell [1, 13].

The average crystallite size, lattice parameter and X-ray density, with copper and zinc content, are shown in table 1.

EM has been provided further insight into the grain sizes of the Nano-composite. Fig. 2 shows the SEM images and EDX analysis of Ni_{0.75-2x} Cu_{1+x} Zn_{1.5+x} Fe₂O₄ (0.01 ≤ x ≤ 0.05). The image shows agglomerates of with an average size of 32.4 nm.

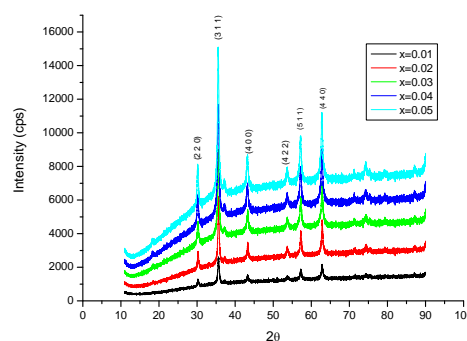


Fig.1. Obtained XRD Pattern of Ni_{0.75-2x} Cu_{0.1+x} Zn_{0.15+x} Fe₂O₄ (0.01 ≤ x ≤ 0.05)

S. NO	Parameters	X=0.01	X=0.02	X=0.03	X=0.04	X=0.05
1	d-spacing (Å)	2.511	2.516	2.528	2.527	2.525
2	Lattice strain	0.0023	0.0018	0.0031	0.0029	0.0037
3	X-ray density (D _x), g/cm ³	5.33	5.35	5.37	5.36	5.34
4	FWHM	0.1728	0.1344	0.2304	0.2112	0.2688
5	Average crystallite size (D), nm	50.47	64.88	37.83	41	32.43
6	Magnetization (M _s), emu/g	54.56	55.314	42.917	56.468	55.726
7	Retentivity (M _r), emu/g	9.2758	9.0768	9.0893	10.642	8.5975
8	Coercivity, H _c (G)	238.26	264.93	414.17	337.14	256.86
9	Curie Temperature, T _c (K)	614	621	654	635	642
10	Effective magnetic moment (μ _{eff}) μB	7.59	8.39	8.7	9.3	11.56

Table 1: Particle size and other characteristics of Ni_{0.75-2x} Cu_{0.1+x} Zn_{0.15+x} Fe₂O₄ (0.01 ≤ x ≤ 0.05)

Fig2: Obtained SEM image of $Ni_{0.75-2x}Cu_{0.1+x}Zn_{0.15+x}Fe_2O_4$ ($0.01 \leq x \leq 0.05$)

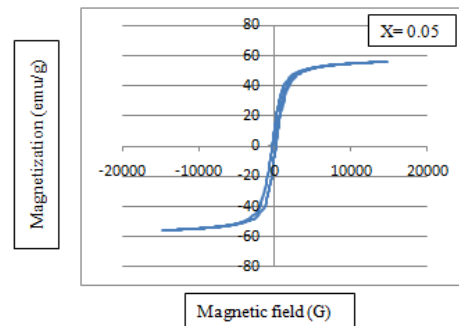
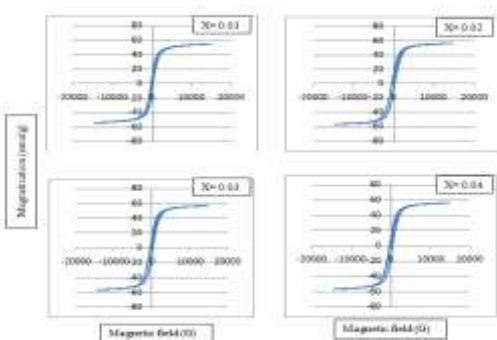
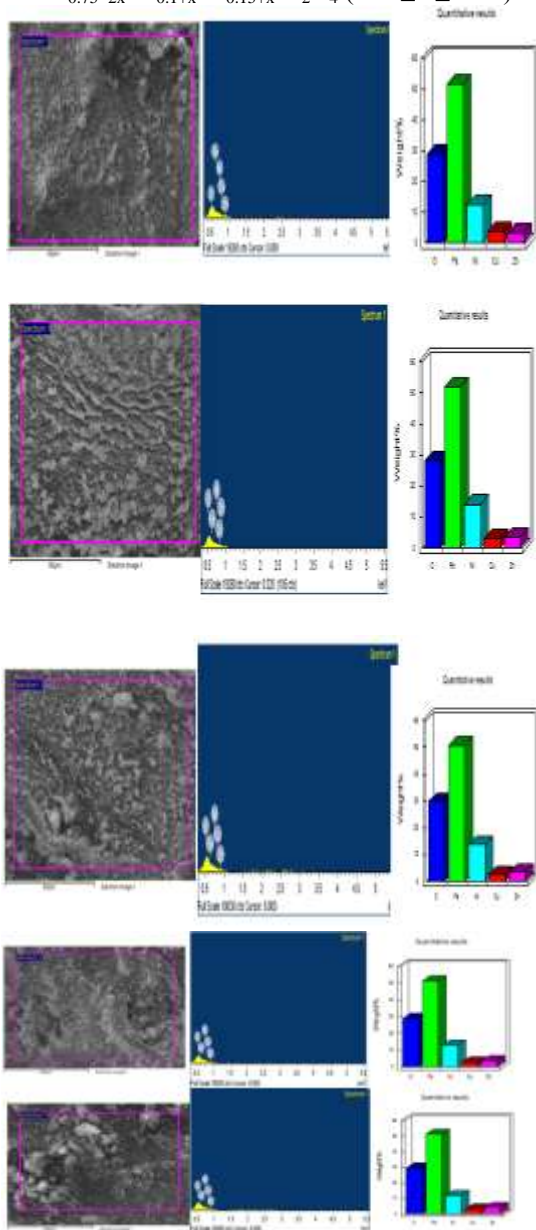


Fig.3. Magnetic hysteresis loop for $Ni_{0.75-2x}Cu_{0.1+x}Zn_{0.15+x}Fe_2O_4$ (where $x = 0.01-0.05$)

IV. CONCLUSION

Nanocrystalline $Ni_{0.752x}Cu_{0.1+x}Zn_{0.15+x}Fe_2O_4$ ($0.01 \leq x \leq 0.05$) has been successfully synthesized using Citrate Precursor Method and Ball milling for grinding the compound. The obtained powders are characterized using XRD, SEM methods. The outcomes indicate that single phase cubic ferrites are obtained after calcining the precursors at $600^{\circ}C$ for two hours. The magnetic properties of the ferrite are obtained using Vibrating Sample Magnetometer at room temperature and magnetic properties measured at different magnetic fields. The hysteresis loops has given that the maximum coercivity is occurred for the Nano-composition with $x=0.03$. An investigation of characteristics and properties, it is observed and greatly affects the magnetic properties.

Note: The authors declare that there is no conflict of interest regarding the publication of this paper.

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