



RESEARCH ARTICLE

INFLUENCE OF QUENCHING ON STRUCTURAL, SIZE AND MAGNETIC PROPERTIES OF
 $\text{Ni}_{0.5+x}\text{Zn}_x\text{Cu}_{0.5}\text{Fe}_{2-2x}\text{O}_4$ FERRITE SYNTHESIZED USING CERAMIC ROUTE

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ABSTRACT

A system of ferrites with formula $\text{Ni}_{0.5+x}\text{Zn}_x\text{Cu}_{0.5}\text{Fe}_{2-2x}\text{O}_4$ ($x=0.0,0.1,0.2,0.3,0.4,0.5$) synthesized through ceramic route. As a special consideration for optimisation the material is quenched from 1137 K to 77 K by dropping it in liquid nitrogen bath in the final stage. XRD patterns present that the crystals formed are in the polycrystalline nature. The structure of ferrite is confirmed as Face Centred Cubic (FCC) phase. The particle size of the ferrite system varies from 20 nm to 40 nm and they are nano particles. Thermal study helps to infer completion of reaction of formation of ferrite. FTIR and Raman Spectroscopy measurements confirm the presence of tetrahedral and octahedral coordination of atoms in spinel structure of ferrite. However a dominating impact of defect states intruded in the structure of ferrite is visualized through FTIR and Raman Spectra. The presence of defect states is attributed to mainly to the quenching of the ferrite by a large range of fall of temperature. The existence of defect states is expected to contribute a big variation in electrical and the magnetic properties.

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INTRODUCTION

Ferrites continue to be candidate substances which find wide applications in microwave devices, magnetic recording and computer memories (Uzgun, 2009; Goldman, 2006; Murugesan, 2005). In view of their importance several researchers have studied the thermal and magnetic properties of ferrite materials in the past few years (Tatarchuk et al., 2017; Golkhatmi, 2017; Kakade et al., 2016; Ahmad et al., 2016; Khalaf, 2016). The magnetic ordering and ferromagnetic resonance properties of Ni Zn (Kurmude et al., 2014; Boon, 2012; Kwon, 2017) and Fe Zn (Yao et al., 2007; Chinnasamy, 2000) spinel ferrites have been studied by a number of workers. These have been satisfactorily explained on the basis of Yafet-Kittel (Murthy et al., 1969) type of spin ordering on the B-sub lattice. It is well known (Kakade et al., 2016; Ahmad et al., 2016; Khalaf et al., 2016) that crystal distortion arising from John-Teller effect occurs whenever an ion like Cu^{2+} is present of the octahedral site. Smit and Wijn (1959) have systematically studied magnetic properties of the ferrites NiFe_2O_4 and CuFe_2O_4 .

They have reported that the Saturation magnetization value of NiFe_2O_4 is 300 Gauss of 0 K and its Curie temperature is 585°C. For the ferrites CuFe_2O_4 the values are 160 Gauss and 455°C respectively. Similarly Arrot and Goldman (Boon, 2002) and Hartmann-Boutron and Imbert (Hartmann-Boutron, 1968) have studied ZnFe_2O_4 magnetically and have found out that it is antiferromagnetic below 263K. Many attempts have been made to lower the curie temperature of NiFe_2O_4 and CuFe_2O_4 and for increasing the transition point of ZnFe_2O_4 there by suitably tailoring the saturation magnetization, resistivity, chemical stability and mechanical hardness (Tatarchuk, 2017; Khalaf, 2016; Guner et al., 2014; Choodamani et al., 2013; Choodamani et al., 2016). Such attempts have been made possible by mixing the end member ferrites namely NiFe_2O_4 , CuFe_2O_4 (Li et al., 2017) and ZnFe_2O_4 (Naghib et al., 2016; Yang et al., 2016; Wu et al., 2016; Zhu et al., 2016). The mixed ferrites of Ni-Zn-Cu have motivated to prepare ferrites of ternary mixtures to provide optimum magnetic properties for some specified applications. It is known that the magnetic behaviour of Ni-Cu ferrites is largely governed by Fe-O-Fe interaction and Ni-O-Fe interaction.

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In the present work it is of interest to substitute Zn ions in A-site by readjustment of Ni and Fe ions in different sites, infuse changes in interactions and novel changes in magnetic properties. Further to investigate the impact of quenching in the nature of crystallite, structure and magnetic properties. Interestingly, the developments of substructures are confirmed through FTIR and Raman measurements.

MATERIALS AND METHODS

Synthesis: A set of six samples of $Ni_{0.5+x}Zn_xCu_{0.5}Fe_{2-2x}O_4$ ($x=0.0,0.1,0.2,0.3,0.4,0.5$) has been synthesized by conventional ceramic pre-sintering method. The samples are prepared by mixing analytical reagent grade Ni_2CO_3 , Fe_2O_3 , ZnO and CuO . They are mixed and ground well in acetone using agate pestle and mortar. The powder is transferred to silica crucible and heated to 1137 K by placing it in a muffle furnace with air atmosphere. The pre-sintering of the powders and of the pellets are done at 1173K for 20 hr and furnace cooled at the rate of 100 °C/hr. The final powder is made as pellet using pelletiser with 2 tons of pressure. The final sintering is also carried out for the same high temperature, whereas the cooling is done by quenching the pellet by dropping into liquid nitrogen bath at 77 K. Hence there is a sudden fall of temperature from 1173 K to 77 K. The powder form of ferrite is subjected to structural studies using XRD, thermal, FTIR, Raman and magnetic measurements.

Characterization

The XRD spectra are recorded Analytical X-ray Diffract meter (Model: X'Pert PRO) with an X-ray radiation source of $Cu-K\alpha$ ($\lambda_{K\alpha1} = 1.54056 \text{ \AA}$; $\lambda_{K\alpha2} = 1.54439 \text{ \AA}$). The specific heat measurement for all the samples have been carried out using METTLER TOLEDO TG-DTA. The FTIR spectra are observed using the SHIMADZU 8700 FTIR spectrometer at room temperature over the range 4000-350 cm^{-1} . Raman scattering spectra are recorded using confocal micro-Raman spectrometer (Renishaw inVia Reflex) having a liquid-nitrogen cooled charge-coupled device multichannel detector (256 pixels X 1024 pixels). The measurements are carried out with an excitation wavelength of 488 nm in the temperatures range of 303 K - 503 K. The acquisition time and illumination power of the laser used for the present study are 20 s and 1mW respectively. Magnetization $M(H)$ measurement were done using a vibrating sample magnetometer (VSM) Lakshore UK.

RESULTS AND DISCUSSION

XRD study: Experimental lattice constants, molecular density, particle size and porosity $Ni_{0.5+x}Zn_xCu_{0.5}Fe_{2-2x}O_4$ ferrite system are determined by recording XRD patterns. The XRD patterns so recorded are shown in Fig1. The pattern shows well resolved peaks indicating that the ferrites are polycrystalline. They are characteristic peaks of a spinel structure which conform the phase formation indicating absence of other impurity phases. All the peaks perfectly match with the crystalline phase of Face Centre Cubic structure of $Ni_{0.5+x}Zn_xCu_{0.5}Fe_{2-2x}O_4$ ferrite (JCPD-Joint committee on powder diffraction). The observed values of structural parameters are given in Table1. in the present system of ferrite, Cu^{2+} , Ni^{2+} , Zn^{2+} and Fe^{2+} are the constituent cations. Using knowledge of site preference of the ions and the ionic size data of the respective ions as in Table.2 substitution of the ions in A and B sites of spinel structure will

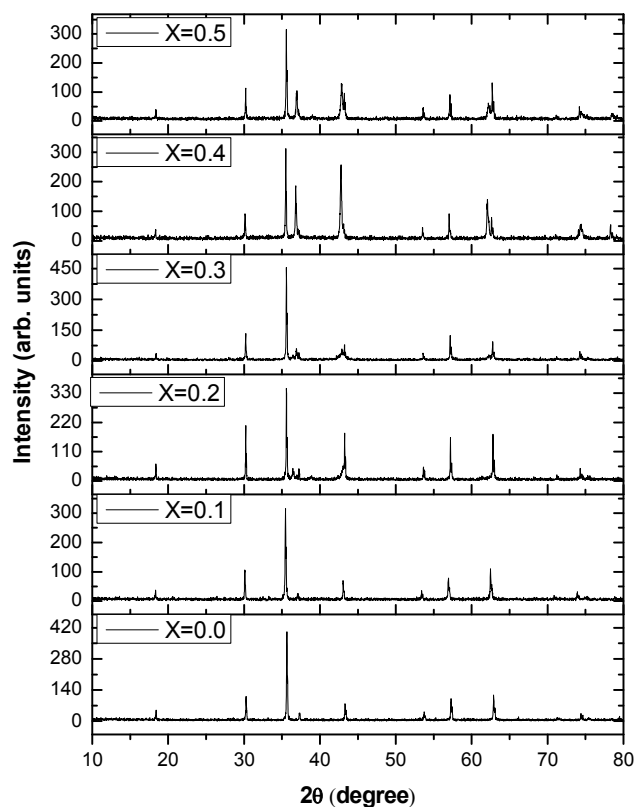


Figure 1. XRD patterns of $Ni_{0.5+x}Zn_xCu_{0.5}Fe_{2-2x}O_4$ system

be elucidated along with lattice parameter. It is seen from Table1. that the lattice constant fluctuates with Zn enhancement. It is expected to increase as the ionic size of Zn^{2+} is the largest. However the fluctuating trend might be assigned to effect of migration of ions mutually between sites and formation of ultrafine fine crystallite size. The crystallite sizes is calculated using

$$\text{Scherer's formula } D = 0.9\lambda / \beta \cos\theta$$

where, λ is the X-ray wavelength, β is the width of the peak and θ is the Bragg's position of the peaks (2 θ). The values of crystallite size evaluated are in a range from 20 nm to 40 nm, Molecular density is also indicative of fluctuating trend with Zn substitution justifying existence of vacancy sites. The high value of observed porosity is substantiation for the existence vacant sites.

Thermal study: The phase transition points are obtained by an analysis done using STAR software for powder sample taken in silica crucible. The temperature variation is set in between 100 and 900 °C in steps 20 c/min. the specific heat curves Ni-Zn-Cu mixed ferrites. The variation of specific heat flow with temperature for different concentration is shown in Fig2. The nature of specific heat curves of these ferrite samples certifies the completion of the chemical reaction meant for the preparation of ferrites. A shallow region of specific heat is a common trend observable in and around the phase transition point of these ferrites. It is expected that there is a phase transition around 550 – 570°C indicative of ferromagnetic to paramagnetic phase (Viswanathan *et al.*, 1955).

FT-IR Spectral Study: In order to conform the formation of the spinel phase the FTIR spectra of the powders were recorded and shown in Fig3. Centre frequencies of the bands of FTIR spectra of the sintered $Ni_{0.5+x}Zn_xCu_{0.5}Fe_{2-2x}O_4$ system are

Table 1. Experimental lattice constant, particle size, porosity and molecular density of sintered $\text{Ni}_{0.5+x}\text{Zn}_x\text{Cu}_{0.5}\text{Fe}_{2-2x}\text{O}_4$ ferrite system

Concentration (X)	lattice constant a (Å)	Particle size (nm)	Molecular density ρ_m (kg/m ³)	Porosity P%
0.0	8.347	25	5.5135	28.52
0.1	8.391	20	5.3514	22.33
0.2	8.360	40	5.4393	19.24
0.3	8.361	29	5.4644	21.12
0.4	8.400	24	5.2976	13.97
0.5	8.379	31	5.4858	22.01

a – Lattice constant (Å); m – Molecular density(kg/m³); nm=nanometer;P – Porosity (%)

Table 2. Centre frequencies of the bands of FTIR spectra of the sintered $\text{Ni}_{0.5+x}\text{Zn}_x\text{Cu}_{0.5}\text{Fe}_{2-2x}\text{O}_4$ system

Concentration (X)	ν_1	ν_2
0.0	453.20	775.27
0.1	495.63	744.41
0.2	503.35	740.56
0.3	543.85	732.84
0.4	518.77	725.13
0.5	522.63	773.34

ν_1 & ν_2 - Frequencies

Table 3. Centre frequencies of the bands of Raman spectra of the sintered $\text{Ni}_{0.5+x}\text{Zn}_x\text{Cu}_{0.5}\text{Fe}_{2-2x}\text{O}_4$ system

Concentration (X)	Centre frequency cm ⁻¹				
	ν_1	ν_2	ν_3	ν_4	ν_5
0.0	195	334	484	566	700
0.1	195	334	484	566	698
0.2	196	366	488	570	705
0.3	168	297	340	534	706
0.4	183	336	495	652	701
0.5	191	331	487	548	698

ν_1 & ν_2 & ν_3 & ν_4 & ν_5 - Frequencies

Table 4. Saturation magnetisation, remanent constant, Mr/Ms ratio and coercive field of $\text{Ni}_{0.5+x}\text{Zn}_x\text{Cu}_{0.5}\text{Fe}_{2-2x}\text{O}_4$ ferrite

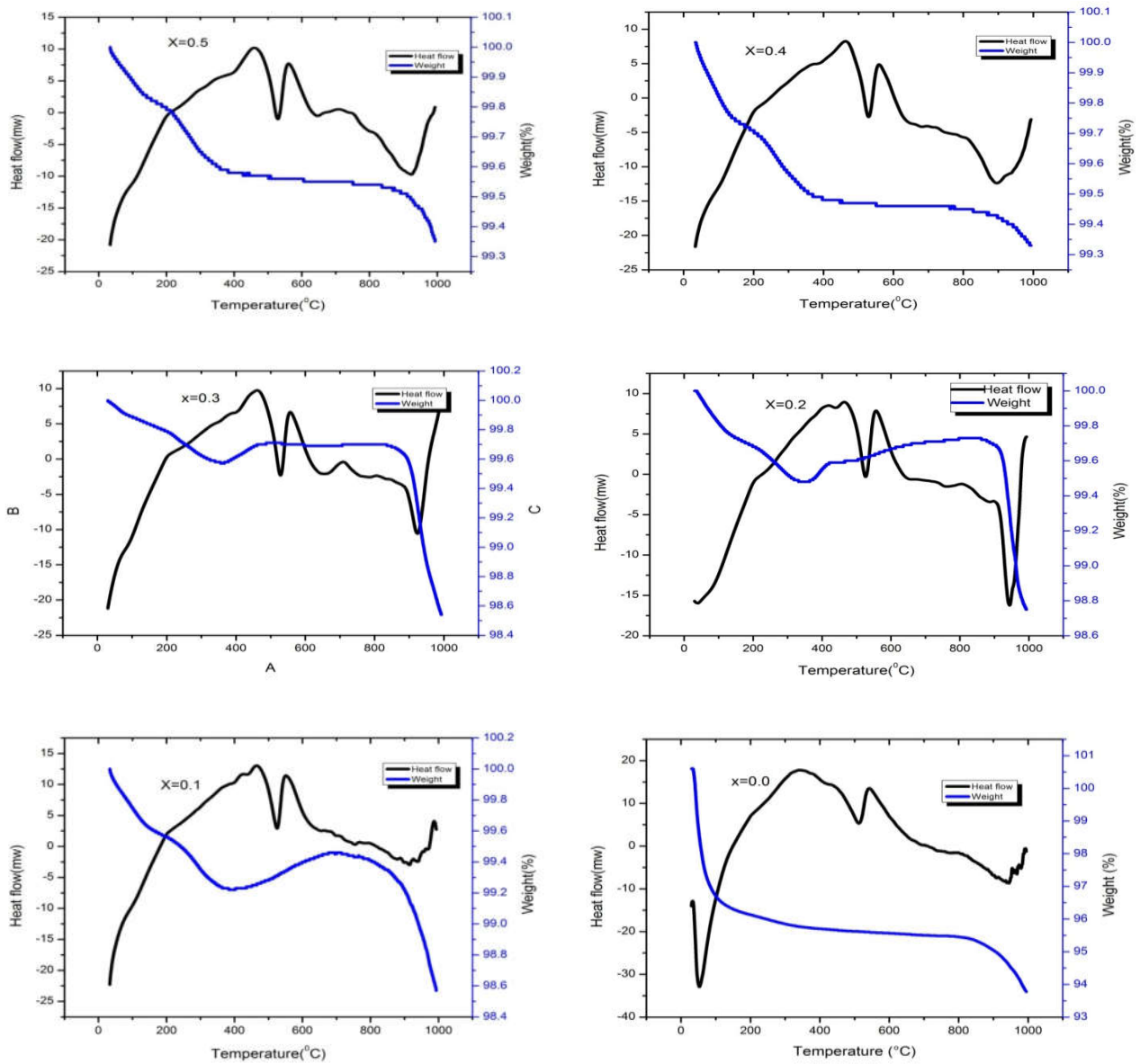
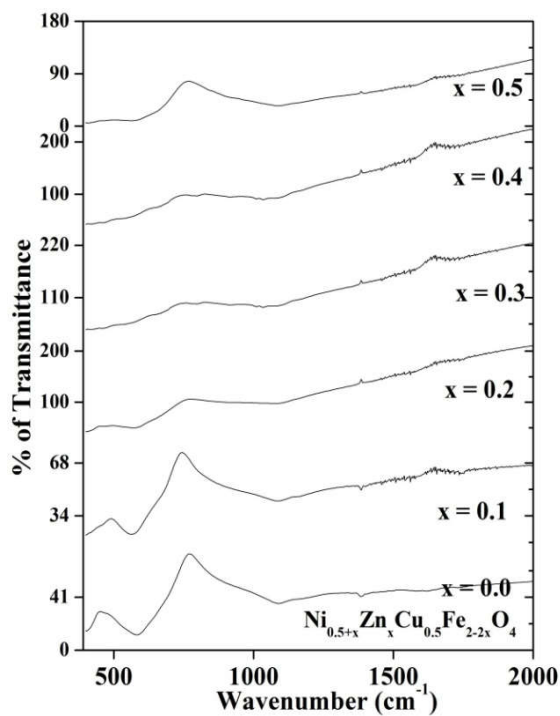
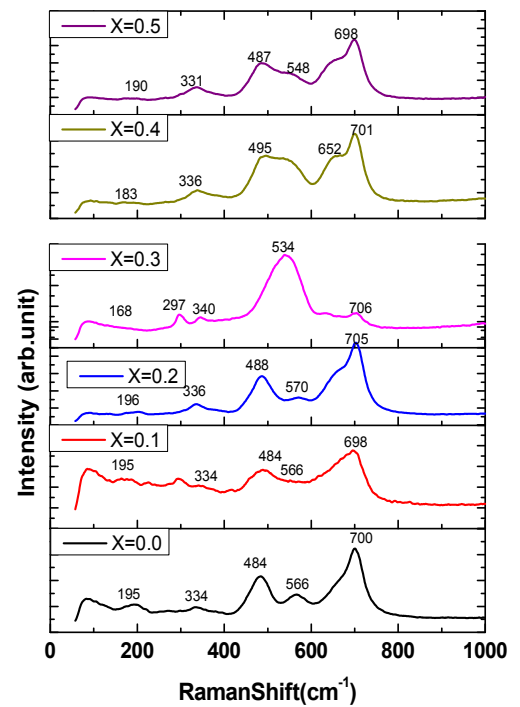
Concentration (X)	Saturation magnetisation (Ms) emu/g	Remanent magnetisation (Mr) emu/g	Mr/Ms Ratio	Coercive field (Hc) Oe
0.0	39.37	1.30	0.0332	15.51
0.1	56.17	1.72	0.0307	6.71
0.2	53.49	1.24	0.0232	18.13
0.3	54.08	1.35	0.0249	20.44
0.4	92.88	4.49	0.0483	10.75
0.5	39.12	0.94	0.0240	33.61

Ms – Saturation Magnetisation (emu/g); (Mr)- Remanent magnetization (emu/g);Hc – Coercive field (Oe); Mr/Ms - Ratio

given in the Table 2. The spectra show characteristic absorption of ferrite phase. The strong absorption around 600cm⁻¹ and weak absorption in the range 480 cm⁻¹would correspond to tetrahedral and octahedral coordination ions respectively (3). This difference in the band position is expected because of difference in the system. $\text{Ni}_{0.5+x}\text{Zn}_x\text{Cu}_{0.5}\text{Fe}_{2-2x}\text{O}_4$ ferrites for the octahedral and tetrahedral compounds. Waldron studied (Waldron, 1955). The vibrational spectra of ferrites and attributed the sharp absorption band around 600 cm⁻¹ to the intrinsic vibrations of the tetrahedral group and 450 cm⁻¹ to the other band of the octahedral group. It is clearly noticed from the spectra and Table2. that the ferrites invariably contain octahedral and tetrahedral coordination of ions which are basic composite substructures of spinel. The shift of the bands with reference to the standard values reported by Waldron (Waldron, 1955) is indicative of encroachment of different ions in the sites when Zn is substituted. Further higher values band around 700 cm⁻¹ and above should indicate the formation of certain defect states in ferrites. It is bound to occur as the post sintering process is quenching. Normally quenching may lead to amorphous or defects state formation.

Raman Spectral Study: Fig 4. shows the Raman spectra of $\text{Ni}_{0.5+x}\text{Zn}_x\text{Cu}_{0.5}\text{Fe}_{2-2x}\text{O}_4$ sample. The persistent bands are observed in the range of 50-1000 cm⁻¹. The phonon modes of present ferrites are found at 195 cm⁻¹, 334 cm⁻¹, 484 cm⁻¹, 566 cm⁻¹, 700 cm⁻¹, since modes above 600 cm⁻¹ corresponds to the tetrahedral sub-lattice and due to the motion of the oxygen atoms in tetrahedral group and below refers octahedral sub-lattice (Murugesan *et al.*, 2015). Broadening of modes observed in some Raman spectral bands is due to the strain developments and reduction in grain size (Murugesan *et al.*, 2015). Further the mass difference between the three ions in the ferrite sample namely, Ni^{2+} , Fe^{3+} and Zn^{2+} splits the mode in three different energy values. The lightest ion Ni^{2+} corresponds to 700 cm⁻¹ for x=0.2 to 0.4 the heaviest one ie Fe^{3+} correspond to 340cm⁻¹ and an intermediate mode of Zn^{2+} ions around 566cm⁻¹. The observed centre frequencies of the bands of Raman spectra of the sintered $\text{Ni}_{0.5+x}\text{Zn}_x\text{Cu}_{0.5}\text{Fe}_{2-2x}\text{O}_4$ system are given in the Table 3.

Magnetic Study: The experimental magnetic hysteresis loops observed are shown in Fig5. The saturation magnetisation,

Figure 2. Thermographs of $Ni_{0.5+x}Zn_xCu_{0.5}Fe_{2-2x}O_4$ systemFigure 3. FTIR spectra of $Ni_{0.5+x}Zn_xCu_{0.5}Fe_{2-2x}O_4$ systemFigure 4. Raman spectra of $Ni_{0.5+x}Zn_xCu_{0.5}Fe_{2-2x}O_4$ system

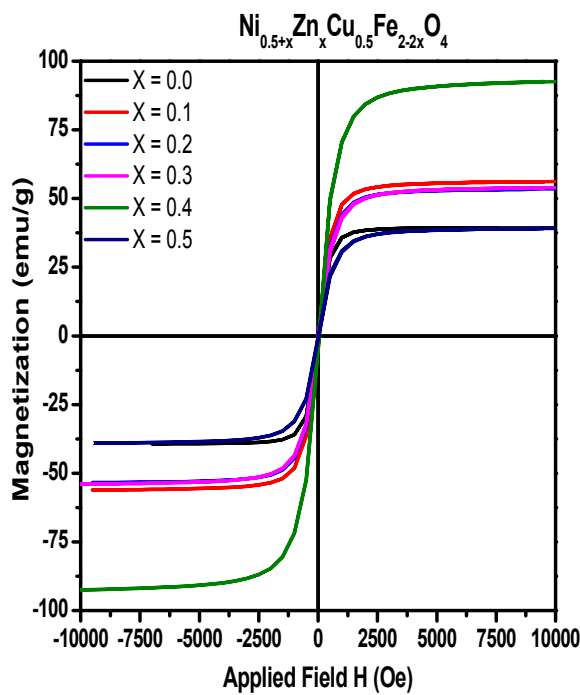


Figure 5. Magnetic hysteresis loop of $\text{Ni}_{0.5+x}\text{Zn}_x\text{Cu}_{0.5}\text{Fe}_{2-2x}\text{O}_4$ system

remanent magnetisation, M_r/M_s ratio and Coercive field are determined. The values of the magnetic parameters are given in Table 4. The shape of hysteresis loop renders explanation for the nature of a magnetic domain and type of the magnetic material (Spaldin, 2010). According to the fairly well established consideration the loops obtained for the $\text{Ni}_{0.5+x}\text{Zn}_x\text{Cu}_{0.5}\text{Fe}_{2-2x}\text{O}_4$ quenched system i.e., in Fig5. are indicative of multi-domains (Cullity, 2011). The value of coercive field varies between 6.714 Oe and 33.61 Oe. It is seen from the Table.4 that the coercive field is found to be fairly low and there is no much change with respect to concentration of Zn. Low value of coercive field is effected due to soft nature of the ferrite. The values of remanent ratio ranges from 0.0240 to 0.0332. Therefore it is understood that the ferrite prepared in the present study have multi-domain (Kwon, 2017; Cullity *et al.*, 2011). The saturation magnetisation (M_s) values range from 39 emu/g to 92 emu/g. These values are in good agreement with the values reported by (Murugesan *et al.* (Murugesan *et al.*, 2015) Gawas *et al.* (2016) Erdemi Baykal *et al.* (2015) for the similar types of ferrite. In these system the magnetic order is mainly due to an indirect exchange interaction mechanism occurring between the metal ions in the A and B sub lattices. The substitution of Zn^{2+} ions, which has a preferential A site occupancy results in the reduction of the exchange interaction between A and B sites. Hence magnetic properties of the particle can be varied by varying the degree of zinc substitution (Neel, 1952). These the present study indicates a possibility for A-B and B-B exchange interaction. As the crystallites are in nano size. However Mossbauer study would give clear information about the magnetic structure of the ferrite to understand in terms of Neel's two sub lattice models and canting of a spin.

Conclusion

In the present work the $\text{Ni}_{0.5+x}\text{Zn}_x\text{Cu}_{0.5}\text{Fe}_{2-2x}\text{O}_4$ system of ferrites is synthesised in solid state route. However interestingly the quenching of the samples in liquid nitrogen bath during second sintering is expected produce novelties. XRD measurements on

lattice parameters show the fluctuating changes for Zn substitution. FTIR and Raman studies confirm spinal structure of the ferrites prepared and show widening of bands indicating the defect states due to quenching. Magnetic studies explain the changes of magnetisation in terms of change in the exchange interaction as there is unusual distribution of ions in sites due to the quenching of ferrites sample. Thus quenching along with wild grinding of sample during synthesis leads to formation of ultra-fine crystallites which causes amusing change in magnetic properties. The results suggest that the process of quenching after second sintering can be attempted for preparation of nano size crystallites. Ultimately it is expected to bring in novel properties as evidenced in the present work.

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