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Structural and Magnetic investigation of Cu^{2+} substituted Mn-Zn spinel ferrite synthesized using hydrothermal route

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Abstract:

Spinel Ferrites are investigated for different applications in the field of electronics such as information storage devices. The properties of these spinel ferrites vary with the changes in the different compositional elements. With this aim super paramagnetic $Mn_{0.25}Zn_{0.5}Cu_{0.25}Fe_2O_4$ and $Mn_{0.2}Zn_{0.5}Cu_{0.3}Fe_2O_4$ ferrite was successfully prepared by using a hydrothermal approach at temperature of 180°C for 20 hrs. The resulting compounds shows different structural and magnetic evolution as inspected from various analytical techniques such as XRD, FTIR, VSM. XRD. The impact on the crystallite size calculated using Debye Scherrer's formula for (311) reflection is discussed in results.The VSM gives magnetic parameters of synthesized SFNPs with a smooth S-shaped MH-curve representing the super paramagnetic nature with minimum squareness ratio 0.13 and 0.08, indicating pseudo-single domain in nature.

Keywords: Hydrothermal approach; Super paramagnetic; Spinel ferrite nanoparticles; XRD; VSM, nanotechnology, High Frequency applications, MRI

1 Introduction:

Spinel ferrites are a class of magnetic materials with the chemical formula AB2O4. These materials have unique magnetic, electrical, and structural characteristics, which make them highly desirable for a range of applications. Soft spinel ferrites, in particular, are of great interest because they contain divalent metal ions such as copper, manganese, nickel, zinc, and cobalt.

The applications of spinel ferrites are widespread and diverse. For instance, they are used in magnetic data storage devices such as hard drives and magnetic tapes. They are also used in microwave devices such as circulators and isolators, as well as in microwave absorbers for radar and communication systems. Additionally, spinel ferrites find applications in the medical field as contrast agents for magnetic resonance imaging (MRI) and in the production of magnetic nanoparticles for drug delivery systems [1-4].

Overall, spinel ferrites have become increasingly important in a range of fields due to their unique properties and diverse applications. Following the short overview of the application of spinel ferrites.

a) Magnetized Instruments:
   - Magnetic Recording: Because spinel ferrites have a high magnetic coercivity and stability, they are frequently employed in magnetic recording medium for hard drives and other storage devices.
   - Due to its exceptional magnetic qualities, magnetic sensors are used in transducers and sensors, which helps to create sensitive magnetic field detectors.

b) The catalytic process:
   - Chemical Reaction Catalysts: Because of spinel ferrites' catalytic activity and stability in a range of reaction conditions, they have been investigated to utilize their ability to increase some catalytic processes' efficiency.

c) Electronics:
   - Magnetic and Microwave Devices: Spinel Ferrites are very useful to design components for microwave devices, such as circulators and isolators. Their unique magnetic properties make them suitable for these applications.
   - Magnetic Resonance Imaging (MRI): In the medical field, certain spinel ferrites are employed as contrast agents in MRI due to their magnetic characteristics.

d) Magnetostrictive Materials:
   - Actuators and Sensors: Spinel ferrites, particularly those with specific compositions, exhibit magnetostrictive properties, making them useful in actuators and sensors that respond to changes in magnetic fields.

e) Electromagnetic Interference (EMI) Shielding:
   - EMI Shielding Materials: Spinel ferrites are employed in EMI shielding applications, providing effective protection against electromagnetic interference in electronic devices.

f) Biomedical Applications:

- Hyperthermia Treatment: Spinel ferrite nanoparticles, which are used to treat hyperthermia, are injected into the targeted tissues once an external magnetic field is created. The treated area experiences localized hyperthermia as a result of the magnetic nanoparticles absorbing energy from the field and creating heat. There may be applications for this phenomenon in cancer treatments to treat hyperthermia. [5].

Spinel ferrite nanoparticles with high surface-to-volume ratio are synthesized by using different methods to investigate their electrical and magnetic properties significant in production of magnetic fluid, storage devices and medical diagnosis [6,7]. There are different methods of synthesis which can be categorised as wet methods such as co-precipitation [8], hydrothermal synthesis [9], micro-emulsion synthesis [7] and solgel method [10] and dry method including grinding [11], mechanical alloying [12] and thermal plasma methods [13].Apart from these methods, very few attempts have been made to apply the hydrothermal/solvothermal strategy.

In this research work, $Mn_{0.25}Zn_{0.5}Cu_{0.25}Fe_2O_4$ and $Mn_{0.2}Zn_{0.5}Cu_{0.3}Fe_2O_4$ SFNPs were synthesized at 180˚C by hydrothermal approach. These spinel nanoparticles are investigated to elaborateon how the Cu^{2+} substitution affects various parameters among which structural, spectroscopic, and magnetic parameters are of key interest. The detailed observations are obtained from X-ray diffraction, Fourier transforms infrared analysis, and vibrating sample magnetometer.

3. Experimental and characterization tools:

Two samples of SFNPs with chemical compositions as $Mn_{0.25}Zn_{0.5}Cu_{0.25}Fe_2O_4$ and $Mn_{0.2}Zn_{0.5}Cu_{0.3}Fe_2O_4$, were prepared by hydrothermal method. The prepared samples have been referred to as SD-1 and SD-2, respectively, hereinafter in the text and graphical representation. The samples are prepared using following analytically pure (AR) grade $MnCl_2$,$ZnCl_2$, $CuCl_2.2H_2O$ and $FeCl_3$ as precursor materials in stoichiometric amounts with the ratio of 1:2 molar solution for divalent to trivalent metal chlorides. Divalent and trivalent metal chlorides were stirred in beakers with 40 ml of double-distilled water to form an aqueous solution, while metal ion precursors were stirred with NaOH up to a pH level of 12. The mixture was refluxed at 80°C for 45-50 minutes, then transfused into a Teflon-coated autoclave. The precipitated MnZnCu SFNPs were filtered and dried at 70°C using a mortal-pestle method.

The X-ray diffraction (XRD) of the prepared sample is done to get detail insight of their crystal structure, size, phase composition, and orientation.The PAN Analytical X'pert PRO diffraction system and Brucker diffract meter were used to record XRD details, while Fourier Transform Infrared Spectroscopy (FTIR) was used to identify functional groups. Vibrational spectra of the samples were recorded using a Perkin Elmer instrument to study saturation magnetization, coercivity, remnant magnetization, and other critical parameters. Lakeshore 7400 series models were utilized for sample characterisation.

4. Results and Discussion:

3.1 XRD

Crystal identification and structural analysis for the $Mn_{0.25}Zn_{0.5}Cu_{0.25}Fe_2O_4$ and $Mn_{0.2}Zn_{0.5}Cu_{0.3}Fe_2O_4$ ferrites, obtained from the XRD pattern is illustrated in **Figure 1**.This pattern consists of a predominantly pure spinel phase diffraction pattern (JCPDS card # 88-1935 & # 74-2082.) with spacing group Fd-3m [14]. The traces of detectable secondary phases of α-Fe_2O_3/Mn_2O_3 were observed in both synthesized samples represented by * which are more favourable in $MnFe_2O_4$ and substituted $MnFe_2O_4$ [15]. Ferrite's peak appears in the locations indexed representing (220), (311), (400), (422), (511), and (440). The angle range of scattering (2θ) was preferred from 20° to 80° [16]. The obtained values of scattering angle (2θ), peak broadening (β), Lattice parameter(a), crystallite size (D), Strain ($\mathcal{E}$) for SD-1, SD-2 SFNPs for most intense (311) reflection have been listed in **Table 1**.

Table 1: Structural parameters calculated using XRD

Sample Name	(2θ)	(β)	(a) (A^o)	(D) (nm)	($\mathcal{E}$)*10^{-3}	(δ) (lines/m^2)*10^{15}	ρ_{X-ray} ' gm/ cm^3
SD-1	35.55	0.47	8.3708	17.7602	6.3937	3.1703	5.3894
SD-2	35.50	0.42	8.3817	19.8704	5.7221	2.53215	5.3781

The lattice dimension (a) of synthesized SFNPs:

$$a = d_{hkl}\sqrt{h^2 + k^2 + l^2} \ldots. (1)$$

The crystallite size estimated by the Scherer equation:

$$D = K\lambda/\beta cos\theta \ldots\ldots\ldots.. (2)$$

Where *D* is the crystallite size, λ is the X-ray wavelength of $Cu_{K\alpha}$; K is 0.9. Full width at half maximum (FWHM) (β) is the peak width of the most intense diffraction peak (311) profile at half maximum height. The calculated crystallite size corresponds to most intense (311) peak of the synthesized SFNPs are found to be 17.7602 nm and 19.8704 nm for SD-1 and SD-2 samples, respectively.

The following relation can be used to compute the induced micro-strain and dislocation density in the sample to assess the size enhancement of crystallites with Cu^{2+} substitution [17].

$$\textit{The}\ \text{microstrain}(\varepsilon) = \frac{\beta}{4\tan\theta} \qquad (3)$$

The dislocation in the lattice site can be estimated by the relation.

$$\textit{Dislocation density}(\delta) = {}^{1}\!/_{D^2} \qquad (4)$$

The X-ray density of synthesized SFNPs:

$$\rho_{X-ray} = \frac{ZM}{N_A a^3} \qquad (5)$$

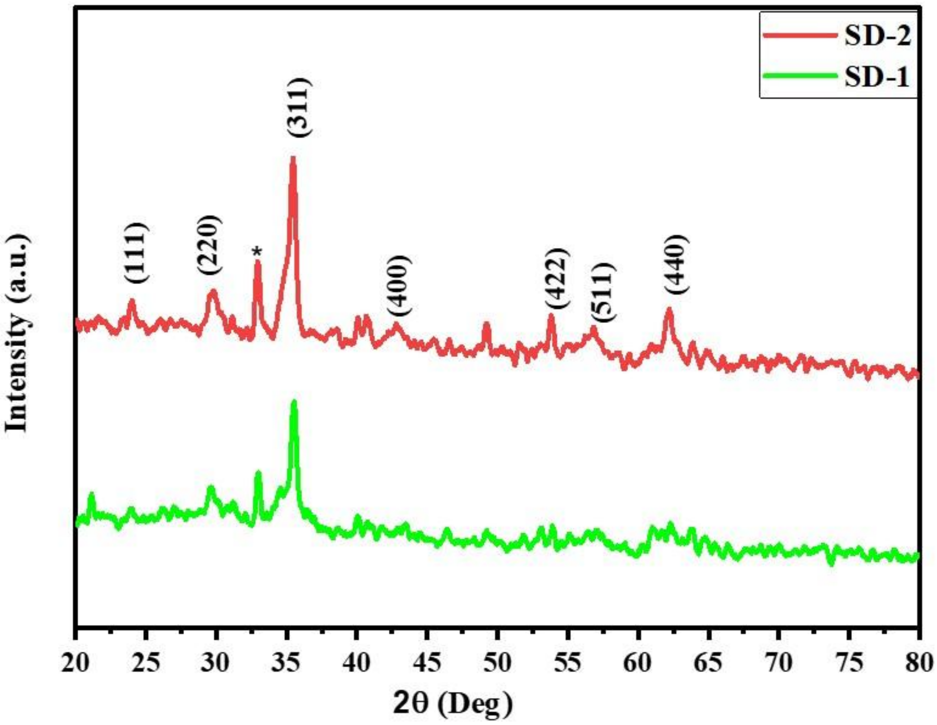

Figure 1 X-ray diffraction pattern for synthesized samples SD1 and SD2 SFNPs.

Table 1 provides a detailed analysis of the structural properties of a sample, which includes X-ray density, dislocation density, and induced microstrain. The findings of the study suggest that the structural parameters of the sample are highly consistent with the previous research conducted on cubical spinel ferrites composed of manganese and zinc[18-20].

3.2 Vibrational Spectroscopy:

Fourier Transform Infrared (FTIR) analysis is a type of spectroscopy that uses infrared light to identify the various organic, inorganic, and polymeric components in a given sample. This technique is widely used in many fields, such as chemistry, biology, and materials science, to identify additives, oxidation, decomposition, new chemicals, and contaminants in various samples.In addition to this, the FTIR analysis is also useful in studying the structural properties of certain materials, such as ferrite spinel formations. Metal ions occupy tetrahedral and octahedral sites in these formations, which makes them an ideal candidate for this type of analysis.

The FTIR spectra of $Mn_{0.25}Zn_{0.5}Cu_{0.25}Fe_2O_4$(SD-1) and $Mn_{0.2}Zn_{0.5}Cu_{0.3}Fe_2O_4$(SD-2), were recorded at ambient temperature. The spectra were depicted in **Figure 2** and covered a range of wave numbers from 400 to 4000 cm^{-1}.Two primary absorption bands (v_1 and v_2) were visible in the spectra, close to 600 cm^{-1} and 450 cm^{-1}. These bands' emergence in these ranges provided evidence that the spinel phase structure had formed [23]. The lower frequency band (v_2) is observed at 469.3 cm^{-1} and 501.9 cm^{-1} and is attributed to the vibration on octahedral site for SD-1 and SD-2 ferrite [24], as noted in **Table 2**. The high frequency band (v_1) is observed at 567.5 cm^{-1} and 564.6 cm^{-1} for SD-1 and SD-2 assigned to the intrinsic lattice vibration of the Fe^{3+} -O^{2-} on tetrahedral site. The strength of the bond in the sample is measured using a force constant K which is obtained by,

$$\boldsymbol{K = 4\pi^2 v^2 C^2 \mu}$$

Where, K is force constant, C is speed of light, ν Is wave number, μ is reduced mass of Fe-O bond. The observed force constant on tetrahedral (K_t) and octahedral (K_o) sites is (2.3607 * 10^2N/m, 2.3371*10^2N/m) and (1.6145*10^2 N/m, 1.8464*10^2 N/m) for SD1 and SD2 which match with earlier reports. The force constant value varies due to changes in positional band and induced stresses in samples [25].

Debye temperature (θ_D) impacts thermodynamic properties like mean square atomic displacement, specific heat, melting temperature, vibrational energy, and elastic constant[26].

$$\theta_D = \frac{(h\ C\ V_{av})}{2\pi K}$$

There is slight increase in θ_D, which suggests enhancement of the lattice vibrations and the obtained values of are consistent with previously published results [27-30].

Table 2 Wavenumber, force constant and Debye temperature of synthesized SD-1 and SD-2 SPNPs

Sr. No	Sample name	ν_1 (cm^{-1})	ν_2 (cm^{-1})	K_t (N/m) * 10^2	K_o (N/m) * 10^2	Average Force Constant $K_{av} = \frac{(K_t + K_o)}{2} * 10^2$	(θ_D) (Kelvin)
1	SD-1	567.5	469.3	2.3607	1.6145	1.9876	118.91
2	SD-2	564.6	501.9	2.3371	1.8464	2.0917	122.321

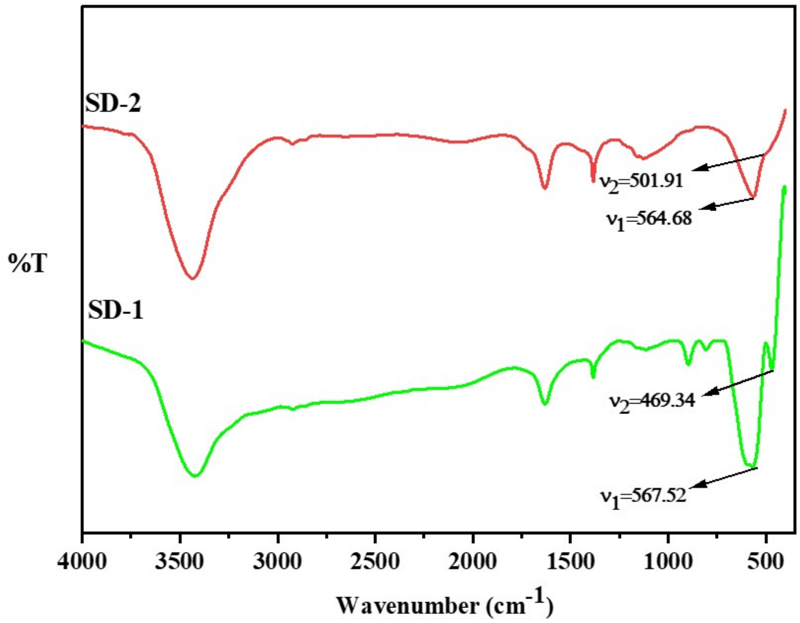

Figure 2 FTIR spectra of synthesized SD-1 and SD-2 SFNPs

3.3 Magnetic study

By analyzing the magnetic behavior of Cu-substituted MnZn spinel ferrite nanoparticles with a vibrating sample magnetometer, these materials can be tailored and optimized for a variety of technological uses. This technique offers an in-depth understanding of the magnetic characteristics of the nanoparticles.

The VSM study revealed a weak ferromagnetic nature of synthesized nanoparticles, with shape and size dependent magnetic properties like coercivity, saturation magnetization, and remnants magnetization. Ferrites have magnetic properties due to super exchange coupling [31].

The study examined the magnetic properties of synthesized spinel Ferrites using VSM, revealing their magnetic hysteresis loops, which are influenced by size, shape, and synthetic conditions as shown in figure 3 [32].

The study analyzed the magnetic properties of $Mn_{0.25}Zn_{0.5}Cu_{0.25}Fe_2O_4$(SD-1) and $Mn_{0.2}Zn_{0.5}Cu_{0.3}Fe_2O_4$(SD-2), finding soft ferrimagnetic behavior with typical S-shaped loops and a low coercive field, indicating a super paramagnetic nature. **Table 3** indicates other parameters such as saturation magnetization, remnant magnetization, coercivity, and squareness ratio.

The anisotropy constants (K) and Magnetic moment or Bohr magneton number (μ_B) are calculated using the following relation [34]

$$K = \frac{H_C M_S}{0.96}$$

$$\eta_B = \frac{M.M_S}{5585}$$

Where, η_B is the magnetic moment (μ_B), Hc is coercivity, M is the molecular weight of the composition, M_S is the saturation magnetization.

Table 3 Magnetic parameters for synthesized SFNPs.

Sample	Hc (Oe)	Mr (emu/g)	Ms (emu/g)	K (erg/cm^3)	SQR	(μ_B)
SD-1	67.63	1.92	14.75	1039.10	0.13	0.63
SD-2	37.16	1.44	16.64	644.10	0.08	0.71

The study reveals that the material is anisotropic, rigid, and single domain when the squareness ratios are large (0.5 <Mr/MS<1). The nanoparticles, which have squareness ratios of 0.13 and 0.08 for the synthesized samples SD-1 and SD-2, are pseudo-single domain in nature. This suggests that the synthetic spinel nano-ferrites have low remanence, coercivity, and superparamagnetic properties, making them potentially useful for biomedical applications [35-36].

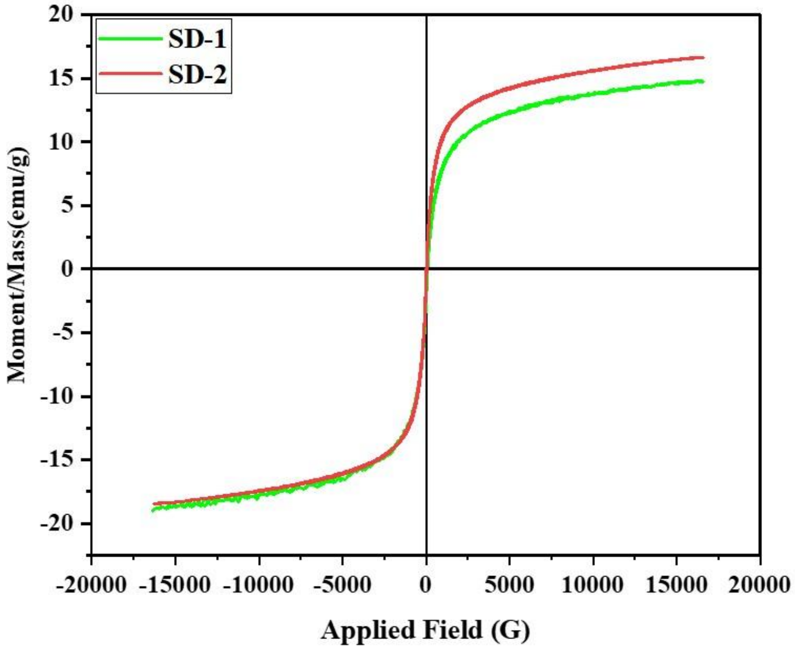

Figure 3: M-H Loop obtained using VSM for synthesized SFNPs

1. Conclusion:

In the present research module, $Mn_{0.25}Zn_{0.5}Cu_{0.25}Fe_2O_4$ and $Mn_{0.2}Zn_{0.5}Cu_{0.3}Fe_2O_4$were successfully synthesized by hydrothermal method. The structural, optical and magnetic behaviour of hydrothermally synthesized nanomaterial has been successfully studied using XRD, FTIR and VSM characterization tools. The spinel structure of hydrothermally synthesized material with pH-12 was confirmed by XRD and FTIR with the crystallite size corresponding to sot intense (311) reflection peak 17.76 nm and 19.87 nm calculated using Scherrer's equation. The magnetic properties of $Mn_{0.25}Zn_{0.5}Cu_{0.25}Fe_2O_4$ and $Mn_{0.2}Zn_{0.5}Cu_{0.3}Fe_2O_4$has been carried out using VSM. The saturation magnetization has inverse dependence with coercivity and the same behaviour has been observed. The negligible value of coercivity, retentivity and squareness ratio indicates the super paramagnetic and isotropic nature of the synthesized nanomaterial. The observed and calculated parameter clearly indicates that these materials may find high density information storage and biomedical applications.

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