

Structural, Morphological, and Magnetic Characterization of Green-Synthesized Cr^{3+} Substituted Mg-Zn Ferrites

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Abstract :

The study reports, the green synthesis of lime juice mediated Cr^{3+} doped $\text{Ni}_{0.8-x}\text{Cu}_{0.3}\text{Cr}_x\text{Fe}_2\text{O}_4$ ferrite nanoparticles ($x = 0.0, 0.2, 0.4, 0.6, 0.8$). The structural, functional, morphological, magnetic properties of the sample were investigated using X-Ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), Scanning electron microscopy (FESEM), energy-dispersive x-ray spectroscopy (EDX), vibrating sample magnetometer (VSM). The XRD studies reveals that the formation of spinal cubic crystal structure. The lattice constant decrease from $8.42 - 8.36 \text{ \AA}$. The crystalline size lies between 36 to 42 nm. FTIR analysis exhibit two strong band in region $508 - 592 \text{ cm}^{-1}$ and $403 - 470 \text{ cm}^{-1}$. The morphological images from FESEM shows the particles are well distributed and spherical in shape with having grain size between 38 to 56 nm. EDX confirm the only element present in the peaks having purity of the sample. VSM measurement estimated from M-H loop the M_s value increase with Cr^{3+} substitution.

1.1 Introduction

In the past years, the nanosized materials have explored due to their unique properties. The magnetic nanomaterial's have a high number of application such as Transformer cores, magnetic storage, magnetic resonance imaging (MRI) contrast agent, drug delivery, catalysis. In these nanomaterial's innovative work is going on the Cr doped ferrite resulting the improved structural and magnetic properties [1,2]. It is well known that doping ions effect on the magnetic behavior of the nanoparticles [3]. Nanomaterial can synthesized using various chemical and physical methods such as milling, electrochemical, photochemical, solgel, and hydrothermal method [4,5]. These methods have many disadvantages while preparation of the nanomaterial's [6]. Apart from this synthesis by using natural resources is safe and environmental friendly by using biological methods [7]. Tremendous work is attempted with green synthesis due to safe and ecofriendly approach in the preparation of nanoparticles. In the present work structural, morphological and magnetic properties of lime juice mediated Cr^{3+} Doped $\text{Ni}_{0.8-x}\text{Cu}_{0.3}\text{Cr}_x\text{Fe}_2\text{O}_4$ Ferrite nanoparticles is reported.

2.1 Preparation of lime juice

Fresh lemon collected from Jadhav Mandi Aurangabad. Lime juice squeezed out and pulp removed and Centrifuged for 15 minute for 5000 rpm. Then filtered with filter paper. The filtered lime juice used for the experiment.

2.2 Chemicals

Nickel nitrate $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, Copper nitrate $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$, Chromium nitrate $\text{Cr}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, Ferric nitrate $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ and double distilled water were purchased from Atmaja chemicals Aurangabad with A grade (99%) purity.

2.3 Synthesis of $\text{Ni}_{0.8-x}\text{Cu}_{0.3}\text{Cr}_x\text{Fe}_2\text{O}_4$

All Chemicals are dissolved in different beakers with 50 ml double distilled water and 50 ml lime juice added in 500 ml BOROSIL glass beaker and placed on the magnetic stirrer with hot plate. All chemicals are added drop wise with constant stirring. Temperature increased up to 300°C for 3 hours then after formation of fluffy powder dried in oven at 200°C for 1 hour. Then grinded in agate motor and calcined at 800°C . The obtained the final product used for the characterization. The green synthesized samples are denoted as $\text{Ni}_{0.8-x}\text{Cu}_{0.3}\text{Cr}_x\text{Fe}_2\text{O}_4$ ferrite nanoparticles ($x = 0.0, 0.2, 0.4, 0.6, 0.8$).

3.1 XRD Analysis

Figure 2. shows the xrd diffraction pattern for lime juice mediated Cr^{3+} Doped $\text{Ni}_{0.8-x}\text{Cu}_{0.3}\text{Cr}_x\text{Fe}_2\text{O}_4$ ($x = 0.0, 0.2, 0.4, 0.6, 0.8$). XRD pattern confirms the spinal cubic structure. The interplanar spacing (d) are well matched with JCPDS card number 96-153-2574. The hkl planes observed in the pattern are (220), (311), (400), (422), (333) and (440) belongs to spinal cubic crystal structure having is group $\text{Fd}3\text{m}$. The lattice constant calculated using interplanar spacing d and hkl values by following relation [9],

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

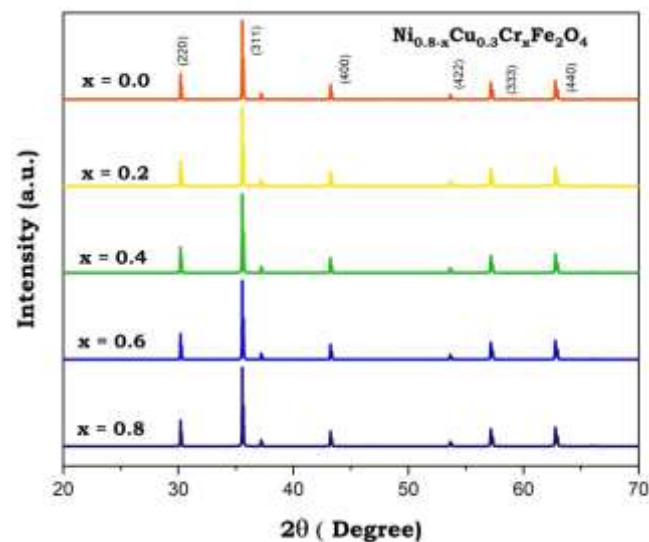


Figure 2. XRD pattern of green synthesized $\text{Ni}_{0.8-x}\text{Cu}_{0.3}\text{Cr}_x\text{Fe}_2\text{O}_4$

The average crystalline size of lime juice mediated $\text{Ni}_{0.8-x}\text{Cu}_{0.3}\text{Cr}_x\text{Fe}_2\text{O}_4$ samples are calculated using the X-ray wavelength (λ), FWHM (β) and diffraction angle (θ) and hkl plane (311) was selected to measure the crystalline size using the following equation [10],

$$D = \frac{k\lambda}{\beta \cos \theta} \dots\dots\dots (2)$$

X-Ray density and Bulk density of the are calculated using the molecular weight of the sample (M_w), Avogadro's number (N_A), lattice parameter (a), mass and volume of the sample using following equations [11],

$$d_x = \frac{8M}{N_A a^3} \dots\dots\dots (3)$$

$$d_B = \frac{\text{mass}}{\text{volume}} \dots\dots\dots (4)$$

the values of crystallite size D , lattice constant a , x-ray density, Bulk density and grain size are listed in table 1 .

Table 1. Lattice parameter (a), crystallite size (D), X-ray density (d_x), Bulk density (d_B), and grain size of $\text{Ni}_{0.8-x}\text{Cu}_{0.3}\text{Cr}_x\text{Fe}_2\text{O}_4$ Ferrite samples

x	a (Å)	D (nm)	d_x (g/cm ³)	d_B (g/cm ³)	Grain size
0	8.4256	36.52	5.3948	4.7655	38.45
0.2	8.4082	37.42	5.3921	4.7865	39.41
0.4	8.3906	40.65	5.3908	4.7892	46.52
0.6	8.3858	41.86	5.3838	4.7920	52.52

0.8	8.3654	42.94	5.3746	4.7964	56.21
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3.2 FTIR Analysis

The formation of functional group have confirmed the factors spectrum of the sample showing in figure 3. Two metal oxygen are observed in the range between 508- 592 Cm^{-1} and 403 - 470 Cm^{-1} with metal stretching at tetrahedral site A and Octahedral site B [12].

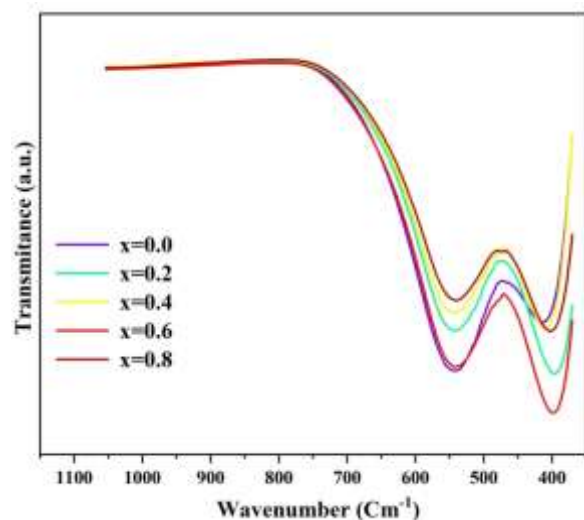
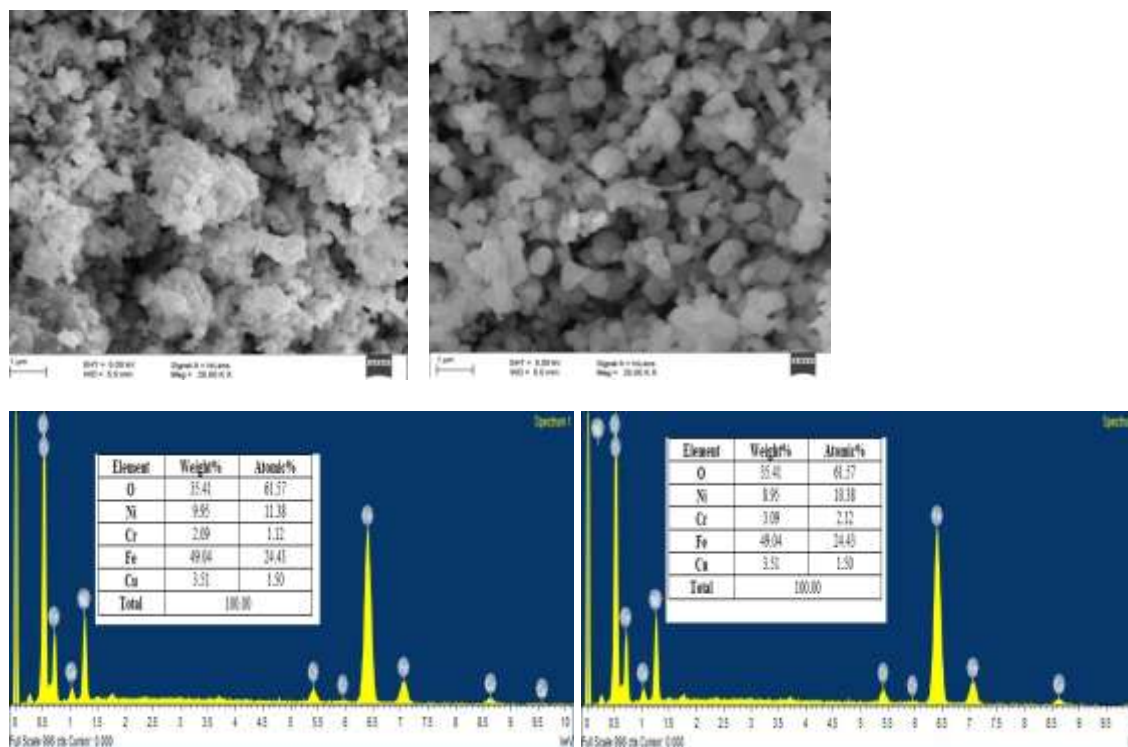


Figure 4 (a). FTIR spectra of of lemon juice mediated $\text{Ni}_{0.8-x}\text{Cu}_{0.3}\text{Cr}_x\text{Fe}_x\text{O}_4$ ($x = 0.0, 0.2, 0.4, 0.6, 0.8$)

3.3 FESEM Analysis

The morphological images are shown in figure 5. shows the spherical shaped with well distributed nanoparticles having the grain size 38 to 56 nm shown in table 1. The EDX pattern contain Ni, Fe, Cu, Cr, O and no extra peak confirming the purity of the samples [13].



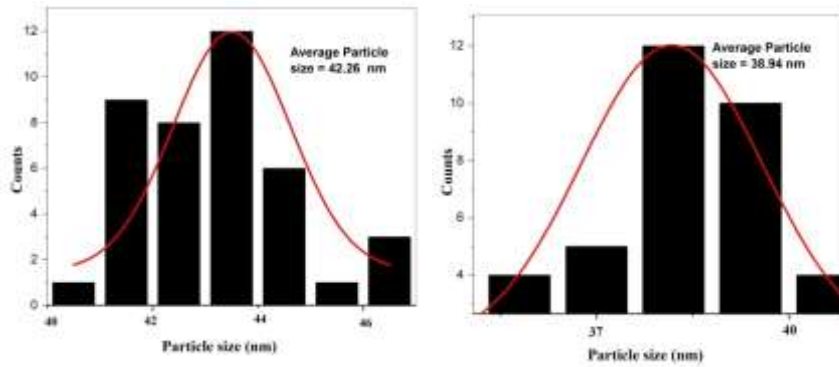


Figure 5 (a). FESEM images showing the microstructures (b) Energy dispersive X-ray Spectra.(c) Particle size distribution of $\text{Ni}_{0.8-x}\text{Cu}_{0.3}\text{Cr}_x\text{Fe}_2\text{O}_4$ ferrite nanoparticles ($x = 0.2, 0.4$).

3.4 VSM Analysis

Figure 5. shows the M-H loops for the lime mediated $\text{Ni}_{0.8-x}\text{Cu}_{0.3}\text{Cr}_x\text{Fe}_2\text{O}_4$ ($x = 0.0, 0.2, 0.4, 0.6, 0.8$) samples. The Hysteresis loop plotted in figure at room temperature 15K Oe. From M-H loop saturation magnetization (M_s), coercivity (H_c), retentivity (M_r) is taken. From this the remanance ratio (M_s/M_r) calculated and bohr magneton number calculated using following relation [14],

$$\eta_B = \frac{M_s \cdot M_w}{5585} \quad (2.1)$$

where M_w is the molecular weight. It is observed that the saturation magnetization (M_s) decrease with increase in substitution of Cr^{3+} ion.

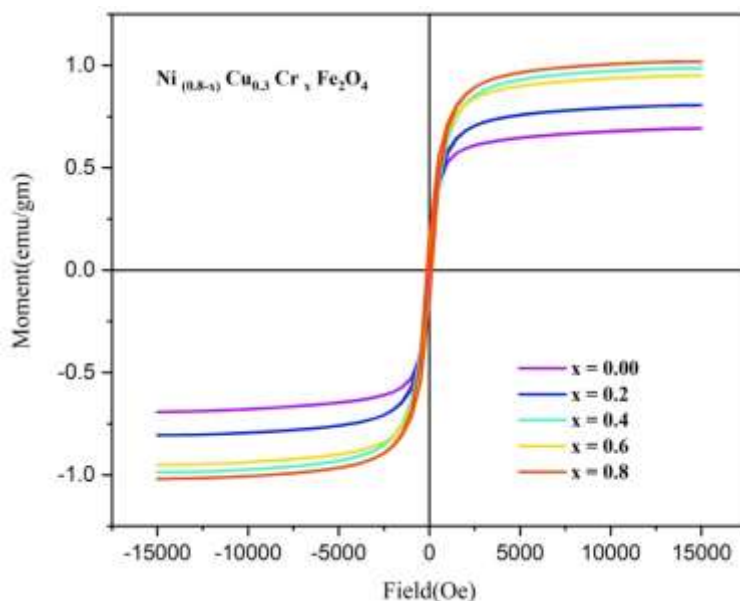


Figure 6.(a) M-H Loop of $\text{Ni}_{0.8-x}\text{Cu}_{0.3}\text{Cr}_x\text{Fe}_2\text{O}_4$ ferrite nanoparticles

Table 2 : Coercivity ,saturation magnetization remanent magnetization, remanence ratio, magneton number . (η_B) $\text{Ni}_{0.8-x}\text{Cu}_{0.3}\text{Cr}_x\text{Fe}_2\text{O}_4$ ferrite nanoparticles

Sample	H_c (Oe)	M_s (emu/g)	M_r (emu/g)	M_r/M_s	η_B (μB)
$\text{Ni}_{0.8}\text{Cu}_{0.6}\text{Fe}_2\text{O}_4$	70.49	72.50	6.32	0.087172	3.0402
$\text{Ni}_{0.6}\text{Cu}_{0.6}\text{Cr}_{0.2}\text{Fe}_2\text{O}_4$	92.56	72.14	6.83	0.094677	3.0343
$\text{Ni}_{0.4}\text{Cu}_{0.6}\text{Cr}_{0.4}\text{Fe}_2\text{O}_4$	114.71	70.60	7.68	0.108782	3.0363

$\text{Ni}_{0.2}\text{Cu}_{0.6}\text{Cr}_{0.6}\text{Fe}_2\text{O}_4$	120.5	70.29	8.82	0.125480	3.0372
$\text{Cu}_{0.6}\text{Cr}_{0.6}\text{Fe}_2\text{O}_4$	121.7	70.08	8.95	0.127711	3.0386

Conclusion

The synthesis of Lemon Juice Mediated Cr^{2+} Doped $\text{Cu}_{0.6}\text{Cr}_x\text{Fe}_2\text{O}_4$ Ferrite Nanoparticles successfully. The structural studies reveals that the formation of spinal cubic crystal structure with crystallite size 26 - 33 nm. lattice constant a lies between 8.3674 - 8.4056. Morphological analysis shows this small spherical particles and grain size observed 29 - 38 nm. EDS confirm the purity of the sample and presence of Cu, Cr, Fe and O elements. FTIR shows two wave number around 541 and 431 cm^{-1} with tetrahedral and octahedral stretching. The magnetic parameters obtained from VSM data shows the increased saturation magnetization (Ms) value by substitution of Cr^{2+} .

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