Impact of Yttrium substitution on structural and optical properties of Ni-Cu ferrites synthesized by Sol –Gel auto combustion method

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Abstract:

Rare earth yttrium (Y^{3+}) doped nanocrystalline Ni-Cu mixed spinel ferrites which are chemically represented as Ni_{0.5}Cu_{0.5}Y_xFe_{2-x}O₄ (x=0, 0.025, 0.050, 0.075, 0.1, 0.125) were synthesized by sol-gel auto-combustion method. The prepared powder was sintered for 5 hours at 600°C to obtain good crystalline phase. The structural and optical properties of samples were characterized by X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), Ultra-Violet spectroscopy (UV-Vis). X-ray analysis confirms the formation of single phase Ni_{0.5}Cu_{0.5}Y_xfe_{2-x}O₄ with cubic spinel structure without any impurity phase. The particle size measured from X-ray diffraction (XRD) is in 51-84 nm range. The lattice parameter calculated

using XRD data was found to be decreasing upto x=0.100 and then increases for x=0.125. FT-IR studies confirmed the spinel structure of compounds formed by this method. The FTIR characterization shows two frequency bands near 530-538cm-1 and 405-433 cm-1 corresponding to the formation of octahedral and tetrahedral clusters of metal oxides. Change in band gap energy was studied with the help of UV spectrometer. The band gap energy of Nickel Copper Yttrium ferrite increases from 1.59 eV to 1.939 eV with an increase in yttrium concentration and is in the range of semiconductor material.

Keywords: Ni-Cu ferrites, Y³⁺ doping, sol –gel method, XRD, FTIR and UV –Vis

Introduction: Ferrites are electrically non-conductive ferrimagnetic ceramic compound materials, consisting of various mixtures of iron oxides such as Hematite (Fe₂O₃) or Magnetite (Fe₃O₄) and the oxides of other metals. Ferrites exhibit various properties in technological and scientific applications. The general formula spinel ferrite MFe_2O_4 , where M represents a divalent metal ion reveals interesting behavior of electric insulator and magnetic conductor. Most ferrites are ionic and have a very stable crystal

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structure. The majority of ferrites contain iron oxides as their major constituents (1,2). Ferrites exhibit various properties, including magnetic, electrical, Electrochemical, magneto-electronic and optoelectronics (3-5). There are several criteria to obtain high-quality ferrites that are preparation techniques, chemical composition, sintering duration and sintering temperature (6-8). Significant variations are seen in the properties of bulk and nanoferrites synthesized by various techniques (9,10).

Granate ferrites are cubic in structure and each cell consists eigth R3Fe5O12 molecules. R3 + ions here are rare elements of the earth like Y,Yb, Pr, Ho, Gd (11). In spinel ferrite crystal structure, the replacement of rare earth metal ions whose ionic radii is greater than that of the Fe3+ions produces microstains. This leads to structural distortion and modification of ferrite properties (12-15). Rare earth doped nano spinel ferrite have a wider range of applications, due to its excellent magnetic and magnetoptic properties, in high frequency microwave, data storage devices and telecommunications sector (16-19). Due to their high resistivity and low dielectric losses, magnesium-based ferrites are also used for producing micro-wave devices.(20-23). Researchers have therefore studied different characteristics of Fe3 + spinel ferrites substituted with trivalent rare earth ions (24-26). Nanoferrites have recently gained significant interest from researchers because of their remarkable electromagnetic properties (27-29). Due to the high reflection losses and wide absorbing bandwidth, nano spinel ferrite could be used as radar absorbing materials in X-band (30). Rare earth metal ions doped spinel ferrite are known to improved photocatalytic ability with multiferroic properties making it ideal for memory devices, high density information storage, gas sensors, optical fibers, multiferroic sensors application and in wastewater treatment plants (31-34).

The effect of yttrium substitution on nickel copper nanoferrite (Ni0.5Cu0.5YxFe2-xO4 where x=0, 0.025, 0.050, 0.075, 0.1, 0.125) synthesized by the sol gel autocombustion method was investigated in the present work. Investigated their structural and optical properties using techniques such as Powder X-ray diffraction (XRD), Fourier Transform Infra-Red (FTIR) spectroscopy and UV-Vis spectrometer at room temperature.

2. Experimental methods and materials: The starting materials for the preparation of Ni $_{0.5}$ Cu $_{0.5}$ Y_xFe_{2-x} O₄ (where x = 0, 0.025, 0.050, 0.075, 0.100, 0.125) are high purity AR grade ferric nitrate (Fe2 (NO3)3.9H2O), Nickel nitrate (Ni (No3)3.6H2O), Copper nitrate (Cu (NO3)3.6H2O), Yttrium nitrate (Y (NO3)3.6H2O), citric acid (C6H8O7) and ammonium hydroxide solution (NH4-OH). All nitrates and citric acid after the stoichiometric calculations dissolved into 100 ml distilled water for the formation of

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solution. As a complexing agent, citric acid was used to obtain a transparent solution with a homogenous metal ion distribution. The solution was then slowly heated at about 80 °C and stirred using a hot plate magnetic stirrer. Then ammonia solution was added to the solution drop by drop to maintain its pH 7. The solution was evaporated with the heating on a hot plate. Continuous stirring forms a continuous gel network of gel , after 4 to 5 hrs auto combustion takes place. The ash was grinded in agate mortar and pestle, converted into fine powder. Then fine powder was sintered in furnace at 600 °C for 5 hrs. The prepared anoferrite powders were characterized by different techniques like XRD, FTIR and UV spectroscopy.

3. Results and discussion:

3.1 X-ray characterization (XRD):-

The X-ray diffraction (XRD) patterns of Ni_{0.5}Cu_{0.5}Y_xfe_{2-x}o₄ (x=0, 0.025, 0.050, 0.075, 0.1,0.125) were recorded with the X-ray diffractometer was shown in figure 1. XRD of all samples were recorded in 2 θ range of 10-80° with cu-k α radiation (λ =1.54178 A°) at room temperature. All peaks were identified by comparing the "2 θ " i.e. diffraction angle with that of the JCPDS data in order to confirm the present crystalline phase. Crystallite size (t) was determined by using Scherrer formula which is given as (2 θ).

$$t=\frac{0.9\,\lambda}{\beta\cos\theta} \quad ,$$

Where, t is the crystallite size (nm), β is a full width at half maxima of diffraction peak, λ = 0.154A⁰ is the wavelength of X-ray and θ is the diffraction angle.



Figure. 1. XRD patterns for yttrium doped Ni-Cu nanoferrites.

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The XRD pattern displays the reflection (220), (311), (222), (400), (422), (333) and (440) all belonging to cubic spinel structure. All peaks of diffraction correspond well to those mentioned in the literature (35). The significant finding of the XRD patterns is that in all compositions there is no evidence of secondary peaks. According to some literature, if rare earth ions are doped, secondary peak occurs in the spinel ferrite. (36,37). This may be due to a low temperature sample synthesis with the Sol-gel auto combustion technique and a lower Y3 + doping concentration. The doping of rare earth in Ni-Cu nano ferrites reveals variations of the crystallite size as shown in Table 1.

Table 1: Lattice constant (A), Crystallite Size (t), X-ray Density (dx) ,dislocation density (δ), of Ni_{0.5}Cu_{0.5}Y_xFe_{2-x}O₄ nanoferrite.

Composition	Lattice	GRAIN SIZE (nm)	X – ray	Dislocation Density
X	Constant	Scherer's formula	Density	δ (Δ lines/m ²)
	A(A ⁰)		(gm/cm ³)	
0	8.421	65.872	5.2675	2.3E+14
0.025	8.361	58.398	5.4005	2.9E+14
0.05	8.357	76.388	5.4271	1.7E+14
0.075	8.354	68.857	5.4517	2.1E+14
0.1	8.341	84.948	5.4961	1.4E+14
0.125	8.344	65.043	5.5091	2.3E+14

The average crystallite size of the $Ni_{0.5}Cu_{0.5}Y_xFe_{2-x}O_4$ was between 51 nm and 84 nm. The variation of particle size with yttrium concentration varies shown in figure 2.



Figure 2: variation of the crystallite sizes with Y content by Scherrer's formula

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W-H plot for prepared Ni_{0.5}Cu_{0.5}Y_xFe_{2-x}O₄ for (x-0.125) nanoferrite is drawn with 4sin θ along the x-axis and β cos θ along the y-axis as shown in Figure 3. The y-intercept of the fitted line gives particle size, . Crystallite size t is obtained by using the relation :

$$t = \frac{\lambda}{y \, intercept}$$



Figure 3 : (W-H plot of $Ni_{0.5}Cu_{0.5}Y_xFe_{2-x}O_4$ Figure 4 : (Variation of Lattice Constant with
Y nanoferrite for x=0.125.)VContent x)

The calculated cystallite size of $Ni_{0.5}Cu_{0.5}Y_xFe_{2-x}$ O₄ nanoferrite for x=0.125 using Scherrer's formula and W-H plot method is 65 nm and 63 nm, respectively, and is in good agreement. The XRD data were used to calculate the lattice parameter for each sample using the following formula,

$$a = d \times \sqrt{h^2 + k^2 + l^2}$$

Where, a is lattice constant, d is the inter planer spacing and hkl are the miller indices of planes. The values of lattice constant are listed in table 1. The lattice constant decreased from 8.421Å (x=0) to 8.341Å (x=0.100) and increased for x = 0.125. The variation of lattice constant 'a' with the Y3+ content is shown in figure 4.

Variations in the lattice constant of the yttrium doped Ni-Cu nanoferrite can also be explained on the basis of the difference in the ionic radii of these doped metal ions. This can be attributed to the larger ionic radii of Y^{3+} (1.02 Å) rare earth ions as compared to Fe³⁺ (0.67 Å) ions. The increased lattice constant for x=0.125 may be due to the increase in the Y^{3+} ion concentration.

The X-ray density was determined using the following formula and table 1 lists the values of X ray density for different compositions.

$$d_x = \frac{8*M}{N*a^3}$$

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Where, M is molecular weight of yttrium doped Ni-Cu nanoferrite, N is Avogadro's number and 'a' is lattice constant. X-ray density increases as yttrium concentartion increases as it mainly depends on the molecular weights of samples. This increase in X-ray density is due to a decrease in lattice constant and an increase in yttrium content. Dislocation density was calculated from crystallite size using the following relation.

$$\delta = \frac{1}{t^2}$$

Dislocation density varies from 2.9E+14 to 1.4 $E^{+14} \Delta lines /m^2$ with Y $^{3+}$ ion concentration. The hoping length between cations on the A-sites (L_A) and cations on the B-site (L_B) can be determined using the lattice parameter of the sample. By using the following equations.

$$L_{A} = \frac{a * \sqrt{3}}{4}$$
$$L_{B} = \frac{a * \sqrt{2}}{4}$$

 $Ni_{0.5}$ Cu_{0.5} Y_xFe_{2-xO4} nanoparticles hoping length was measured at tetrahedral and octahedral sites and shown in Table 2. The hoping length at both tetrahedral and octahedral sites initially decreases up to yttrium concentration (x=0.1) and increases for (x=0.125). It is because the hopping length of the sites were directly proportional to the samples lattice parameters. The increase in hoping length for x =0.125 can be attributed to increase in Y³⁺ ion concentration. This can be interpreted by the differences in ionic radii of Y³⁺ and Fe^{3+.}

Table 2: hoping length values at the tetrahedral and octahedral sites of $Ni_{0.5}Cu_{0.5}Y_xFe_{2-x}O_4$ nanoferrite.

Composition (X)	Hoping length at	Hoping length octahedral	
	tetrahedral A site (d _A)	at B site(d_B)	
0.0	3.646399	2.977273	
0.025	3.620419	2.956059	
0.050	3.618687	2.954645	
0.075	3.617388	2.953585	
0.100	3.611758	2.948988	
0.125	3.613057	2.950049	

Tetrahedral (A-site) and octahedral B- site hopping length variation with Y content were shown in Fig. 5 (a) and (b) respectively.



Figure 5: (a) Variation of hoping length at tetrahedral site with Y Content (b) Variation of hoping length at octahedral site with Y Content.

3.2. Fourier transform infrared spectroscopic analysis :

Figure.6 shows the FTIR spectra of $Ni_{0.5}Cu_{0.5}Y_xFe_{2-x}O_4$ nanoferrite (x=0, 0.025, 0.050,0.075,0.1,0.125) Samples were recorded in the wave number range of 400–4000 cm-1. . The absorption frequency values v1 and v2 are mentioned in the table 3. FTIR spectra display two main absorption bands below 1000 cm⁻¹ corresponding to ferrite metal oxide (M-O) vibration modes. The higher frequency band in the range of 530-538cm⁻¹ is attributed to the tetrahedral-cluster-stretching mode, while the lower frequency band in the range of 405-433 cm⁻¹ is attributed to the octahedral cluster stretching mode. No other group peaks were found, which meant the synthesized material fingerprint free of organic compounds.



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Fig.6. FTIR spectra of Ni0.5Cu0.5YxFe2-xO4 (x=0, 025.0.050. 0.075, 0.100, 0.125)

samples.

Composition X	υ ₁	υ2
0	530.42	433.98
0.025	536.21	432.05
0.050	534.28	418.55
0.075	532.35	406.98
0.100	530.42	405.92
0.125	538.14	418.55

Table No 3: FTIR Absorption frequency bands

The absorption frequency v1 was slightly shifted to higher frequencies and v2 to lower frequencies with increasing Y^{3+} content is shown by Table 2. This shift in v_1 and v_2 can be caused by the substitution of yttrium for iron at octahedral sites because its atomic weight is higher than iron, affecting distances of Fe³⁺ – O²⁻ at octahedral sites. Literature (38-40) has reported similar reports.

3.3 Optical absorption spectra: To identify the given material is conductor, semiconductor or insulator, UV –Vis absorption spectroscopy is very useful. Figure 8 shows the UV absorption spectra of the prepared sample Ni_{0.5}Cu_{0.5}Yfe₂O₄ for its different compositions. The band gap energy is one of the fundamental characteristic to identify type of the given material. The optical energy gap is calculated by using formula: E = hc/v

Where h represent Planck's constant, c is the velocity of light in vacuum and E is Energy absorbed in an electronic transition between valence band and conduction band. The optical band gap of Ni_{0.5}Cu_{0.5}Yfe₂O₄ is found to increase from 1.56 eV to 1.7146 eV with increasing yttrium concentration. Experimentally, to get the value of band gap energy E_g , we usually use of the **Tauc relation**, which is given by this equation:

$\alpha h v = A(hv - Eg)^n$

where, α is the coefficient of absorption and E_g is the optical energy gap. The graph of $(\alpha hv)^2$ versus (*hv*) of the prepared sample Ni_{0.5}Cu_{0.5}Yfe₂O₄ for its different compositions is shown in Figure 9 is a straight lines. This line intersects the X-axis at $(\alpha hv)^2 = 0$ The

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values of E_g are found from Y - intercept and it increases from 1.724 eV to 1.939 eV as concentration of Y⁺³ increases are listed in table 4.



Figure 7: UV spectral of Ni_{0.5}Cu_{0.5}Y_xFe_{2-x} O₄ nanoferrites (x=0, 0.025, .050,0.075,0.1,0.125)



Figure 8 : Band gap energy by Tauc method $Ni_{0.5}Cu_{0.5}Y_xFe_{2-x}O_4$ nanoferrites

(x=0, 0.025,

0.050,0.075,0.1,0.125)

Table 4: Band gap energy of Ni_{0.5}Cu_{0.5}Y_xFe_{2-x}O₄ nanoferrites

(x=0, 0.025, 0.050, 0.075, 0.1, 0.125)

COMPOSITION	WAVELENGTH (nm)	ENERGY GAP (eV)	ENERGY GAP (eV)
х			By Tauc plot method
0	781	1.59	1.724
0.025	795	1.56	1.796
0.05	845	1.47	1.862
0.075	833.5	1.49	1.896
0.1	827.5	1.5022	1.761
0.125	725	1.7146	1.939

Conclusion:

A series of Y^{3+} doped Ni-Cu spinal ferrite (Ni_{0.5}Cu_{0.5}Y_xFe_{2-x} O₄ where x=0, 0.025, 0.050,0.075,0.1,0.125) was successfully prepared using the Sol-Gel auto-combustion method. XRD, FTIR and UV spectroscopy were used for the study of prepared garnet ferrites samples. X-ray diffraction showed sample phase purity and provided evidence of single phase garnet structure of all synthesizing materials. The crystallite size of synthesized nanomaterial was determined using Scherrer's formula within 51-84 nm range and W-H plot particle are same. FTIR and XRD studies confirmed the ferrite powder formation. The optical band gap energy of Ni0.5Cu0.5Yfe2O4 is found to be increases from 1.59 eV to 1.939 eV with increasing yttrium concentration and it is in the range of semiconducting material. The optical band gap determined from the UV absorption spectra and the tauc plot method are in close agreement.

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