Study of Cu^{2+} and Gd^{3+} Doping Effect on Magneto-Optical Properties of Ni Zn Fe_2O_4 Spinel ferrites

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ABSTRACT

This work is based on synthesis and study of magneto-Optical properties of $Ni_{0.4}Zn_{0.6}Fe_2O_4$, $Ni_{0.4}Cu_{0.3}Zn_{0.3}Fe_2O_4$ and $Ni_{0.4}Cu_{0.3}Zn_{0.3}Gd_{0.075}Fe_{1.925}O_4$ nano spinel ferrites. All samples of mixed spinel ferrites were successfully synthesized using sol-gel auto combustion technique. The structural, optical and magnetic properties are investigated by using X-ray Diffractometer, FTIR Spectrometer, Scanning Electron Microscope and Vibrating Sample Magnetometer (VSM) respectively. XRD data analysis confirmed that formation of nanocrystalline cubic spinel structure of prepared samples. The crystallite size of synthesized nanoparticles was in 35 to 45nm range. The lattice constant shows increase with substitution of Cu^{2+} and Gd^{3+} ions in the host of $Ni_{0.4}Zn_{0.6}Fe_2O_4$. The absorption bands in IR spectrum around 448.2331 cm^{-1} and 560.23 cm^{-1} are attributed to υ_1 and υ_2 frequencies respectively. This is concluded that nano material exhibit in the form of spinel ferrite structure. SEM image shows that spinel ferrites were systematically arranged in the form of wires of larger sized diameter. Band gap energy E_g obtained using UV-Visible absorption data was increased with Cu^{2+} and Gd^{3+} substitution. The values of magnetic field H_c and saturation magnetization M_s was increased with Cu^{2+} and Gd^{3+} substitution as compared to values of H_c and M_s for $Ni_{0.4}Zn_{0.6}Fe_2O_4$.

Keywords: Mixed spinel ferrite, Ni-Cu-Zn spinel, NiFe2O4UV, VSM

INTRODUCTION

Iron (Fe) based mixed spinel ferrites are extraordinary, key materials and used in field of electromagnetic devices such as in phase shifters, sensors, recording media, and in high-density data storage devices[1−4]. The spinel ferrite materials have been classified into normal spinel ferrite, inverse spinel ferrite,and mixed inverse spinel ferrite. The nickel ferrite ($NiFe_2O_4$, NFO), was classed as inverse spinel structure, has gained much more attention because of its peculiar physical properties such as low-grade Curie temperature, high electrical resistivity, high permeability, low magnetostriction, etc. [5]. The main structural units in mixed spinel ferrites are divalent metal oxides and trivalent ferric oxide. The chemical formula for inverse spinel structure is A [B_2] O_4 where A is divalent cations like Mn, Zn. Cu, Ni, Cd etc. occupies at tetrahedral site and B is trivalent cation Fe^{3+} and occupies tetrahedral and octahedral sites respectively. The divalent metal ion can be replaced by other divalent metal ions and Fe^{3+} can also be replaced by other trivalent cations like Al, Cr, Gd, La etc. [6]. In case of mixed ferrites divalent metal ions occupied atA site and Fe^{3+} ions occupied in the A as well B sites [7-8].

Mixed ferrites (NiCuZn fe_2O_4) exhibit vital magnetic, electric, dielectric and optical properties which made them extensively useful in technological and industrial applications such as magnetic storage in microwave dives [9]. Recently Rare Earth (RE) ion doped spinel ferrites in nano form have got extensive importance due to their tremendous applications such as in transformer core, in magnetic memory, in antenna rods in biomedical field, catalysis, and solar cells [10-11].

Various methods have been used in the synthesis of spinel ferrite nano particles. Most commonly the Sol-gel method [12], Co-precipitation method [13], Hydrothermal[14], Spray drying [15], etc. However these techniques requires sophisticated instruments however Sol-gel auto combustion technique is comparatively very economic and involve easy steps in preparation of mixed spinel ferrites [16-17]. Hence in present work a Sol-gel auto combustion technique have been used. The raw materials are in the form of Metal nitrates and citric acid were used as oxidizing salts and combustion fuel. All chemicals were of high purity analytical reagent grade [16]. In the view of various applications the present paper reported the synthesis of $NiZnFe_2O_4$, $NiCuZnFe_2O_4$ and Cu2+ doped, rare earth element (Gd^{3+}) doped

NiCuZnFe2O4 mixed nano spinel ferrites. The influence of Cu^{2+} and Gd^{3+} doping on structural optical and magnetic properties of $NiZnFe_2O_4$ were discussed and reported.

MATERIAL AND METHODS

$Ni_{0.4}Zn_{0.6}Fe_2O_4$, $Ni_{0.4}Cu_{0.3}Zn_{0.3}Fe_2O_4$ and $Ni_{0.4}Cu_{0.3}Zn_{0.3}Gd_{0.075}Fe_{1.925}O_4$ nano spinel ferrite particles were synthesized by using cost effective sol-gel auto combustion technique and citric acid is used as a fuel. The analytical grade (AR) nickel nitrate (Ni $(NO_3)_2.6H_2O$), cupper nitrate (Cu $(NO_3)_2.6H_2O$), gadolinium nitrate (Gd $(NO_3)_3.9H_2O$), ferric nitrate (Fe $(NO_3)_3.9H_2O$), Zinc nitrate $Zn(NO_3)_2.6H_2O$ and citric acid ($C_6H_8O_7$) were used as raw materials (99.9% pure) in proper proportion. The metal nitrates to citric acid ratio was taken as 1:3. Ammonia solution was added to maintain the pH 7. Temperature required for the synthesis of ferrite nanoparticles is kept around 110 ˚C. The as-synthesized powder is sintered at 800 ˚C for 4 hour. The sintered samples are charectrized using X-ray diffractometer (XRD), Fourier Transmittance infrared spectrometer (FTIR), Scanning Electron Microscopy (SEM), UV-Visible spectrophotometer and Vibrating sample magnetometer (VSM). The data obtained is analyzed and reported in this report.

RESULTS AND DISCUSSION

Structural Properties

The XRD spectra of sintered samples are recorded over the range 2θ= 20-80 degree by using Mini Flex II X-ray Diffractometer. XRD patterns ofthese three spinel ferrites namely, Ni0.4Zn0.6Fe2O4, Ni0.4Cu0.3Zn0.3Fe2O4 and Ni0.4Cu0.3Zn0.3Gd0.075Fe1.925O4 are shown in Figure 1. The XRD pattern exhibit (111), (220), (311), (222), (400), (422), (511) and (440) peak. The peaks in the XRD pattern are attributed to cubic spinel structure of all three samples. Similar cubic spinel structure for $Ni_{0.4}Zn0.2Mn_{0.4}Fe_2O_4$ was reported in the literature [17]. However $Ni_{0.4}Zn_{0.6}Fe_2O_4$ sample show extra impurity peaks related to Zn metal in the 20° to 30° degree range. No impurity peaks are observed for the samples $Ni_{0.4}Cu0._3Zn0.3Fe_2O_4$ and $Ni_{0.4}Cu_{0.3}Zn_{0.3}Gd_{0.075}Fe_{1.925}O_4$ indicate that Cu, Zn, and Gd completely dissolve in the $NiFe_2O_4$ lattice. The intensity of (311) peak shows significant decrease with low content Gd^{3+} doping. The average grain size was calculated using well known Scherer's [18] formula (1),

$$D = \frac{0.94\lambda}{\beta \cos \theta} \text{ ---------- (1),}$$

Here λ is the wavelength of the x-ray and β is the full width at half maximum intensity in radians.
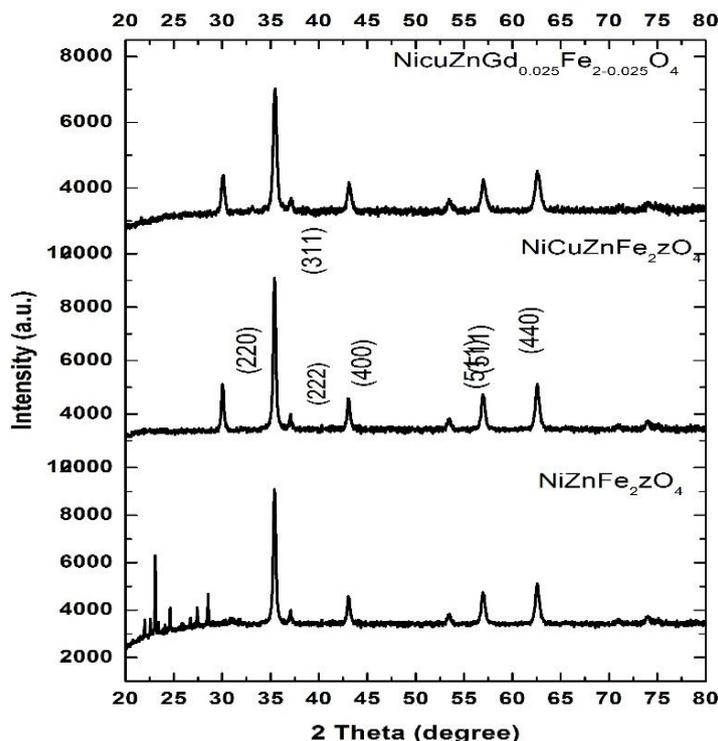
Figure 1. XRD pattern of Prepared three samples of spinel ferrites

The values of lattice constant 'a' (Å) for cubic sample [18] is determined by using the relation,

$$a = d_{hkl}(h^2 + k^2 + l^2) \text{-------- (2)}$$

where (hkl) are the lattice constants. The unit cell volume (V) was calculated by using the following equation;

$$V = a^3 \text{-------- (3)}$$

where, V is the unit cell volume a is the lattice constant. The X-ray density [19] (d_X) was calculated by using the following relation,

$$d_x = \frac{Z \times M}{V \times N_A} \text{ gm/cm}^3 \text{----------(4)}$$

where, Z s the number of molecules per formula unit (Z = 8 for spinel system), M is molecular mass of the sample, V= a3 is the unit cell volume and NA is the Avogadro's number. The estimated crystallite size, lattice constant 'a', unit cell volume 'V'and X ray density were displayed in table 1.The effect of Cu2+ and Gd3+ doping on variation of structural parameters was clearly illustrated in the table 1.

Table 1: Estimated structural parameters from XRD Data

Sample Name	2θ (degree)	d (radians)	β (radians)	D (nm)	Lattice parameter 'a'	Volume (V)	X-ray Density
$Ni_{0.4}Zn_{0.6}Fe_2O_4$	35.600	2.5011	0.346	41	8.36	584.6	6.356
$Ni_{0.4}Cu_{0.3}Zn_{0.3}Fe_2O_4$	35.436	2.5311	0.400	38	8.4013	592.99	6.045
$Ni_{0.4}Cu_{0.3}Zn_{0.3}Gd_{0.075}Fe_{1.925}O_4$	35.360	2.5364	0.423	35	8.4122	595.3	6.072

Infrared (FTIR) Spectroscopy Investigations

IR spectrum of sample $Ni_{0.4}Cu_{0.3}Zn_{0.3}Gd_{0.075}Fe_{1.925}O_4$scanned in the renge 400 to 4000 cm[-1] was presented in figure 2. The IR spectrum exhibits various vibrations corresponding to different functional groups. However absorption bonds observed at 448.23 cm[-1] and at 560.231 cm[-1] which shows the characteristic bands of frequencies v_2 andv_1 related to intrinsic lattice vibrations of octahedral and tetrahedral coordination compounds of spinel ferrite structure respectively[23]. The peaks appeared around 880 cm[-1] attributed to deformation of the iron oxide lattice into the bonding of Fe_3O_4 particles with surface OH groups. Similar results have been reported in the litrature [24]. The absorption bands in the ragne 1600 cm-1 to 3400 cm[-1] are attributed to stretchable vibration of the H–O–H group due to the presence of residual water [25]. The absorption peaks due to C–O and C-O_3 ion vibration are depicted in FTIR spectrum at arround 1000 to 1500 cm[-1] [26-27]. The FTIR spectrum reveals a peak at 1000 cm[-1] that is reported in literature due to the incomplete decomposition of the products. Two prominent peaks appeared around 795-800 cm[-1]are attributed to (Gd-O) strtcheing vibrations [28].
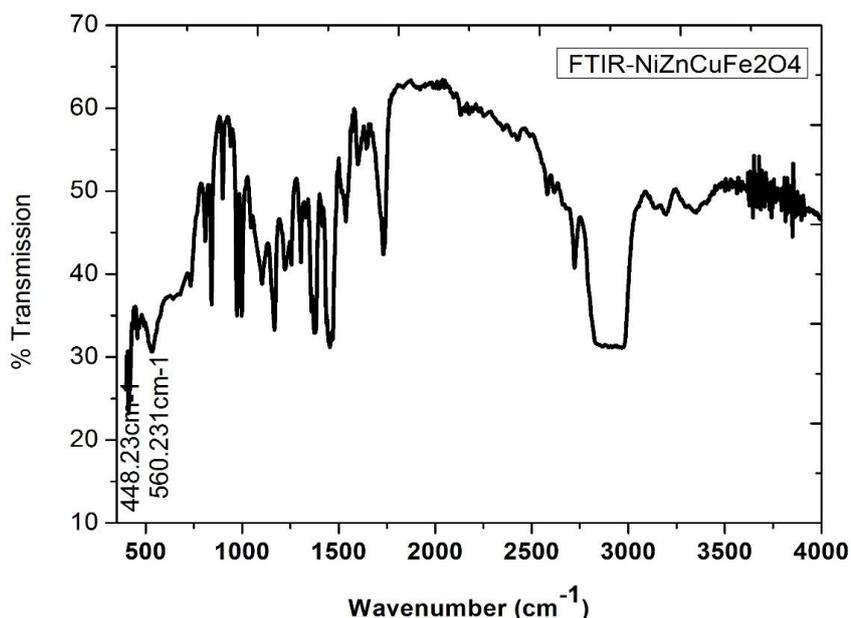
Figure 2. IR Spectrum of $Ni_{0.4}Cu_{0.3}Zn_{0.3}Gd_{0.075}Fe_{1.925}O_4$of spinel ferrite

Surface Morphology Of $Ni_{0.4}Cu_{0.3}Zn_{0.3}Gd_{0.075}Fe_{1.925}O_4$ Spinel Ferrite

Figure 3 shows the SEM images of the Ni0.4Cu0.3Zn0.3Gd0.075Fe1.925O4 spinel. The SEM scan illustrate that micrograph composed of largely agglomerated nanoparticles of the sample. The large clusters of Ni0.4Cu0.3Zn0.3Gd0.075Fe1.925O4 ferrites formed by assembling of small spherical grains of nearly consistent in size. The spinel ferrites were systematically arranged. The dipole-dipole interactions among the uncapped nanoparticles results in agglomeration of nanosized particles.
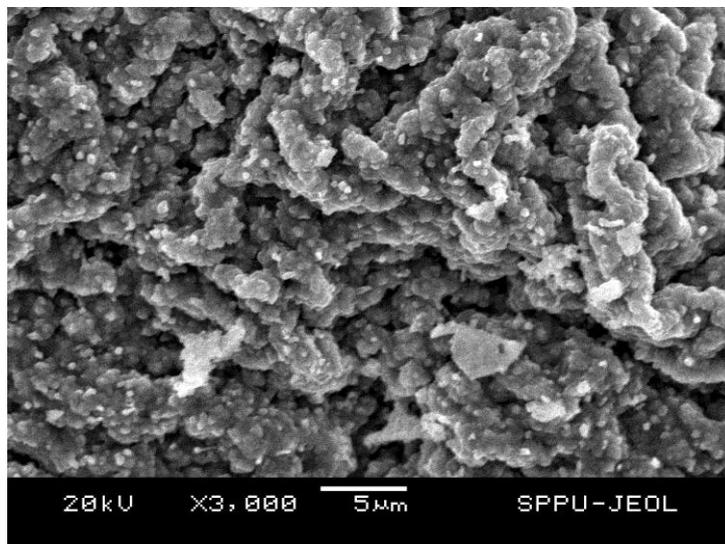
Figure 3. SEM image of of $Ni_{0.4}Cu_{0.3}Zn_{0.3}Gd_{0.075}Fe_{1.925}O_4$ of spinel ferrite

Effect of Cu^{2+} and Gd^{3+} Doping On Absorption And Optical Band GAP

By using double distilled water as a solvent collide solution of prepared ferrites was prepared and absorption spectra was recorded in the range 200 to 800 nm by using Sistronics Double Beam (2021) photo spectrometer. UV-Visible absorption spectra of $Ni_{0.4}Zn_{0.6}Fe_2O_4$, $Ni_{0.4}Cu_{0.3}Zn_{0.3}Fe_2O_4$ and $Ni_{0.4}Cu_{0.3}Zn_{0.3}Gd_{0.075}Fe_{1.925}O_4$ nano spinel ferrite was shown in figure 4.
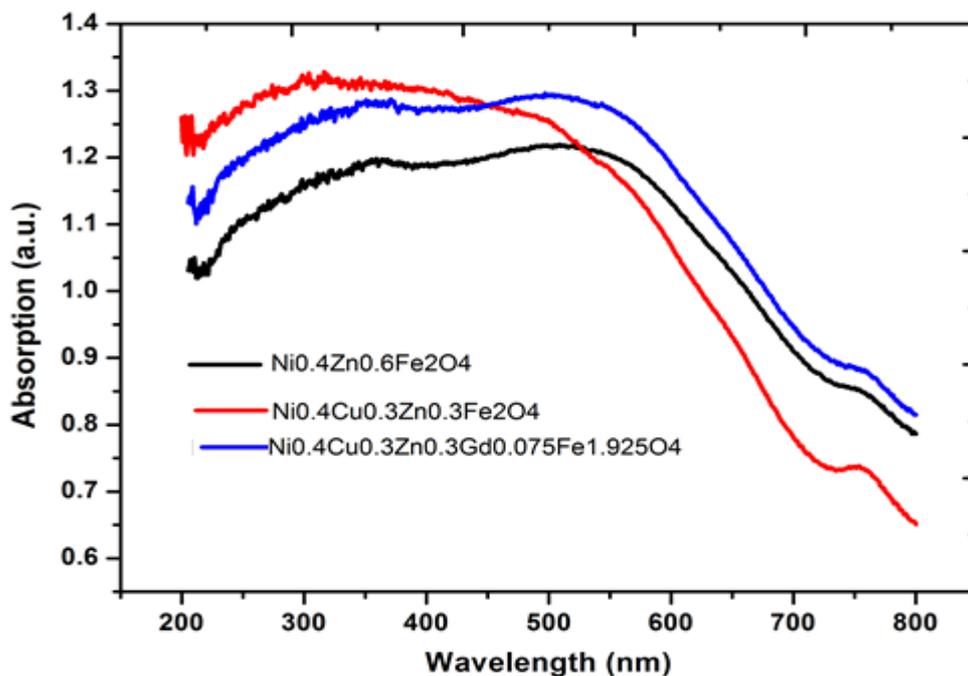
Figure 4. Absorption spectra of prepared spinel ferrite

The spectra show absorption at around 492 nm in the visible region for $Ni_{0.4}Zn_{0.6}Fe_2O_4$, ferrite sample similar results have reported by Elangbam C. Devi et al. (2017) in optical investigation of $MnFe_2O_4$ nanoferrites [28]. The absorption peak in visible region was shifted to lower side around 470 nm for $Ni_{0.4}Cu_{0.3}Zn_{0.3}Fe_2O_4$ doping of sample $Ni_{0.5}Cu_{0.5}Zn_{0.5}Gd_{0.075}Fe_{2-0.075}O_4$ and at around 467 nm for $Ni_{0.4}Cu_{0.3}Zn_{0.3}Gd_{0.075}Fe_{1.925}O_4$ sample on doping of Cu2+ and Gd2+ in $Ni_{0.4}Zn_{0.6}Fe_2O_4$. Spinel. The optical band gap energy (E_g) is estimated by using Tauc relation [12] as given below;

$$\alpha h\nu = B(h\nu - E_g)^n$$

where, α is the linear absorption coefficient of the material, B is the proportionality constant, h is Planck's constant $(6.6260\times10^{-34}$J.s), ν is the photon energy, E_g is the optical energy band gap n is a constant associated with different kinds of electronic transitions (n = 0.5 for a direct allowed, n = 2 for an indirect allowed, n = 1.5 for a direct forbidden and n = 3 for an indirect forbidden). The Tauc plots for $Ni_{0.4}Zn_{0.6}Fe_2O_4$, $Ni_{0.4}Cu_{0.3}Zn_{0.3}Fe_2O_4$ and $Ni_{0.4}Cu_{0.3}Zn_{0.3}Gd_{0.075}Fe_{1.925}O_4$ are presented in figure 5.
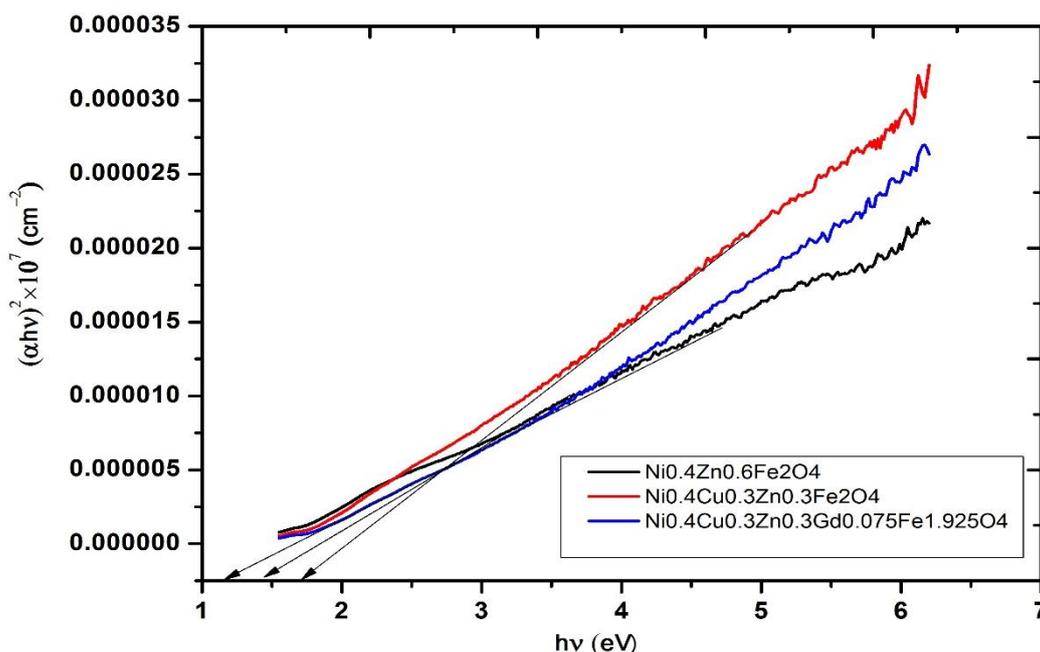
Figure 5. Tauc plotsof spinel ferrite

The optical band gap estimated by using Tauc plots is presented in table 2. The band gap energy was increased on doping of Cu^{2+} for Zn^{2+} and Gd^{3+} for Fe^{3+}. The increase in band gap was related to decrease in crystallite size.

Magnetic Properties Prepared Spinel Ferrites
Vibrating sample magnetometer (VSM) technique was employed for magnetization measurement of $Ni_{0.4}Zn_{0.6}Fe_2O_4$, $Ni_{0.4}Cu_{0.3}Zn_{0.3}Fe_2O_4$ and $Ni_{0.4}Cu_{0.3}Zn_{0.3}Gd_{0.075}Fe_{1.925}O_4$ spinel ferrite samples. Figure. 6 shows the M-H plots of the samples.

The ferrite Samples $Ni_{0.4}Zn_{0.6}Fe_2O_4$, $Ni_{0.4}Cu_{0.3}Zn_{0.3}Fe_2O_4$ and $Ni_{0.4}Cu_{0.3}Zn_{0.3}Gd_{0.075}Fe_{1.925}O_4$ exhibit narrow magnetic hysteresis loops which indicate the ferromagnetic behavior of these samples. Characteristics magnetic parameters such as saturation magnetization, remnant magnetization, coercivity and remanence ratio were measured from M-H loops and their values are given in Table 2. The results revealed that saturation magnetization and magnetic remanence were increased due to cupper (Cu^{2+}) and Gadolinium (Gd^{3+}) doping in $Ni_{0.4}Zn_{0.6}Fe_2O_4$. However the values of magnetic remanence are very low and low coercivity this suggest that $Ni_{0.4}Zn_{0.6}Fe_2O_4$, $Ni_{0.4}Cu_{0.3}Zn_{0.3}Fe_2O_4$ and $Ni_{0.4}Cu_{0.3}Zn_{0.3}Gd_{0.075}Fe_{1.925}O_4$ are soft ferrite material, they can change easily their magnetization and work as magnetic conductors. These type of ferrites were useful in making magnetic cores for high frequency inductors, transformers and in many microwave devices [29]. This increase in M_s could be explained on the basis of Neel's model [30].

$$M = M_B - M_A --------- (6)$$

Here, M_A and M_B represent the A and B sublattice magnetic moments in μ_B respectively. The total magnetic moment of ferromagnetic materials depends on the number of magnetic ions sharing at tetrahedral (A) and octahedral (B) positions

[19-20,]. In Ni-Zn-CuF$_2$O$_4$ material, equal distribution of Fe^{3+} ions at tetrahedral and octahedral sites, whereas the octahedral sites are occupied by diatomic ions. However Gd^{3+} doped into Ni-Cu-Zn F$_2$O$_4$ lattice will result in the substitution of Gd^{3+} ions replacing theFe^{3+} ions and Cu^{2+} at the B site in place of Zn $^{2+}$. The magnetic moment of Cu^{2+} islarge 1.7 Bohr Magneton (B.M.) and that of Zn $^{2+}$ is zero due to unpaired d^{10} electrons. The Gd^{3+} ions possess a large spin magnetic moment of 7.9 B.M. as compared to that of Fe^{3+} ion of 5M.B. [31].Therefore, a small amount Cu^{2+} and Gd^{3+}substitution could lead to an increase in the total magnetic moment to enhance saturation magnetization.
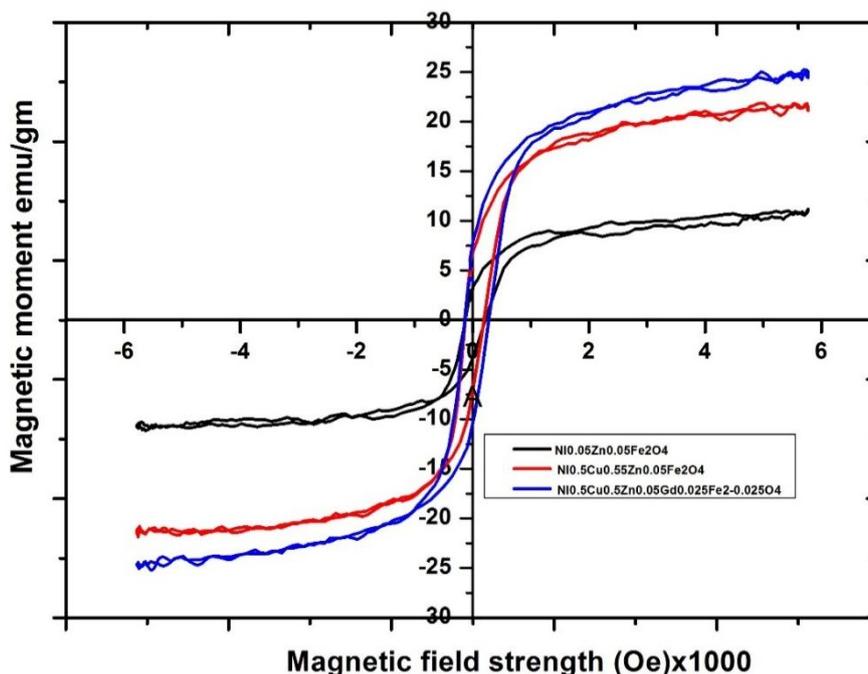
Figure 6. M-H plotsof prepared spinel ferrite

Table 1:Estimated structural parameters from XRD Data

Sample	HC (Oe)	Ms (emu/gm)	Mr(emu/gm)	Ms/Mr	Band Gap (eV)
Ni$_{0.4}$Zn$_{0.6}$Fe$_2$O$_4$	37.778	43.712	7.008	0.1603	1.25
Ni$_{0.4}$Cu$_{0.3}$Zn$_{0.3}$Fe$_2$O$_4$	40	50.4960	8.1280	0.16096	1.46
Ni$_{0.4}$Cu$_{0.3}$Zn$_{0.3}$Gd$_{0.075}$Fe$_{1.925}$O$_4$	50	49.2520	7.1680	0.14553	1.75

CONCLUSION

In Conclusion, Ni$_{0.5}$Cu$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ and Ni$_{0.5}$Cu$_{0.5}$Zn$_{0.5}$Gd$_{0.075}$Fe$_{2-0.075}$O$_4$ mixed spinel ferrites were prepared via a sol−gel combustion method. XRD pattern revealed that formation of a cubic spinel ferrite structure. The substitution of Cu^{2+} and Gd^{3+} ions induced the marked influence that lattice parameter, unit cell volume and optical band gap were increased and Xray density and crystalline size was decreased. Besides, the study indicated that Gd^{3+}doping had important impacts on the magnetic property of Ni$_{0.5}$Cu$_{0.3}$Zn$_{0.3}$Fe$_2$O$_4$ spinel ferrite. The saturation of magnetization, coercive field increased, and Remanence magnetization was decreased due to the effect of Cu^{2+} and Gd^{3+}doping, due to exchange interaction and redistribution of cations. The present work recommend that low content doping of cu^{2+} and Gd^{3+} in Ni$_{0.}$Zn$_{0.6}$Fe$_2$O$_4$ is useful for potential applications in multifunctional materials.

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