

# Synthesis Of $\text{Ni}_{1-(X)}\text{Zn}_{(X)}\text{Fe}_2\text{O}_4$ Nanoparticles And Study Of Their Structure, Magnetic And Optical Properties At Different $\text{Zn}^{2+}$ -Content

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## Abstract

Composition of  $[\text{Ni}_{1-(X)}\text{Zn}_{(X)}\text{Fe}_2\text{O}_4]$ ; (X) = 0.00, 0.3, 0.5, 0.7, and 1.00] spinel nanoparticles (NPs) has been produced using sol gel auto-combustion process hardened at 800°C for 4 hours in air. The XRD data confirm that every sample possess a spinel structure of cube phase with the lattice constant increasing from 8.340 to 8.455 Å as  $\text{Zn}^{2+}$  concentration increases because of higher ion radii of  $\text{Zn}^{2+}$  replacing the lower ion radii of  $\text{Ni}^{2+}$ . FT-IR results revealed two strong absorbing bands of ferrite spinel structure and its dependence on Zn-concentration is explained and investigated. FE-SEM images revealed a nano-sized in nearly spherical-shaped element with the formation of agglomeration. The magnetic characteristics of the samples have been measured by (VSM) at room temperature. The outcomes showed that  $\text{Ni}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$  nanoparticles present the highest  $M_s$  (saturation magnetization) of 63.3 emu/g. The direct band gap ( $E_g$ ) was estimated in UV-Visible

absorption spectra and the results showed an increase in  $E_g$  with an increase of zinc content from 1.46 to 1.95 eV.

**Keywords:** Ferrite NPs, nano structured materials, reaction combustion, magnetic properties, and Optical band gap energy

## 1. Introduction

Mixed nickel zinc ferrite has attracted the researchers because of its categories such as high magnetic permeability, low magnetic coercivity, higher Curie temperature, high magnetization, strong resistance, lower dielectric loss, and superior mechanical strength loss, and chemical stability which makes it proper for several applications such as microwave device, magnetical storage system, magnetic fluids, telecommunication equipment's, magnetically guided drug delivery, high-frequency devices, photocatalyst, antenna rods, and gas sensor, etc. [1-6]

The mixed spinel ferrite (Ni-Zn) has the chemical formula  $[(Zn_a^{2+} Fe_{1-a}^{3+})^A (Ni_{1-a}^{2+} Fe_{1+a}^{3+})^B O_4^{2+}]$  ( $0 \leq a \leq 1$ ) in which the first bracket indicates (A) tetrahedral site and the second bracket indicates the (B) octahedral site. Besides that dissemination on the cations among A and B-site, the properties of such mixed ferrite are also affected by the grain size, method of preparation, chemical composition, type of substituents, holding time, and annealing temperature [7-9]

Facile combustion technique [10], co-precipitation technique [11], sol gel process [12], microwave sintering technique [13], sol gel auto-combustion technique [14], thermal deposition technique [15] and hydrothermal technique [16], are some various approaches that is used to synthesize Nickel-Zinc spinel ferrite NPs. Among all of these techniques sol gel auto-combustion, the technique has more attention due to the involvement of high purity, simple steps, homogeneous and small particle size, and low cost [17]. In this work, we have synthesized the composition of  $[Ni_{1-(x)}Zn_{(x)}Fe_2O_4]$ : (X) = 0.00, 0.3, 0.5, 0.7, and 1.00] nanoparticles and reviewed the effectiveness of using  $Zn^{2+}$  ions on the structural, morphological, magnetical, as well as optical assets of nickel ferrite.

## 2. Experimental

### 2.1. Synthesis of NP samples

The sol-gel auto-combustion approach has been utilized to make  $[Ni_{1-(x)}Zn_{(x)}Fe_2O_4]$  (X) = 0.00, 0.3, 0.5, 0.7, and 1.00], Zinc ferrite spinel doped with nickel. The starting materials of nickel nitrate  $[(Ni(NO_3)_2 \cdot 6H_2O)]$  - 98%, zinc nitrate  $[(Zn(NO_3)_2 \cdot 6H_2O)]$  - 96%, ferric nitrate  $[(Fe(NO_3)_3 \cdot 9H_2O)]$  - 98%, citric acid  $[(C_6H_8O_7 \cdot H_2O)]$  - 99% and ammonia ( $NH_3$ ) each of the analytical grade. The metal nitrates all absorbed by distilled water in a glass beaker and agitated for 20 minutes in their stoichiometry. Then the metal nitrate (M) solution was added to (C) Citric acid (fuel) which should be stirred for 20

min. The ratio of metal nitrates (M) to total moles of citric acid (C) is adjusted at M: C = 1:1. At 70 °C and below constant stirring, and to maintain the (PH) value to 7 the ammonia solution is added dropped drop and then stirred continuously, inroom temperature 100°C later, viscous gel is formed. The sol was further heated up to 200°C. Then the auto combustion reaction occurred and loose powder was formed. Finally, the burnt powder was grounded well and toughened with in air 800°C lasting four hours.

## 2.2. Characterization

The ferrite structure were studied through X-ray diffractometer (Rigaku:Smartlab 3kW) by step of 0.02° using Cu-K<sub>α</sub> radiation (1.5406Å) in 2θ diffraction angle range from 25-70°. At room temperature, the bond structure was recorded by FTIR (Bruker: Alpha Two). Surface morphology, elemental composition characterization were investigated by FESEM (FEI: Nova Nano SEM-450) configured with EDS. At 300K the magnetic properties were passed out using vibrating sample magnetization (Quantum Design: Versa Lab-3Tesla). Optical absorption spectra were studied using UV-Visible (VIS) spectrometer (Shimadzu: UV\_3600 Plus).

## 3. Result and Discussion

### 3.1. XRD study

The XRD pattern of all samples annealed at 800°C indicated in Fig.1. Every peak of pure and Zn substituted Ni ferrites corresponds to JCPDS (Card-No. 44-1485 for Nickel-ferrite and Card-No. 22-1012 for Zinc-ferrite), indicating that the spinel cubic structure of ferrite has been developed. Impurity phases of Fe<sub>2</sub>O<sub>3</sub> were observed for X = 0.3, 0.5, and 0.7 annealed sample (shown as ^ in Fig.1).

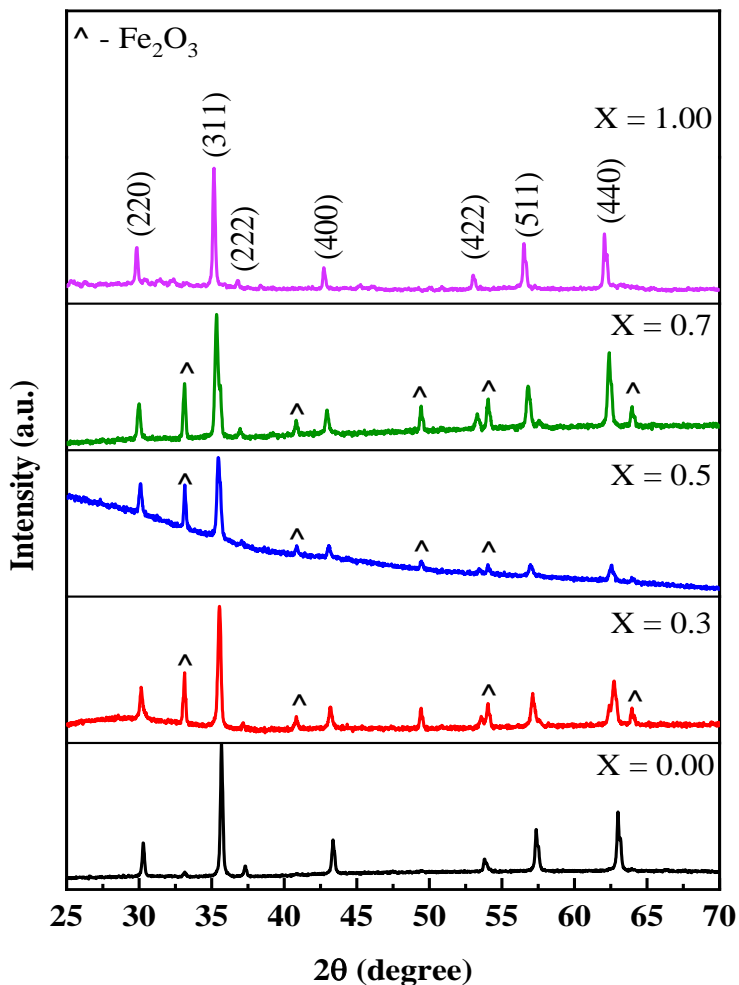


Fig. 1. XRD patterns of  $\text{Ni}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4$  annealed at  $800^\circ\text{C}$

The crystallite size for the highest (311) peak for all ferrite sample compositions calculated by Debye-Scherrer's formula [18] is charted in table 2. Fig. 2 demonstrated the difference in crystallite size (D) as a function of Zn concentration, that lies in the range 25.61–43.95nm for different Zn content. As the concentration of the zinc rises there will be no monotonic distribution of average crystallite and this could cause an inhomogeneous structure and for a large number of times various gels are formed and so on [19].

Zinc Content (X)	(311) peak position $2\theta^\circ$	Size of Crystallite "D"(nm)	Lattice variable "a" (Å)
0	35.16	43.51	8.340
0.3	35.34	35.45	8.371
0.5	35.44	29.94	8.392
0.7	35.54	25.61	8.415
1.00	35.68	43.95	8.455

Table 1. Size of Crystallite 'D' (nm), and lattice variable 'a' (Å) for  $\text{Ni}_{1-(X)}\text{Zn}_{(X)}\text{Fe}_2\text{O}_4$  NPs

Using the following equation the lattice parameter 'a' is measured by miller indices (hkl) values and inter-planar spacing (d):

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

The calculated lattice parameter 'a' for all NPs samples are listed given Table 1. The composition differentiation of lattice variable 'a' as the function of zinc content is given in Fig. 2. Fig. 2 shows a monotonic increase of lattice constant from 8.34Å to 8.455 Å by raising concentration of Zinc and ascribed to the difference of ion radii of  $\text{Zn}^{2+}$  (0.83Å) which is larger than of  $\text{Ni}^{2+}$  (0.65Å)[6, 20, 21].

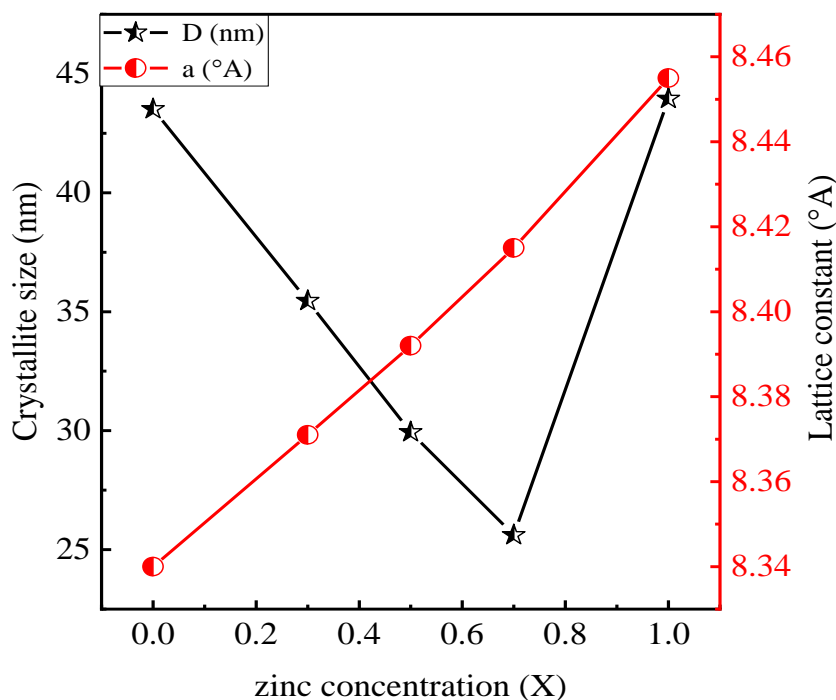


Fig. 2. Compositional variation of size of crystallite ‘D’ (nm) and lattice constant ‘a’ (Å) of  $\text{Ni}_{1-(X)}\text{Zn}_{(X)}\text{Fe}_2\text{O}_4$  NPs annealed at  $800^\circ\text{C}$

### 3.2. FT-IR measurements

Figure 3 shows FTIR measurements of all composition nanoparticles in the scale  $350\text{--}4000\text{ cm}^{-1}$ . The weak absorption bands between  $3300\text{--}3420\text{ cm}^{-1}$  also  $1612\text{--}1629\text{ cm}^{-1}$  are equivalent to widening and bending vibrations of hydroxyl (O–H) group because the surface absorbs the water molecules [22, 23]. The bands seeming in the range ( $\nu_1 = 560\text{--}590\text{ cm}^{-1}$ ) and ( $\nu_2 = 468\text{--}477\text{ cm}^{-1}$ ) are submitted to the stretching vibrations of tetrahedral ( $\nu_1$ ) and octahedral ( $\nu_2$ ) of metal ion - oxygen (M–O) bonds [24]. The frequency band at around  $471\text{ cm}^{-1}$  is the cause of the presence of divalent ( $\text{Ni}^{2+}$ ) metal ions in octahedral (B-sites) with different forms of lattice vibration [25, 26]. The FT-IR frequency bands position of increased Zn concentration are listed in table 2. It also found that the peak position value ( $\nu_1$  and  $\nu_2$ ) were identified to be decreased and increased, respectively, with the increase  $\text{Zn}^{2+}$  contents except for the  $\text{ZnFe}_2\text{O}_4$  sample. By adding  $\text{Zn}^{2+}$  ions to tetrahedral (A- sites) that has larger radius and higher atomic mass, it causes migration in  $\text{Fe}^{3+}$  to octahedral (B- sites), also it decreases the tetrahedral vibration frequency ( $\nu_1$ ). This implies increases the octahedral vibration frequency ( $\nu_2$ ) as a result

of the exchange in  $\text{Fe}^{3+}$  ions to the octahedral (B-site) [27,28]. The existence of these two basic bands proved the development of spinel ferrite structure of synthesized ferrites NPs [29].

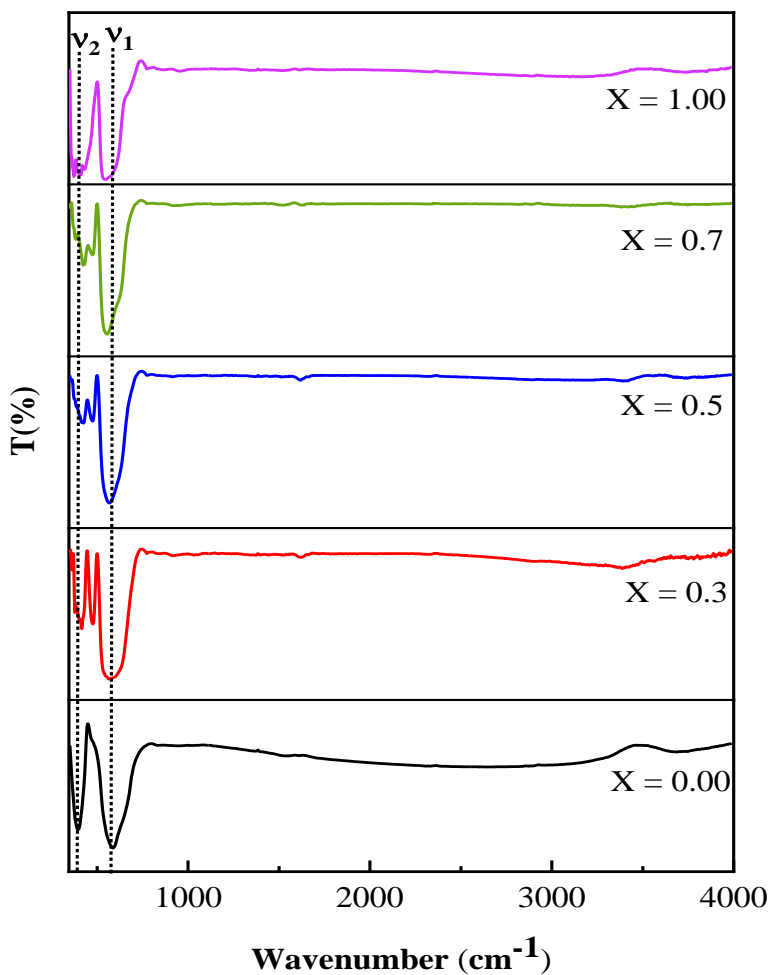


Figure 3. FT-IR spectra of  $\text{Ni}_{1-(X)}\text{Zn}_{(X)}\text{Fe}_2\text{O}_4$  [ $X = 0.00, 0.3, 0.5, 0.7, 1.00$ ] NPs

Zinc Content $X$	$\nu_1$ [1/cm]	$\nu_2$ [1/cm]
0.00	586	395
0.3	570	420
0.5	564	428

0.7	551	428
1.00	544	412

Table 2. FT-IR frequency bands position for tetrahedral ( $\nu_1$ ) and octahedral ( $\nu_2$ ) sites

### 3.3.FE-SEM with EDS

Figure 4(a-c) shows FE-SEM pictures of  $\text{Ni}_{1-(X)}\text{Zn}_{(X)}\text{Fe}_2\text{O}_4$  [(X) = 0.00, 0.5, 1.00] NPs. The FESEM images of the pure nickel ferrite sample (X) = 0 show the least spherical and irregular, non-symmetrical particles and formation of agglomeration (fig4(a)). For Ni– Zn mixed ferrite sample (X) = 0.5 the agglomeration shows uniform particles with spherical shape (fig 4(b)). From fig. 4(c), the FESEM pictures indicates, that pure zinc ferrite nanoparticles (X) = 1 possess uniformity and spherical symmetry with few agglomerations. However, it is also observed that at (X) = 1 the crystallite size increase which is in decent agreement with XRD results. Figure 5 (a)-(c) shows EDS pattern of (X) = 0.00, 0.5, and 1.00 NPs. Peaks of Ni, Fe, and O were observed in pure nickel ferrite (X) = 0 sample (fig. 5(a)), and peaks corresponding to Ni, Fe, O, and Zn elements are observed in Zn-doped  $\text{NiFe}_2\text{O}_4$  sample (X) = 0.5 as given in fig. 5(b). Fig. 5(c) demonstrated the peaks of Zn, O, and Fe in a pure zinc ferrite sample (X) = 1., and no other peaks were observed, which was confirmed that the annealed samples are pure and there is no other impurity.

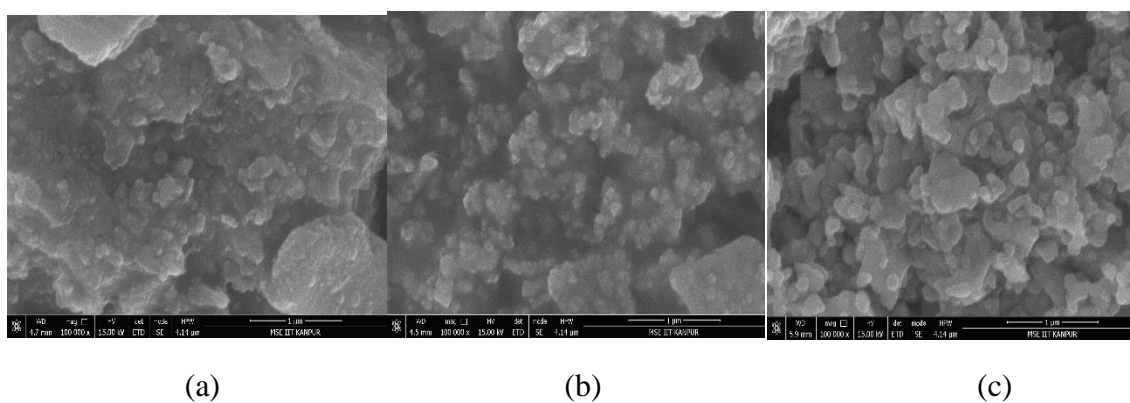


Figure 4. FE-SEM images for (a-c)  $\text{Ni}_{1-(X)}\text{Zn}_{(X)}\text{Fe}_2\text{O}_4$  [(X)=0.00, 0.5 and 1.00] nanoparticles.



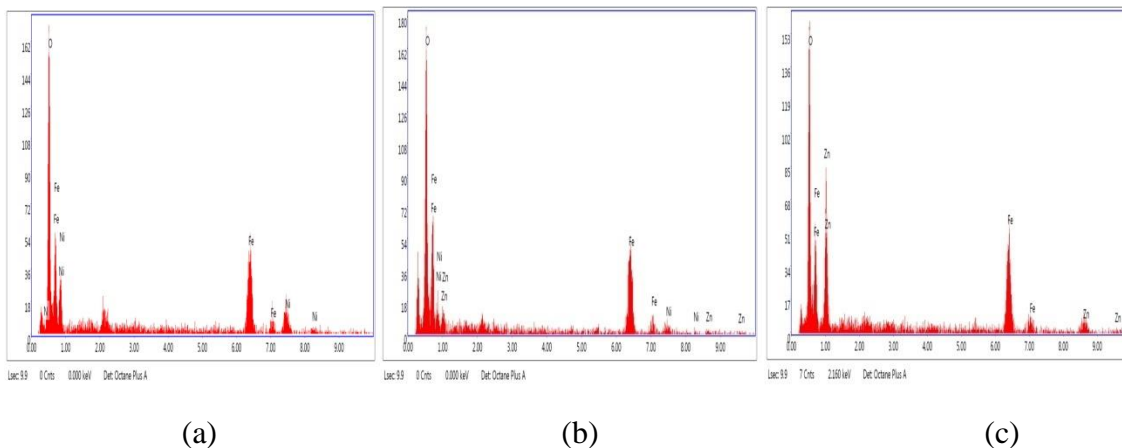


Figure 5. EDS spectra for (a-c)  $\text{Ni}_{1-(X)}\text{Zn}_{(X)}\text{Fe}_2\text{O}_4$  [(X) = 0.00, 0.5 and 1.00] nanoparticles.

### 3.4. VSM (magnetic study)

Magnetic properties were performed through VSM which is an applied magnetic field of  $\pm 20\text{kOe}$  at  $300\text{K}$ . Figure 6 shows (M-H) curve of synthesized all composition NPs of various Zn content. The remanent magnetization ( $M_R$ ), coercive field ( $H_C$ ) and saturation magnetization ( $M_S$ ), values were calculated from the curves and given for different Zn concentrations in Table 3. It is noted that the  $M_S$  has no monotonic behavior as Zn content increases. It is initially increase up to (X) = 0.5 from  $27\text{ emug}^{-1}$  to  $60\text{ emug}^{-1}$  later decrease to  $44\text{ emug}^{-1}$  at (X) = 0.7 and again increases to  $46\text{ emug}^{-1}$  at (X) = 1.

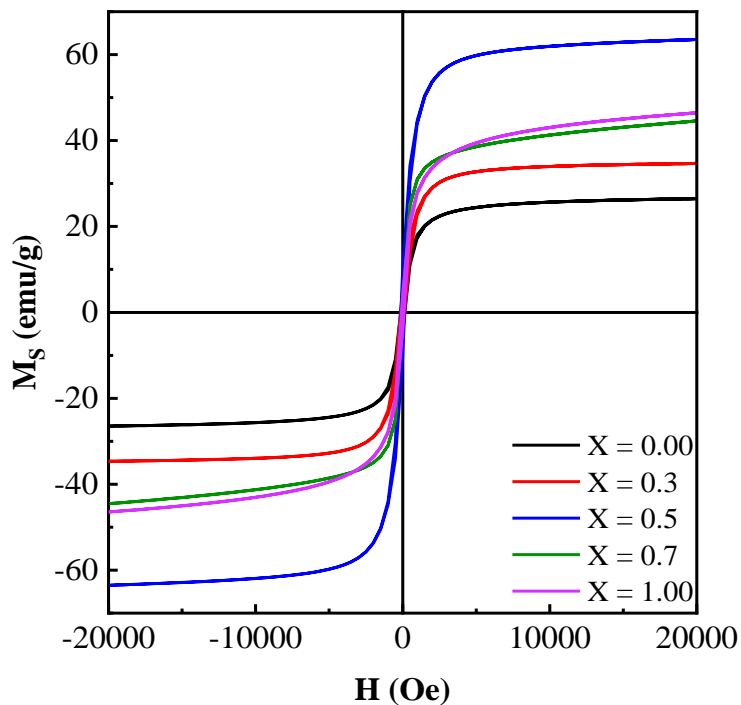


Figure 6. (M-H) loops of  $\text{Ni}_{1-(x)}\text{Zn}_{(x)}\text{Fe}_2\text{O}_4$  NPs sample

Zn Content (X)	$M_s$ ( $\text{emu g}^{-1}$ )	$M_R$ ( $\text{emu g}^{-1}$ )	$H_c$ (Oe)
0.00	26.5	4.2	139
0.3	34.5	5.8	149
0.5	63.3	10.3	124
0.7	44.2	2.7	50
1.00	46.5	4.3	89

Table 3. Magnetic characteristics ( $M_s$ ,  $M_R$  and  $H_c$ ) of  $\text{Ni}_{1-(x)}\text{Zn}_{(x)}\text{Fe}_2\text{O}_4$

The variation in saturation magnetization ( $M_s$ ) was impacted by various factors which are crystallite size, spin canting and the exchange interaction (B-B, A-B) changes between tetrahedral (A) and octahedral (B) sub lattice [30, 31]. The magnetic moment of  $\text{Zn}^{2+}$ ,  $\text{Ni}^{2+}$ , and  $\text{Fe}^{3+}$  are 0, 2, and 5  $\mu_B$ , y only considering spin contribution. The occupancy of non-

magnetic  $Zn^{2+}(0\mu_B)$  substituted ions in tetrahedral (A-site) cause's transfer of  $Fe^{3+}(5\mu_B)$  ions from (A- site) to (B-site). This leads to the magnetic moment in tetrahedral site (A) decreasing and the increases of magnetic moment in octahedral site (B). Thus increase the net magnetization up to  $(X) = 0.5$ . This magnetic performance can be learned through Neel's model [27, 32]. However the  $M_S$  decreases at  $(X) = 0.7$ . The reason for the decrease in saturation magnetization for  $Ni_{0.3}Zn_{0.7}Fe_2O_4$  NPs is that when the Zn content increase in the compound the super-exchange interaction A-B were damaged also the octahedral spins are not held rigidly comparable to the little spins of tetrahedral sites, and thus canted spin structure occurs which causes the  $M_S$  to decrease at  $(X) = 0.7$  [33, 34]. The sample  $ZnFe_2O_4$  with concentration  $(X) = 1$  showed again an increase in  $M_S$ . The increase in  $M_S$  at  $(X) = 1$  was attributed as it raises the crystallite size of the sample. Generally, the surface to volume ratio decreases and increases the crystallite size which will lead to an increase in saturation magnetization [30, 35]. Figure 7 shows the ( $M_S$ ) as a function of concentration of Zinc. The difference of coercivity with increased  $Zn^{2+}$  content was attributed to different factors, like domain structure, crystallite size, and magnetic crystallite anisotropy [12, 36].

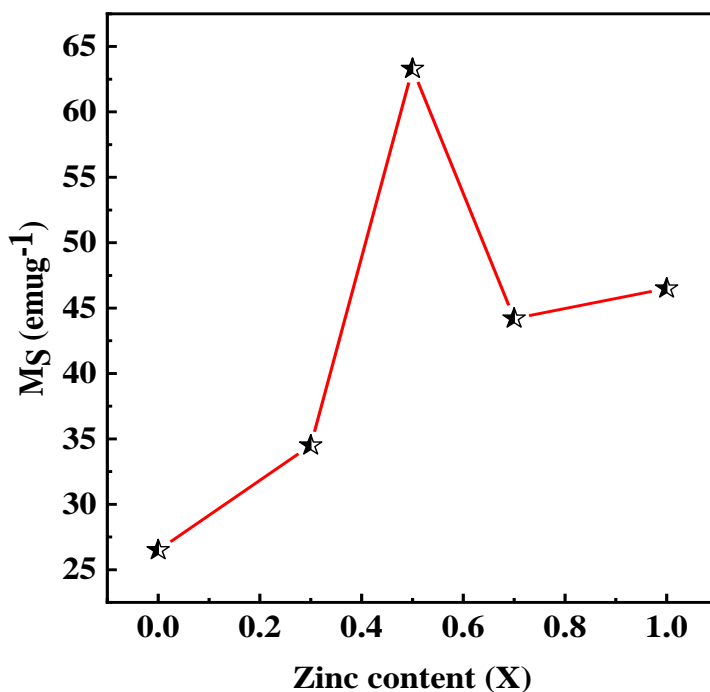


Figure 7. ( $M_S$ ) as a function of zinc concentration

### 3.5. Optical properties

The energy band gap of all NPs were determined from optical absorption spectra using UV-Vis spectrometer at RT as given in figure 8.

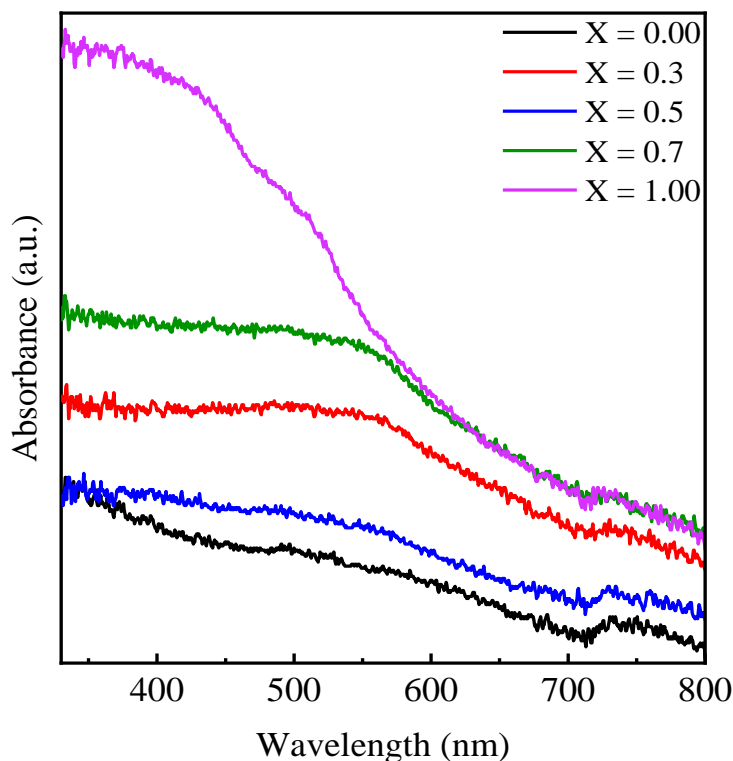


Figure 8. Absorption spectra of Ni<sub>1-(X)</sub>Zn<sub>(X)</sub>Fe<sub>2</sub>O<sub>4</sub> [(X)= 0.00, 0.3, 0.5, 0.7 and 1.00] NPs

The band gap energy ( $E_g$ ) values of all the samples were calculated using Taucs relation [37]:

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

Where  $h$ ,  $\alpha$ ,  $\nu$ ,  $A$ ,  $E_g$ , and  $n$  are constant, light frequency, band gap energy, absorption coefficient, Planck's constant, as well as exponent depend on the type of electron transition. The direct band gap ( $n = 1/2$ ) of different Zn concentrations was estimated by linear extrapolation of plotting  $(\alpha h\nu)^2$  against photon energy ( $h\nu$ ) to the energy axis as shown in figure 9.

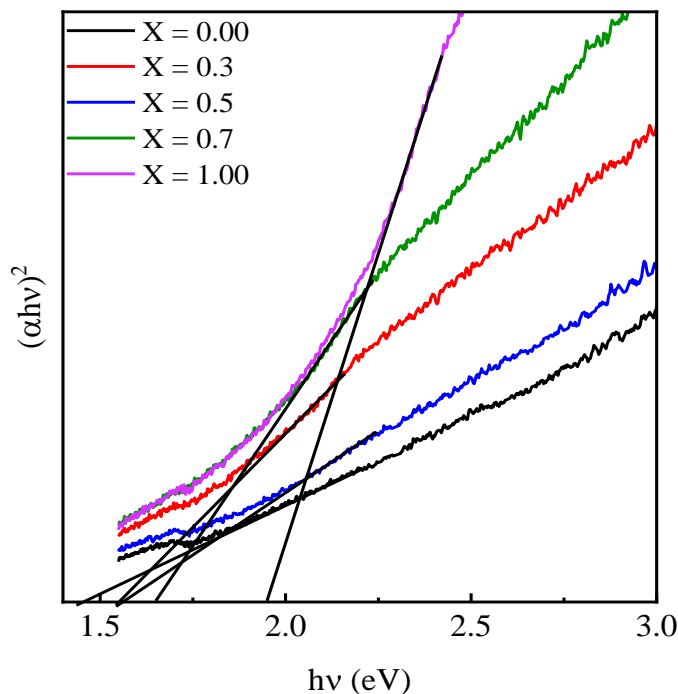


Figure 9. Tauc plot of  $[\text{Ni}_{1-(X)}\text{Zn}_{(X)}\text{Fe}_2\text{O}_4$  ( $X = 0.00, 0.3, 0.5, 0.7$  and  $1.00$ )] NPs samples

The calculated  $E_g$  values of ( $X = 0.00, 0.3, 0.5, 0.7$  and  $1.00$ ) nanoparticles are  $1.46, 1.55, 1.56, 1.65,$  and  $1.95$  eV, correspondingly. The energy band gap ( $E_g$ ) amount is found to increase with increasing Zn content that may be related to several factors which are crystallite size, interface effect, presence of impurity as well as structure parameter [22, 32, 38]. Increasing the direct band gap for these samples may attribute to a decrease in crystallite size (quantum size effect) [37, 39]. The increase with band gap energy might be the cause of (sp-d) interchange interaction among the localized (d) electrons of ( $\text{Zn}^{2+}$ ) ions along with band electrons of  $\text{NiFe}_2\text{O}_4$  [40, 41]. Note that the narrowed band gap values with ( $\text{Zn}^{2+}$ ) dopant in  $\text{NiFe}_2\text{O}_4$  lattice specify the development in sub-bands amongst the energy band gap and inclusion of its sub-bands along the conduction band (CB) to create a continuous band [42].

#### 4. Conclusion

By using the sol-gel combustion approach the nano ferrites samples  $[\text{Ni}_{1-(X)}\text{Zn}_{(X)}\text{Fe}_2\text{O}_4$ : ( $X = 0.00, 0.3, 0.5, 0.7,$  and  $1.00$ )] were formed. All synthesized NPs samples were annealed for 4 hours at  $800^\circ\text{C}$  in the air. The development of cubic spinel ferrite structure for all samples are established by XRD and FT-IR analyses. The lattice constant is identified to

be increased as zinc content is increasing from 8.240 Å to 8.455 Å. Magnetic properties through (VSM) indicated that  $\text{Ni}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$  has the maximum saturation magnetization ( $M_s$ ) value of 63.3 emu/g. UV-visible (Vis) spectroscopy study revealed the direct band gap of the undoped  $\text{NiFe}_2\text{O}_4$  is 1.46 eV, and by increasing the Zn -content is increasing from 1.55 to 1.95 eV.

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