

Effect of Copper Doping on the Structural, Morphological and Optical Properties of Zinc Ferrite Nanoparticles

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ABSTRACT

The $\text{Cu}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$ ($x = 0, 0.05, 0.1$) nanoparticles were prepared by co-precipitation method. For preparation iron chloride $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$, zinc chloride ZnCl_2 and copper chloride $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ were used. The synthesized copper doped ZnFe_2O_4 nanoparticles were characterized by techniques such as XRD, High-resolution SEM, FT-IR spectroscopy and UV-visible spectroscopy. The single phase cubic spinel structure of $(\text{Cu}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4 \ x = 0, 0.05, 0.1)$ nanoparticles was revealed by XRD. The results of XRD confirmed the crystallite size and lattice parameter determined as $18\text{nm} - 10\text{nm}$ and $8.4880\text{\AA} - 8.4135\text{\AA}$ respectively. The SEM analysis described spherical morphology of samples. FTIR spectroscopy was done for the investigation of functional group analysis and vibrational characteristics. The presence of metal oxygen bond was detected by FTIR analysis. The UV-Visible spectroscopy was performed to find the band gap of all the samples. The value of band gap energy as calculated by using Tauc plot method was found to be 2.0eV to 2.7eV . The impact of cation distribution can effectively tuned and has potential application in photocatalysis, sensing optoelectronic devices and electromagnetic technologies of various emerging fields of nanomaterials.

Keyword: $(\text{Cu}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4 \ x = 0, 0.05, 0.1)$ Co-Precipitation, XRD, FTIR, SEM, UV–Visible, Spectroscopy Tauc Plot, Sensing, Electromagnetic Technologies.

Introduction

The name “ferrite” is taken from the Latin word “ferrum,” which means iron. It means iron oxide is the basic component of ferrites, which are uniform ceramic materials made of different oxides[1]. Ferrites with spinal structure are the intriguing material due to the application in sensors, catalysts and magnetic storage devices, because of their well-known electrical and magnetic properties. By doping zinc ferrites with copper Cu^{+2} , one may modify their optical and structural properties, customizing their performance for certain applications. The composition, ion valence state, magnetic characteristics and distribution within the real structure all affect the functional qualities of ferrites [2]. Spinal ferrites are excellent choice owing to photocatalysis, eco-friendly and light sensitivity [3]. A normal or direct spinel system is one in which every divalent cation is found in the tetrahedral position. There should be an equal number of trivalent cations must settle tetrahedral sites when octahedral sites are filled by a portion or the totality of divalent cations under certain conditions [4]. The general relation for spinal ferrite is AFe_2O_4 , where A represents divalent metal cation such as copper, nickel, manganese, zinc and cobalt etc on the tetrahedral site. Iron represent octahedral site The examples included are MnFe_2O_4 , CoFe_2O_4 , CuFe_2O_4 , NiFe_2O_4 , and ZnFe_2O_4 [5]. The spinal ferrites are seen in the form of hard, soft magnetic material and their composites. These material reflects their magnetic properties like saturation magnetization coercivity, retentivity, permeability and susceptibility [6]. However, because of their excellent corrosion resistance, high electrical resistivity, high Curie temperature, soft and hard ferrite composites are advantageous for advanced permanent magnetic materials [7].

On the basis of crystal structure ferrites included are spinal ferrite, hexagonal ferrite, garnet and ortho-ferrites. The spinal category of ferrites is one of the best that have also three types such as normal, inverse and mixed ferrites. The spinal ferrites have Fd-3m space group or space number 227 represents the face centred cubic crystal structure [8]. Doping of copper in zinc ferrite nanoparticles may alter the structural, morphological and optical properties. Keeping in view the prospectus of these characteristics, they have many potential uses in current emerging science and technology[9]. The copper doped zinc ferrites nanoparticles have applications in advance sensors, biomedical, high frequency electronics and photocatalysis [10].

Synthesis methodology of $\text{Cu}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$ ($x = 0, 0.05, 0.1$)

Cu^{2+} -doped and pure zinc ferrite nanoparticles were produced by using the low cost and eco-friendly co-precipitation method. In order to get a product free of contamination, beakers, flasks and burettes were cleaned with deionized water before to starting the experiment. Both zinc chloride (ZnCl_2) solution and iron chloride ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$) solution were mixed in a ratio 1:2 for pure sample (ZnFe_2O_4). To get the copper doped preparation copper chloride ($\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$) solution was added drop-wise with the help of burette. After Mixing and continuous stirring, the NaOH is added to maintain pH(9-10). This stage enhanced homogeneity and encouraged full precipitation. The sample is then moved to water bath for 2 hours keeping temperature at 90 degree. Then samples undergo the filtration washing and drying at 80 degree until 7pH. Now obtained sample is grind to get fine powder of $\text{Cu}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$ nanoparticles and kept in furnace at 800 degree for 4 hours [11].



Figure 1. $\text{Cu}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$ Nanoparticles synthesis process [11].

Results and Discussion

The synthesized samples are brought to characterization techniques such as X-ray Diffraction (XRD), Fourier Transform Infrared spectroscopy (FTIR), High Resolution Scanning Electron Microscopy (SEM) and Ultraviolet-Visible Spectroscopy (UV-Vis). The XRD determined the crystal structure and lattice parameters such as crystallite size, lattice constant and texture coefficient. SEM was used to get surface morphology of all the compositions. FTIR identified the functional group and bonds. UV-Vis analysis lead to optical properties.

Structural Analysis with XRD.

The XRD analysis of pure and copper doped zinc ferrites is shown in figure 2. The XRD patterns revealed the following diffraction peaks are labelled with (hkl) values (220), (311), (400), (511) and (440). These crystallographic planes of pure and that of copper zinc ferrites are with approximate 2 theta positions 30, 35, 43, 57 and 62 degrees respectively. The diffraction data peaks are well agreement with JCPDS 01-077-0011 for pure sample of ZnFe_2O_4 . The doped samples diffraction analysis $\text{Cu}_{0.05}\text{Zn}_{0.95}\text{Fe}_2\text{O}_4$ and $\text{Cu}_{0.1}\text{Zn}_{0.9}\text{Fe}_2\text{O}_4$ are consistent with JCPDS 01-077-13 and 01-077-12. As shown below in Figure 2. The presence of most intense peak is (311) and weak one is (400). The XRD diffraction patterns confirmed the spinal structure of all the compositions. Because the indexed reflections are consistent with the characteristics single-phase formation in ferrite structure as reported in previous literature [12].

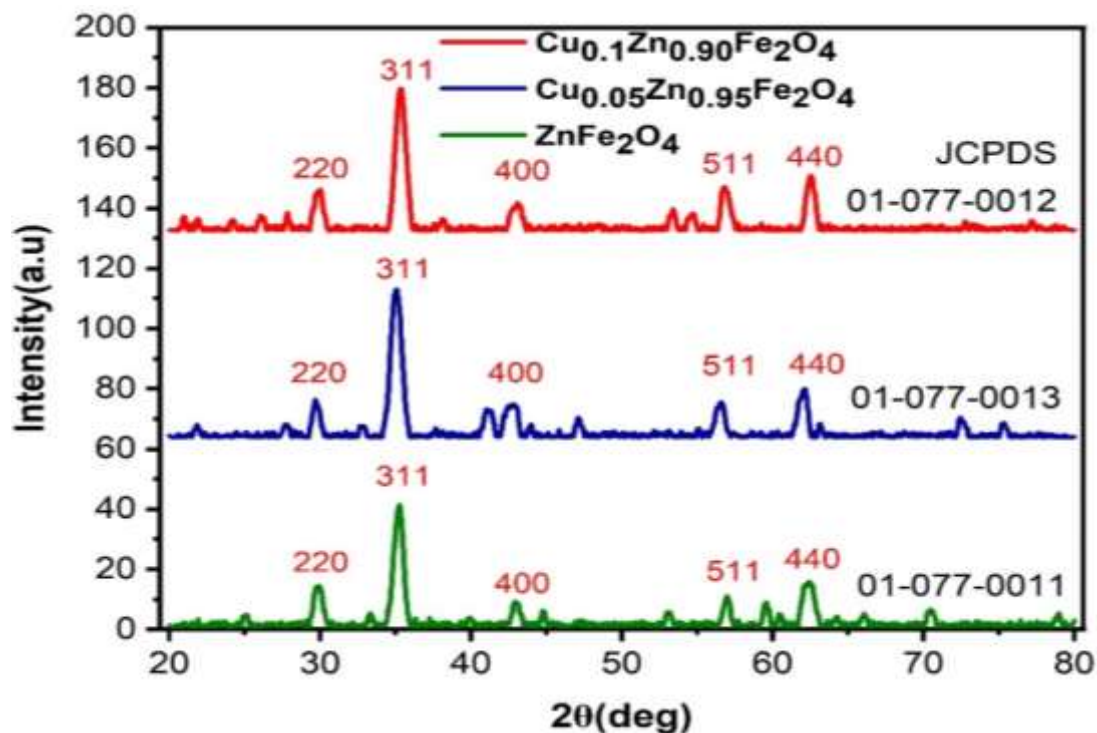


Figure 2. XRD Patterns of $\text{Cu}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$ ($x = 0, 0.05, 0.1$).

The copper doping produced a slight variation in peak position and relative intensity due to modification in lattice structure and cation distribution because the Cu^{+2} are being substituted with Zn^{+2} ions. No additional phase is observed and the spinel phase is retained with copper incorporation [13]. The crystallite size D , the lattice constant a and texture coefficient calculated with following formulas,

$$D = \frac{0.9\lambda}{\beta_{hkl} \cos \theta} \dots\dots\dots 1$$

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

$$TC = \frac{N(I(hkl)/I_0(hkl))}{\sum(I(hkl)/I_0(hkl))} \dots\dots\dots 3$$

The crystallite size values changes from 18nm to 10nm and the lattice constant values ranges between 8.4880 to 8.4135 Å.

Table 1. Phase shift analysis in $\text{Cu}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$ ($x = 0, 0.05, 0.1$).

Miller Indices (hkl)	0% Cu 2Theta(deg)	5% Cu 2Theta(deg)	10%Cu 2Theta(deg)
220	29.84	29.70	29.93
311	35.18	35.02	35.29
400	42.78	42.61	42.93
511	56.62	56.40	56.85
440	62.18	61.94	62.45

It has been observed that the crystallite size and lattice constant decreases gradually but this trend may not be universal for higher concentration of doping material [14].

Online ISSN

3007-3197

Print ISSN

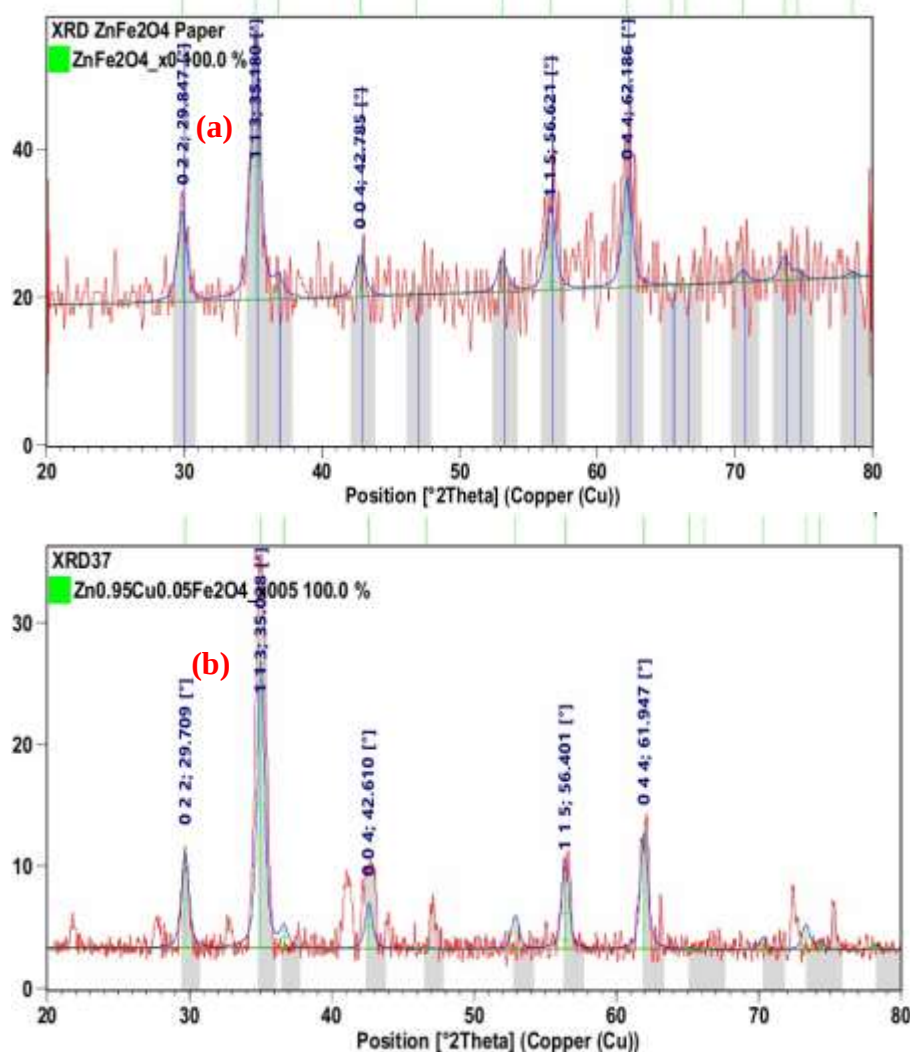
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Table 2. Texture coefficient values overview

Miller Indices (hkl)	ZnFe ₂ O ₄	Cu _{0.05} Zn _{0.9} Fe ₂ O ₄	Cu _{0.1} Zn _{0.9} Fe ₂ O ₄
220	1.01	1.04	1.01
311	0.95	0.94	0.95
400	0.92	0.90	0.94
511	0.95	0.92	0.92
440	1.17	1.19	1.19

The plane (440) has the highest texture coefficient among all of three samples suggesting a preferred orientation of planes in this direction.



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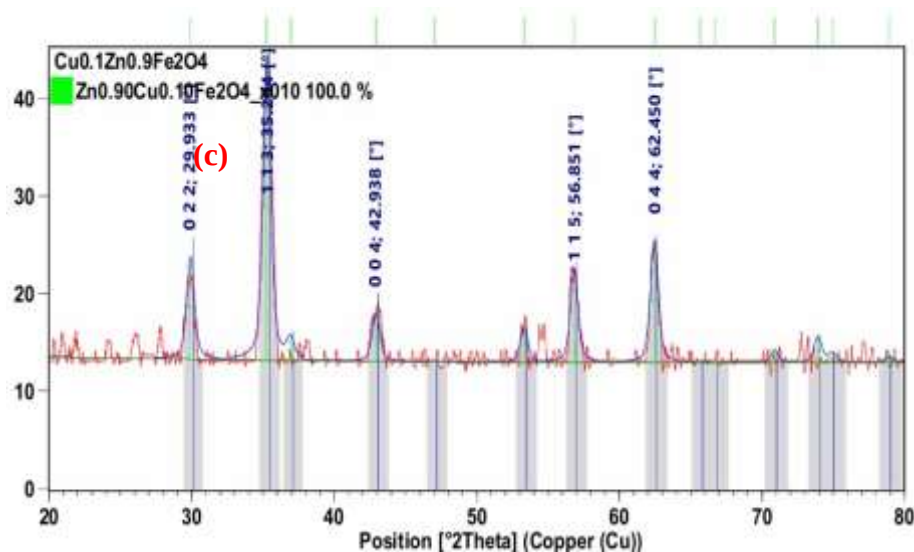


Figure 3. Rietveld refinement of $\text{Cu}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$ (a) $x=0$, (b) $x=0.05$, (c) $x=0.1$

Morphological Analysis (SEM)

The SEM analysis defined the surface morphology of prepared nanoparticles, grain shape and their characteristics. The spherical/quasi spherical shape of nanoparticles is obtain with agglomeration [15].The SEM pictures at x10K magnification are shown in Figure 4.

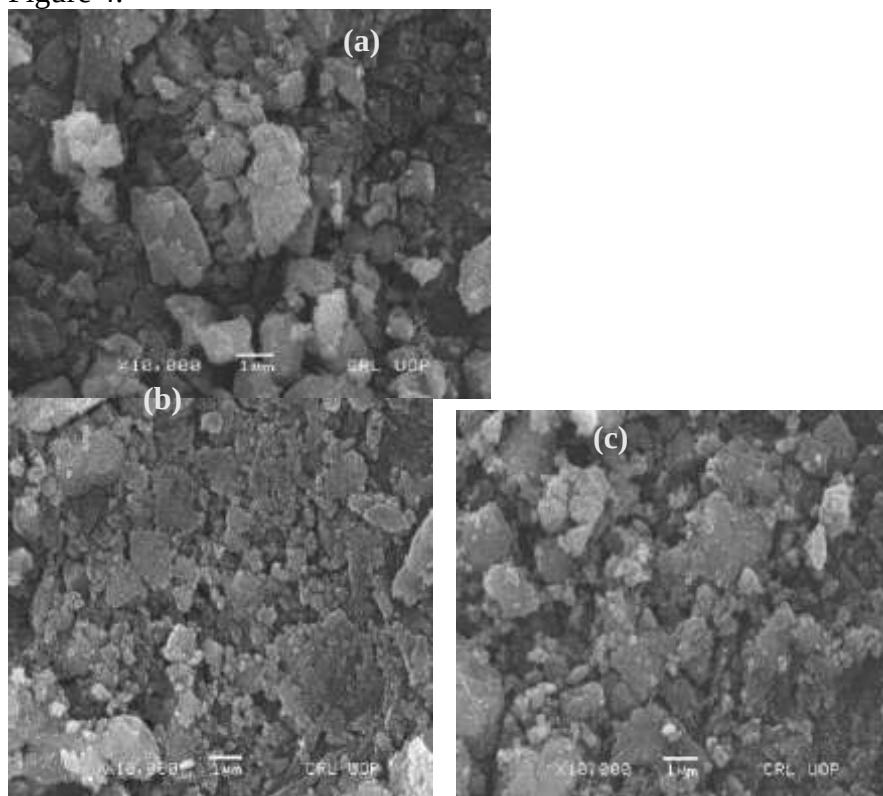


Figure 4. SEM of $\text{Cu}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$ (a) $x=0$, (b) $x=0.05$, (c) $x=0.1$

Functional group analysis (FTIR)

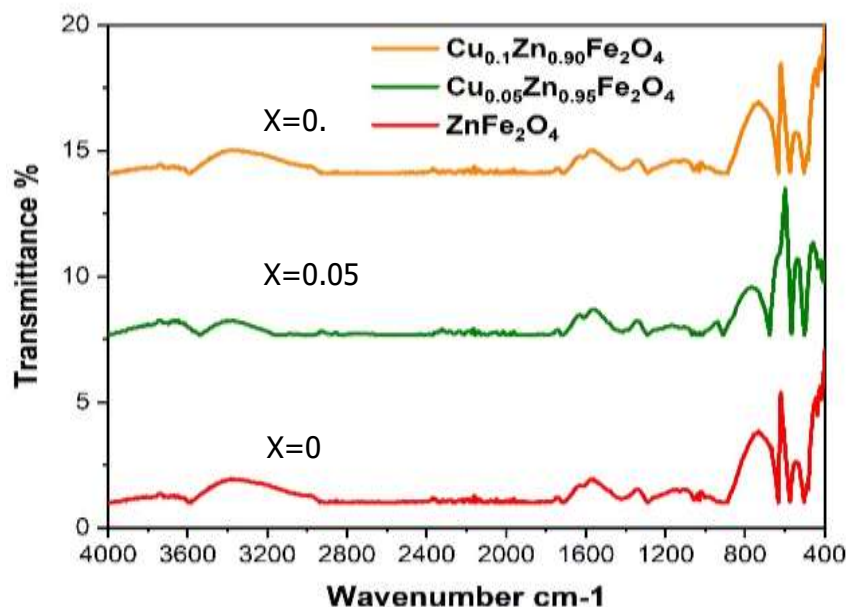
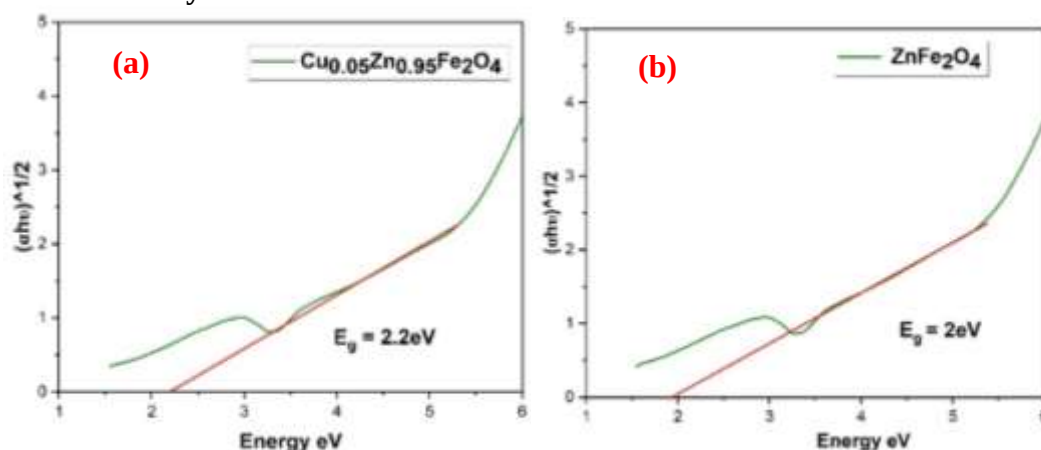


Figure 5. FTIR of $\text{Cu}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$ ($x = 0, 0.05, 0.1$).

The Fourier Transform infrared Spectroscopy is a vital tool for bonds and functional group analysis on the basis of wavenumber analysis corresponding to a specific bond or a group. The vibrational characteristics are also determined with this analysis. The FTIR was conducted in the range from 400cm^{-1} to 4000cm^{-1} . The part of pattern from 450cm^{-1} to 650cm^{-1} is characteristic region that exhibits metal oxygen bond and confirmed the spinel ferrites. The metal oxygen band is retained after the doping of copper. The highest band observed around 3600cm^{-1} absorption corresponds to the O-H stretching [16]. The FTIR patterns show two characteristic absorption bands in low wavenumber region associated with spinel ferrite lattice. The upper range of this region $500\text{--}650\text{cm}^{-1}$ lies in metal oxygen bonds at tetrahedral site A, and the lower range of this region $400\text{--}500$ corresponds to metal oxygen vibration at octahedral sites [17].

UV- Vis Analysis.



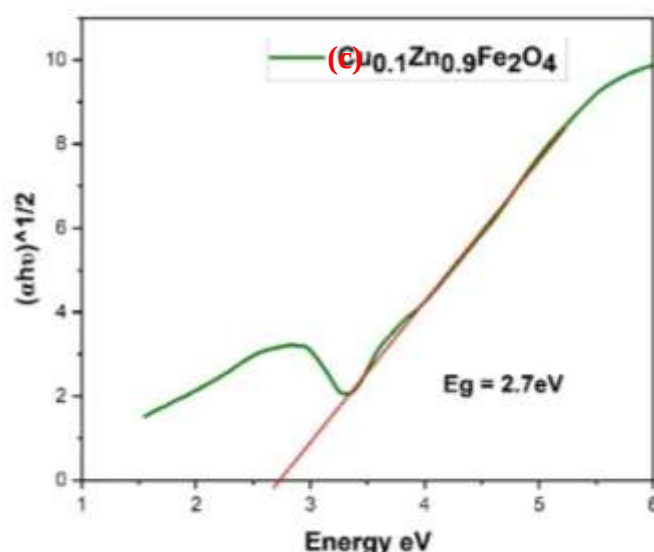


Figure 6. Tauc plots of $\text{Cu}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$ (a) $x=0$, (b) $x=0.05$, (c) $x=0.1$

The UV visible spectroscopy is a technique that is used to investigate the optical characteristics of synthesized nanomaterial. The Tauc plot method was used to estimate the energy band gap with relation 4 and 5.

$$E_g = 1240/\lambda \dots\dots\dots 4.$$

$$(\alpha h\nu)^{1/n} = B(h\nu - E_g) \dots\dots\dots 5.$$

$1/n$ depends on the type of electronic transition either direct or indirect. For $n=1/2$ a direct transition and if $n=2$ then it's indirect transition [18]. The optical properties enabled one to check the suitability of material for potential application in various field of science and technology. The energy band gap of the material get increased by the incorporation of dopant material [19].

Conclusion

Co-precipitation method based $\text{Cu}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$ ($x = 0, 0.05, 0.1$) nanoparticles were prepared. Copper doping in ZnFe_2O_4 leads to variation in the structural, morphological and optical properties. XRD analysis revealed changes in lattice constant and crystallite size was determined. The lattice constant and crystallite size gradually decreases with the increase of dopant material. The structural analysis confirmed the single phase spinel structure for all the compositions. As calculated the crystallite size decreases from 18nm – 10nm, and the lattice constant from 8.4880 Å – 8.4135 Å accordingly. This decrease is due to copper doping, also redistribution cation at tetrahedral and octahedral site, because Zn^{+2} is being substituted with Cu^{+2} . The SEM analysis revealed the spherical/quasi spherical morphology of synthesized nanomaterial. The FTIR technique confirmed the tetrahedral region 500cm^{-1} - 650cm^{-1} and octahedral 400cm^{-1} – 500cm^{-1} region present in spinel ferrite. The presence of metal oxygen bond, O-H stretching and vibrational characteristics at different wavenumber are shown in the FTIR spectra. The UV visible spectroscopy investigation gave the optical energy band gap of the nanoparticles. The band gap get widen by the incorporation of copper. The energy band gap changes from 2eV to 2.7eV as calculated by using Tauc plot method. The increase of band gap could yield

the way for copper doped zinc ferrite nanoparticles for potential application in photocatalysis, optoelectronic devices and sensing technology.

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