



# INVESTIGATIONS ON STRUCTURAL AND FUNCTIONAL PROPERTIES OF ZINC-COBALT FERRITE NANOPARTICLES

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**Abstract:** In this study, the hydrothermal method has been used in the Co–Zn spinel ferrites with the chemical formula  $\text{Co}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4$  ( $0.1 \leq x \leq 0.9$ ) nanoparticles, which have been characterized using X-ray diffraction (XRD), Fourier transform Infrared spectroscopy (FTIR), UV-Vis spectrometer, and photoluminescence properties. XRD investigation confirms the cubic spinel structure and the Debye-Scherrer formula was used to determine the crystallite size, showing values of 4.839 to 5.896 nm. FTIR confirms the presence of all functional groups and A, B-site wave numbers are studied and these are responsible for the characteristic of spinel ferrites. The UV-visible analysis was used to explain the variation of optical energy bandgap as Zinc doping was increased and Photoluminescence intensity was found to vary from 420-425 nm for CZF nanoparticles.

**Key words** - XRD, FTIR, UV-Vis, PL and crystallite size.

## 1.INTRODUCTION

Nanotechnology has revolutionized various scientific and industrial fields by offering a novel avenue for designing and engineering materials at the nanoscale. Co-Zn nanoferrite is a type of magnetic nanoparticle that has garnered significant attention in recent years due to its unique properties and potential applications in various fields. These nanoparticles are composed of cobalt (Co), zinc (Zn), and iron (Fe) oxide, with the composition  $\text{Co}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4$ , where  $x$  represents the Co to Zn ratio. The unique composition and structure of Co-Zn nanoferrites make them promising materials for a wide range of applications in biomedical, environmental, and industrial fields. One of the most significant advantages of Co-Zn nanoferrites is their high magnetic anisotropy, which means they have a preferred direction of magnetization. This property makes them ideal for various magnetic applications such as magnetic data storage, magnetic hyperthermia, and magnetic resonance imaging (MRI). Co-Zn nanoferrites can be used as contrast agents for MRI due to their high magnetic moment and excellent biocompatibility. Co-Zn nanoferrites have also been studied for their potential applications in the field of environmental remediation. They can be used as adsorbents for removing heavy metals and organic pollutants from water and soil due to their high surface area and magnetic properties. Additionally, they have been shown to have excellent photocatalytic activity, making them potential candidates for the degradation of organic pollutants under visible light irradiation. In the field of industrial catalysis, Co-Zn nanoferrites have been studied for their potential applications as catalysts in various chemical reactions. Due to their high surface area and unique magnetic properties, they have shown excellent catalytic activity in various reactions such as oxidation, reduction, and hydrogenation. The spinel ferrite materials are good candidates for distinct scientific and industrial fields because of their high saturation magnetization, high magnetic permeability, low electrical conductivity, and inexpensive [1]. However, these parameters mainly depend upon the type of cations, charge carriers (electrons or holes), and cationic distribution at A & B-sites

(tetrahedral & octahedral respectively) [1]. Many scientists have reported different properties including structural, optical, and magnetic, that are ideal for high-frequency applications. Therefore, these ferrite materials can be extensively useful for electromagnetic device applications [2]. In the literature it is found that several scientists worked on zinc ferrite (ZF) & cobalt ferrites (CF) and doped nanoparticles for electrical, magnetic, photocatalytic, and superparamagnetic properties [3-6]. Herein, for the synthesis of nanoparticles, citric acid worked as an added precursor along with the stoichiometric materials. In addition to these, spinel cobalt ferrite materials exhibited magnetic recording and magneto-optical recording media applications as noticed in the literature [7-10]. Therefore, the magnetic spinel structured materials preserved be expected for various technological field applications [11]. Additionally, zinc ferrite showed a normal spinel structure with an attractive structural, morphological, and magnetic properties [12]. On the other hand, the CF exhibited inverse spinel structure in nanoform [5-6]. Therefore, it can be expected that the zinc addition to the CF may reveal different electrical, magnetic, thermal, and antimicrobial properties. In addition, it was also seen that previous scientists prepared the Co-Zn ferrites in both bulks as well as nanoform via different synthesis methods such as low-temperature hydrothermal technique, starch-assisted sol-gel auto combustion method, microwave combustion method, coprecipitation method followed by high-temperature sintering, and organic solution techniques [13-17]. The obtained results of these studies were confined to only structural, optical, magnetic, cation distribution, and catalysis activity properties. Besides, the literature survey expressed the fact that the  $\text{CoFe}_2\text{O}_4$  and iron oxide superparamagnetic nanomaterials were studied for magnetic hyperthermia therapy, core/shell structures, and different magnetic properties [18-24]. As a whole, this evidenced a fact that the X-ray photoelectron spectroscopy, thermal, antimicrobial, and magnetic properties were not studied at length for the Co-Zn ferrite system via the citrate gel auto combustion method. Therefore, an intention was carried out to investigate the above-mentioned properties of the Co-Zn ferrite system to advance the scientific community. The general formula  $\text{Co}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4$  represents a solid solution of transition metal ferrites where the composition of cobalt ( $\text{Co}^{2+}$ ), zinc ( $\text{Zn}^{2+}$ ), and iron ( $\text{Fe}^{2+}$ ) ions can vary systematically. The most common values of  $x$  in this formula are 0.1, 0.3, 0.5, 0.7, and 0.9, which correspond to different ratios of cobalt and zinc relative to iron. This controlled variation allows researchers to tailor the physicochemical properties of these nanoparticles to suit specific applications.

In this paper, we report the influence of Zn doping on the structural, morphological, optical, electrical, and magnetic properties of  $\text{Co}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4$  nanoparticles. The substitution of non-magnetic Zn ion is expected to modify crystal as well as other properties by altering the cation distribution between the two crystallographic A and B-sites.

## 2. MATERIALS AND EXPERIMENTAL METHODS

For the synthesis of  $\text{Co}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4$  ( $0.1 \leq x \leq 0.9$ )/CZF nanoparticles, we selected the starting materials as Cobalt nitrate [ $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  (99.8% purity, Sigma-Aldrich)], Zinc nitrate [ $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  (99.8% purity, Sigma-Aldrich)], and ferric nitrate [ $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$  (99.9% purity, Sigma-Aldrich)]. The sodium hydroxide (NaOH) pellets and their aqueous solution were also used to work as the solvent, in the hydrothermal reaction.

At the outset, the stoichiometric equation of CZF nanoparticles was prepared as  $\text{Co}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4$  ( $0.1 \leq x \leq 0.9$ ). Using this stoichiometric formula, the raw materials (in nitrate form) were weighed on a digital sensitive balance and mixed in a fresh glass beaker. Afterward, the nitrate materials were dissolved in distilled water contained in a glass beaker. The mixed solution was then kept on a magnetic stirrer with a stirring rate of 500 rpm. In the meantime, the NaOH aqueous solution was added drop by drop to the nitrate solution. As a result of the stirring of the solution for 2 h, the white and delicious solution was obtained. This solution was further shifted to a Teflon bowl of 500 ml capacity and kept in a stainless steel autoclave. Afterward, the screws of the autoclave were made tight to prevent the explosion of the solution at the time of hydrothermal reaction. Then the autoclave was kept in a programmable hot air oven. In the oven, the hydrothermal reaction was performed at  $160^\circ\text{C}/8$  h. After completion of the reaction, the oven was cooled down to room temperature. In the next step, the sample was removed from the Teflon bowl and then cleaned 10–12 times until the pH reached 7. Later on, the powder sample was dried at  $60^\circ\text{C}$  for 2 h in the hot air oven. The dried CZF nanopowder was ground in an agate mortar for 15 min to achieve homogeneous powder particles.

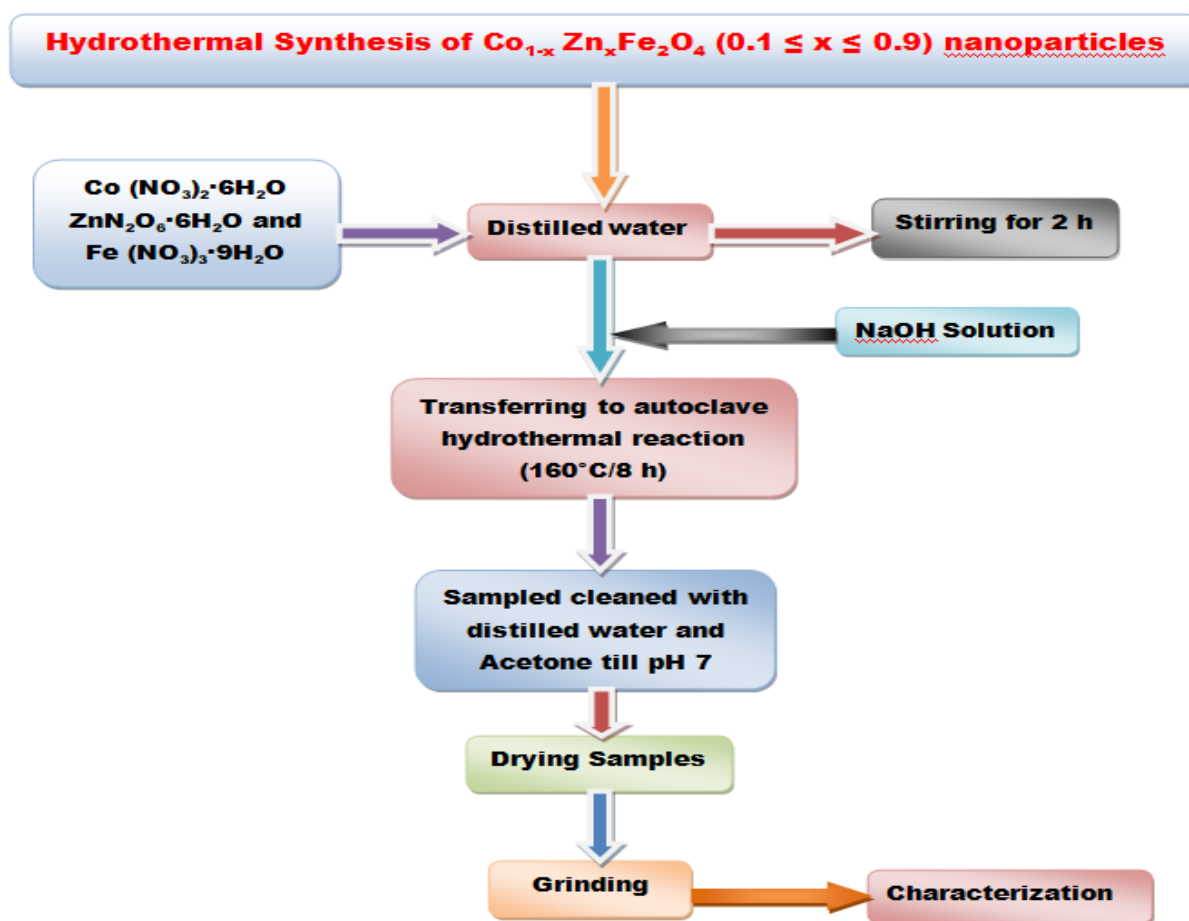


Fig.1. Flow chart for preparation of CZF nanoparticles

### 3. RESULTS

#### 3.1. X-ray diffraction (XRD)

The X-ray diffraction (XRD) patterns of  $\text{Co}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4$  ( $0.1 \leq x \leq 0.9$ ) powder nanoparticles prepared are shown in Fig.2. The results showed the present CZF nanoparticles were crystal structures of cubic spinel phase. The XRD patterns were confirmed (JCPDS Number 22-1086). The diffraction pattern typically shows characteristic peaks at  $2\theta$  values  $30.1^\circ$ ,  $35.8^\circ$ ,  $43.2^\circ$ ,  $47.3^\circ$ ,  $53.7^\circ$ ,  $56.9^\circ$ ,  $62.7^\circ$  and  $68.0^\circ$ . These peaks correspond to the (220), (311), (400), (331), (422), (511), (440) and (620) planes, respectively of the prepared CZF nanoparticles were cubic spinel phase [25]. Based on the diffraction peaks and using the Debye–Scherrer formula:  $D = k\lambda/\beta\cos\theta$ , where  $\beta$  is FWHM,  $\lambda$  is the wavelength of  $\text{CuK}\alpha$  source (0.15406 nm) and  $\theta$  is the diffraction angle [26-27]. The average crystalline size was found to vary from 4.839 to 5.896 nm with the increase in zinc content. It may happen microstrain  $\epsilon$  varies from 0.0113 to 0.0412 with a doped zinc content of 'x'. The maximum intense reflection plane was noted to be (311). In addition, the lattice constants (a, b & c) were found to be equal ( $a = b = c$ ) and the achieved value was observed to vary with the increase of zinc content varying from 8.329 to 8.401 Å [28-29]. This implied that the variation trend of the lattice constant obeys Vegard's law [30]. This kind of manner is obtained because of the occupation of  $\text{Co}^{2+}$  (0.58 nm) cations of smaller ionic radii by the  $\text{Zn}^{2+}$  (0.60 nm) cations of larger ionic radii [31]. Subsequently, the unitcell volume (V) was also increased from 577.97 to 591.31 Å<sup>3</sup>. This indicated the fact that the expansion of the unit cell took place in the CZF nanoparticles with Zn-content. Besides, the X-ray density ( $\rho_x$ ) of CZF nanoparticles was found to vary from 5.34 to 5.41 g/cm<sup>3</sup> using the formula:  $ZM/Na^3$ , herein 'Z' indicates the effective number of atoms per unit cell (8), 'M' is associated to the compositional molecular weight, 'N' is the Avogadro's number and 'a' is lattice parameter of content [32]. The present behavior of  $\rho_x$  is obtained as a result of the decrease of molecular weight with an increase in zinc concentration. Moreover, this kind of nature was noticed in the literature [33]. In a few cases, the linear increase of 'a' with Zn-content can also be expected as another reason for the behavior of X-ray density [34]. The bulk density ( $\rho_b$ ) of CZF nanoparticles was estimated to vary from 4.021 to 4.071 g/cm<sup>3</sup> using equation  $\rho_b = M/\Pi r^2 h$ . Further, the porosity of the CZF nanoparticles was found to vary from 0.2469 to 0.2477 using the equation: Porosity (P %) =  $[1 - (\rho_x/\rho_b)] * 100$ . The CZF nanoparticles were determined dislocation density ( $\rho$ ) varying from 2.877E+16 to 4.271 E+16 m<sup>-2</sup>, a specific surface area (S) varying from 190.57 to 231.76 m<sup>2</sup>/g listed in Table 1.

### 3.2. WILLIAMSON- HALL ANALYSIS

To calculate approximately the strain and crystallite size, we have preferred the W-H plot for CZF nanoparticles shown in Fig.3 and expressed in equation [35]:  $B \cos\Theta = \frac{K\lambda}{D} + 4\epsilon \sin\Theta$ , Where  $\lambda$ = wavelength,  $\beta$ =Full-Width Half Maxima (FWHM),  $\Theta$ = Bragg's diffraction angle,  $\epsilon$ = Micro-strain and  $D$ = crystallite size. The slope of the W-H plot gives the Micro-strain value. The CZF nanoparticles Micro-strain ( $\epsilon_{W-H}$ ) varied from 0.0299 to 0.0519 and the crystallite size ( $D_{W-H}$ ) was calculated to be varying from 2.679 to 4.62 nm of zinc content 'x'. The average crystallite sizes estimated by the two methods are close to each other. Further, a good agreement is perceived between the values expressed from the W-H Plot based on Scherrer's formula and the values  $\epsilon_{W-H}$  and  $D_{W-H}$  in Table 1. [36].

#### 1.1.OPTICAL PROPERTIES

FTIR spectra of  $Co_{1-x}Zn_xFe_2O_4$  ( $0.1 \leq x \leq 0.9$ ), the sample codes are C1, C2, C3, C4, and C5 of room temperature in a wave number range from 400 to 4500  $cm^{-1}$  are represented in Fig.4. All FTIR spectra in the described range demonstrate two well-defined peaks, first one around 462.72  $cm^{-1}$  to 475.03  $cm^{-1}$  ( $\nu_b$ ) and the other near 539.67  $cm^{-1}$  to 557.11  $cm^{-1}$  ( $\nu_a$ ), which are related to intrinsic stretching. The metal-oxygen (M-O) vibration bands like Co-O, Zn-O & Fe-O can be noticed in CZF samples [36] as it is known that the  $Zn^{2+}$ ,  $Fe^{3+}$ ,  $Co^{2+}$  ions can occupy both A and B-sites of spinel structure [1]. Vibrations of the oxygen bond with metal cations in positions of the tetrahedral sites (A- sites) and the octahedral sites (B-sites). The weak frequency peak ( $\nu_b$ ) attributed to the intrinsic vibration of the metal-oxygen bond at the B- sites and the high-frequency peak ( $\nu_a$ ) shown the metal-oxygen vibration of the A- sites [37] are shown in Fig.4.

Table 1. Data on the XRD structural and physical parameters of CZF nanoparticles

Parameters	C1 (x=0.1)	C2 (x=0.3)	C3 (x=0.5)	C4 (x=0.7)	C5 (x=0.9)
Lattice Parameter (a)	8.329	8.374	8.372	8.401	8.397
Crystallite size( $D_{ave}$ ) in nm	4.886	4.839	4.840	5.896	5.582
Strain $\epsilon$	0.0412	0.0354	0.0173	0.0113	0.0128
$D_{W-H}$	4.62	2.679	3.136	3.044	4.09
$\epsilon_{W-H}$	0.0299	0.0517	0.044	0.045	0.0339
FWHM in radians	0.04747	0.0596	0.0265	0.0235	0.0240
Volume V ( $A^{03}$ )	577.97	587.35	587.62	594.72	592.31
MW g/mol	235.265	236.55	237.84	239.13	240.42
Surface Area S ( $m^2/g$ )	226.98	231.76	230.42	190.57	199.42
Dislocation density ( $m^{-2}$ )	4.189 E+16	4.271 E+16	4.269 E+16	2.877 E+16	3.209 E+16



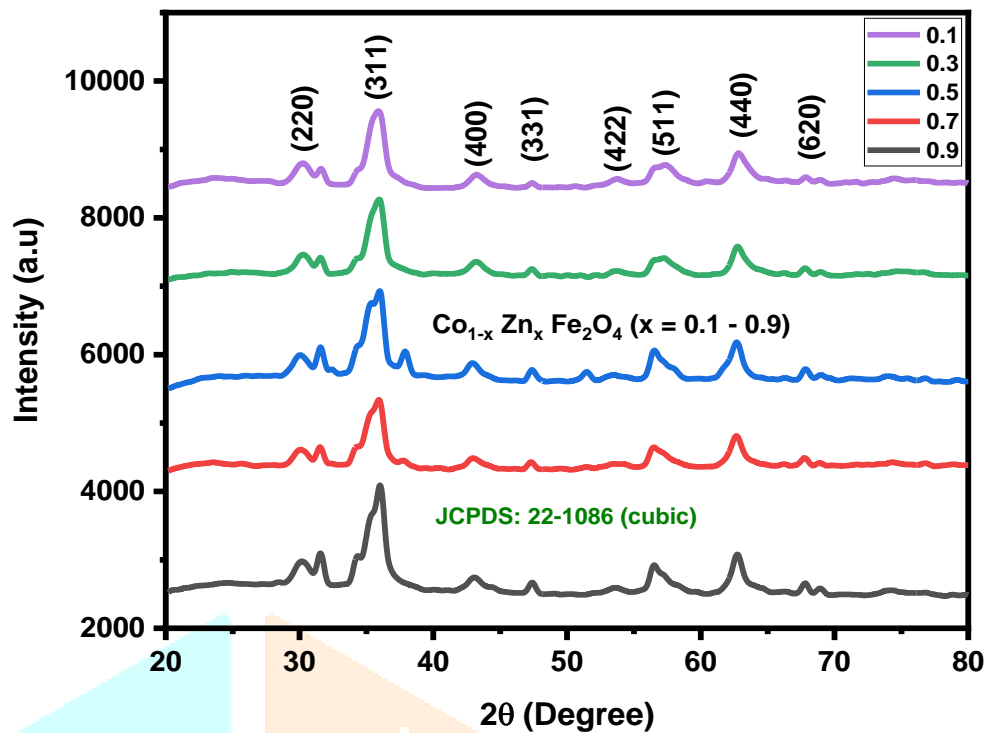
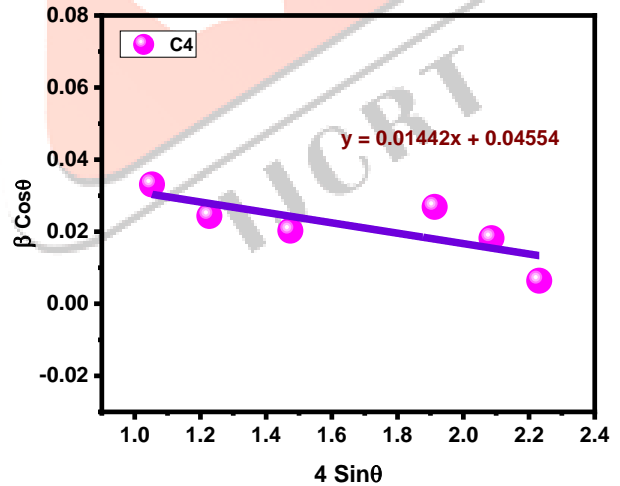
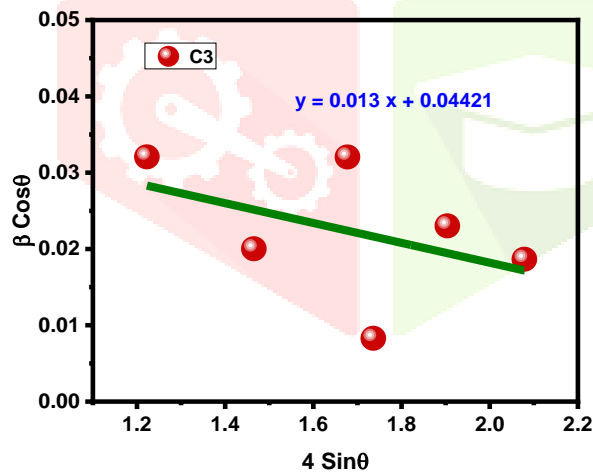
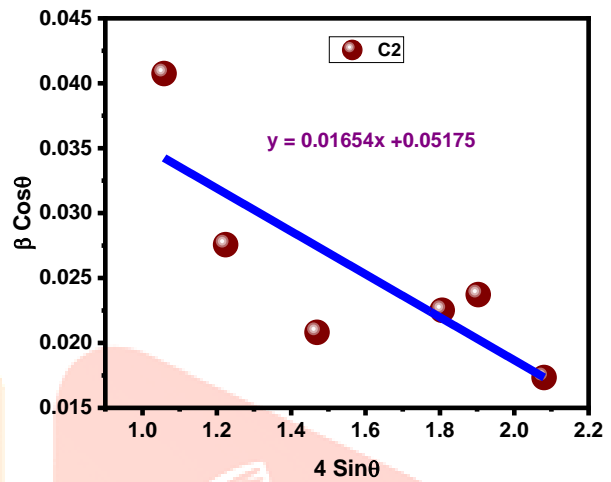
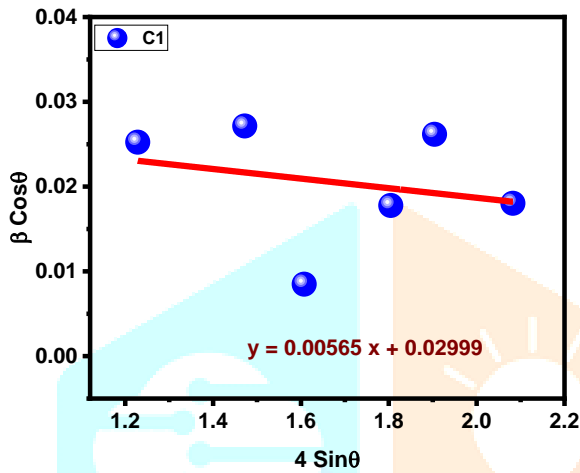


Fig.2. X-ray diffraction (XRD) pattern of CZF nanoparticles

To calculate the optical energy gap ( $E_g$ ), the Kubelka-Munk function was considered. Using this, the function of reflectance  $F(R)$ :  $(1-R)^2/2R$ , (where  $R$  = reflectance which was recorded from diffuse reflectance spectra as shown in the inset of Fig.5) was computed [33]. Here, the  $F(R)$  was observed to be directly proportional to the absorptivity ( $\alpha$ ), and therefore, in place of ' $\alpha$ ', we can substitute  $F(R)$ . Further, using the equation:  $(\alpha h\nu)^n = m(h\nu - E_g)$  and the  $(\alpha h\nu)^n$  versus photon energy ( $h\nu$ ) plots, we calculated the  $E_g$  values of CZF. The exponent ( $n$ ) in the band gap equation was taken as 2 for the direct transition of charge carriers between two energy bands [33, 38]. The  $(\alpha h\nu)^n$  versus  $h\nu$  plots of  $x = 0.1-0.9$  contents showed the linear portions. The linear portion was extrapolated towards the  $h\nu$ -axis. Then, using the intersecting position of the straight line at the  $h\nu$ -axis, wherein  $\alpha$  approaches zero, the  $E_g$  values were calculated. These  $E_g$  values were indexed in Fig.4. The results indicated that the  $E_g$  values were varying from 0.935 to 1.524 eV with an increase of Zn-content from  $x = 0.1$  to 0.9. This kind of behavior is mainly attributed to the oxygen vacancies present in each composition. That is, the  $E_g$  value will be high for the lower number of oxygen vacancies and vice versa [38]. In the case of CZF nanoparticles, the numbers of oxygen vacancies were varying with the increase of Zn-content. Therefore, the concerned distance between the valence and conduction bands was varied (from 0.935 to 1.524 eV). However, the  $x = 0.1-0.9$  contents showed wide  $E_g$  values suggesting optoelectronic and sensor applications [38].

The photoluminescence spectra were found in the visible region and were independent of the particle size. The PL spectra of  $\text{Co}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4$  ( $0.1 \leq x \leq 0.9$ ) /CZF nanoparticles are shown in Fig.6. The Photoluminescence intensity was found varying from 420-425 nm for CZF nanoparticles. The most well-known peaks in visible regions were only one which is dependent on particle size. The PL emission of light from a material under optical excitation is an essential trend. If light of sufficient energy is incident on a material, photons are absorbed important near electronic excitations. These excitations then relax and the electrons return to the ground state. Photoluminescence occurs when the radiative relaxation occurs. Since the excitation wavelength might impact the photoluminescence of any material, the absorption of a material depends strongly on the energy of an incident light. This makes the selection of the excitation light consequently critical in the photoluminescence study of material. The excitation wavelength controls a density of photo-excited electrons and holes and this governs the behavior of these carriers. Here, the photoluminescence spectrums of CZF nanoparticles at room temperatures have been observed at the excitations varying from 420nm - 425nm. The spectrum consists of emission peaks varying from 420 nm (2.95 eV) - 425 nm (2.92 eV). It is also observed that the emission intensity of the peaks depends on the excitation wavelength. Concerning the mechanism of PL, it may be attributable to quantum confinement. This

confinement can be explained in terms of the shortening of a super-exchange interaction bond length in the nano-crystalline ferrite materials, which modifies the electronic structure of ferrite, or in terms of the occurrence of the fast non-radiative relaxation channels in nano-crystals which are enchanting part at the surface. The PL around 3 eV is indeed generally due to quantum confinement although the peak values approximately agree with an energy band gap of ferrite nanomaterials. The effect of the surface oxide and non-homogenous size was furthermore important if there is a difference within the energies, which is associated with the Stokes shift between the absorption surface defects or surface oxide. The mechanism of the charge transfer between the trivalent ions appears to involve a non-radiative super-exchange process via the intervening oxide ions that support the ferromagnetic ordering [39]. The PL is such that their intrinsic and extrinsic bands were within the visible range [40].



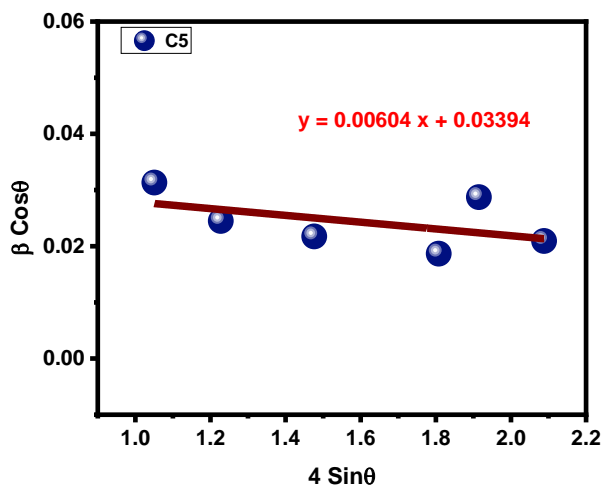
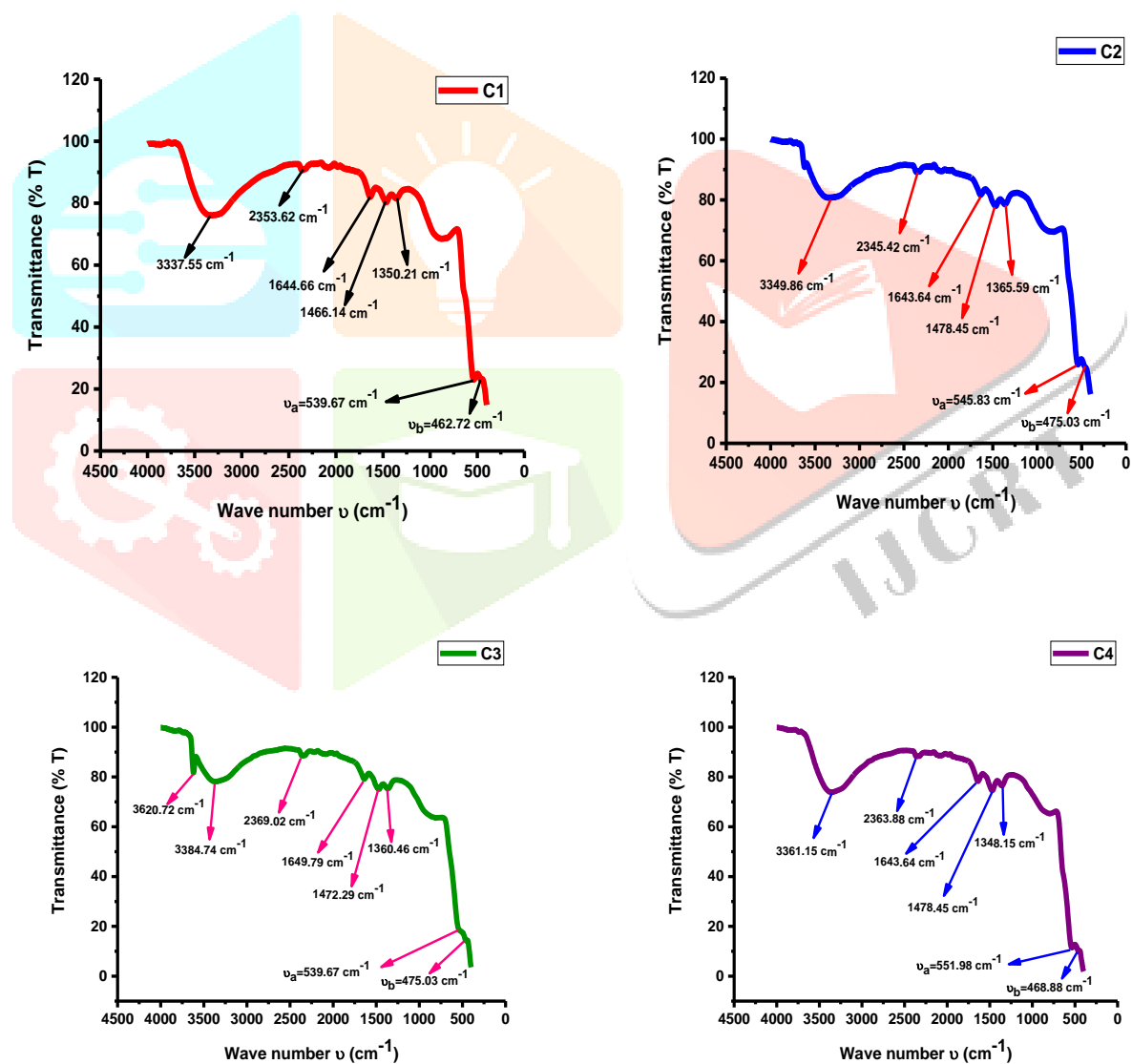


Fig.3. W-H Plot of CZF nanoparticles



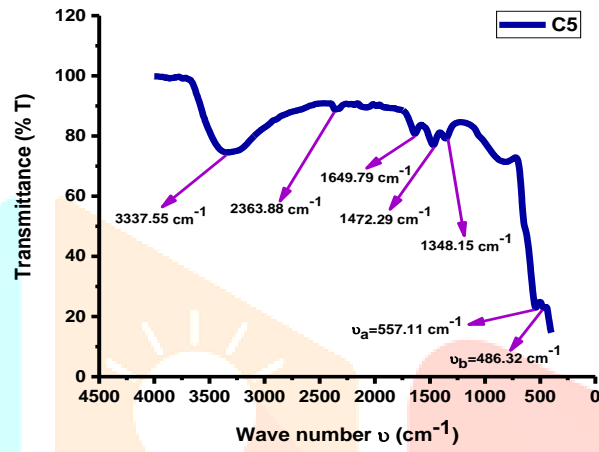
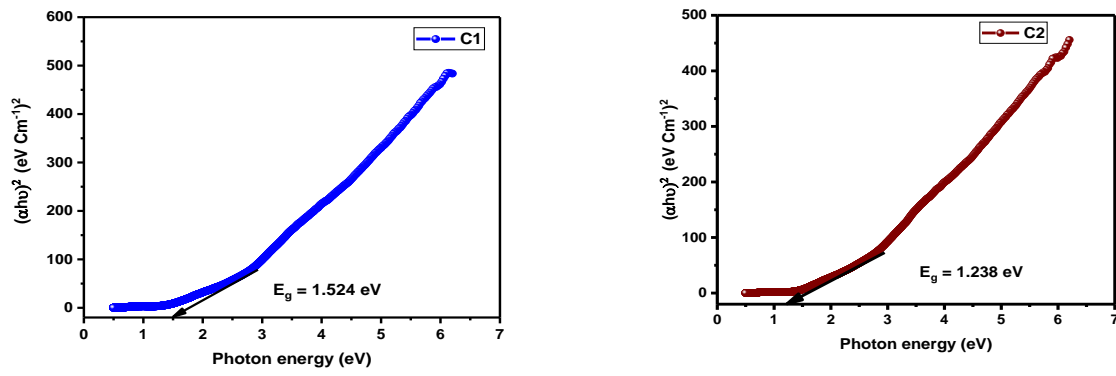


Fig.4. FTIR plots of CZF nanoparticles



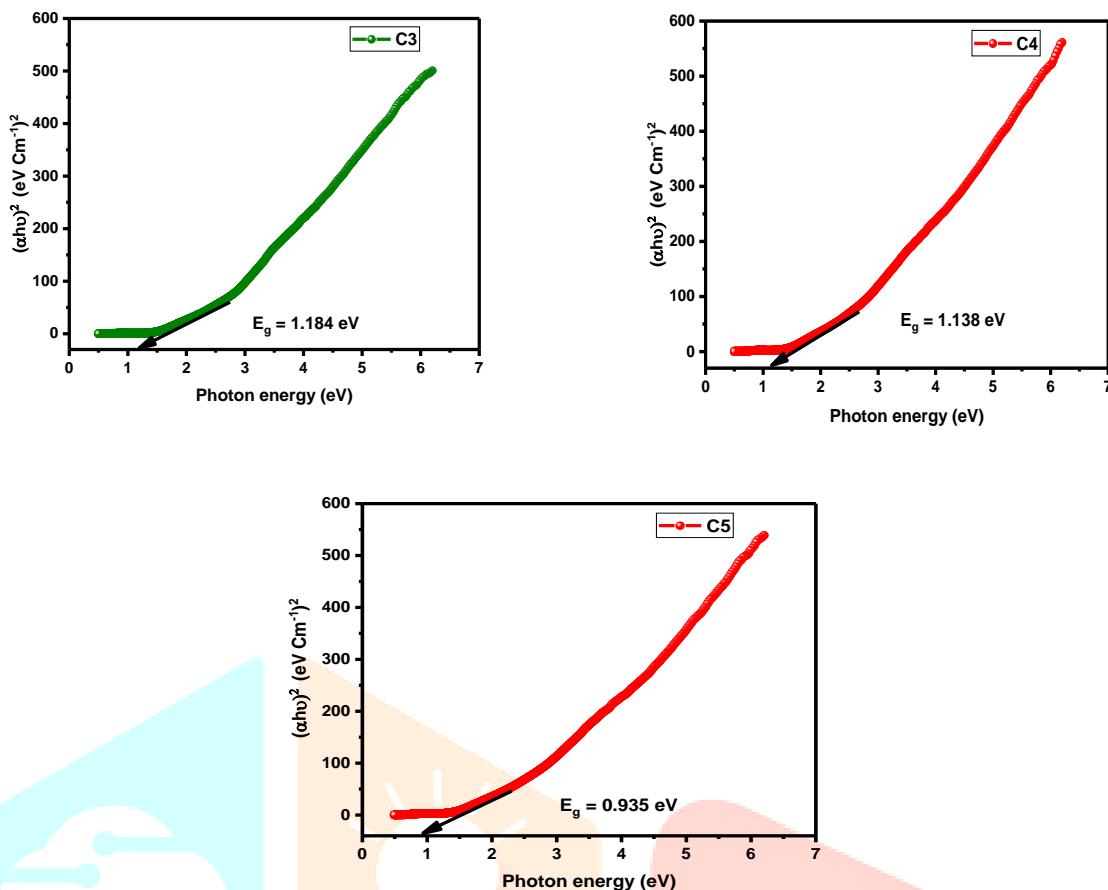


Fig.5. UV-Vis spectra of CZF nanoparticles

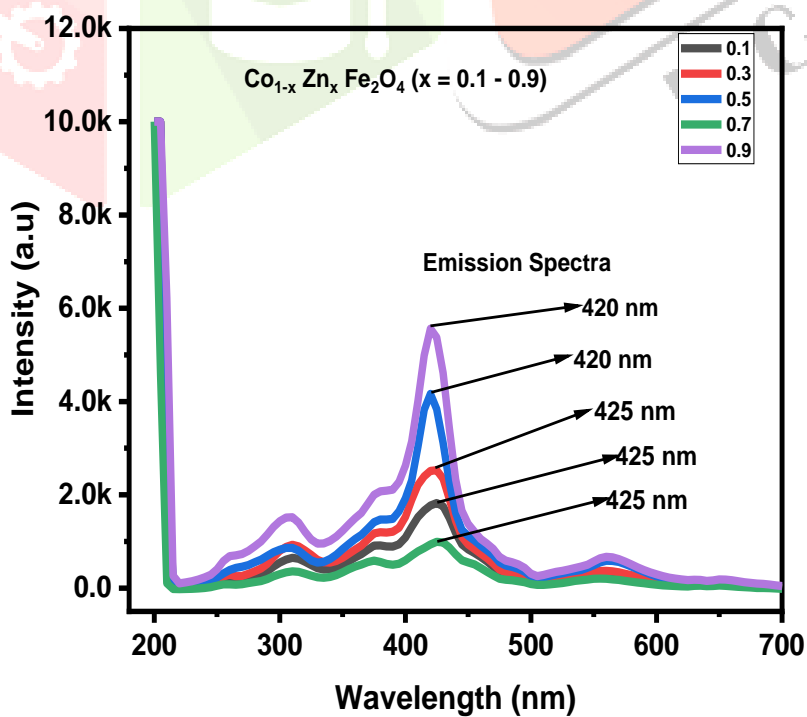


Fig.6. PL spectra of CZF nanoparticles

## 2. CONCLUSIONS

The hydrothermal method is found to be successful in the preparation of the spinel ferrite  $\text{Co}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4$  ( $0.1 \leq x \leq 0.9$ ) nanoparticles. The XRD has confirmed the formation of a cubic spinel structure. The lattice constants ( $a=b=c$ ) vary from 8.329 to 8.401 Å with an increase in zinc content. Using Scherrer's formula and W-H analysis, the average crystallite size and the lattice strains were determined, and average crystallite sizes estimated by the two methods are close to each other. Two main absorption peaks in the range from  $462.72 \text{ cm}^{-1}$  -  $475.03 \text{ cm}^{-1}$  ( $\nu_b$ ) for octahedral sites, and the range  $539.67 \text{ cm}^{-1}$  to  $557.11 \text{ cm}^{-1}$  ( $\nu_a$ ) for tetrahedral sites observed in FTIR spectra which confirmed the ferrite nature of the sample. The intense emissions of 420nm - 425nm occurred mainly due to radiative defects and oxygen vacancies in the room-temperature PL studies. The energy band gap of prepared samples was calculated from varying between 0.935 to 1.524 eV with increasing Zn content 'x'. These  $\text{Co}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4$  ( $0.1 \leq x \leq 0.9$ ) nanoparticles are promising for suggesting optoelectronic and sensor applications.

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