

# Chemical Synthesis and Characterization of Zn-doped Cadmium Oxide Based Nanoparticles: As Nanofluids for Thermophysical Applications

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## ABSTRACT

Semiconductor nanoparticles are studied worldwide at present because of their good optical, physical and chemical properties. In this research, a set of Zn doped cadmium oxide ( $Cd_{1-x}Zn_xO_{1-\delta}$ ) nanoparticles were synthesized by simple chemical precipitation route. The precursor materials used in this work were cadmium nitrate and zinc nitrate (as basic materials) and sodium hydroxide (as a precipitator material). The formed hydroxides of Cd/Zn were washed with water and ethyl alcohol (9:1 v/v) and dried at 75° C overnight. The dried powder was then heat treated for 1 h each at 150, 300, 450° C, respectively and then cooled down to room temperature. The prepared nanoparticles were characterized by XRD where the peaks are well matched with the phase purity of cubic structure. FTIR analysis confirmed the presence of the functional groups present in the pure and Zn doped CdO nanoparticles. The SEM results showed that the obtained nanoparticles are polycrystalline in nature, having the average grain size of 100-200 nm. The molecular interactions with respect to the different dopant concentrations (Zn) and their thermo-physical characteristics of CdO based nanofluids ( $Cd_{1-x}Zn_xO_{1-\delta}$ ; where  $x=0, 0.05, 0.10, 0.15$  and  $0.20$ ) prepared in ethylene glycol were also studied and reported. It was found that the intermolecular free length of the present nanofluids seemed to be increasing with the higher incorporation of the  $Zn^{2+}$  ions in CdO nanoparticles.

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## 1. Introduction

Nanofluids have been introduced as a novel route for heat transfer between the fluid and the surface that considerably reduces the heat transfer rates. There is a high demand for coolants with high performance and thermal conductivity. In the past few years, this has been achieved by dispersing the micrometer sized

metal or metal oxide nanoparticles into the base fluids [1]. These nanofluids can be synthesized by suspending a very small amount of nanoparticles (1 to 100nm) into the carrier fluid which can be either water or polymeric solutions or organic liquids which improves the thermal conductivity of these fluids [2]. When compared to the bulk fluids, nanofluids acquire efficient

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heat transfer properties, making them applicable to many industrial sectors such as power generation, microelectronics, transportation, solid state lighting, manufacturing, and so on [3 - 5]. These nanofluids can even surpass the conventional fluids by enhancing the molecular interactions, thermal conductivity, viscosity, and adiabatic compressibility [6]. However, these enhancements of nanofluids depend on several factors such as type of the particle, size, base fluids, molar concentrations, temperature and sonication of nanoparticles [7]. Lee et al. have measured and reported the thermal conductivity of fluids containing metals or metal oxides of conductivity higher than the carrier fluids (e.g. water, ethylene glycol, etc.) [8]. Experiments were performed on a variety of nanofluids with the effect of temperature combined with variation of volume fraction and reported that there is an effect in the thermal conductivity of the nanofluids [9].

Presently, semiconductor based nanoparticles have a greater importance for their physical properties and for their industrial applications such as photocatalysis, microelectronics, manufacturing, etc. They occupy the state in the transition regions between the molecules [10]. Cadmium oxide is a II-VI n-type semiconductor used in optoelectronic applications such as photovoltaic cells, solar cells, phototransistors, transparent electrodes, gas sensors and a variety of other materials depending upon its optical and electrical properties [11-13]. Several methods have been developed to synthesize cadmium oxide nanoparticles with various morphologies and structural aspects. It was reported that cadmium oxide nanoparticles have many advantages over the conventional materials because of their large surface area [14-16]. Dispersing the nanoparticles into the base fluid must be a challenging task in order to avoid agglomeration. Many researchers had used a two-step process for preparing stable nanofluids. In the two-step process, nanoparticles were first synthesized and then dispersed into the base fluid which effectively increased the thermal conductivity of the base fluid [17]. This method is simple and economical, too.

In this research paper, we report a simple chemical synthesis route for the preparation of the Zn – doped CdO nanoparticles. Chemical precipitation process is developed to avoid any sequential precipitation of metal ions in order to achieve desired stoichiometry [19]. This research proposes an experimental analysis on the behaviour of the Zn-doped CdO based nanoparticles as nanofluids in the ethylene glycol medium (EG) which has been used as the base fluid, a proven lubricating coolant in many industrial sectors and which has been found to have appreciable thermal conductivity proportions [7,18]. By dispersing the CdO based nanoparticles into the ethylene glycol medium, thermo-physical properties and the intermolecular interaction of the nanofluids at room temperature have been studied and analyzed.

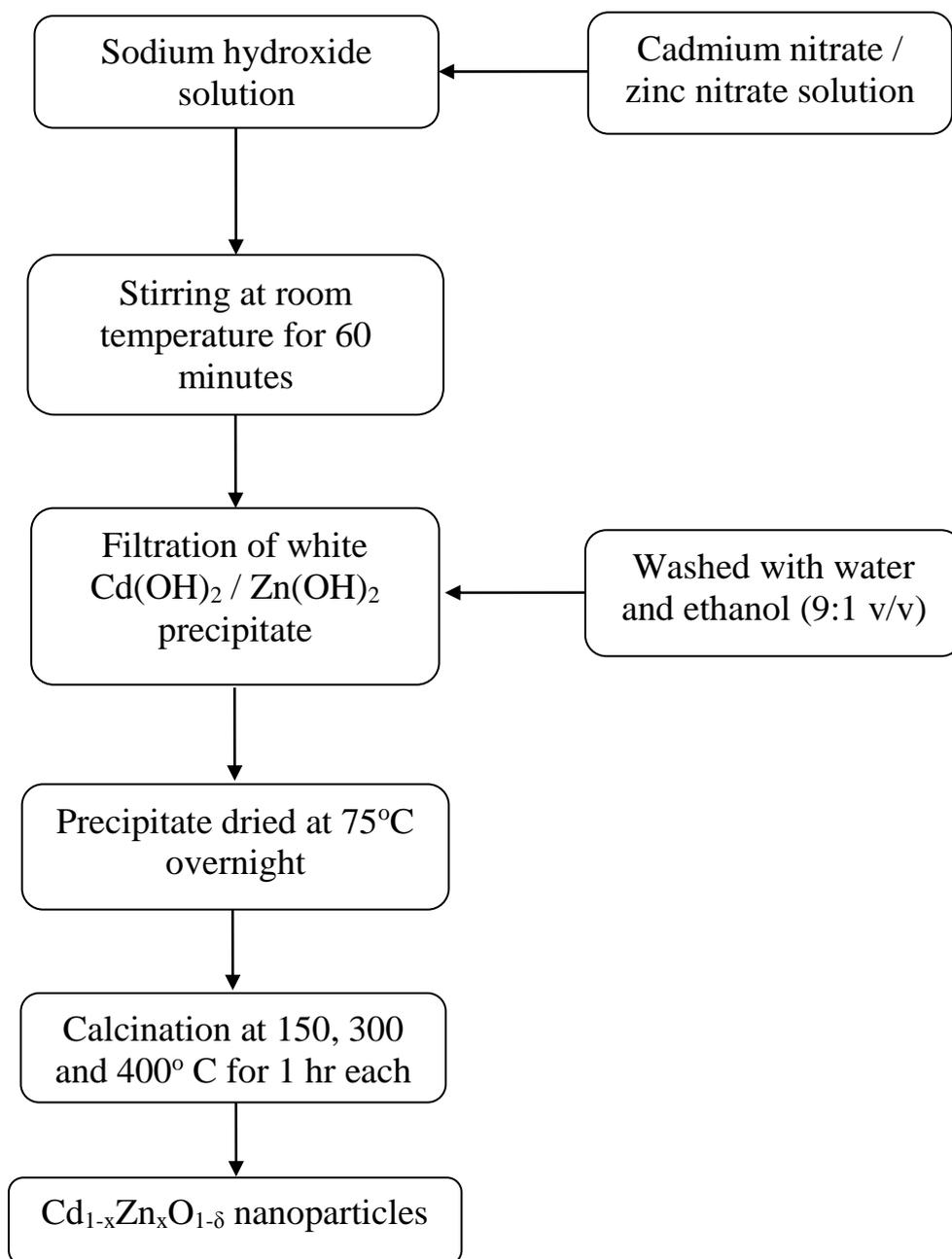
## 2. Materials and methods

### 2. 1. Chemical synthesis of pure and Zn-doped CdO nanoparticles

The precursor materials used in this research work were cadmium nitrate  $\text{Cd}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$  and zinc nitrate  $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  (as basic materials) and sodium hydroxide NaOH (as a precipitator material). In a typical experiment, the aqueous solution containing  $\text{Cd}^{2+}/\text{Zn}^{2+}$  ions was mixed with the alkaline solution. The mixed solution was stirred by magnetic stirrer at 1000 rpm at room temperature for 15 to 20 min. White precipitate thus formed was then filtered and dried at  $100^\circ\text{C}$  for about 2 hours in a hot air oven. The dried product was calcined at 150, 300 and  $450^\circ\text{C}$  for one hour to obtain the CdO and Zn doped CdO nanoparticles. Fig. 1 shows a schematic illustration of the synthesis method.

### 2. 2. Preparation of the CdO based nanofluids

In this research, CdO based nanofluids were prepared by a two-step method. The synthesized CdO based nanoparticles were then dispersed in the ethylene glycol. Ethylene glycol is used as the base fluid because of its coolant property and higher efficiency. In the typical experiment, 0.005g of CdO / Zn- doped CdO nanoparticles were dispersed in 100 ml of



**Fig. 1.** Schematic illustration of the synthesis of  $\text{Cd}_{1-x}\text{Zn}_x\text{O}_{1-\delta}$  nanoparticles by chemical precipitation process

ethylene glycol separately and sonicated for  $\frac{1}{2}$  an hour through ultrasonication. The prepared nanofluids were then used for determination of the acoustic parameters and their thermophysical characteristics for heat transfer applications. The experimental procedure to prepare the CdO based nanofluids is indicated in Fig. 2.

### 2. 3. Characterization techniques

Thermogravimetical studies of the precursor material were performed with TGA instrument between 25 and  $1000^\circ\text{C}$  in air. The powder XRD studies were carried out using Shimadzu XRD6000 X-ray diffractometer at a scan speed of 5 deg/min using  $\text{Cu-K}\alpha$  radiation. The FTIR spectra for the samples were taken using

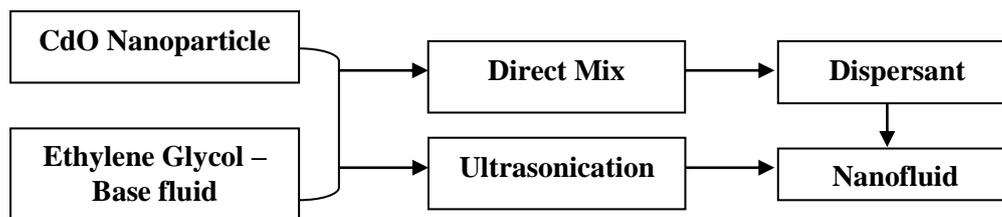


Fig. 2. Experimental procedure to prepare CdO based nanofluids

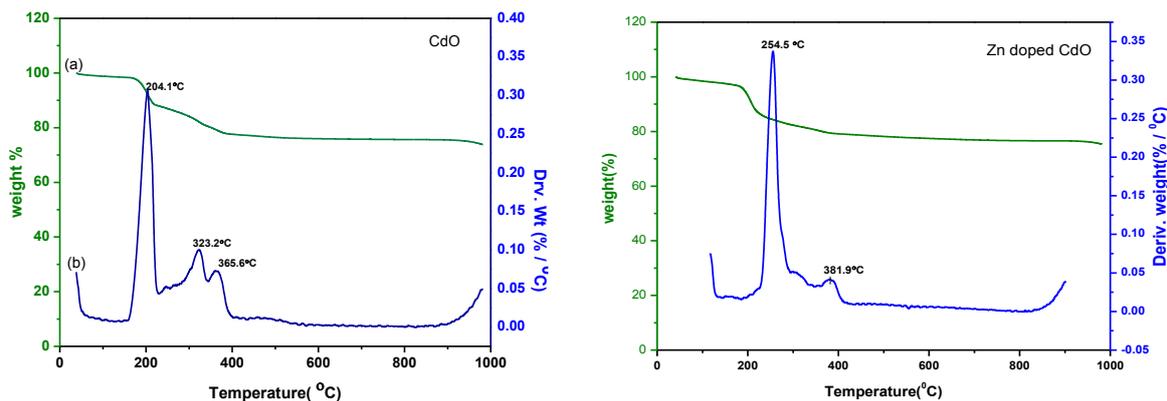


Fig. 3. Thermal analysis pattern obtained on the precursor of (a) the un-doped CdO (b) Zn doped CdO

Shimadzu IR Prestige – 21 model FTIR spectrometer. The crystallite sizes of the ceramic powders were calculated by Scherrer's formula. The surface morphology of the particles was studied by means of JEOL JSM-6360 scanning electron microscopy (SEM). Thermal conductivity of the nanofluids was measured by KD2 pro thermal properties analyzer containing sensor needles. The Viscosity measurement of the prepared nanofluids was carried out by using Ultra III programmable Rheometer.

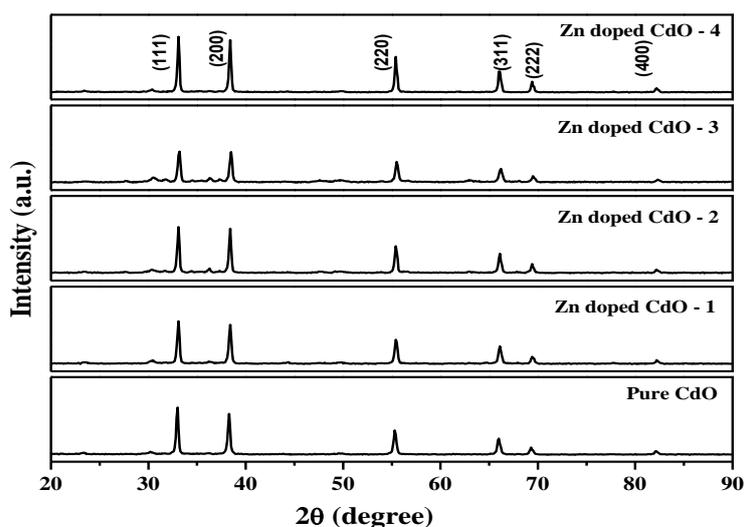
### 3. Results and discussions

#### 3.1. Thermal Gravimetric Analysis

The thermal decomposition study was performed in the 25–1000 °C temperature range at a heating rate of 10 °C/min. The thermal analysis pattern obtained on the precursor of pure cadmium oxide is shown in Figure 3 (a). The figure shows the gradual thermal decomposition as the temperature increases. The weight loss occurred in the range of 0–165° C may be due to the removal of water

molecules from the sample. The significant weight loss was found at 180 °C and 300 °C, respectively. The weight loss found above 165 °C may be due to the removal of organics present in the sample. Three exothermic peaks shown by the DTA curve at 200°C, 320°C and 362°C indicate the formation of the cadmium-oxide phase through oxidation decomposition process [20]. No significant weight loss was found above 450 °C, which shows the formation of pure crystalline cubic phase above 450 °C. The thermal stability of CdO at the range of 565 – 880 °C was analyzed. There was an apparent decrease in mass at 982°C indicating 73.78% weight loss.

The thermal analysis pattern obtained on the precursor of the zinc doped cadmium oxide is shown in Figure 3(b). It was understood that the first weight loss of 15% is observed in the temperature range of 190 to 220 °C, indicating the evaporation of water molecules. The second weight loss started in the range of 344 to 380°C belongs to the decay of the organic components present in the precursor material [21]. The



**Fig. 4.** Combined XRD pattern of (a) pure CdO and Zn doped CdO nanoparticles, (b)  $\text{Cd}_{0.95}\text{Zn}_{0.05}\text{O}_{1-\delta}$ ; (c)  $\text{Cd}_{0.90}\text{Zn}_{0.10}\text{O}_{1-\delta}$ ; (d)  $\text{Cd}_{0.85}\text{Zn}_{0.15}\text{O}_{1-\delta}$ ; (e)  $\text{Cd}_{0.80}\text{Zn}_{0.20}\text{O}_{1-\delta}$

oxidation decomposition is observed from the exothermic peaks at 206, 265 and 365 °C, respectively. The sample was found to be stable above 450 °C. Nearly 75% weight loss is observed at 980 °C by the continuous decrease in mass.

The thermogravimetric analysis obtained on the precursor samples of pure cadmium oxide and Zn doped cadmium oxide helped to understand the heat treatment pattern to be followed to synthesize pure phase compounds.

### 3. 2. X-Ray diffraction studies

X-ray diffraction (XRD) was carried-out for the well ground powder samples. X-Ray diffraction patterns were recorded from 20° to 90°. It exhibited three strong diffraction peaks at (38.336°, 33.040°, 55.336°) which are associated with planes (200) (111), (220) and other peaks assigned for the (311), (222) and (400) planes, respectively. The resulted XRD patterns of the pure and Zn doped CdO nanoparticles were compared with the standard JCPDS data (65-2908). The predominant peaks were matched well except for very few impurity phases. Also, the crystalline structure of the all the samples was found to be cubic. The combined XRD pattern of the samples is shown in the Figure 4.

The crystallite size of the powder can be calculated from the X-ray diffraction peak intensity analysis using the Scherrer's formula:

$$D = \frac{0.9\lambda}{\beta \cos \theta} \quad [1]$$

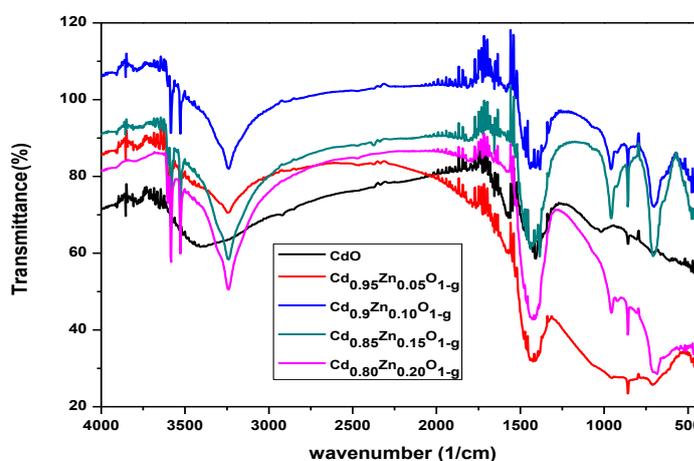
where D is the crystallite size in nm,  $\lambda$  is the radiation wavelength (for Cu-K $\alpha$  radiation,  $\lambda = 1.5418 \text{ \AA}$ ),  $\theta$  is the diffraction peak angle and  $\beta$  is the broadening of the line ("half width") (Fig.3) measured at half its maximum intensity (in radians) [11]. The Theoretical X-ray density has been calculated (in gcm<sup>-3</sup>) using the lattice parameters with the formula:

$$D_{th} = z \frac{M}{N \times V} \quad [2]$$

where M (in atomic-weight units) is the mass of atomic ensemble constituting one unit of the chemical formula, z is the number of such chemical units in one unit cell of the crystal, N is the Avagadro's number and V (in  $\text{\AA}^3$ ) is the volume of the crystalline unit cell as determined by X-ray diffraction. The average crystalline size of the pure and doped CdO nanoparticles was found to be around 5.3 nm. The crystalline parameters obtained on pure and zinc doped CdO nanoparticles are presented in Table. 1. The crystalline characteristics were found to be uniform in all the samples.

**Table 1.** Crystalline parameters obtained on un-doped and zinc doped CdO nanoparticles

Sample	Average crystalline size (nm)	Average d-spacing (Å)	Unit cell parameter[a] (Å)	Density [D <sub>th</sub> ] (g/cm <sup>3</sup> )
CdO	32.64	1.7746	4.688	1.2440
Cd <sub>0.95</sub> Zn <sub>0.05</sub> O <sub>1-δ</sub>	30.21	1.8711	4.697	1.2234
Cd <sub>0.90</sub> Zn <sub>0.10</sub> O <sub>1-δ</sub>	29.83	1.7745	4.614	1.2592
Cd <sub>0.85</sub> Zn <sub>0.15</sub> O <sub>1-δ</sub>	33.63	1.7758	4.686	1.1157
Cd <sub>0.80</sub> Zn <sub>0.20</sub> O <sub>1-δ</sub>	28.50	1.7734	4.686	1.1564

**Fig. 5.** FTIR Spectra for Pure and Zn doped CdO nanoparticles

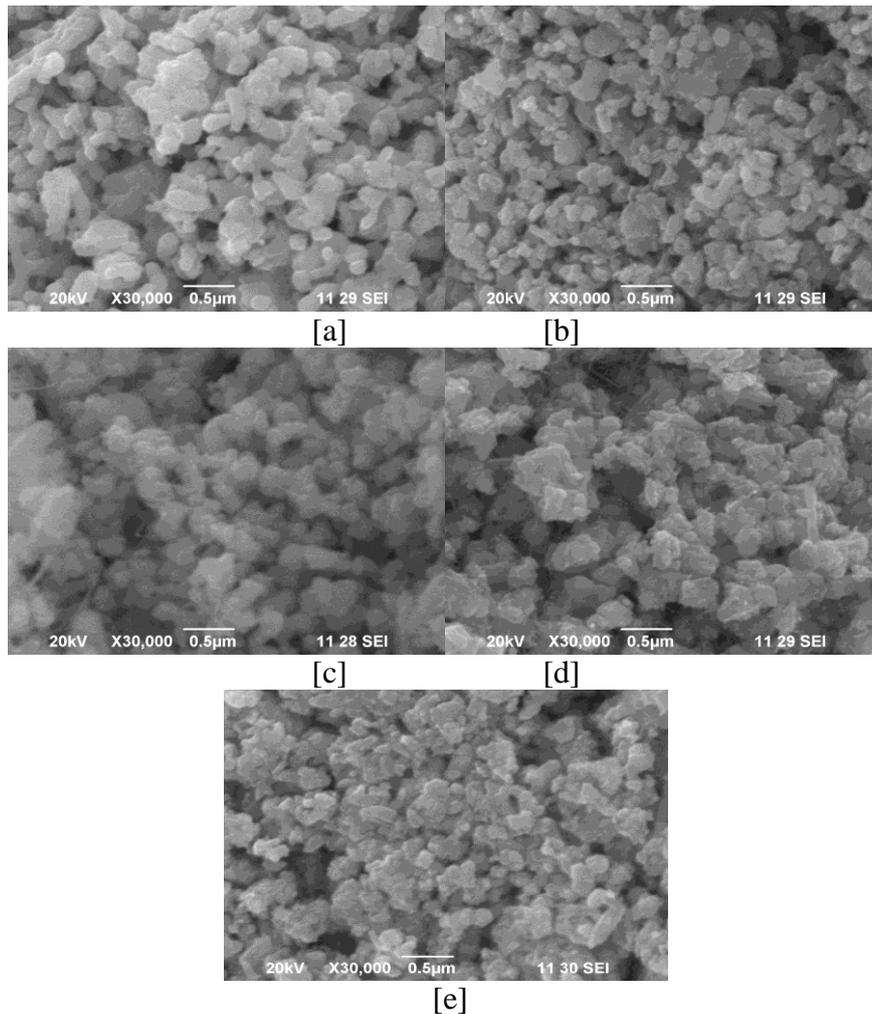
### 3. 3. Fourier transform infrared spectroscopy analysis

From the FTIR spectra, the formation of organic functional groups of the pure CdO and Zn doped CdO nanoparticles have been predicted. The peaks appeared at around 3580 to 3515  $\text{cm}^{-1}$  are attributed to the stretching and bending vibrations of the absorbed water molecules in the samples. 3234  $\text{cm}^{-1}$  attributes to O-H stretching bands. The peak appeared at around 1420  $\text{cm}^{-1}$  indicates the vibration of symmetric  $\text{CO}_2$  molecule. The sharp peaks at about 715  $\text{cm}^{-1}$  are attributed to the metal-oxygen stretching of CdO [22]. Upon incorporating  $\text{Zn}^{2+}$  in the CdO lattice site, shifting of the stretching band to the lower wave number in the FTIR spectra is observed. This may be due to the bond weakening that arises because of the interaction of the additionally added  $\text{Zn}^{2+}$  in the cadmium oxide

lattice site [15]. The combined FTIR spectra obtained on the samples is shown in Figure 5.

### 3. 4. Scanning electron microscopy analysis and the EDAX analysis

The SEM images obtained on pure and Zn doped cadmium oxide nanoparticles are indicated in Figure 6. The average grain size of the particles is found to be 100 – 200 nm. Presence of larger particles in the sample may be due to the high temperature treatment. The compositional analysis given by EDAX confirmed that the samples are around the nominal composition for various doping concentrations as shown in Figure 7. The atomic percentage of the elements present in pure CdO and Zn doped CdO nanoparticles is indicated in Table 2. The EDAX data confirmed the presence of the atomic elements as per the requirement in the samples.



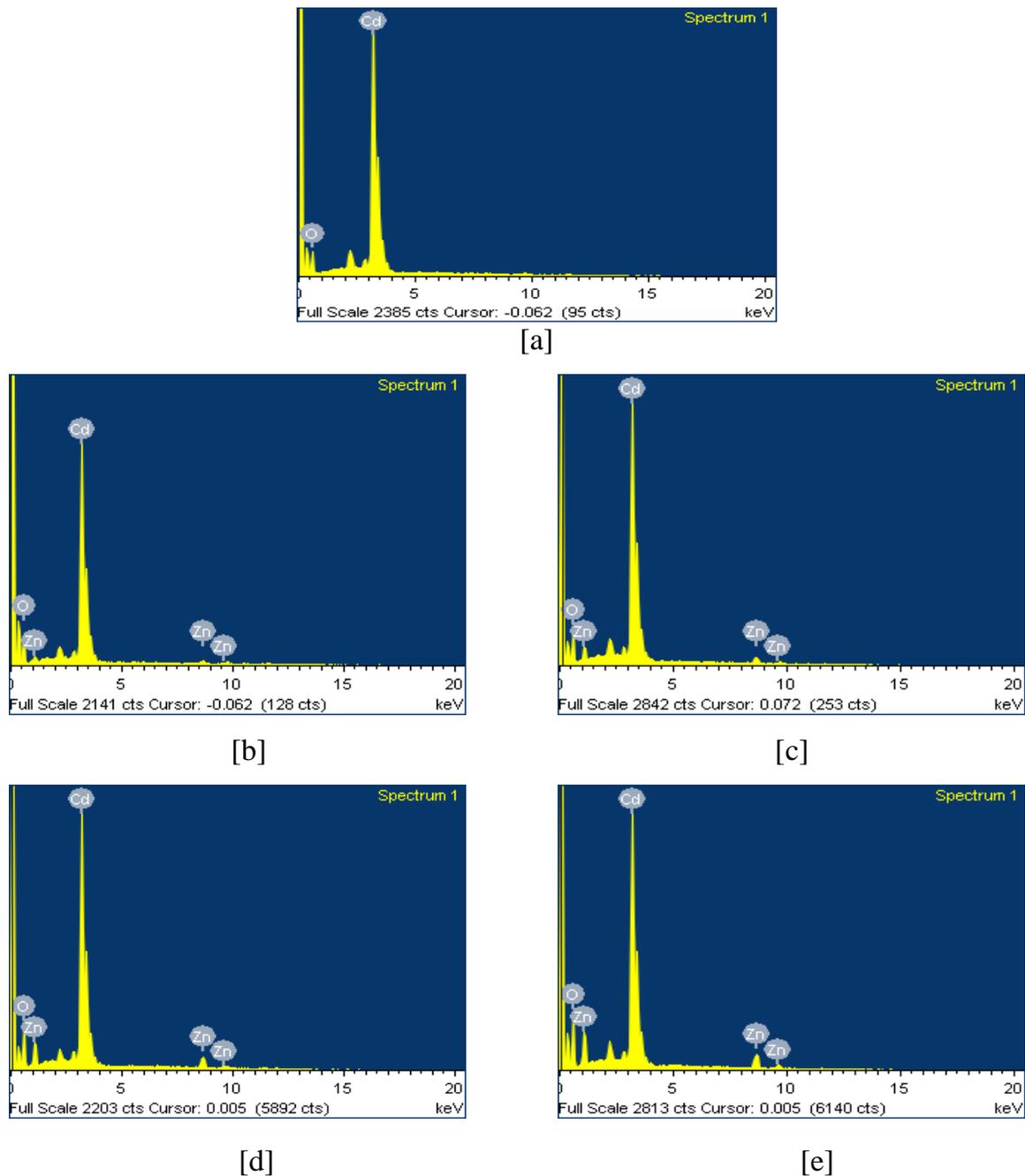
**Fig. 6.** SEM images obtained on (a) Pure CdO (b)  $\text{Cd}_{0.95}\text{Zn}_{0.05}\text{O}_{1-\delta}$  (c)  $\text{Cd}_{0.90}\text{Zn}_{0.10}\text{O}_{1-\delta}$  (d)  $\text{Cd}_{0.85}\text{Zn}_{0.15}\text{O}_{1-\delta}$  and (e)  $\text{Cd}_{0.80}\text{Zn}_{0.20}\text{O}_{1-\delta}$ . Nanoparticles

### 3. 5. Analysis of thermophysical properties obtained on CdO and Zn-doped CdO nanofluids

From the thermo-physical studies, various thermal and physical characteristics of nanofluids are obtained for heat transfer applications. The thermo-physical data obtained on pure and zinc doped CdO nanofluids are indicated in Table 3. The graph mentioned in Figure 8 shows the variation of thermal conductivity with different mole % of Zn in CdO. From the curve, it was found that the thermal conductivity decreased as the doping concentration increases. This decrease in the thermal conductivity may be due to the particle coalition and settling of the nanoparticles in the nanofluid [7, 23, 24].

However, the thermal conductivity for  $x=0$  and 0.05 molar concentrations has increased the thermal conductivity of Ethylene Glycol (0.258w/mk). This also predicts that there is a high stability for the obtained CdO based nanofluids due to the presence of Ethylene Glycol (base fluid) at the initial time after sonication.

The viscosity measurements obtained on the prepared nanofluids are indicated in Figure 9. From the curve, it was understood that the viscosity decreased with increase in the dopant concentration. This shows that the agglomerate size has increased after 1 h of ultrasonication which decreases the viscosity of the obtained nanofluid [25]. This also predicts the rheological Newtonian behaviour of the



**Fig. 7.** EDAX spectra obtained on (a) Pure CdO (b)  $\text{Cd}_{0.95}\text{Zn}_{0.05}\text{O}_{1-\delta}$  (c)  $\text{Cd}_{0.90}\text{Zn}_{0.10}\text{O}_{1-\delta}$  (d)  $\text{Cd}_{0.85}\text{Zn}_{0.15}\text{O}_{1-\delta}$  and (e)  $\text{Cd}_{0.80}\text{Zn}_{0.20}\text{O}_{1-\delta}$  nanoparticles

Cadmium Oxide–Ethylene Glycol nanofluids [26]. Hence, the viscosity of the nanofluid is found to be dependent on the Zn doped CdO concentrations. However, the viscosity of the pure CdO is appreciably higher than the conventional fluid.

The acoustical parameters were calculated using the measured experimental values of

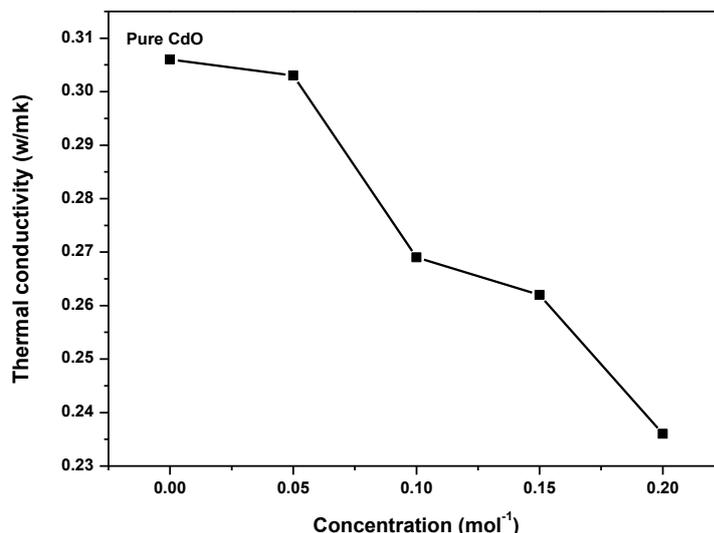
velocity ( $v$ ) and density ( $\rho$ ). The effect of compressibility has been pronounced by the electrostatic field produced by interacting atoms in liquid influences the structural arrangement of molecules. Adiabatic compressibility is calculated using the following formula [27, 28]

**Table 2.** Atomic percentages of pure CdO and Zn doped CdO nanoparticles obtained from the EDAX analysis

Sample	Atomic weight percentage of Cd	Atomic weight percentage of Zn	Atomic weight percentage of O
CdO	29.95 %	-	70.05 %
Cd <sub>0.95</sub> Zn <sub>0.05</sub> O <sub>1-δ</sub>	23.02%	1.52%	75.46%
Cd <sub>0.90</sub> Zn <sub>0.10</sub> O <sub>1-δ</sub>	24.47%	2.22%	73.31%
Cd <sub>0.85</sub> Zn <sub>0.15</sub> O <sub>1-δ</sub>	21.34%	4.23%	74.44%
Cd <sub>0.80</sub> Zn <sub>0.20</sub> O <sub>1-δ</sub>	18.12%	4.03%	77.85%

**Table 3.** Thermo-physical characteristics obtained on pure and zinc doped CdO nanofluids

Sample	Velocity (m/s)	Thermal conductivity (w/mk)	Viscosity (cP)	Density (g)
CdO	1643.927	0.306	15.0	16.3736
Cd <sub>0.95</sub> Zn <sub>0.05</sub> O <sub>1-δ</sub>	1640.286	0.303	14.8	16.3950
Cd <sub>0.90</sub> Zn <sub>0.10</sub> O <sub>1-δ</sub>	1634.480	0.269	14.4	16.3862
Cd <sub>0.85</sub> Zn <sub>0.15</sub> O <sub>1-δ</sub>	1630.152	0.262	13.9	16.3814
Cd <sub>0.80</sub> Zn <sub>0.20</sub> O <sub>1-δ</sub>	1625.891	0.236	13.6	16.3760



**Fig. 8.** Variation of thermal conductivity with different dopant concentration of zinc in the CdO nanofluids (0, 5, 10, 15 and 20 mole% of Zn doped in CdO)

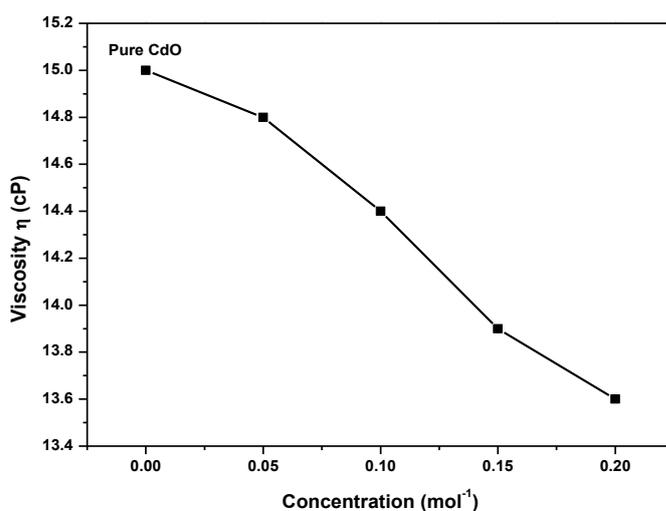
$$\beta = \frac{1}{U^2 \rho} \quad \text{m}^2\text{N}^{-1} \quad [3]$$

where U is the measured value of ultrasonic velocity and ρ is density of the nanofluid. The distance between the surfaces of two neighboring molecules in a liquid is called ‘intermolecular free length (L<sub>f</sub>)’. Intermolecular free length is calculated using the following formula [29],

$$L_f = K \sqrt{\beta} \quad \text{meter} \quad [4]$$

$$K^T = 199.5 \times 10^{-8}$$

where β is the adiabatic compressibility value and K<sup>T</sup> is temperature dependant Jacobson constant. Sound propagation generated by the vibration of particles and liquid molecules of acoustic medium at a given frequency is called ‘acoustic impedance’. Acoustic impedance is calculated using the following formula [30],



**Fig. 9.** Variation of viscosity with different dopant concentration of zinc in the CdO nanofluids (0, 5, 10, 15 and 20 mole% of Zn doped in CdO)

**Table 4.** Variation of acoustic parameters with pure and doped CdO nanofluids

Sample	Adiabatic Compressibility $\beta$ (m <sup>2</sup> N <sup>-1</sup> )	Intermolecular Length, $L_f$ (m)	Acoustic Impedance $Z$ (kgm <sup>2</sup> s <sup>-1</sup> )
CdO	$3.397 \times 10^{-7}$	$1.1628 \times 10^{-9}$	1.790
Cd <sub>0.95</sub> Zn <sub>0.05</sub> O <sub>1-<math>\delta</math></sub>	$3.399 \times 10^{-7}$	$1.1631 \times 10^{-9}$	1.794
Cd <sub>0.90</sub> Zn <sub>0.10</sub> O <sub>1-<math>\delta</math></sub>	$3.428 \times 10^{-7}$	$1.1681 \times 10^{-9}$	1.784
Cd <sub>0.85</sub> Zn <sub>0.15</sub> O <sub>1-<math>\delta</math></sub>	$3.449 \times 10^{-7}$	$1.1717 \times 10^{-9}$	1.778
Cd <sub>0.80</sub> Zn <sub>0.20</sub> O <sub>1-<math>\delta</math></sub>	$3.471 \times 10^{-7}$	$1.1755 \times 10^{-9}$	1.771

$$Z = U\rho \text{ kgm}^{-2}\text{s}^{-1} \quad [5]$$

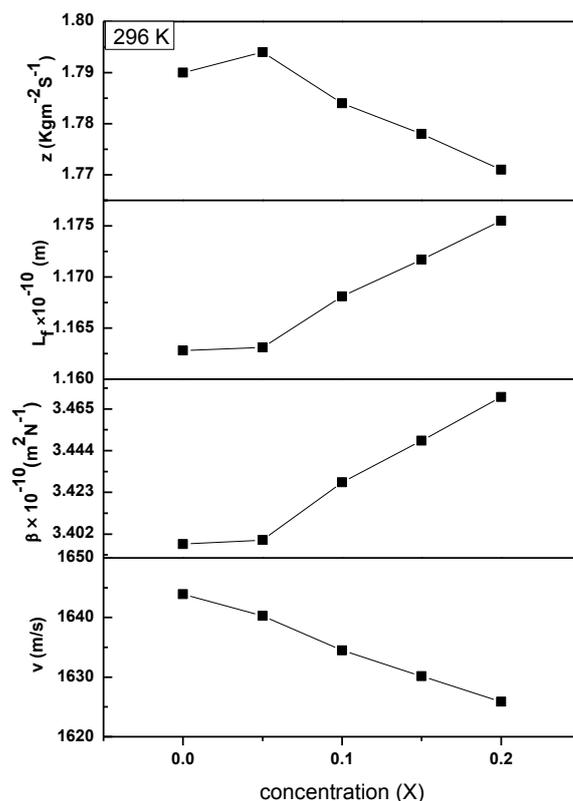
where  $U$  is the measured value of ultrasonic velocity and  $\rho$  is density of nanofluid. From the ultrasonic velocity and the density, the acoustic parameters were calculated and mentioned in Table 4.

From Figure 10, it is observed that with the effect of doping concentration, the adiabatic compressibility gradually decreases. With the addition of pure and doped cadmium oxide nanoparticles to the base fluid (ethylene glycol), weak intermolecular forces occur which in turn reduce the ultrasonic velocity with increase in dopant concentration. This evidences that the particle-fluid interaction is more than the particle-particle interaction [30, 31]. Thus, the intermolecular free length of the CdO based nanofluid increases with the increase in the adiabatic compressibility

through strong force between nanoparticles and the base fluid. The reduction observed in the acoustic impedance is attributed to the variation in density and compressibility [32].

#### 4. Conclusion

Chemical precipitation process can be effectively used to synthesize undoped CdO and zinc doped CdO nanomaterials. The optimum temperature required by each sample for the formation of pure phase is identified by thermal analysis. The XRD studies revealed the presence of cubic crystalline structure in all the samples. Through the FTIR spectra, the Cd-O stretching bond formation and the other functional groups have been observed. The SEM images confirmed the presence of nanoparticles (100 – 200 nm) in the samples. The EDAX data confirmed the presence of atomic elements as per the requirement in the



**Fig. 10.** Geometrical view from the acoustic parameters for different dopant concentration of zinc in the CdO nanofluids (0, 5, 10, 15 and 20 mole% of Zn doped in CdO)

samples. The thermophysical properties for the undoped and doped ZnO samples conveyed that the particle coalition affects the thermal conductivity as the dopant concentration increases. The Newtonian behavior of the CdO based nanofluids can be predicted from the reduced viscosity. The intermolecular free length of the present nanofluids observed seemed to be increasing with the higher incorporation of  $\text{Zn}^{2+}$  ions in CdO nanoparticles. This indicates that the particle – fluid interaction is more in the prepared CdO based nanofluids.

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