IOSR Journal of Applied Physics (IOSR-JAP)
e-ISSN: 2278-4861,
PP 80-86
www.iosrjournals.org

Structural, Optical and Antibacterial Activity of Pure and Cadmium Doped Zinc Oxide Nano Particles

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Abstract: *Metal oxide nanoparticle is a versatile material with much scientific and industrial application. Zinc Oxide (ZnO) and Cd doped Zinc Oxide were prepared by Co-precipitation method. ZnO nanoparticle play important role in the enhancement of dielectric properties of nano composites. The crystalline nature of the sample were also confirmed by X-ray diffraction pattern. The crystalline size of the pure and Cd doped nano particle were determined as 21 nm and 18 nm. FT-IR spectra identify the functional groups present in the molecular structure. There is a shift in the low frequency region confirmed the presence of the dopant (Cd). The surface morphology of the prepared materials were also analysed by SEM with EDAX. The photo luminescence analysis spectrum of these samples were also recorded. It shows blue shift at 366 nm. The energy gap value for pure and Cd doped ZnO were also determined. Anti-bacterial studies show that the zone of inhibition is high for the ZnO and Cd doped ZnO nano particle. ZnO nanoparticle shows significant antibacterial activity against bacterial species.*
Keywords: *Antibacterial activity, Nanoparticle, SEM with EDAX, X-ray diffraction, Zinc oxide.*

I. Introduction

Nanoparticles are of great interest for many technical applications and fundamental research due to the dependent physical properties[1-4]. Nanostructure materials have received broad attention due to their distinguished performance in electronics, optics and photonics. Zinc oxide is a unique material that exhibits semiconducting and peizoelectronic dual properties. From the 1960s, synthesis of ZnO thin films has been an active field because of their applications as sensors, transducer and catalyst [5]. ZnO is an interesting chemically and thermally stable-type semiconductor with wide direct band gap 0f 3.37 eV at room temperature and high sensitivity to toxic and combustible gases [6].Compared with other semiconductor materials ZnO has higher excitation binding energy (60 meV) and has been studied as an optoelectronic, transparent conducting, and piezoelectric material. Thus, the synthesis of ZnO nanoparticles by direct precipitation method is simple, fast and eco-friendly in nature [7]. Recently, Mn-doped ZnO nanoparticles was used in the optical device in diluted magnetic semiconductor materials[8].The Cu-doped ZnO has sensor RT sensitivity, faster response time, and good selectivity. Miniaturized Cu-doped ZnO rod based sensor can server as a good candidate for effective H2 detector with low power consumption.[9]. Different size of ZnO nanoparticles were formed because of different environment of laser pulse generated and the blue shift in the absorption edge indicated the quantum

confinement property of nanoparitcles.[10]Motivated by the above work in the present investigation pure and Cd doped ZnO have been prepared by Co-precipitation method and characterized by powder X-ray diffraction method and FT-IR spectrum. The morphology of the nanoparticles were studied by scanning electron microscopy (SEM) method. The optical properties were also determined from UV-Visible spectroscopy and photoluminescence spectra. The antibacterial activity of pure ZnO and Cd-doped ZnO nanoparticles were also studied.

II. Experimental

2.1. Synthesis of pure ZnO and Cd doped ZnO Nanoparticles

ZnO nanoparticles were prepared by economically less Co-precipitation method [11,12] . A 0.2 M of zinc acetate (Zn C4H6O4) was dissolved in 20 ml of absolute ethanol and stirred at room temperature for 30 min. Then 36 mg of NaOH was dissolved in 20 ml of ethanol. The NaOH solution was added to the zinc acetate solution in drop wise in constant stirring. The pH value of the solution was measured to be 12. The solution was continuously stirred for 2 hours at room temperature. The solution became turbid form which indicated the ZnO nano particles were formed. The solution was filtered and precipitates were collected and washed with distilled water and absolute ethanol and acetone for several times. The final product was annealed at 200°C for 1 hour.

The resultant ZnO nanoparticles were collected and sealed in plastic container. Cd doped ZnO Nanoparticles were prepared by the above said procedure with the addition of 0.002 M cadmium sulphate in 20 ml ethanol.

III. Results and Discussion

3.1. Structural characterization

a) XRD Pattern

The phase composition and the crystalline size of the prepared ZnO sample were evaluated by X-ray diffraction analysis. Fig.3.1 presents the XRD pattern of pure ZnO nanoparticles annealing at temperature of 200°C. The sharp and high intensity diffraction peak are corresponding to the (1 0 1) plane. The various crystal planes are namely, (1 0 0), (0 0 2), (1 0 1), (1 0 2), (1 1 0), (1 0 3) ,(1 1 2) . The narrow line widths indicate high crystalline nature of the synthesized material. The scan range of 20-80 degrees contained a strongest line for hexagonal phase[13] as shown in Fig.3.1. The X-ray diffraction very well agreed with hexagonal phase of ZnO in JCPDS 36-1451. The lattice parameters a and c were calculated using Eq. (1),

$$\frac{1}{d^2} = \frac{h^2+k^2}{a^2} + \frac{l^2}{c^2} \qquad (1)$$

The average crystalline size of all the samples was calculated using Debye-Scherrer formula

$$D= \frac{0.9\lambda}{(\beta \ cos\,\theta)} \qquad (2)$$

Where λ is the wavelength of X-ray radiation, β is the full width at half maximum (FWHM) of the peaks and θ is the angle of diffraction. The calculated crystallite size was found to be 21 nm for ZnO nanoparticles. The lattice parameter are a=b= 3.2417 Å and c=5.1850 Å. The same procedure was adopted to calculate the particle size of Cd-doped ZnO and it is found to be 18nm .The crystal planes of Cd-doped ZnO are as follow (1 0 1), (1 0 2), (2 0 2). The XRD pattern of Cd-doped ZnO are shown in fig 3.1

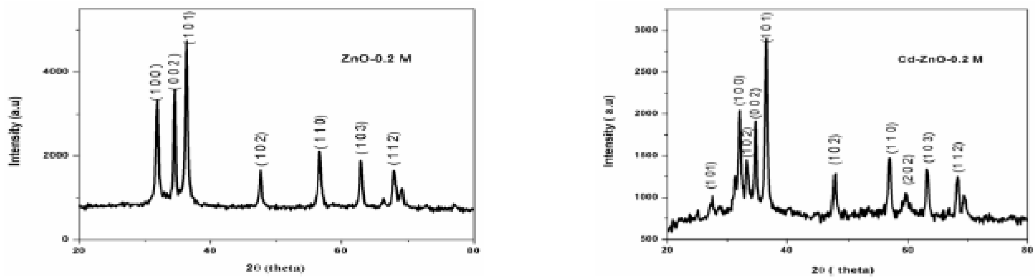

Fig.3.1.X-Ray diffraction patterns of pure ZnO and Cd doped ZnO nanoparticles

3.2 FT-IR analysis

Infrared spectroscopy was used to detect the functional group absorbed on the surface of synthesized nanoparticles during precipitation process. Fig 3.2 represents the FT-IR spectra of ZnO nanopowder recorded in the range 3200-3500 cm-1 using JASCO-FTIR 460 plus spectrometer. The peak centred at 3318 cm-1 corresponding to the stretching vibration of intermolecular hydrogen bond (O-H) existing between the absorbed water molecules and indicates the higher amount of hydroxyl group. The results were well matched with the observation done by samirabagheri et al [14]. The two weak bonds at 2996 and 2927 cm-1 corresponding to the C-H stretching modes. The bend near at 2108 cm-1 bending vibration of the intercalated O=C=O species produced by the structure group. The weak bend at 645-831 cm-1 appearing in IR spectrum of annealed (200°C) compound indicates the presence of stretching and bending vibration of the intercalated C-O species. The characteristic peak around 462 cm-1 becomes stronger, indicating the formation of stretching mode of ZnO. This indicates the presence of ZnO nanoparticles in annealed compound. The vibrational frequency 564 cm-1 is absents in pure ZnO. It also confirm the presence of Cd-doped in ZnO angle lattice as shown in the fig 3.3. the vibrational frequency of ZnO (464 cm-1) is shifted to the lower frequency side (430 cm-1) in Cd-ZnO indicates the presence of the Cd in the Zno nano lattice.

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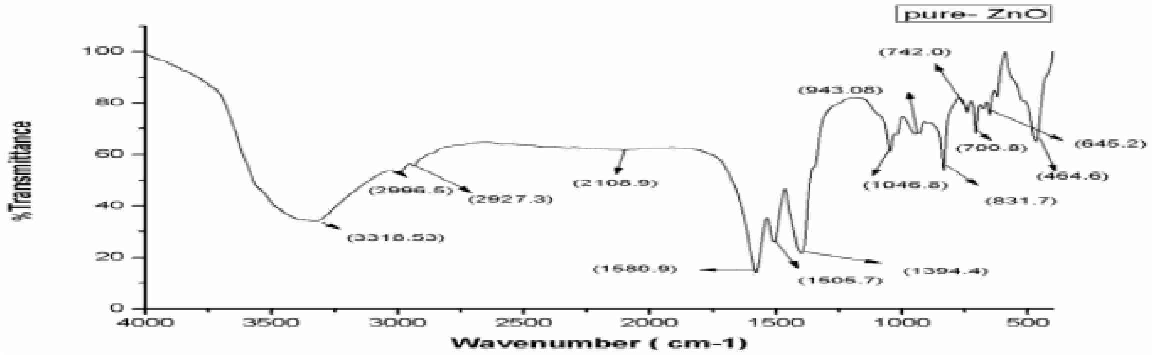

Fig:3.2 FT-IR spectra of pure ZnO nanoparticles

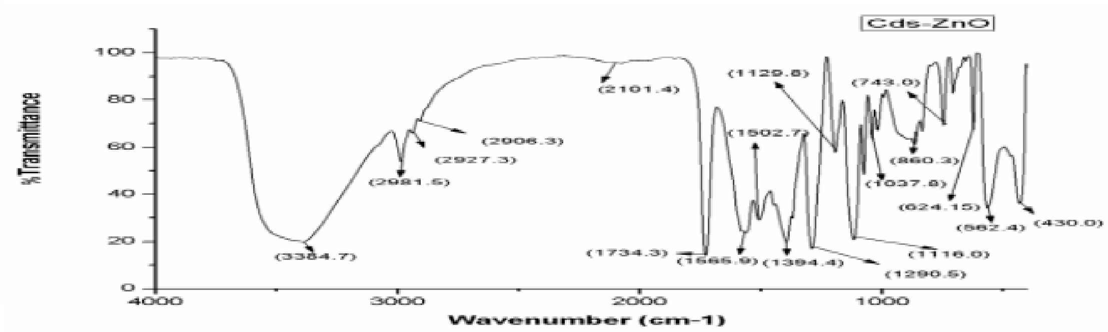

Fig: 3.3 FT-IR spectra of Cd-doped Z nO nanoparticles

3.3 Scanning electron microscopy (SEM)

Fig 3.4 shows scanning electron microscopy (SEM) micrograph of pure ZnO nanoparticles scanned by 10 kV electron beam at 20,000 x magnification[15]. The SEM image reveals the polycrystalline, porous morphology with the inter-connected grains present on the pure ZnO nanoparticles. The small crystalline agglomerated to form nano fused clusters seen in the surface morphology of pure ZnO. Fig 3.5 (a) shows scanning electron microscopy (SEM) micrograph of Cd-doped ZnO nanoparticles scanned by 20 kV electron beam at 20,000 x magnification reveals the hexagonal shaped nanorods and pencil shaped nano particle. Similarly fig 3.5 (b) shows the image scanned by 20 kV electron beam at 10,000 x magnification reveals the Nano crystalline structure of Cd-doped ZnO. The magnification power increases as 55,000 x and 30,000 x exhibits the nano plates of Cd-doped ZnO nanoparticles (fig 3.5 (c) and (d)). The EDAX confirm the element present in ZnO shown in fig (3.6).

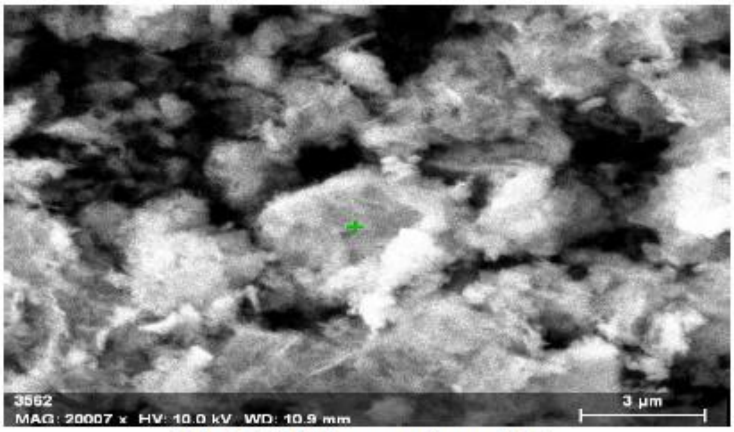

Fig 3.4 SEM image of pure ZnO

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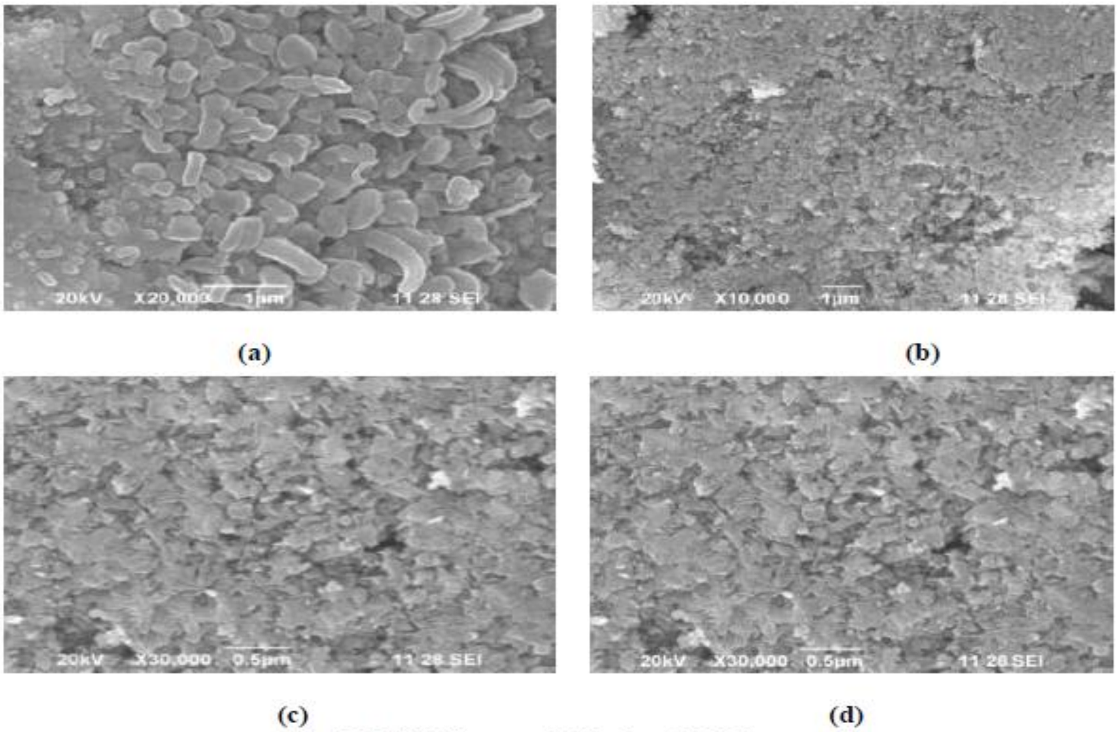

Fig 3.5 SEM image of Cd-doped ZnO

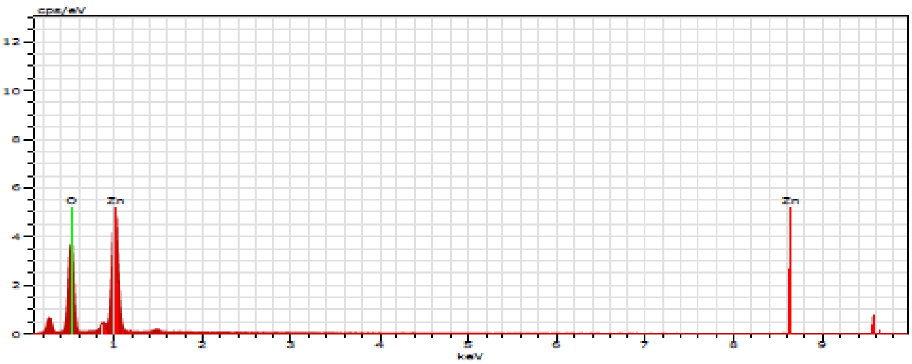

Fig 3.6 EDAX spectrum of ZnO

3.4 UV-Vis Spectra Optical Studies

Optical properties like optical transmittance and optical absorption are the two important parameters to be considered in the fabrication of optical devices[12]. The UV-Vis absorption spectra recorded using LAMDA UV-spectra photometer. The UV-vis spectra of pure ZnO shows the absorption band at 357 nm shown in the fig 3.7 after that its shows less absorption, i.e (highly transparent).and also Cd-doped ZnO shows the absorption band at 345 nm as shown in fig 3.8.The Pure ZnO and Cd-doped ZnO has large optical transmittance window in the region 400-800 nm. There is no remarkable absorption band found in the region. Wide transparency region is the one of the criteria of the nano device fabrications and applicable for opto-electronics. The band gap (Eg) of pure ZnO and Cd-doped ZnO nanoparticle were calculated by the formula Eg= hc/λ,where h= plancks constant, c= velocity of the light and λ= wavelength. The corresponding band gaps were found to be 3.1eV and 3.2eV as shown in fig 3.9 (a) and (b).

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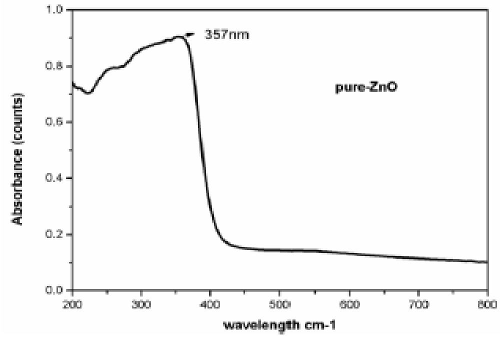

Fig 3.7 UV-Vis spectra of pure ZnO

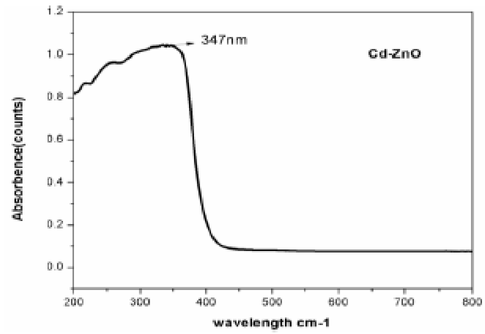

Fig 3.8 UV-Vis spectra of Cd-doped ZnO

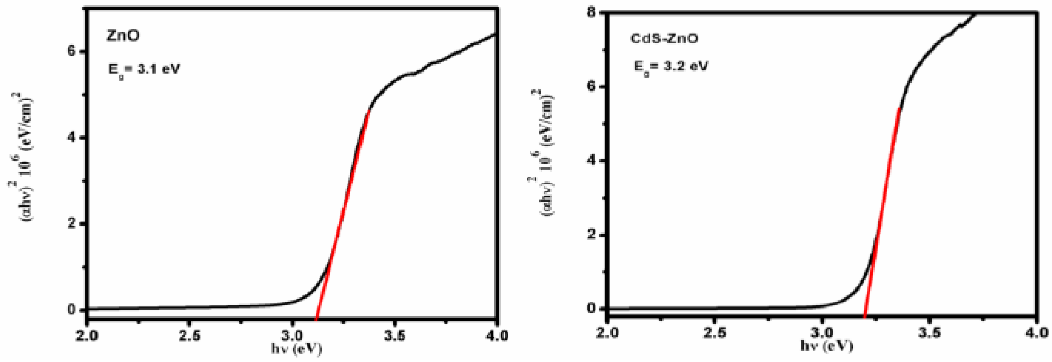

Fig 3.9 (a) Band gap graph for pure ZnO (b) Band gap graph for Cd-doped ZnO

3.5 Photoluminescence

Photoluminescence spectrum (PL) of these sample were recorded at room temperature and the excitation wavelength of pure ZnO and Cd-doped ZnO are 366 nm and 387nm. Compared with pure ZnO the peak position Cd-doped ZnO nanoparticles shows a blue shift. The vacancy related emission was observed at 524 nm implies that Cd-doped ZnO nanoparticles have abounded with oxygen vacancy [fig 3.10]. the oxygen vacancies are occupied by Cd atoms in the ZnO lattice.

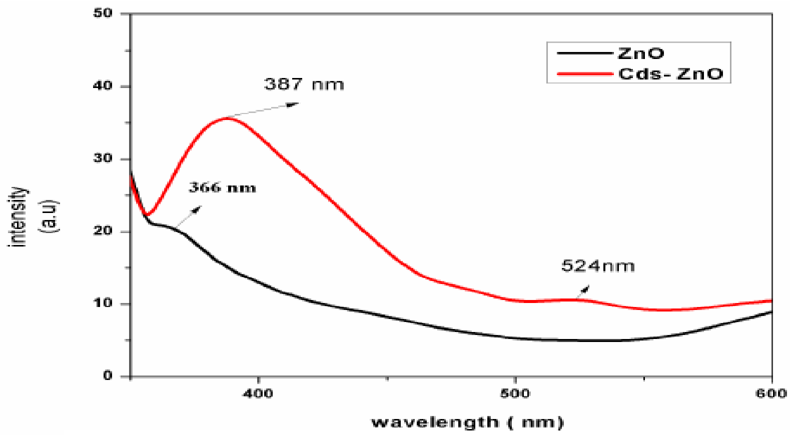

Fig 3.10 PL spectrum of pure and Cd doped ZnO nanoparticles

3.6 Antibacterial activity

Many antibacterial studies were made using different nanoparticles. The reason for the bactericidal activity is due to presence of reactive oxygen species (ROS) generated by different nanoparticles. Chemical interaction between hydrogen peroxide and membrane proteins or between the chemical produced in the presence of pure and Cd doped ZnO nanoparticles and the outer bilayer of bacteria could be the reason for the antibacterial activity of pure and Cd doped ZnO. The hydrogen peroxide produced enters the cell membrane of bacteria and kills them. It is also noted that nanoparticles continue to be in interaction with dead bacteria once the hydrogen peroxide is generated, thus foiling further bacteria action and continue to produce and release hydrogen peroxide to the medium.[16]. In fig 3.11 (a-d) and fig 3.12 (a-c) bacteria is completely destroyed and

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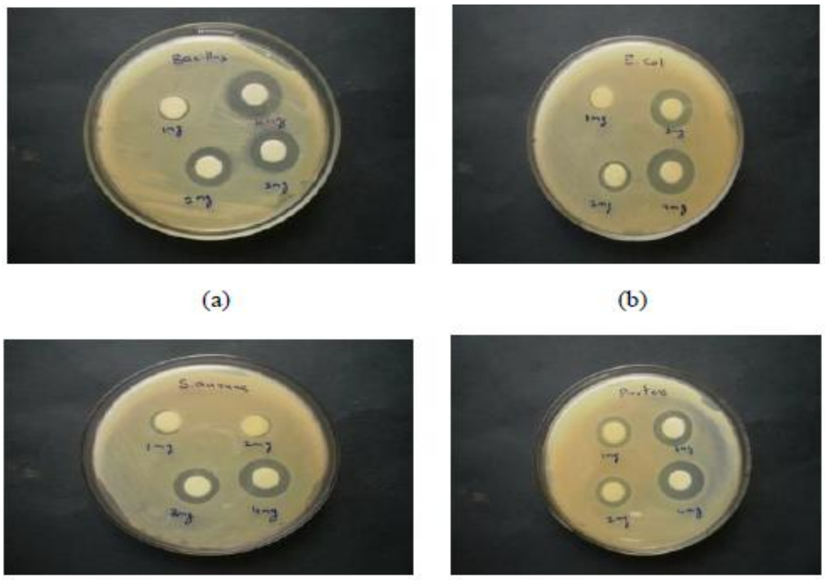
antibacterial activity is still active in pure and Cd doped ZnO nanoparticles. The various bacteria's like E.coli , bacillus ,S.aureus,and Proteus interacts with pure and Cd doped ZnO nanoparticles and the results were shown fig 3.11 (a-d) and fig 3.12 (a-c) and the zone of inhibition is given in table 3.1 & 3.2 respectively.



(a) (b) (c) (d)

Fig 3.11 Antibacterial activity of different extracts of pure ZnO

(a) (b) (c)

Fig 3.12 Antibacterial activity of different extracts of Cd doped ZnO

Table 3.1 Antibacterial activity of different extracts of ZnO

Zone of inhibition															
Escherichia coli				***Proteus vulgaris***				***Staphylococcus aureus***				***Bacillus subtilis***			
1 mg	2 mg	3 mg	4 mg	1 mg	2 mg	3 mg	4 mg	1 mg	2 mg	3 mg	4 mg	1 mg	2 mg	3 mg	4 mg
10 mm	14 mm	19 mm	22 mm	16 mm	17 mm	19 mm	23 mm	10 mm	12 mm	18 mm	22 mm	11 mm	18 mm	22 mm	24 mm

Table 3.2 Antibacterial activity of different extracts of Cd doped ZnO

Zone of inhibition in mm		
Name of the sample	**Zno + CdS**	
Name of the microorganisms	**5 mg**	**10 mg**
Staphylococcus aureus	**12**	**24**
Salmonella typhi	**13**	**24**

IV. Conclusions

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Pure and Cd doped ZnO nano particles were synthesised by Co-precipitation method. The powder X-Ray diffraction pattern of synthesized pure and Cd doped ZnO confirmed that the materials are nanomaterials of particles size 18 nm and 21 nm respectively. The lattice parameter were determined and tabulated. The (h k l) planes are also identified. The functional groups present in the pure and Cd doped ZnO nanomaterials have confirmed by FT-IR spectral analysis. The surface morphology of the samples were determined by scanning electron microscopy (SEM). The optical transmittance lie in the visible region is the advantage of ZnO used in optoelectronic application like display devices and LEDs. Low cost production is the main advantage for any product to compete in the market and ZnO nanostructure being their solution provides much potential to become the choice for cheap devices In addition, devices with ZnO nanorods exhibit better performance due to a better interfacial contact, having fewer defects and improved light extraction. The optical energy gap of the pure and Cd doped ZnO nanomaterials (Eg= 3.1 eV , Eg=3.2 eV) were confirmed by UV-spectra and photoluminescence spectra. The vacancies related emission at 524 nm is suitable for the preparation of photovoltaic device fabrication.

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