# 9. Study of Physical and Optical Properties of Pure ZnO and Cd Doped ZnO Nanostructures and Antibaterial Activities

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#### Abstract

Chemical Synthesized ZnO nano-structure using Sol-Gel chemical precipitation method Zinc acetate and Sodium hydroxide (Merck Mumbai) 1: 2 used in the experiments. To synthesize pure and 5% Cd doped ZnO nanoparticles at room temperature 37°C. Wurtize hexagonal crystal structure of pure and 5% Cd doped ZnO nanoparticles have been confirmed by X-ray diffraction (XRD). The crystalline size have been estimated from XRD data. The particles size calculated by using Debye's-Scherrer formula was found 30-42 nm. varying with increasing reaction temperature. Pure and Cd doped ZnO has been investigated by analysis of FTIR 437-473 cm<sup>-1</sup>. The direct band gap of nanoparticles has been estimated using optical absorption UV spectra 3.42 to 3.47 ev.EDAX estimate atomic percentage of samples and purity. The significant morphological changes of the ZnO nanoparticles were investigated from SEM images 52 nm. Defect due to oxygen deficiency and interstitial vacancy have been confirmed using PL spectra. The Photoluminescence spectrum exhibits two emission peaks one at 368nm and excitation emission 488nm shows that highly blue shifted. The antibacterial activity of pure and Cd doped ZnO nano-materials have been tested Agar well diffusion disc method. Bacillus thuringiensis NCIM2130 and Pseudomonos cf. monteilli 9 cultures are used to shows antibacterial activity of Pure and Cd doped ZnO nanomaterials. The samples shows only antibacterial activity against Bacillus Thuringiensis but Cd doped shows more effect as compared to pure ZnO.

Keywords: ZnO nanostructures, XRD Spectra, FTIR, SEM, UV visible, EDAX, PL Spectra Antibacterial activity, Bacillus thuringiensis NCIM2130 and Pseudomonos cf. monteilli9. Introduction

ZnO has also been confirmed as a promising functional material in other nanodevices such as field emitters and gas sensors. The synthesis of ZnO nanostructures is thus currently attracting intense worldwide interest. Other favourable aspects of ZnO include its broad chemistry leading to many opportunities for wet chemical and biocompatibility..

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In the manufacture of optoelectronic devices, one should have concrete knowledge about the properties of the material that can be used as impurities like donors, acceptor and isoelectronic impurities. The isoelectronic trapping state can enhance the efficiency of the free recombination of electrons and holes [1]. Isoelectronic properties were created by doping a host atom by an atom from the same priod of the periodic table. The impurity doping can cause the lattice deformation due to difference in atomic size between impurity and host atom [2]. The replacement of host atom by proper add ion of impurity atom to change the various extensive and intensive properties of host material was reported by many reports.

Synthesis and characterization of ZnO(Cd) semiconducting material is one of the important new combination to modify the physical and optical properties of ZnO semiconductor.

Vijaylaxmi et al. (2008), Tabet- Derraz et al. (2002) and Vinodkumar et al.(2010) [3-5] have been reported on Cadmium doped ZnO but no work is reported so far to the best of our knowledge on Cadmium dope ZnO nanostructure calcined at different temperatures, and to study its photoconductive and photoluminescence properties. Present chapter deals with 5% Cd doping in ZnO nanostructure and study the effect of temperature of calcinations on structure, morphology, Uv-Visible absorption and photoluminescence properties. Antibacterial activity also shows ZnO nanostructure and Cd doped ZnO.

## Synthesis of Pure and Cd Doped Zno Nano Powder

Economically cheap and simple Chemical precipitation called as Sol-gel preparation is used for synthesis of pure Cd doped zinc oxide nanoparticles ZnO(Cd). In the synthesis of pure ZnO nanopwder the Sol-Gel parameters such as pH of the reaction mixture, Molarity of reagents, reaction time and reaction temperature play crucial role in formation of navel ZnO nanostructure.

Initially appropriate molarity (0.50M) solutions of Zinc acetate, (0.50M) sodium hydroxide and (0.50 M) cadmium salt solutions were prepared. Similar procedure was used to synthesis of Cd doped ZnO. The white colored residue of Cd doped ZnO was dried in oven at 100°C for two hours. The dried product was ground to fine powder using agate mortar and pestle. The samples were sintered at different temperatures (200, 400, 600°C). The as-prepared and sintered samples were characterized and results were discussed.

1. Zinc acetate and sodium hydroxide in distil water preparation ZnO.

 $Zn(CH_3COO)_2$ :  $2H_2O + 2NaOH \rightarrow Zn(OH)_2 + 2CH_3COONa + 2H_2O$ 

 $Zn(OH)_2 \rightarrow ZnO + H_2O_1$  (1).

2. Cadmium acetate and sodium hydroxide in distil water preparation CdO. Cd(CH<sub>3</sub>COO)<sub>2</sub>: 2H<sub>2</sub>O + 2NaOH  $\rightarrow$  Cd(OH)<sub>2</sub> + 2CH<sub>3</sub>COONa + 2H<sub>2</sub>O

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 $Cd(OH)_2 \rightarrow CdO + H_2O_1$  (2).

### **Results and Discussion**

### 1. Structural Investigations Pure ZnO as prepared and Cd doped ZnO

XRD pattern of Cd doped ZnO nanoparticles were have been shown in figure 1. The Xray diffraction (XRD) data were recorded and variations of intensity counts were collected over a  $2\theta = 20-80^{\circ}$ range. The as-prepared Cd doped ZnO nanopowder represents somewhat different crystal structure as compared to pure ZnO and calcined ZnO(Cd) exhibit wurtzite crystal symmetry similar to pure ZnO. The crystallity was observed improved on increasing calcination temperature. As the calcined temperature increased to 200 °C the diffraction peaks at reflection planes (100), (002), (101), (102), (110), (103) and (200) start emerging. The prominent peak was (101) direction. The similar results have been reported in the earlier literature [6]. Further on increasing temperature of calcinations crystallity gradually increased and XRD pattern exhibit wurtzite hexagonal crystal structure as like that of pure ZnO. At temperature of calcinations increased to 600 °C the diffraction peaks become sharp and strong showing improved in crystallity. On increasing the calcination temperature some peaks assigned to CdO phase were noticed in the XRD spectra. This concluded that Cd was substituted at the ZnO lattice.

Figure2. The average particles size of the wurtzite structure was estimated with the help of equation 1 using the full width at half maxima of (100), (002), (101), and (102) of the x-ray diffraction peaks and displayed in table1. The plot of variation of particles size with the calcinations temperature of was presented in figure 2. The particle size of as-prepared Cd doped ZnO as observed smaller as compared to particle size of pure ZnO, as-prepared and as-prepared ZnO(Cd) samples. The average crystalline size was increased with increase in calcination temperature from 30 nm (as-prepared ZnO(Cd)) to 42 nm( calcined at 600 °C). The lattice constants of pure and as-prepared and calcinied ZnO(Cd) was calculated by using equation (1) shown in table1. The values of lattice constants **\*a\*** was observed increased and **\*c\*** was found to be decreased as sintering temperature increased. This may be due to deformation of ZnO lattice by incorporation of large Cd ion than Zn ion. The ionic radius of Cd is larger than Zn.

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calcined at different temperature

ZnO-Cd	2 Theta (deg)	d- spacing	hkl	FWHM (deg)	Partic leSize	Grain Size	Lattice Parameters	
Sample		('A)			(nm)	from SEM (nm)	'a'	ʻb'
ZnO –Cd 27 <sup>°</sup> C	31.4621	2.84931	100	0.4723	30	60	3.172	5.486
	34.4932	2.63196	002	0.4723				
	35.9446	2.54004	101	0.3149				
	47.2408	1.91409	102	0.3149				
	58.4111	1.57866	110	0.3149				
ZnO –Cd 200 <sup>0</sup> C	31,4621	2.84351	100	0.3149	32		3.283	4.990
	34.0990	2,62941	002	0.3936		68		
	35.9877	2,49563	101	0.3936				

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	47.2814	1.92254	102	0.3149				
	31,4591	2.84377	100	0.3149	36	70	3.283	4.990
ZnO-Cd	34.1157	2.62816	002	0.3936				
$400^{0}C$	35.9918	2,49536	101	0.3936				
	47.2729	1.92286	102	0.3149		_		
	31.5720	2.83385	100	0.4723	42	42 72	3.272	5.231
ZnO –Cd	34.2536	2.61789	002	0.3149				
$600^{0}$ C	36.0813	2.48937	101	0.3149				
	47.3652	1.91933	102	0.3149				



verses calcinations temperature

### 2. FTIR Study of Pure ZnO As-prepared and calcined ZnO(Cd)

The FTIR spectroscopy is an important tool used to detect various functional groups trapped on the surface of the nanoparticles and helps to investigate the chemical composition of the material. The FTIR of the particular substance is the finger print of the molecules present in the compound as shown in figure 4.

FTIR spectra of pure ZnO, as-prepared and calcinied ZnO(Cd) were recorded in the wave number range 400-4000 cm<sup>-1</sup> and presented in figure 4. The modes of vibrations and their band assignment were shown in table2. The absorption band assigned in the region 443.64 to 476.43 cm<sup>-1</sup> assigned to the ZnO stretching mode in pure ZnO. This absorption band related to metal oxide region [7-9]. The effect of Cd doping in ZnO show that band become stronger and deformed in the region 437.86 to 488.01 cm<sup>-1</sup>[10].

When material was calcined at higher temperature this absorption band was observed shifted to lower wave number region. This shows that Cd is substituted at site of Zn ions by replacement of Zn ion. The absorption bands in the region 511.15 to 588.31cm<sup>-1</sup> associated to Cd-O vibration was not appearing in pure ZnO structure. The absorption band in the wave

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number region 667.39 to 626.89 cm<sup>-1</sup> assigned to Zn-Cd-O vibration was observed spread over the wave number region 611.45 to 669.32 cm<sup>-1</sup> due to Cd doping in ZnO lattice. The effect of calcinations can be summarized in table 2. The similar assignment has been reported in earlier literature [10].

The vibration band at 833.28 cm<sup>-1</sup> related to ZnO vibration which was broaden in the region frequency 731.05 to 895.00 cm<sup>-1</sup> due to Cd doping. The calcinations process affects the band position and bands are shifted to lower wave number value. The absorption peak present at 1020.38 cm<sup>-1</sup> is assigned to C-O-C this peak was appeared to be dispersed from 1020.38 to 1047.38 cm<sup>-1</sup> on calcinations and doping. Symmetric and asymmetric bending mode of C=O assigned at 1415.80 and 1624.12cm<sup>-1</sup> and the peak located at 1745.64 and 1815.08 assigned to C=O [11]. The absorption band at 2181.56 and 2387.85 related to CO<sub>2</sub> the atmospheric absorption of the carbon dioxde on the surface of ZnO and CdZnO nano particles. The effect of calcinations was clearly observed form the table 2.

The band located in the region 2924.18 and 3026.41 cm<sup>-1</sup> was related to symmetric and asymmetric C-H stretching bonds which was disappeared in pure ZnO lattice. The absorption band located in the region 3433.71 to 3471.98 cm<sup>-1</sup> was attributed to the presence of hydroxyl bond O-H due to water molecules absorbed from atmospheric moistures. There were some bands originated from the presence of water moisture and  $CO_2$  in the air in the process of making powder. The shift in the position of the band towards lower and higher frequency can be associated with change in bond length due to the partial substitution of Cd at the ZnO lattice The analysis IR data confirmed that Cd was doped in the ZnO matrix.





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Functional Group	As prepared ZnO	ZnO-Cd 0 <sup>0</sup> C	ZnO-Cd 200 <sup>0</sup> c	ZnO- Cd400 <sup>0</sup> C	ZnO-Cd 600 <sup>0</sup> C
ZnO	443.64	451,36	432.07	437.86	437.86
	476.43	464.86 499.58	464.86 453.29	464.86 488.01	464.86 478.36
		513.08	511.15	511.15	511.15
		561.30	588.31	559.38	588.31
Zn – O - Cd	667.39	611.45	669.32	669.32	611.45
	626.89	669.32	655.82	611.45	669,32
87	833.28	731.05	711.76	719,47	719.47
		761.91	759.98	767.69	771.55
Zn – O - Cd		858.35	821,70	875.71	779.27
		895.00	871.85	823.63	877.64
	1020.38	1020.38	1020.38	1020.38	1020.38
C – C	Control of the Property Control of Control o		1045.45	1045.45	1047.38
C - C		1111.03	1114.89	1114.89	1109.11
		1274.99	1232.55	1234.48	
	1383.01	1383.01	1327.07	1325.14	1377.22
C - O			1383.01	1375.29	
C = O	1415.80	1442.80	1438.94	1442.80	1442.80
	1624.12	1564.32	1560.46	1585.54	1562.39
		1629,90	1629.90	1629.90	1620.26
		5447 (K1.45-03 (S-638)		and the second s	1629,90
		1764.93	1726.35	1724.42	1763.00
		1745.64	1786.14	1774.57	1774.57
$\mathbf{C} = \mathbf{O}$		1815.08	1818.93	1818.93	1892.23
					1928.88
COO -	2181.56	2181.56	2343.59	2343.59	2345,22
	2387.95	2387.95	2499.83	2492.11	2416.89
		2490.18		1	
С-Н	C 1990 C	2924.18	2924.18	2808.45	2808.45
		3026.41	2958.90	2924.18	2997.48
0 - H	3433.71	3389.04	3437,26	3433.41	3433.41
	3471.98	3450.77	3533.71	3531.78	3531.78

## 3. Surface Morphology Study of Cd doped ZnO

The SEM images of as-prepared and calcined Cd doped ZnO-NPs were Shown in figure 5 the scan image of as-prepared Cd doped ZnO nanopowder exhibits polycrystalline, porous morphology with interconnected grains of average particles size 52 nm present on ZnO (Cd)

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nanoparticles. The particles size of the grain is auto visualized in the micrographs at the time of scanning the samples. The Cd doped ZnO cleined at 200 °C shows similar morphology with hexagonal nanorod stick on the surface. The small crystalline agglomerated to form nano fused clusters seen in the surface morphology of ZnO(Cd). The microstructure was observed changes to hexagonal nanorods at high temperature, the sample calcined at 600 °C exhibit excellent nano rod arranged like plates of rocks. The size of nanorods and nanograins is vary from 32 to 86 nm which is visualized in SEM images. The average grain size ranges from 30nm to 42 nm was displayed in table 1 above. The increase in grain size as compared to obtained from XRD data is due to agglomeration of the particles. ZnO(Cd) nanostructures exhibit significant varying morphology and may be used as antibacterial agent. It is scope for future work.



Figure 5.SEM Image of As prepared and calcined Cd doped ZnO Nonostructure

## 4. EDAX Analysis of Cd Doped ZnO

The EDAX spectra of ZnO(Cd) was shown in figure 6. EDX spectra concluded that present samples contain Cd, Zn and O elements. Their initial and final atomic percentage was also investigated and presented in table 3. Although the 5% doping of Cd in ZnO was confirmed form the EDAX spectra however the Zn percentage was less than initial atomic percentage. This may be due to in homogeneity of the samples. As prepared and calcined ZnO(Cd) samples show Oxygen and Zinc rich. The sulphur peak appeared in the spectra are due to the impurity in the carbon tape pasted on copper grids used for the measurement in the machine.





Figure 6. EDAX Spectro of asprepored and calcined Cd doped ZnO Nanostructure

Table 3.1	ndicating t	he weight %	6 and atom	ic <u>% of</u> Zu	O(Cd) Nan	ostructures.		
	Weight	Atomic	Weight	Atomic	Weight	Atomic	Weight%	Atomic
Content	%	%	%	%	%	%		%
	27°C		200 °C		400 °C .		600 °C'	
OK	31.95	66.24	28.83	62.98	26.37	59.24	31.67	65.77
SK	6.46	6.68	7.67	8.36	7.56	8.48	9.61	9.96
Zn L	41.95	21.28	39.89	21.33	48.45	26.64	32.49	16.51
Cd L	19.64	5.80	23.61	7.34	17.62	5.64	26.23	7,76
	100.00		100.00		100.00		100.00	

#### 5. UV-Visible Absorption Study for Cd doped ZnO

The absorption spectrum of pure ZnO, and as prepared and calcined ZnO(Cd) nanopowder is shown in fig 7. Uv-Visible absorption spectra of undoped ZnO sample shows t a strong absorption band at about 363 nm. The absorption is maximum in the Uv region and after that sample shows less absorption in other words the material is highly transparent. The as prepared ZnO(Cd) sample shows absorption band at 361 nm. However intensity of absorption was tremendously decreased as compared to pure ZnO [12]. The intensity of calcined ZnO(Cd) was gradually increased as the temperature of calcinations increased. The effect of calcinations on the absorption band is significant that the absorption bands shifted to the blue side of the electromagnetic spectrum. The as prepared Cd doped ZnO has large optical window as compared

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to pure ZnO as there was no any absorption bnd in the region 360 to 1100 nm. The optical window region increased on calcinations the ZnO(Cd) since absorption peaks shifted to lower wavelength region. This attributed to grain size and change in structure, morphology and change in defect states of the material on doping and calcinations. The estimated band gap and near band edge wavelength is tabulated in table 4. On sintering at different temperatures effectively tuned the band gap. This is attributed to change in structure and grain size. The band gap decreased on increasing grain size. As prepared ZnO(Cd) exhibit 3.44 eV significant band gap as compared to pure ZnO nanostructure (3.42 eV). The band gap was observed decreased on increasing sintering temperatures (200 to 600 °C). The decrease of band gap is attributed to oxygen and Zn site vacencies. The pure and Cd- doped ZnO nanostructure exhibiting higher band gap and lower particle size may be used in optoelcectronics, photoelectrochemical cells, and for biological applications.



Nanostructure calcined at different temperature

Table 4. Variation betwe	en band gap(Eg) and	calcined temperature
Samples ZnO-Cd Temperature	Wavelength (nm)	Band gap (eV)
Pure ZnO	363	3.42
As-prepared ZnO(Cd)	361	3.44
200°C ZnO(Cd)	360	3.45
400°C ZnO(Cd)	358	3.47
600°C ZnO(Cd)	358	3.47

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Figure 8 Variation of Energy band gap with calcinations of ZnO(Cd) Nonostructure

#### 6. Photoluminescence Investigations for Cd doped ZnO

Photoluminescence Spectra (PL) of ZnO(Cd) samples calcined at 27 °C and 600°C have been studied in reference to the luminescence intensity of emission spectra. PL spectra of excitation and emission of as prepared ZnO(Cd)27°C and ZnO(Cd) calcined at 600 °C were recorded at room temperature, and shown in figure 9. The PL intensity in excitation at 360 nm for pure ZnO sample is three times more than as- prepared ZnO(Cd) sample. The intensity of ZnO(Cd) calcined at 600 °C was observed decreased as compared to as-prepared ZnO(Cd) and pure ZnO samples. The effect of decrease in excitation intensity reveals that defects produced due to oxygen deficiency or interstitial defects were reduced on doping and on sintering the samples. The Photoluminescence spectrum exhibits two emission peaks one at 368nm and excitation emission 488nm, shows that highly blue shifted The amplification of the UV emission indicates that the crystalline defects in the ZnO nanoparticles have changed after Cd doped. On the other hand, at moderate doping (5%) Cadmium atoms acts as surfactant and changes emission intensity in blue green region. The intensity of blue green emission bands (428-599 nm) was observed decreased leads to the change in surface morphology. On calcinations Cd is substituted at the regular site of the ZnO matrix on replacing Zn. PL emission in green region (420 to 600 nm) was decreased on sintering the sample.

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Figure 9.FL for us prepared InD and coloined at 600°C ZnO(Cd)

#### 7. Antibactrial Activity of pure ZnO and Cd-doped ZnO

In order to study the antibacterial activity of Cd-doped ZnO nanoparticles against Bacillus thuringiensis NCIM2130 and Pseudomonas cf. monteilii 9 cultures slightly modified Agar Well Diffusion method was used. The pure ZnO and Cd doped ZnO nanopowder were calcined at 600 °C and used for antibacterial study. The inhibition zone size was measured. Pure ZnO shows inhibition zon size of 16 mm while Al doped ZnO shows increase in zone size up to 42mm. The pure ZnO and Cd doped ZnO never show antimicrobial activity against Pseudomonas cf.monteilii 9 and not presented in form of photoplate. While comparing inhibition zone of pure ZnO with respect to ZnO(Cd)) against Bacillus thuringiensis NCIM2130 and Pseudomonas cf.monteilii 9, Cd doped compound is more active than pure ZnO. The photo plate of Cd doped ZnO nanostructures calcined at 600 °C have been shown in figure 10.



Figure 10. Photo plate of Incubation of Bacillus Thuringiensis Comparision by ZnO as prepared with ZnO(Cd)

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Table 5. Inhibition Zone (mn calcinations Temperatures	n) Zones produced by ZnO nar	io particles at 600 °C
Bacterial Culture	Inhibition Zone (mm)	
	ZnO Cd	ZnO Pure
Bacillus thuringiensis NCIM2130	42	16
Pseudomonas cf. monteilii 9	0	0

#### Conclusion

Wurtize hexagonal crystallity pure and Cd doped ZnO nanostructure was synthesized by using simple low cost Sol-Gel chemical precipitation method. Estimated grain size for Cd doped ZnO was 33 nm and for pure ZnO was 43 nm. hence it was concluded that preset prepared materials of pure and Doped ZnO have nanoscale dimension. The presence of absorption bands in the frequency range 412 to 491 cm-1 indicate the incorporation of Cd into ZnO nanocrystals. Band gap estimated for pure and Ag doped ZnO using near band edge wavelength was found to be 3.28 eV for pure ZnO and for Cd doped ZnO it was 3.43 eV. Increase in band gap was related to decrease in grain size on incorporation of Cd in ZnO matrix. The low percentage Cd doping in ZnO matrix absorption band edge was shifted to low wavelength side. The knowledge of emission peaks green region of the PL spectrum revealed the oxygen and inertial Zn vacancy. Antimicrobial study reveal that the calcinations of material may change structure and surface morphology of ZnO(Cd) that could affect the microbial growth. It was concluded that Cd doped ZnO nanostructure calcined at 600 °C can be used against Bacillus thuringiensis NCIM2130. To obtain excellent results Cd doping percentage should be increased.

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