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Optimizing ZnO/CdS Nano Composite Controlled by Fe Doping Towards Efficiency in Water Treatment and Antimicrobial Activity

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Abstract

Nanocrystalline composite zinc oxide (ZnO) and CdS with Fe doping thin films grown on glass substrate by chemical method. The parameterslike temperature of the solution, UVexposure, pH of solution, immersion time, immersion cycles, have been controlled and standardized for nanocrystalline film. The synthesis NPs were analyzed by X-ray diffraction (XRD). Rietveld method shows that Fe-doped composite ZnO/CdSis a single pure phase and wurtzite structure. Samples were analyzed bysophisticated various instrument like XRD, UV- Visible spectrometer, HRTEM, HRSEM and composition was analyzed by EDX attached with HRTEM. The band gap was calculated by absorption spectroscopy and found that absorption wasblue-shifted. The electron structure shows that doping changes the crystal structure and transition level create better efficiency and creates octahedral symmetry. The antibacterial studies showed that the 5.0 wt% Fe-doped exhibited maximum antibacterial effect.

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Introduction

Nano based Zinc oxide and Cadmium sulphide with doped thin films has been gaining much attention due to potentialapplications in largeareas. It is used as important material in optoelectronic application and highefficient antimicrobial activity.[1,2] ZnO/CdS-NPs exhibit potential antibacterial activities due to large surface to volume ratio. Among the various compound semiconductors, ZnO/CdS with Fe dopped nanoparticles have shown interesting antibacterial activity. The ZnO/CdSNPs are stable and relatively low toxicity and attractive

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antimicrobial properties.[3,6] ZnO nanocomposite are used in the wallpapers in hospitals as antimicrobials. Investigations on antibacterial efficiency of ZnO/CdS-NPs would enhance the research area of nanotechnology fromthe development of novel antibacterial agents against pathogenic bacteria strains like *Escherichia coli*, *Staphylococcus aureus*, *Clostridium perfringens*, *Pseudomonas aeruginosa*, *Enterococcus faecalis*, *Salmonella sp*. etc has become of utmost demand now-a-days.[7,9]

Due to the large band gap of the composite material,it has attracted lots of scientific attention.[10]

The paperdiscusses the reporton preparation of composite Zinc oxide (ZnO)and Cadmium Sulphide (CdS) with Fe doped thin films and studies the structuralproperties, surface morphology, optoelectronic and antimicrobial properties with water treatmentalong with thin films forms.

Experimental

Materials

ZnO, CdS ZnS, NaOH , $CdCl_2$ and Na2S were used from Merck, India.The materials used were in high analytical grade.

Synthesis of Fe doped Composite zno/cds Nanoparticles

The composite ZnO/CdS nanoparticles is doped 0.5, 1.0, 1.5, and 2.0 wt% with Fe.The chemical used are ZnS, NaOH, $CdCl_2$ and Na_2S.The constituent are stirred 12 hours until the solution become transparent. After washing the samples were dried with alcohol in a hot air oven and maintain temperature 700C for duration of 2 hours. After preparation solution was caste in glass substrate and kept 24 hours for adhesion.[11]

Preparation for Antibacterial Activity Test

Escherichia coli (MTCC 739), *Staphylococcus aureus* (MTCC-740), *Klebsiella pneumonia* (MTCC 432) and *Pseudomonas aeruginosa* (MTCC-424) were used for the study of antibacterial efficacy. The microbial cultures were used from the "Microbial Gene Bank" (MTCC). The bacterial formationwasperformed at -4°C.

Antibacterial processdone in three samples like un-doped, Fe-doped ZnO/CdS NPs and UV treated doped ZnO/CdSNPs by Well Diffusion Assay.[12,14] The nutrient agar plates were inoculated by spreading the swab over the region. 6mm diameter were cut on the agar plates and the wells areplaced with the composite ZnO/CdSnanoparticles. For optimizing the activity in positive control Standard antibiotic (Tetracyclin) was used. All plates were keptovernight at 37^{0}C.The inhibition diameters were measured usinghigh resolution travelling microscope.

Results nd Discussion

Rietveld Refinement Analysis of Xrd

X-ray diffraction (XRD) analysis of Fe dopedZnO/CdS nanoparticles was prepared for structural analysis and diffractometer was maintained withstandard voltage of 40kv and a current was maintained at 30mA. The diffraction peaks are obtained at 2θ=31.73^0, 34.38^0, 36.22^0,47.56^0, 56.59^0, 62.79^0, 66.43^0, 67.95^0, 69.08^0, 72.67^0, and 77.050. The diffraction peaks are fitted and foundhexagonal wurtzite type structure. The peaks observed at 2θ = 22.99^0, 26.5^0, 29.15^0, 44.81^0 and 53.92^0 are corresponding to the planes (1 0 0), (0 0 2), (1 0 1), (1 1 0) and (1 1 2) of hexagonal phase of both ZnO and CdS. From the XRD patterns, it was observed that Fe^{2+} ions were substituted systematically in vacancy of Cd and Zn without changing the hexagonal structure. The peaks are shifted to higher diffraction angle due to the formation of strain.Using DebyeeScherrer equation,the average crystallite size was calculated.[15,16] Theparticle size was found in the range of 8-12nm.

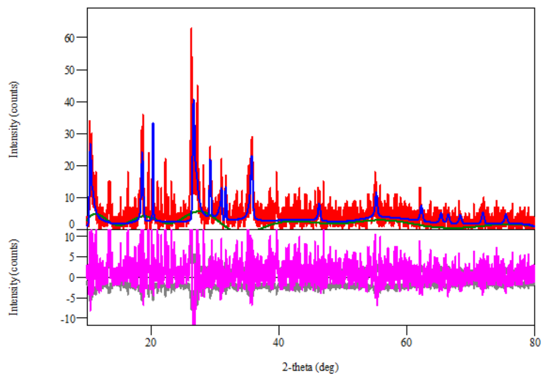


Fig.1: XRD Rietveld refinement spectrum of Fe doped ZnO/CdS

The Rietveld refinement plots of Fe-doped ZnO/CdS nanoparticles was fitted with standard mathematical functions.[17,18] The structural analysisis a good agreement with pure hexagonal wurtzite ZnO and Cd Sphase. From the table it was seen that with increase of doping concentration due to the stress the lattice parameter decreases.

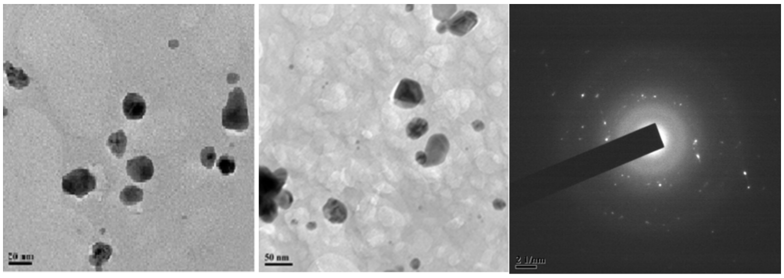
The Rietveld refinement analysis gives information about the variation of lattice constant. The lattice parameter are shown in table. The c/a parameter value is 1.603 which is a close packed hexagonal structure.

Tem Result Analysis

Composite nanoparticles variation was confirmed by TEM analysis (Model- JEM-100 CX II, Jeol) of the Fe doped ZnO/CdS nanoparticles. It is clear that there was formation of the distinct, disaggregated and uniform size i.e. monodispersed nanoparticles. Fe doped ZnO/CdS nanostructure showed that the sample had an average diameter of 40 nm with mix spherical and hexagonal shape.

Table 1: Angular correction

Data name	a(A)	b(A)	c(A)	alpha(deg)	Beta (deg)	gamma (deg)
Fe doped ZnO/CdS	3.29 (6)	3.294(6)	5.27 (11)	90	90	120



A B C

Fig. 2: TEM Photograph of ZnO/CdS nanoparticle (A) without doping, (B) after Fe doping and (C) SAED pattern of the one-dimensional Fe doped ZnO/CdS

Table 2: Elemental analysis of ZnO/CdS nanocomposite with Fe-doped

ement	line	K factor	Weight %	Weight % sigma	Atomic %
O	K- SERIES	1.353	9.02	1.08	33.06
Si	K- SERIES	1.000	1.47	0.35	2.06
S	K- SERIES	1.069	9.86	0.94	6.07
Fe	K- SERIES	2.363	4.87	0.83	3.42
Zn	K- SERIES	3.907	48.07	1.70	38.73
Cd	K- SERIES	2.987	26.71	0.10	16.66
Total			100		

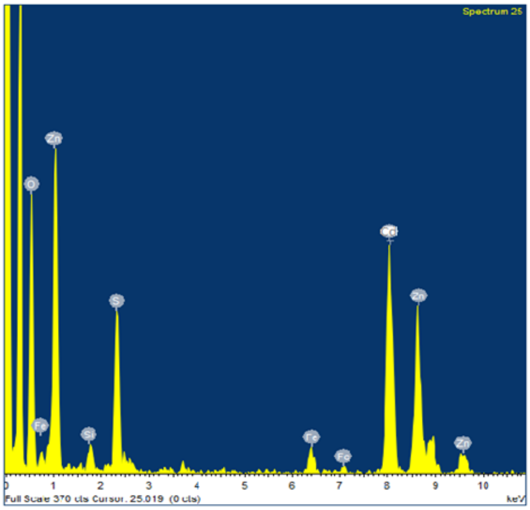


Fig. 3: EDX spectrum of ZnO/CdS nanocomposite with Fe-doped

Data analysis from the graph and table it is clear that the sample contains S, Fe, Zn and Cd and which is identified only the target elements and found without any impurities. From the above studies it is found that doping of Fe plays vital role for size and properties of the composite nanostructure.

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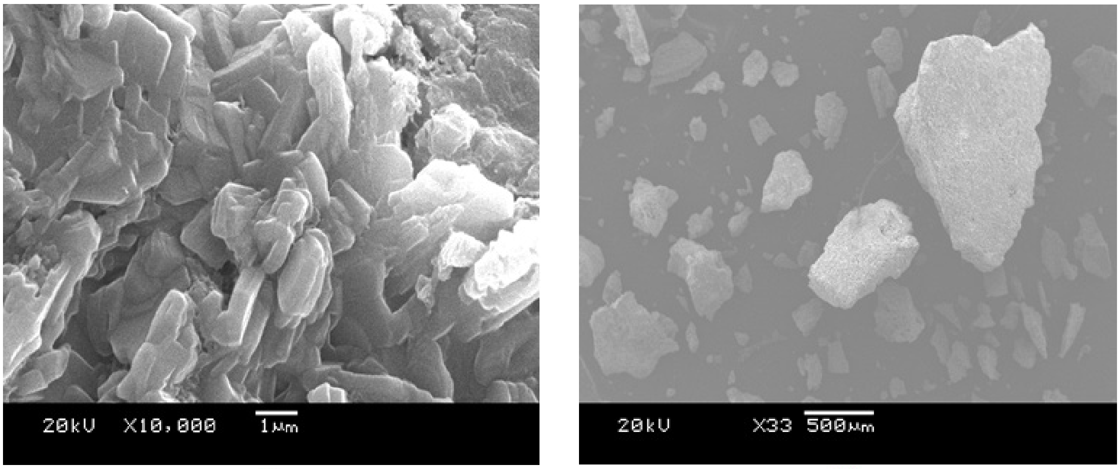
Sem Result Analysis

The morphology of both the dopped and un-dopped with Fe in composite ZnO/CdS NPs were studied by SEM (JSM-6360) (JEOL) Electron microscope. As shown in fig. 4A, the SEM analysis confirmed the size range of 22- 40nm, a clear indication of the formation of ZnO/CdS nanoparticles. Fig 4 (A). shows the formation of NPs that are self-aggregated. Fig. 4(B) shows the random distribution of ZnO/CdS NPs with 3.0 wt% Fe-doping.The average size found in between 37 to 42 nm. From the SEM analysis it is found that doping concentration changes the particle size.



A B

Fig. 4: SEM image of ZnO/CdS nanoparticle (A) without doping (B) 3.0 wt% Fe-doped ZnO/CdS

Uv Spectrometric Analysis

The UV-Vis spectrum was recorded in the range 300 to 800 nm and shown in Figure 5. For the prepared material ZnO/CdS-doped with Fe, the optical band gap value found to be 3.83-4.1 eV. It was also found higher doping decreases band gap. Table shows the variation. The change of lattice mismatch caused by incorporation of Fe^{2+} the presence of ZnO/CdS matrix may be the possible reason for the variation of band gap.[19,20]

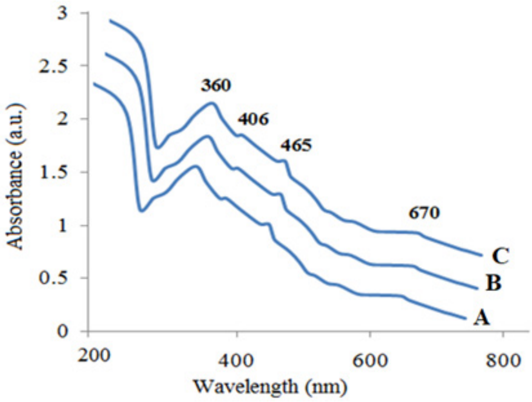


Fig. 5: UV-Vis Absorption Spectrum of Fe doped ZnO/CdS nanoparticle

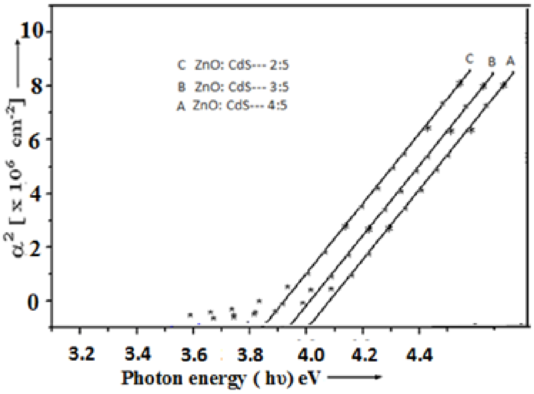


Fig. 6: Band gap energy of composite ZnO / CdSat different ratio with Fe doping

The absorption spectrum shows four characteristic bands at 360, 406 , 465 and 670 nm are due to the transitions from band to band. The position of bands identifies the characteristic of Fe^{2+} doping.

From the absorption spectra the possible transition, $(\alpha h\upsilon)^2$ versus $h\upsilon$ were plotted and calculated the band gap by extrapolating the straight-line. It is found that band gap is shifted and from this shifting data using established optical model from equation 1 band gap is calculated

$$E_{gn}= [E^2_{gb}+2\hbar^2E_{gb}(\pi/R)^2 / m^*]^{1/2} \quad ...(1)$$

Where m* is the average effective mass of the composite, Egbis average bulk band gap andEgn is the estimated band gap energy. With the change of band gap energy the particle size were estimated with Hyper Band Model (HBM). It is found that with increase doping concentration of Fe^{2+} the band gap increases the particle size decreases according to HBM. The particle size was found in the range 4-9 nm.[21,25]

Antimicrobial Assay

Antibacterial activity of the Fe doped ZnO/CdScomposite nanoparticles was carried out in water borne pathogenic bacteria species. The antibacterial activity was measured by zone inhibition area by high resolution travelling microscope. Tetracyclin of 1mg/ml, concentration was used as a control antibacterial agent. The results shown in the Table-2 depict that Fe doped ZnO/CdS nanoparticles are efficiently giving zone inhibition.[26]

Table 3: Antibacterial efficacy of Fe doped ZnO/CdS nanoparticles with standard antibiotic bacterial strains

Bacterial Strains	Zone of inhibition (diameter in nm) Fe doped ZnO/CdS nanoparticles			Tetracyclin ($1mgml^{-1}$) 100 µl
	50 µl	80 µl	100 µl	
E. coli	6±0.12	10±0.13	18±0.11	35±0.11
K.pneumonia	9±0.09	11±0.18	20±0.13	32±0.12
P.aeruginosa	3±0.13	8±0.16	17±0.09	31±0.11
S. aureus	6±0.09	12±0.11	20±0.09	34±0.13

Conclusions

Fe doped ZnO/CdS nanocomposite were successfully synthesized by chemical method. The prepared sample is composed of mix phases. Due to mismatch of radii of Fe^{2+} and Zn^{2+}/Cd^{2+} ions, diffraction peaks shift in the main characteristic peaks of XRD pattern. Optical band gap was calculated for different Fe and ZnO/CdS compositions and modification of band gap from 3.83 to 4.1 eV was observed. Different transitions were indicated by the four optical spectrum peaks. The results are improved for water purification methods including removal of the contaminants from water and destruction or immobilization of toxic compounds and pathogens. The size of the nanoparticles are obtained in different range because shape plays an important role and we have considered only spherical shape from TEM result and also depends on interface.

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Conflict of Interest

The author(s) declares no conflict of interest

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