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Characterization of Bivalent Zn (II) and Cd (II) Nanoparticles / Nanocomposites by XRD

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Abstract:- Herewith we discuss about the characterization of **ZnS/CdS** and **CdS/ZnS NPS,** composite materials were performed by Energy Dispersive X-ray Spectroscopy. ZnS and CdS nano particles have versatile applications in light –emitting diodes. The XRD pattern of **CdS-ZnS** showed mixed peaks for both cubic CdS and hexagonal ZnS suggesting the formation of both CdS and ZnS nanocomposites. In case of **CdS-ZnS** sandwich type NC which consisted of a mixture of two different nanoparticles in close contact in a sandwich type fashion, all the electrons and holes were directed towards CdS. The increased amount of photo-excited electrons in CdS ultimately led to enhanced photo-activity. The close contact of CdS and ZnS in the nanocomposite was confirmed by XRD. The formation of core shell **ZnS/CdS** was further supported by the presence of sharp peaks and absence of mixed peaks. The estimated crystallite size for **ZnS/CdS** based on the FWHM of the (111) peak was 5.58nm.

Keywords ZnS, CdS, nanoparticles, XRD spectroscopy.

1. INTRODUCTION:

ZnS and CdS nano particles can be prepared in the forms of powder, thin film using different synthesis techniques such as sol gel, thermal evaporation [1], MWI method. ZnS NP doped with transitional metals may possess improved properties [2]. The optical properties of various semiconductor QDs may also be improved by coating them with a shell of a second higher band gap semiconductor, resulting in core-shell type-I systems. Core-shell nanocrystals contain at least two semiconductor materials in an onion-like arrangement. Various semiconductor photocatalysts can be used for this purpose, among all the concerned photocatalysts, ZnS has been widely investigated as an efficient photocatalyst for the photocatalytic degradation of organic pollutants such as dyes, p-nitrophenol and halogenated benzene derivatives in wastewater treatment. CdS is an effective photocatalyst in visible region but faces a major drawback of poor quantum efficiency due low stability in solution as a result of Cd^{2+} ions leaching.

2. MATERIALS AND METHODS:

Energy Dispersive X-ray Spectroscopy (EDS):

The synthesis procedure for nanoparticles and their composites are cited in reference 1.We have investigated the chemical composition of ZnS and CdS in the **ZnS, CdS-ZnS**, **ZnS/CdS** and **CdS/ZnS,** the elemental analysis of samples were performed by Energy Dispersive X-ray Spectroscopy. The EDS spectra of the synthesized ZnS nanoparticles and its nanocomposites with CdS. The spectrum in **Figure 1: (a)**, shows the EDS spectrum of **ZnS**, it revealed the presence of only Zn and S peaks confirming the formation of pure zinc sulphide with no other elemental impurity. The average atomic percentage ratio of Zn:S was 54.00:46.00. **Figure 1: (b)** shows the EDS spectrum of **CdS-ZnS**, it shows the presence of Zn, Cd and S peaks confirming the formation of both CdS and ZnS. The average atomic percentage ratio of Zn:S:Cd was 18.97:50.00:31.02 which were in good agreement with the stiotiometric ration used for the synthesis of this nanocomposite, further confirming the formation of both ZnS and CdS.

The EDS patterns of **ZnS/CdS** and **CdS/ZnS** core shell nanoparticles with low shell thickness are shown in **Figure 1: (c)** and **(d)**. The results showed that the weight percentage of Cd in the **ZnS/CdS** is 10% and the weight percentage of Zn in the **CdS/ZnS** is about 9%. **Figure 1: (c)** shows the EDS spectrum of **ZnS/CdS**. It contained peaks for Zn and S along with weak peaks for Cd supporting the formation of CdS shell over ZnS core. The average atomic percentage ratio of Zn:S:Cd was 40.69:49.08:9.32. **Figure 1: (d)** shows the EDS spectrum of **CdS/ZnS**, it revealed the presence of Cd and S peaks along with weak peaks for Zn supporting the formation of ZnS shell over CdS core. The average atomic percentage ratio of Zn:S:Cd was

8.40:51.50:40.00. Other peaks in this figure corresponded to carbon, oxygen and silicate which were due to sputter coating of glass substrate on the EDS stage and were not considered in elemental analysis of Zn, Cd and S.

The XRD pattern shows the presence of pure ZnS nanoparticles, indicating the formation of hexagonal, W-type crystal structure with seven prominent characteristic diffraction peaks for hexagonal ZnS at 2 =24.316˚ (100), 25.963˚ (002), 27.611˚ (101), 36.018˚ (102), 43.160˚ (110), 47.364˚ (103) and 51.383˚ (112)[3-6]. Few weak peaks at 67.879˚ (203), 71.254˚ (211) and 75.120˚ (105) were also observed. The estimated crystallite size for **ZnS** was 1.98nm based on the FWHM of the (101) peak. The crystal structure, FWHM peak value and the calculated average particle sizes are listed in **Figure 2:** and **Table 1.**

The XRD pattern for **CdS-ZnS** (**Figure 3:**) showed mixed peaks of cubic CdS at 2 =25.992˚ (111), 30.069˚ (200), 43.333˚ (220), 51.482˚ (311)[7-8] and hexagonal ZnS nanoparticles at 2 =24.333˚ (100), 25.992˚ (002), 27.800˚ (101), 36.254˚ (102), 43.333˚ (110), 47.565˚ (103), 51.482˚ (112) [3-6]. This ruled out the presence of core-shell structure and predicted the formation of CdS-ZnS colloidal nanocomposites, having cubic CdS and hexagonal ZnS NPs in a s andwich type fashion. The estimated crystallite size for **CdS-ZnS** based on the X-ray diffraction peak was not possible due to the presence of mixed peaks of hexagonal ZnS and cubic CdS.

No peaks similar to (100), (002), (101), (102), (110), (103) and (112) corresponding hexagonal W-type ZnS core was observed. The presence of diffraction peaks corresponding to only CdS and complete absence of diffraction peaks due to ZnS core predicted the core shell structure of the **ZnS/CdS** sample. The presence of diffraction peaks corresponding to only CdS and complete absence of diffraction peaks due to ZnS core, was in agreement with earlier reports on core-shell structures, that as the shell thickness increases, the diffraction pattern shifts from core to that of the shell material[9-11].

3. RESULTS AND DISCUSSION:

The structural studies were performed using Diffraction Spectroscopy.

Table 1: The crystalline phase and average crystallite size obtained by XRD data

Sample	Crystalline Phase	FWHM (in degree)
ZnS	Hexagonal	2.74
CdS-ZnS	Mixed	-
ZnS/CdS	Cubic	1.46
CdS/ZnS	Hexagonal	1.74

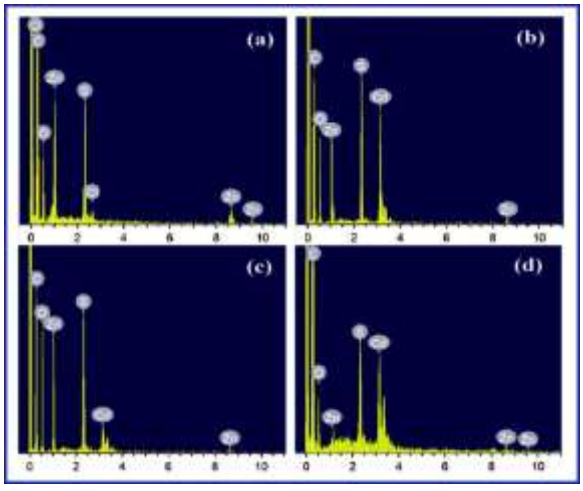

Figure 1: (a-d) The EDX Spectra of synthesized **ZnS** nanoparticles and its nanocomposites with CdS (**CdS-ZnS**, **ZnS/CdS** and **CdS/ZnS**).

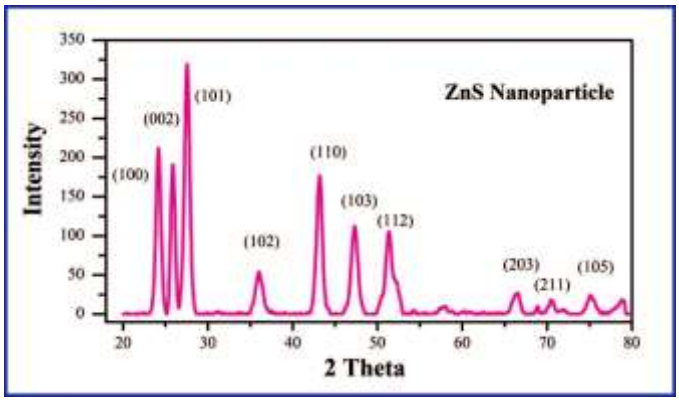

Figure 2: The XRD pattern for ZnS sample showing hexagonal-wurtzite type structure.

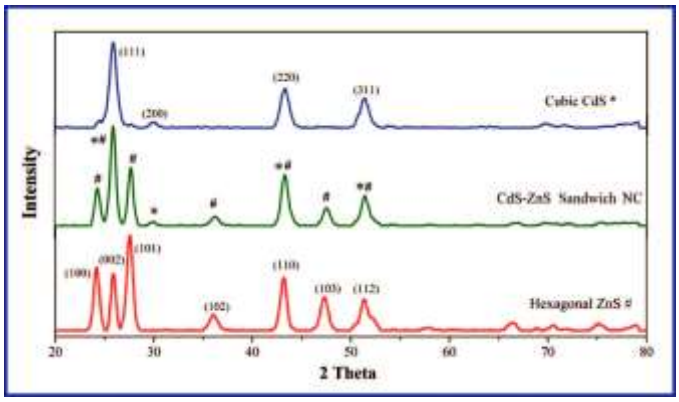

Figure 3: The XRD pattern for **CdS-ZnS**, showing mixed peak of pure cubic CdS

(*) nanoparticles and pure hexagonal ZnS (#) nanoparticles CdS.

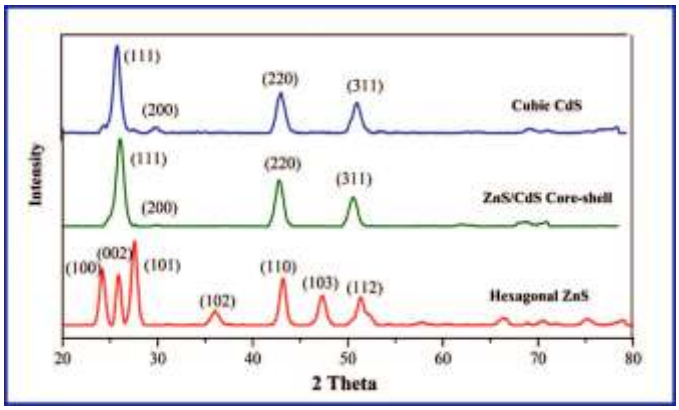

Figure 4 : The XRD pattern for **ZnS/CdS** sample showing similar peaks to the XRD pattern of pure CdS & the XRD pattern for **CdS/ZnS** sample showing similar peaks to the XRD pattern of pure ZnS .

CONCLUSION:

Zinc Sulphide nanoparticles (**ZnS**) were synthesized using single pot chemical precipitation method under ambient conditions. The synthesized **ZnS** showed good thermal stability, crystalline nature having hexagonal crystal structure, good optical property following effective mass approximation and quantum confinement effect. The XRD pattern of **CdS-ZnS**

showed mixed peaks for both cubic CdS and hexagonal ZnS suggesting the formation of both CdS and ZnS in closer and which was further confirmed The XRD spectra for **CdS/ZnS** showed similar pattern to that of hexagonal ZnS shell.

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