

## Characterization of nano-sized CdS-Ag<sub>2</sub>S Core-Shell Nanoparticles Using XPS Technique.

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### Abstract

This paper reports the use of X-ray photoelectron spectroscopy (XPS) method for the characterization of surface composition of core and shell nanoparticles. The core and shell nanoparticles were synthesized using sodium bis(2-ethylhexyl) sulfosuccinate (AOT)/n-heptane/water micro-emulsion technique. The post-core and partial microemulsion methods have shown promise for core and shell formation and enhanced stability and sphericity of the semiconductor nanoparticles. In addition to above technique, the core-shell nanoparticles have also been characterized using SEM- EDAX and results are discussed. The XPS studies of CdS, Ag<sub>2</sub>S, and CdS-Ag<sub>2</sub>S nanoparticles prepared by post core/partial microemulsion methods were carried out and results are compared with physical mixing of CdS and Ag<sub>2</sub>S nanoparticles. The Ag/Cd atomic ratio is found to be 5 and 10 as determined by XPS methods for nanoparticles prepared by using post core and partial microemulsion methods respectively. These results show the formation of core and shell nanoparticles.

*Key words:* Core-shell, Nanoparticles, w/o microemulsion, XPS, CdS-Ag<sub>2</sub>S, AOT

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## 1. Introduction

Nanophase and nanostructured materials as a component of nanotechnology have attracted a great deal of attention of today's research [1]. The synthesis of inorganic nanocrystals and nanoparticles in a controllable fashion has been the goal of much active research over the past decade [2, 3]. Semiconductor nanocrystals and nanoparticles have been studied extensively as their properties are dependent on size, morphology, and surface composition [4-10].

The synthesis and characterization of core and shell nanoparticles is an important research area at the frontier of advanced material chemistry [11, 12]. These core and shell composite material can increase the luminescence quantum yield due to improved passivation of the surface and tend to be more physically robust than the "bare" organically passivated clusters [13, 14]. Recent research interest arises from the diverse attributes of core and shell nanoparticles as model building blocks towards functional materials, including 1) monodispersity, 2) core and shell processability 3) solubility, 4) stability and tenability, 5) capability of self assembly and 6) functional activities in involving optical, electronic, magnetic, catalytic and chemical/biological phenomenon. Examples of core and shell structures include CdS-ZnS [15], Au-SiO<sub>2</sub> [16], Ag-SiO<sub>2</sub> [17], CdS-SiO<sub>2</sub> [18], CdS-HgS [19], CdSe-CdS [20], SiO<sub>2</sub>-TiO<sub>2</sub> [21], Fe<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub> [22], and CdS-Ag<sub>2</sub>S [23]. From these examples, we can note that these are potentially useful in a broad range of application. The controlled synthesis of novel uniformly coated stable nanoparticles, however, for many years has remained as a technical challenge.

The CdS-Ag<sub>2</sub>S coated nanoparticles were synthesized by Han *et al.* [23], by inverse microemulsion technique and were characterized by means of EDAX and TEM technique. The TEM micrographs of these particles do not clearly show the core and shell structure because of the nanometer size and similarity in core and shell materials. Therefore, a better characterization technique is needed for characterizing these kinds of core and shell nanoparticles. In general, it is not clear how core and shell nanoparticles can be clearly documented and adequately characterized. It is not certain that some of TEM micrographs showing good contrast to discern core from shell can be used for other materials or for that matter, at lower size level. Overall, the research on the formation and characterization of core and shell nanoparticles is in its infancy

and focused studies are called for, to develop better formation methods as well as better characterization techniques.

X-ray photoelectron spectroscopy is a surface analytical technique with a sampling depth of 2-5 nm [24] and it is well suited for interrogation of the surface composition of the colloidal particles [25]. Recently it has been used for surface characterization of gold nanoparticles [26]. In a perfectly coated “core-shell” particle the core material would be completely buried under a uniform layer of the shell material. If the thickness of the shell were in between 3-6 Å, XPS and X-ray excited Auger spectroscopy techniques whose sensitivities lie in this range may be reasonably used to demonstrate the layering of shell materials [25, 27, 28].

In this study, we have used two different methods, post core and partial microemulsion methods to prepare CdS-Ag<sub>2</sub>S core and shell nanoparticles. We report a detailed X-ray photoelectron spectroscopy study of these core and shell nanoparticles. These results are compared with SEM-elemental detection analysis as well. In this paper, we have discussed the various features of Cd3d, Ag3d and S2p core levels, which provide an understanding of the elemental surface composition and formation of core-shell morphology of prepared CdS-Ag<sub>2</sub>S nanoparticles.

## **2. Experimental**

### ***Materials***

The surfactant, Dioctyl Sulfosuccinate Sodium Salt (Aerosol OT, or AOT) was purchased from Sigma with 99% purity. Silver nitrate (AR) and Cadmium nitrate (LR) were purchased from S.D. Fine Chemicals, India. Ammonium sulfide [(NH<sub>4</sub>)<sub>2</sub>S, 25%] was purchased from SISCO, India. Heptane was purchased from SISCO, India and was used after refluxing over sodium wire for two hours to remove moistures. Double distilled water was used throughout the experiments.

### ***Sample preparation***

Aqueous solution of Cadmium nitrate (0.1M), Silver nitrate (0.1M) and Ammonium sulfide (0.2 M) were freshly prepared. Neat and clean previously dried 15 ml centrifuge tubes with stopper and small glass bottles (Scam, India) were used to carryout the experimental work.

Surfactant solution (stock solution) of concentration 0.1M was prepared by mixing appropriate amount of AOT in n-heptane as a continuous medium.

### ***Preparation of core-shell nanoparticles***

AOT capped CdS and Ag<sub>2</sub>S nanoparticles were prepared using w/o microemulsion as reported by Hota *et al.*, [29]. In an attempt to prepare core-shell/composite nanoparticles, we have used two mixing methods using w/o microemulsions. These methods are as follows.

- a. Post-core method:** In this method, a microemulsion,  $\mu\text{E}$  of AgNO<sub>3</sub> solution was added to the  $\mu\text{E}$  containing CdS nanoparticles, which shall act as cores. The microemulsions containing core nanoparticles of CdS also contains the S<sup>-2</sup> ions since excess amount of (NH<sub>4</sub>)<sub>2</sub>S was added while preparing the core nanoparticles.
- b. Partial microemulsion method:** This method is similar to the post-core method except the fact that the AgNO<sub>3</sub> solution was not added in the microemulsion form. It was directly added in aqueous form and in a drop wise manner followed by proper shaking.
- c. Physical mixing method:** In this method, two individual microemulsion, containing CdS and Ag<sub>2</sub>S nanoparticles have been mixed physically for the reference point of view.

The nanoparticles of CdS, Ag<sub>2</sub>S, and core-shell CdS-Ag<sub>2</sub>S prepared by post core/partial microemulsion methods were separated from microemulsions by addition of acetone followed by ultracentrifugation and washed 3-4 times with double distilled water and then ethyl alcohol in order to remove the surfactants. The nanoparticles were dried using the vacuum desiccator and used for XPS studies.

### ***X-ray Photoelectron Spectroscopy***

XPS measurements were performed on a V. G. Microtech Unit ESCA 3000 Spectrometer with twin anode, AlK <sub>$\alpha$</sub> (1486.6 eV) and MgK <sub>$\alpha$</sub>  (1256.6 eV). The vacuum in the analyzing chamber was  $<1 \times 10^{-9}$  Torr. Multichannel detectors were used to detect the photoelectron and a spherical sector analyzer was used to detect the photoelectron's energy. The X-ray flux used was 200 Watts. The spectrometer was calibrated by determining the binding energy values of Au<sub>7/2</sub> (84.0 eV) Ag 3d<sub>5/2</sub> (368.8 eV) and Cu 2p<sub>3/2</sub> (932.4 eV) using spectroscopically pure metals obtained from Johnson-Matthey, London. The binding energy values (measured to an accuracy of 0.2 eV)

are good agreement with the literature values. The XPS spectra were acquired at 50 eV pass energy, 5 mm slit width and take off angle (angle between electron emission direction and surface plane) of 55°. Due to a large number of channeltrons the detection limit has been increased. The instrumental resolution under these conditions is 1.6 eV full width at half maximum (FWHM) for the Au 4f<sub>7/2</sub> level. The sample material was mounted in a thin layer film on sample holder and used for recording the XPS spectra. All spectra were recorded with similar spectrometric parameters.

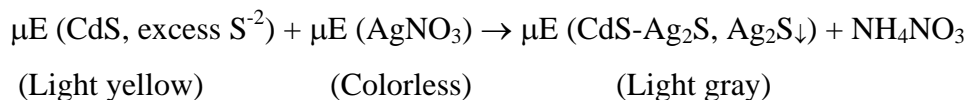
The general scan and C1s, O1s, S 2p, Cd 3d and Ag 3d core levels were recorded with referring un-monochromatized AlK<sub>α</sub>(1486.6 eV) radiation at 50 eV pass energy and take off angle (55°). The peak shift due to charging was corrected using the C1s level at 285 eV as an internal standard.

The XPS peaks assumed to have Gaussian line shape and were resolved into individual components after proper subtraction of baseline using Shirley background subtraction method with the help of software supplied by V.G. Microtech.

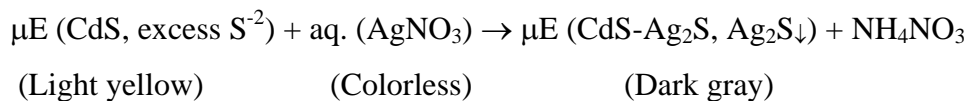
### 3. Results and Discussion

The formations of nanoparticles via two addition techniques are as shown below.

#### *Post-core method*



#### *Partial microemulsion method*



The UV-VIS spectra obtained from the addition techniques to obtain core-shell/nanocomposite are indeed, difficult to interpret as reported earlier by Hota *et al* [29]. It should be mentioned here that the nanoparticles are formed due to intermicellar exchange. This intermicellar exchange being random in nature, four different types of particles formation might take place such as CdS,

Ag<sub>2</sub>S, and CdS-Ag<sub>2</sub>S core-shell/composite nanoparticles. The absorption spectra of samples containing these particles may turn out to be a complex combination. Nevertheless, we believe that in the mixing methods used, the formation of core-shell nanoparticles will be dominant while the single core CdS and Ag<sub>2</sub>S nanoparticles will be comparatively less. To the best of authors' knowledge there is no technique available for the quantitative analysis of these mixtures of nanocomposites.

### ***SEM- EDAX Analysis***

The elemental composition of CdS-Ag<sub>2</sub>S nanoparticles is also determined using SEM energy dispersive analytical -X-ray (EDAX) spectroscopy by performing the spot measurements on nanoparticles powder. The major peaks we found due to presence of Cd and Ag along with sulfur and negligible amount of carbon and oxygen. The elemental ratios of Ag to Cd in the CdS-Ag<sub>2</sub>S core and shell nanoparticles were obtained using EDAX data. The table 1 summarizes the results of elemental composition EDAX analysis of the nanoparticles prepared by post core/partial microemulsion methods.

**Table 1: Elemental composition of CdS-Ag<sub>2</sub>S core-shell nanoparticles by EDAX**

Sample	Elemental Composition ( Atomic% )	
	<b>Ag</b>	<b>Cd</b>
Post core CdS-Ag <sub>2</sub> S	80	20
Partial emulsion CdS-Ag <sub>2</sub> S	88	12
Physical mixture (50:50) CdS and Ag <sub>2</sub> S	52	48

We observe from table-1 that the percentage compositions of Ag and Cd vary with in the range of 80 to 90 % and 10 to 20 % respectively for post core/partial microemulsion method. This result clearly shows that the materials in the outer portion of the nanoparticles primarily compose of Ag. This observation is also evidenced by Tougaard analysis in the post core method.

## *XPS Analysis*

XPS is very sensitive to the chemical composition and environment of the elements in a material. In XPS spectra, each element will give rise to a characteristic set of peaks in the photoelectron spectrum at kinetic energies determined by the photon energy and the respective binding energies. The presence of peaks at particular energies therefore indicates the presence of a specific element in the sample under study - furthermore, the intensity of the peaks is related to the concentration of the element within the sampled region. Thus, the technique provides a quantitative analysis of the surface composition. Arising out of the fact that the chemical surrounding slightly influences the inner energy levels of the atoms, it is possible to distinguish the signals originating from the atoms in different chemical surrounding like those located at the surface or the volume phase of the particles. It was used to evaluate the chemical composition of CdS, Ag<sub>2</sub>S and CdS-Ag<sub>2</sub>S nano-materials.

The survey scans were carried out to search for the presence of particular elements in the sample showed the presence of C1s, O1s, S 2p, Cd 3d and Ag 3d core levels. The C1s core levels was resolved in to two components at binding energies, CI = 285 eV and CII = 287.8 eV and are assigned to the hydrocarbon CH<sub>2</sub> and the presence of higher binding energy with C II carbon attached to oxyfunctionalities of AOT respectively. These CdS particles possess an inorganic crystalline CdS core surrounded by organic AOT growing in to macroscopic phase and preventing agglomeration during nanoparticle formation.

The Cd3d, Ag3d and S2p core levels of nanoparticles of CdS, Ag<sub>2</sub>S are shown in Fig 1. The Cd 3d spectrum in each case has a doublet feature due to spin orbit splitting resulting in to 3d<sub>5/2</sub> and 3d<sub>3/2</sub> peaks with spin orbit separation 6.7 eV. The characteristic binding energy of Cd 3d<sub>5/2</sub> and 3d<sub>3/2</sub> of CdS agree with those reported in the literature [6]. Figure 1a represents the XPS of CdS nanoparticles. Both peaks (Cd3d<sub>5/2</sub> and Cd3d<sub>3/2</sub>) samples have FWHM 1.8 eV and the position of the maxima at 405.4 and 412.3 eV respectively. The Cd 3d core level could be satisfactorily fit to single spin-orbit pair at 405 eV (Cd3d<sub>5/2</sub>) and at 412 eV (Cd3d<sub>3/2</sub>). These values are in good agreement with the published values for CdS nanoparticles [6]. Although the relative cross section of sulfur is small in comparison to the other elements, the sulfur peaks are sensitive to the chemical environment. The lower binding energy peak at 161.5 eV is indicative

of metal sulfide while the other peak at 168.8 eV has been assigned to the more oxidized form of sulfur from AOT molecule.

Figure 1b represents the XPS of Ag<sub>2</sub>S nanoparticles. The peak position (Ag3d<sub>5/2</sub> at 368.eV and S2p<sub>3/2</sub> at 161.2 eV) corresponds to Ag<sub>2</sub>S. These values are in good agreement with the reported values for Ag<sub>2</sub>S nanoparticles [7]. The Ag3d core level could be satisfactorily fit to a single spin-orbit pair at Ag3d<sub>5/2</sub> at 368.eV and Ag3d<sub>3/2</sub> at 374 eV. The Ag<sub>2</sub>S nanoparticles are cleaned thoroughly till free from adsorbed AOT molecules. The Ag<sub>2</sub>S cleaned sample free from AOT molecules showing only one species at 161.2 eV (SI) which is a characteristic binding energy for the sulfide components and absence of second species (SII) corresponding to C-SO<sub>3</sub>H at ~168.5 eV from AOT molecules.

The S2p level was resolved into a single spin orbit splitting of Gaussian component. The spin orbit splitting separation was allowed to vary during fitting. The binding energy value of S2p<sub>3/2</sub> is 161.2 eV and S2p<sub>1/2</sub> is 162.4 eV with separation of 1.2 eV and the ratio in intensity of the two components was 2:1. Thus surface composition of CdS and Ag<sub>2</sub>S nanoparticles are presented in Table-2.

**Table 2: B.E. (eV) of different core levels**

Sample	Cd3d	Ag3d	S	Ag/Cd
CdS	405.4, 412.3	-	161.5, 168.8	-
Ag <sub>2</sub> S	-	368, 374.1	161.2	-
Post core CdS-Ag <sub>2</sub> S	405.5, 412.3	368, 374	161.3, 168.9	5
Partial emulsion CdS-Ag <sub>2</sub> S	405.4, 412.3	368, 374.1	161.3, 168.9	~10
Physical mixture (50:50)	405.4, 412.3,	368.14, 374.14	161.9, 168.4	~1

***The nanoparticles of CdS-Ag<sub>2</sub>S by post core method***

The CdS-Ag<sub>2</sub>S nanoparticles prepared by post core method show the presence of C1s, O1s, S2p and Cd3d, Ag3d core levels with no significant impurities. The FWHM of Cd<sub>5/2</sub> and Cd<sub>3/2</sub> is increased from 2.2 to 2.8 eV and maxima are observed at binding energy of 405.5 and 412.3 eV respectively. Figure 2a shows the result obtained by using a fit procedure. The Cd core level



could be satisfactorily fit to a single spin-orbit pair of CdS nanoparticles. The Ag3d core level could also fit to a single spin-orbit pair of Ag3d<sub>5/2</sub> at 368 eV and Ag3d<sub>3/2</sub> at 374 eV as shown in Fig 2b. The XPS of S2p<sub>3/2</sub> level is shown in the Fig 2c. It is clear that there are two distinct species SI and SII at in the spectrum at 161.3 eV and 168.9 eV.

### *Tougaard analysis*

Much effort has been devoted to the developments of new and more accurate methods in the view of high and rapidly growing technological importance of reliable information on the in-depth composition of the surface region of solids on the nanometer scale. This observation has been applied in the formulation of the new method for quantification that is based on the quantitative analysis of measured peak shape and peak intensity. This technique is sensitive on the ~1-10 nm depth scale and it is non-destructive. Due to elastic and inelastic scattering of photo-excited core electrons the ratio of the peak areas to the increase in the background signal associated with a single XPS peak depend strongly in depth composition as reported by Tougaard et al., [27, 28].

Preliminary examination of Tougaard background analysis of Cd 3d signal indicates that the thickness of Ag<sub>2</sub>S shell on CdS core is in the order of 2-3 nm as supported by TEM analysis [29]. Fig. 3 shows the XPS spectrum of (a) CdS nanoparticles and (b) core-shell CdS-Ag<sub>2</sub>S nanoparticles prepared by post core method. There is clear evidence in spectra indicating a shell or coating over CdS nanoparticles. These two XPS spectra clearly demonstrate the inelastic background and are extremely sensitive to the depth of the location of Ag<sub>2</sub>S layer over CdS core nanoparticles. The ratio of the background/peak intensity is a strong function of the depth of core species. This ratio is 0.6 for CdS nanoparticles as compared to 0.98 for core-shell nanoparticles by post core method. The extent to which the peak intensity differs from the background depends on the variations in the concentration profile with in a thin layer of materials at the surface of the CdS-Ag<sub>2</sub>S nanoparticles.

The formation of Ag<sub>2</sub>S shell layer on CdS core is also supported by the fact that in the Tougaard background analysis the shift in base line after Cd core level is indeed, due to the coating of Ag<sub>2</sub>S over CdS species (shown in Fig 3b). In this study the peak shape and peak intensity are used for quantitative analysis of core and shell nanoparticles by XPS method.

### ***The nanoparticles of CdS-Ag<sub>2</sub>S by partial microemulsion methods***

The nanoparticles CdS-Ag<sub>2</sub>S prepared by partial emulsion method shows the presence of C1s, O1s, S2p and Cd3d, Ag3d core levels with no significant impurities. The peaks of Cd (Cd3d<sub>5/2</sub> and Cd3d<sub>3/2</sub>) samples have FWHM 2.8 eV and the position of the peak maxima are observed at 405.4 and 412.3 eV respectively shown in Fig 4a. The Ag3d core level could be satisfactorily fit to a single spin-orbit pair of Ag3d<sub>5/2</sub> at 368 eV and Ag3d<sub>3/2</sub> at 374 eV as shown in Fig 4b. The XP spectrum of S2p<sub>3/2</sub> level is shown in the Fig 4c. In this spectrum also there are two distinct species SI (161.3 eV) and SII (168.9 eV) observed in the spectrum as in case of post core method. The second binding energy value of 168.9 eV is a characteristic value of C-SO<sub>3</sub>H group of AOT surfactant. The atomic concentration of Ag/Cd ratio is ~ 10, where as this ratio is 5, for post core sample, as presented in Table 2. The high Ag/Cd atomic ratio suggests that the surface of partial microemulsion sample contain Ag<sub>2</sub>S molecule twice as compare to that of post core method. This result is also supported by SEM – EDAX spot analysis.

### ***Physical mixture of CdS & Ag<sub>2</sub>S nanoparticles***

The physical mixture of CdS and Ag<sub>2</sub>S (1:1) was used as a reference material for quantitative analysis of composition of Cd and Ag core levels to correlate with the XPS results of samples prepared by post core / partial microemulsion method. The general scan spectrum of the physical mixture shows the presence of C1s, S2p and Cd3d, Ag3d and O1s core levels as observed in previous samples.

The Cd 3d and Ag3d core levels of single spin-orbit pair at binding energy values of 405 eV, 412 eV and 368.eV, 374 eV respectively are in agreement with the CdS and Ag<sub>2</sub>S materials (As shown in Fig 5) . In this spectrum also S2p level of SI and SII species observed at respective binding energy values {Fig 5 (c)}, indicates the presence of C-SO<sub>3</sub>H group of AOT molecule. The Ag to Cd atomic ratio as estimated from the intensity of the spectra of physical mixture is found to be unity as expected and are comparable to sample of similar atomic compositions, as determined by Energy Dispersive X-ray Analysis (Table 1).

This comparison demonstrates that the expected core-shell structure is present, i.e. the material intended to be localized in the shell is indeed concentrated near the surface and the material from

core is indeed buried in the coating layer particles. Evidence of strong dependence of the electron attenuation length on the overall particles composition is seen.

#### **4. Conclusions**

The CdS-Ag<sub>2</sub>S core and shell nanoparticles were synthesized by post-core and partial microemulsions addition method using AOT/n-heptane/water microemulsion system. The quantitative elemental surface composition of the core-shell nanoparticles were characterized by using X-ray photoelectron spectroscopy (XPS). The XPS data are also comparable with the SEM elemental detection X-ray analysis (EDAX). The Ag/Cd ratio as determined by the XPS spectra of core-shell nanoparticles are (~5) and (~10) for post core and partial microemulsion methods respectively. However, the Ag/Cd ratio is found to be unity (~ 1) for physical mixing sample. These results indicate the formation of core-shell nanoparticles i.e. CdS nanoparticles are coated with a layer of Ag<sub>2</sub>S atoms.

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## Figure Captions

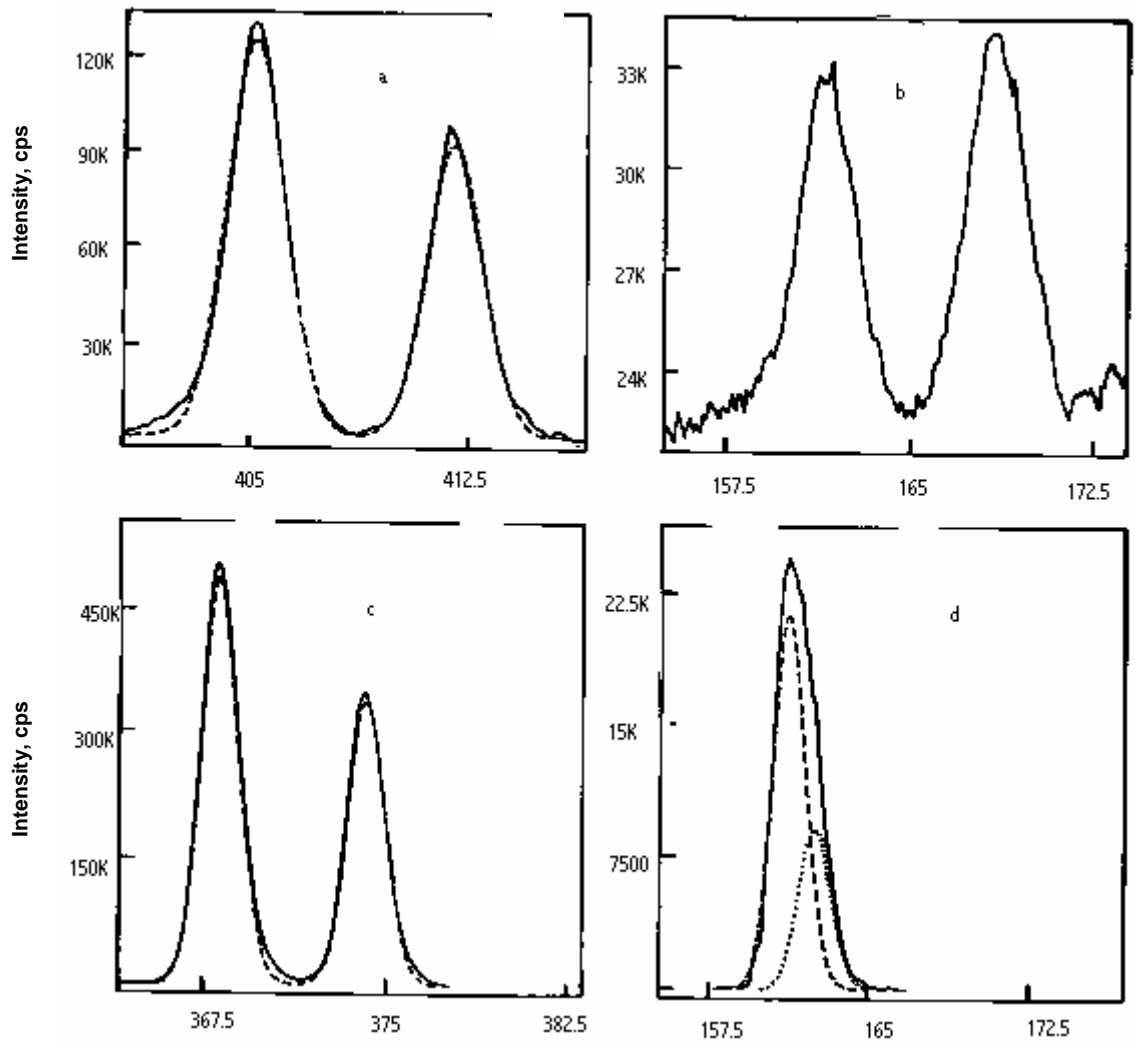
Fig. 1 : XPS Spectra of CdS and Ag<sub>2</sub>S nanoparticles (a) Cd 3d and (b) S2p (c) Ag3d and (d) S2p core level

Fig. 2 : XPS Spectra of CdS - Ag<sub>2</sub>S core-shell nanoparticles (post core method) (a) Cd 3d and (b) Ag3d and (c) S2p core levels

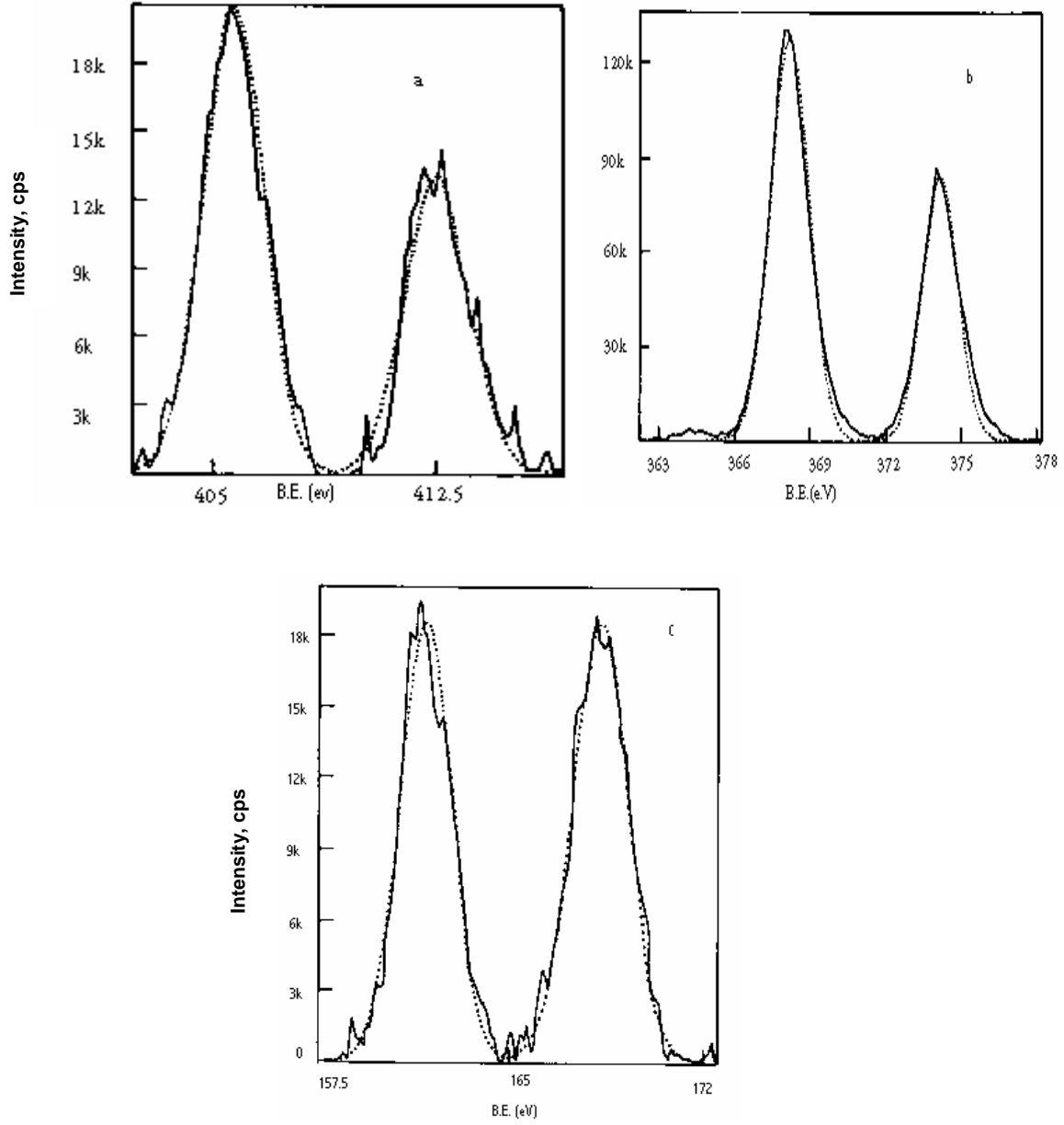
Fig. 3 : XPS Spectra of Cd 3d core level (a) CdS nanoparticles (b) CdS - Ag<sub>2</sub>S (post core method) core-shell nanoparticles

Fig. 4 : XPS Spectra of CdS - Ag<sub>2</sub>S nanoparticles Partial microemulsion method (a) Cd 3d (b) Ag3d and (c) S2p core levels

Fig. 5 : XPS Spectra of CdS and Ag<sub>2</sub>S nanoparticles (Physical mixture 50 :50) (a) Cd 3d (b) Ag3d and (c) S2p core levels

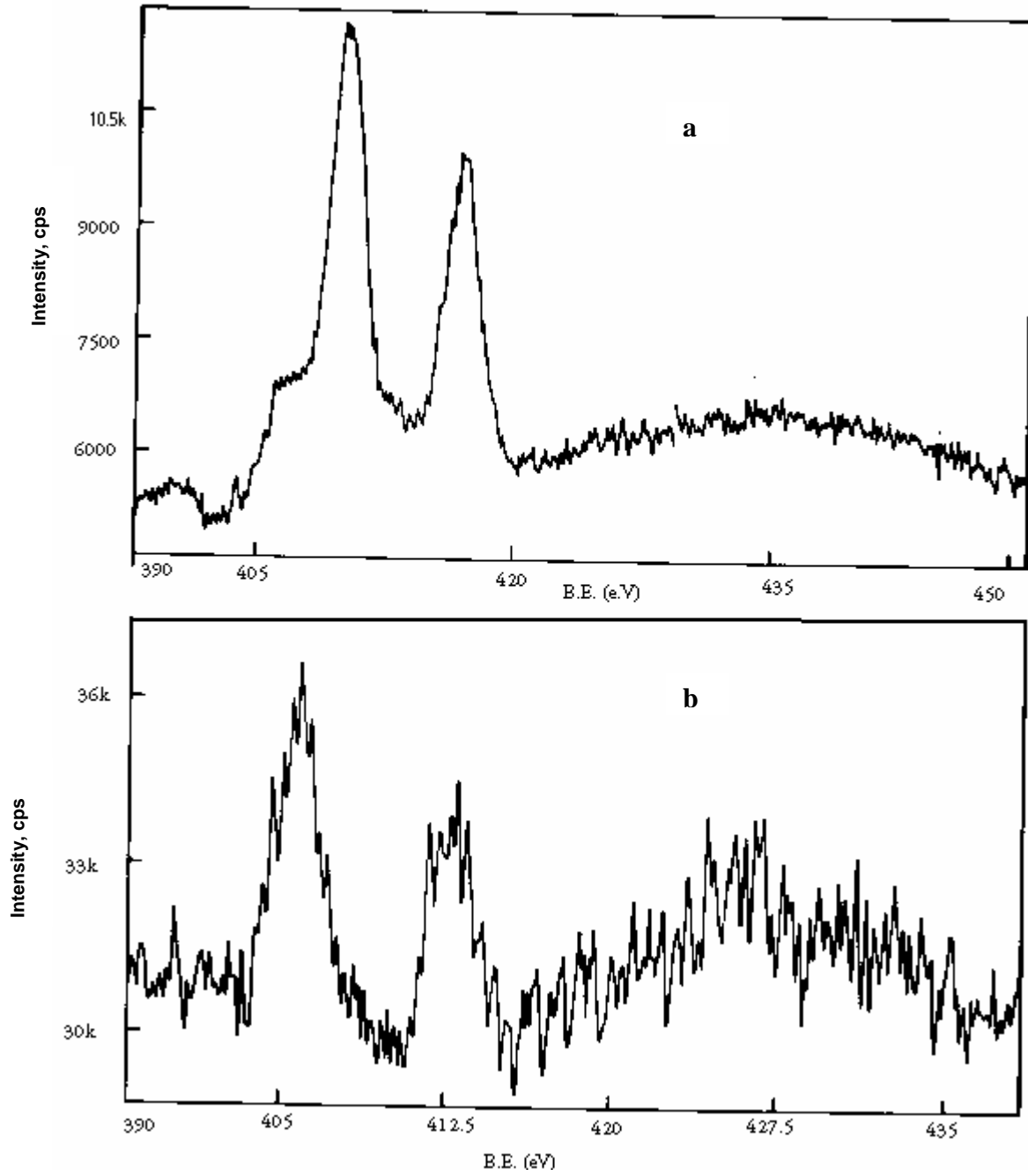


**Fig. 1 : XPS Spectra of CdS and Ag<sub>2</sub>S nanoparticles (a) Cd 3d and (b) S2p (c) Ag3d and (d) S2p core level**

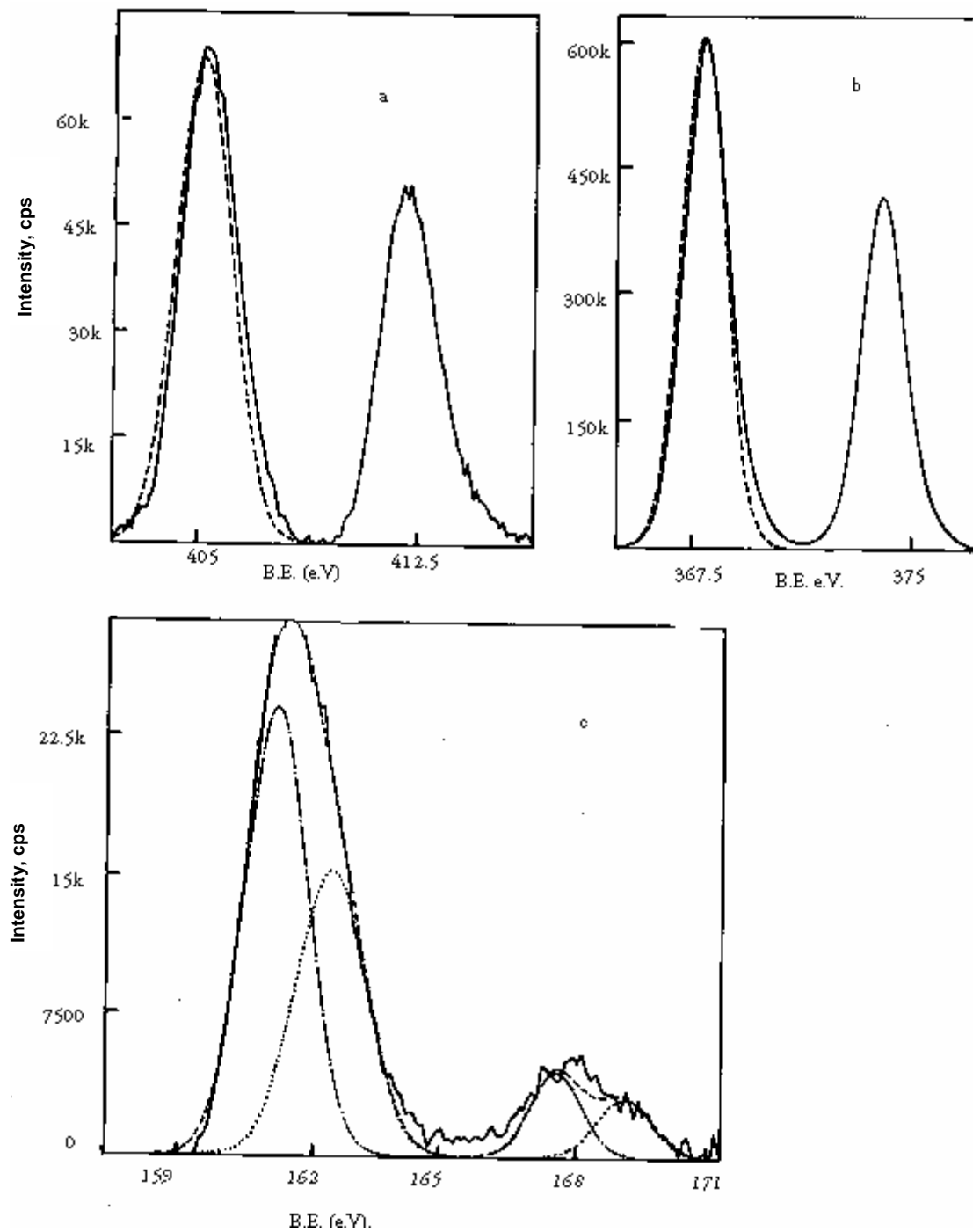


**Fig.2 : XPS Spectra of CdS - Ag<sub>2</sub>S core-shell nanoparticles (post core method) (a) Cd 3d and (b) Ag3d and (c) S2p core levels**

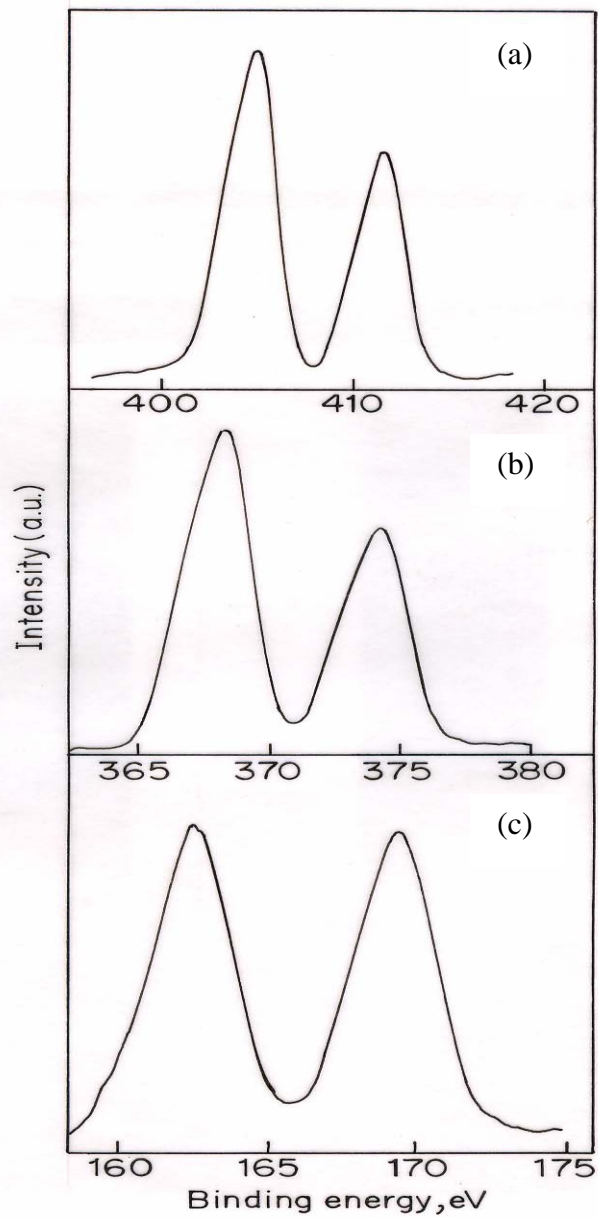




**Fig. 3 : XPS Spectra of Cd 3d core level (a) CdS nanoparticles (b) CdS - Ag<sub>2</sub>S (post core method) nanoparticles.**



**Fig. 4 : XPS Spectra of CdS - Ag<sub>2</sub>S core-shell nanoparticles prepared by Partial microemulsion method (a) Cd 3d (b) Ag3d and (c) S2p core levels**



**Fig. 5 : XPS Spectra of CdS and Ag<sub>2</sub>S nanoparticles (Physical mixture 50:50)**  
(a) Cd 3d (b) Ag3d and (c) S2p core levels