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Structural and Optical Characterization of Cds Nanoparticles by Chemical Precipitation Method

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ABSTRACT

Synthesis of CdS nanoparticles by a conventional simple chemical co-precipitation technique is reported here. Cadmium acetate and sodium sulphide is used as the starting materials and N-N dimethyl formamide (DMF) as a stabilizing agent. The prepared sample have been analyzed by XRD, TEM, EDAX, and UV-Vis. XRD study reveals that the CdS particles have cubic structure and crystalline nature. The particle size calculated from the most intense broad peaks in the diffraction pattern, found to be about 5.11 nm. The optical band gap energy of the sample calculated from UV-Vis absorption spectra is found to be about 3.8 eV. TEM micrograph also reveals the nano sized particles of CdS. The elemental analysis by EDAX confirms that the prepared sample contains the appropriate cadmium and sulphur weight percentages in the reaction compound.

Keywords: CdS, nanoparticles, XRD, TEM, Optical band gap, and EDAX.

I. INTRODUCTION

Recently, many researchers have been attracted towards semiconductor nanoparticles due to their interesting optical, electrical and catalytic properties. Significant growth is found in research on semiconductor nanoparticles during past 20 years, due to their interesting and exciting novel properties [1, 2]. Among various nanoparticles, sulphides and selenides studied enormously due to their interesting opto-electronic properties. Cadmium sulphide (CdS) is II-VI group n-type semiconducting material with a direct band gap of 2.42 eV at room temperature (RT) has the promising applications for solar cell, LEDs, photoconductors, gas sensor, detectors for laser and infrared. Cadmium sulphide has the excellent light detecting properties compared to other semiconductors [3, 4]. Various techniques such as sonochemical method, chemical vapour deposition, sol-gel technique, electro deposition, electrochemical method, spray pyrolysis technique, thermal decomposition of precursors and other chemicals techniques and so on have been reported earlier for the synthesis of nanocrystalline CdS [5-7]. In the present study we have synthesized CdS nanoparticles using chemical precipitation technique which is simple and economic compared to those reported earlier. The synthesized samples were characterized by XRD, TEM, EDAX and UV-Visible Spectrometer.

II. MATERIALS AND METHODS

A. Experimental:

CdS sample were prepared using cadmium acetate [$(CH_3COO)_2$ Cd, $2H_2O$] (extra pure Loba Chemie) and sodium sulphide [Na_2S, H_2O] (extra pure Loba Chemie) as the starting materials. DMF $(CH_3)_2NC(O)H$ (Merck) is used as stabilizing agent. All the chemicals used in this investigation were of analytical reagent (AR) grade and used as received without further purification. The doubled distilled water was used throughout the experiment as solvent for all the solutions referred in the present synthesis.

0.1 M Cadmium acetate [Cd^{2+}] solution is prepared by dissolving cadmium acetate in 100 ml double distilled water and 0.1 M Sodium sulphide [S^{2-}] is also prepared in 100 ml double distilled water. Cadmium acetate solution was mixed with specific amount of DMF and

stir for 10 minutes. Then 100 ml sodium sulphide solution is added in the mixture drop by drop with constant stirring for 2 hours with electromagnetic stirrer which results in cloudy yellow solution. This solution was kept overnight for settlement and then it was centrifuged several times with deionised water and acetone to eliminate the unreacted molecules. The obtained materials were filtered and dried in vacuum oven at 60^0 for 8 hours. The prepared sample then crushed in to fine powder and collected in a sample bottle for characterization.

B. Characterization of CdS nanoparticles

The structural analysis of CdS nanoparticles was carried out by using X-ray powder diffractometer (Model: D-8 Advance) with CuKα radiation (λ = 0.15406 nm) scanning 2θ in the range 10^0- 90^0. The X-rays were detected using a fast counting detector based on Silicon strip technology (Bruker Lynx Eye detector). The morphology of the nanoparticles was characterized by transmission electron microscopy (TEM) using model Tecnai 20 G^2 (FEI) makes under 200 KV. EDAX spectra were recorded using Model JEOL JSM 5600. All these three characteristics were carried at UGC-DAE CSR, Indore (M. P.) India. A UV-Vis absorption spectrum was recorded using Jasco spectrometer, (Model name: V-770, Serial No. A013161801) for the wavelength range 200-1100 nm at the department of physics D. K. A. S. C. College, Ichalkaranji (M. S.).

III. RESULTS AND DISCUSSION

A. X-ray Diffraction

The XRD pattern of synthesized CdS (figure 1) has three strong peaks at the angles 2θ = 26.68^0, 43.57^0 and 52.06^0 which could be assigned with (111), (220) and (311) planes respectively of cubic CdS crystal lattice which is in good agreement with **JCPDS card file no. (80 - 0019).**

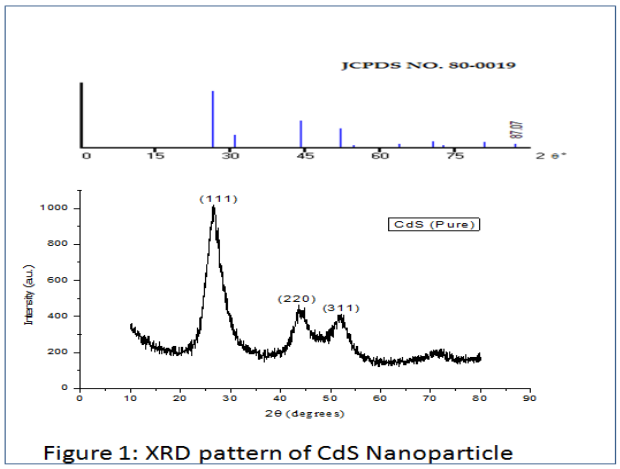

Figure 1: XRD pattern of CdS Nanoparticle

The broadness of the diffraction peaks as obtained in the XRD spectra gives the direct consequence of the reduced particle size in nano range and the sharp peaks indicates the crystalline nature of the material. The most intense peak is considered to calculate the particle size by using Debye-Scherer's formula [8]:

$$D = \frac{0.9\,\lambda}{\beta \cos\theta}$$

Where λ = 0.1541 nm is the wavelength of X-ray diffraction, β is the FWHM in radian of the most intense XRD peak and θ is the angle of diffraction. The particle size for CdS nanoparticles as calculated, found to be 5.11 nm.

The lattice parameter **'a'** for CdS nanoparticles is calculated using equation

$$\mathbf{a} = \left[\frac{\lambda}{2\sin\theta} \times \sqrt{h^2 + k^2 + l^2}\right] \text{ Å}$$

and is found to be **5.8077 Å.**

The d-spacing for cubic system for $2\theta_{(111)}$ is calculated by using equation

$$d = \frac{a}{\sqrt{h^2 + k^2 + l^2}}$$ Å and is found to be 3.3530 **Å.**

B. Transmission Electron Microscopy (TEM)

TEM is used to study the surface morphology of the materials. TEM micrograph with SAED pattern is as shown in figure 2(a). The TEM image shows that the surface morphologies are in the form of assemblies of nanoparticles uniformly distributed over the entire surface. TEM image shows the crystalline structure in scale of nano range. As seen in figure 2(a) the particles are very narrow and spherical shaped.

111 numbers of particles have been selected for calculating the particle size distribution and histogram

drawn is as shown in figure 2(b). The average particle size calculated from histogram is found to be about 4.2 nm.

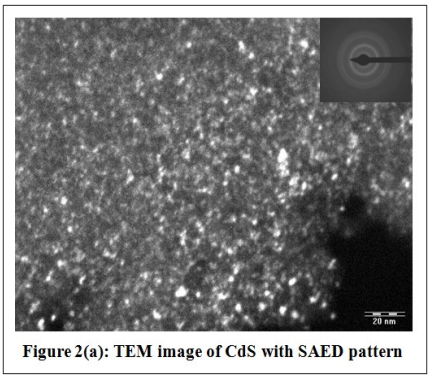

Figure 2(a): TEM image of CdS with SAED pattern

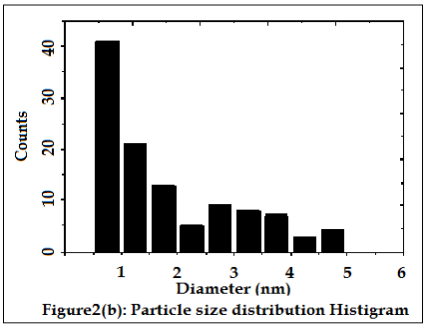

Figure2(b): Particle size distribution Histigram

C. EDAX Analysis:

Figure 3 shows the EDAX plot of CdS nanoparticles which shows the clear peaks of Cadmium (Cd) and Sulphur (S), the elemental analysis by EDAX confirms that the prepared sample contains the appropriate Cadmium and Sulphur weight percentages in the reaction compound.

EDAX data shown in table1 gives the compositions of the prepared sample in weight percentage.

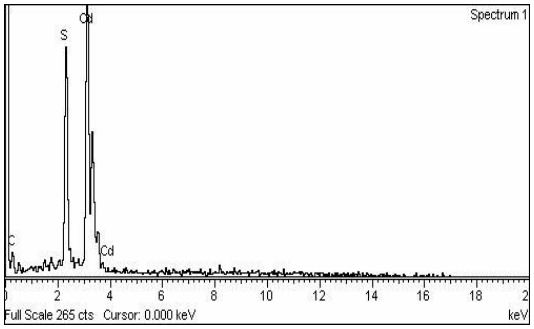

Figure 3. EDAX plot of CdS nanoparticles

Table 1. EDAX data of CdS

Compound name	Element	Weight percentage %
CdS	S K Cd L	45.90 54.10 Total = 100.00

UV-Visible Absorption Spectra

The most dramatic property of semiconductor nanoparticles is the size evolution of the optical absorption spectra. Hence UV-Visible absorption spectroscopy has been used to study the optical properties of nano sized particles. Figure 4(a) shows the optical absorption spectra of CdS nanoparticles. The material of the present study is of direct band gap nature. The bulk band gap of CdS is 2.42 eV as reported by earlier researchers. In the present investigation optical band gap is calculated using Tauc relation [9-11] given by

$$(\alpha h\nu)^{1/n} = A\,(h\nu - E_g)$$

Where α is Absorption Coefficient, A is constant and E_g is band gap of material and the exponent n depends on the type of transition. For direct and indirect allowed transitions, n = ½ and n = 2 respectively. For direct and indirect forbidden transition n = 3/2 and n = 3 respectively. $(\alpha h\nu)^2$ Vs hν plot is used to determine the possible transitions. The corresponding band gap is obtained by extrapolating the linear portion of the graph on hν axis as shown in figure 4(b) and is found to be 3.8 eV. This value is increased compared with the bulk value and this could be the because of a size quantization. The grain size of CdS nanoparticles can be calculated using following Brus equation

$$E_{g\,(nano)} = E_{g\,(bulk)} + \frac{h^2\,\pi^2}{8R^2}\left(\frac{1}{m_e^*} + \frac{1}{m_h^*}\right) - \frac{1.8\,e^2}{4\pi\epsilon_0\,\varepsilon_r}$$

Where $E_{g\,(nano)}$ = 3.9 eV, $E_{g\,(bulk)}$ = 2.42 eV, m_e^* = 0.21 m_e is the effective mass of electron, m_h^* = 0.8 m_e is the effective mass of hole, m_e is the free electron mass and R is the particle radius, ε_r is the dielectric constant and ϵ_0 is the permittivity of free space.

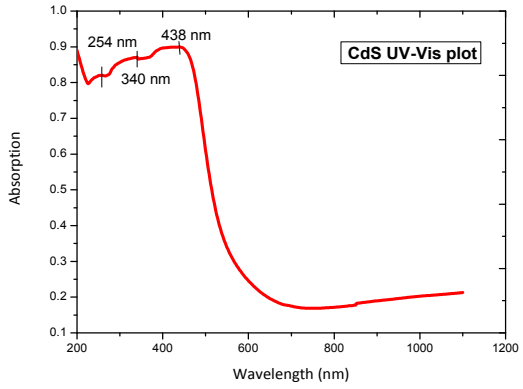

Figure 4(a). UV-Vis Absorption Spectra

The first term in above equation indicates the confinement effect and the second term is the Coulomb term. The second term can be neglected as it is small due to strong confinement [12]. Thus the the particle size estimated is 3.24 nm by using above equation. All three methods used to determine the particle size confirms the size of prepared CdS particles in the range of 4-5 nm.

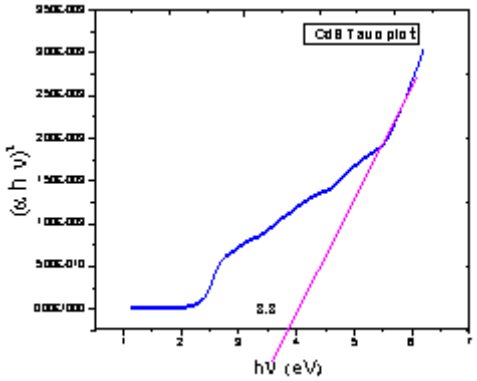

Figure 4(b). Tauc plot of CdS

IV. CONCLUSIONS

Precipitation method used here for the synthesis of CdS nanoparticles is successful, economic and eco-friendly. The crystal structure and the grain size of the particles are determined using XRD which is also confirmed by TEM micrograph. Broad peaks in XRD pattern and blue shift in absorption maxima clearly indicates the formation of nanoparticles. TEM micrograph reveals as uniformly distributed fine particles, which form crystalline aggregates. Compositional analysis by EDAX confirms that the prepared sample contains the appropriate cadmium and sulphur weight percentages. UV-Vis absorption spectrum shows a blue shift indicating quantum confinement of charged particles. Energy band gap increased due to nanoparticle size which is capable in emitting light of shorter wavelength in the blue range. Hence, prepared CdS nanoparticles are found to be novel nanoparticles which can be used in optical devices [13].

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