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Synthesis and Characterization of CdS Nanostructures

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Abstract

We have successfully synthesized CdS nanoparticles by Cadmium sulphate, Thiourea and Ammonia solution as the complexing agent by chemical route method. The formation of CdS was confirmed with the help of infrared (IR) spectroscopy by observing bands corresponding to the multi phonon absorption. The diffraction pattern of the material was obtained using X-ray diffractometer, which revealed the crystallinity of CdS. The average grain size was determined as 42.49 nm using Debye-Scherrer's formula. The absorption spectrum of the material was obtained using UV–Visible spectrophotometer and shows a high absorption in the visible region. The direct optical band gap is obtained as 2.0 eV from the Tauc's extrapolation. The structural morphology was also investigated using Scanning Electron Microscope (SEM) and the images shows agglomeration of grains in form of islands. Cadmium sulfide is one of the most promising materials for solar cells and of great interest for their practical applications in electronics and photonics.

Keywords: Cadmium sulfide, X – Ray Diffraction, SEM, Band gap, Crystalline, Grain size.

1. Introduction

Cadmium sulfide (CdS) is an important II–VI semiconductor (Eg = 2.42 eV at room temperature) (Wang et al., 2005) with many excellent physical and chemical properties (Nie et al., 2004), which has promising applications in multiple technical fields including photochemical catalysis, gas sensor, detectors for laser and infrared, solar cells, nonlinear optical materials, various luminescence devices, optoelectronic devices and so on (Ma et al., 2005). Modifications of the optical, electrical, magnetic and physical properties of semiconductor materials strictly depend on the sizes, structures and morphologies of the material (Wang et al., 2006). Due to these changes in properties with the crystal size, researchers interest turn towards the synthesis of few nanometer particles with dimensions comparable to the Bohr radius (Romano et al., 2006), such particles could be used in a lot of

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applications as quantum dot lasers, single electron transistors and also have biological applications (Morales-Acevedo, 2006). It is important to synthesize nanoparticle at the desired size within a narrow size distribution and in an easy to handle conditions of precursor, solvent and temperature (Tai et al., 2010). In the last few years, researchers have been devoted to the preparation of high-quality CdS nanoparticles and the investigation of their various properties (Hu et al., 1998).

In this study, Cadmium Sulphide nanoscaled crystalline particles have been synthesized via a simple chemical route to achieve a large surface to volume ratio, which has enormous advantages in device fabrication. The chemical constituents, crystal phase, the grain size, morphology and optical properties of the synthesized material have been studied using Fourier transform Infrared (FTIR), X-ray diffractometer (XRD), Scanning Electron Microscope (SEM) and UV-Visible spectrophotometer. The properties reveal the importance of the synthesized material and application for which it can be used.

2. Materials and Methods

CdS was synthesized using wet chemical method. 0.05M of Cadmium Sulphate was mixed with 0.10M Thiourea under vigorous stirring by the magnetic stirrer. Ammonia solution was also introduced to adjust the pH of the reaction mixture as a complexing agent. The material was characterized by Scanning Electron Microscope (SEM), X-ray Diffractometer (XRD), Fourier Transform Infra-Red (FTIR) and UV-Visible Spectrophotometer (PVE UNICAM SPE-400UV/VIS) in order to confirm the successful synthesis of nanostructures CdS, the morphology, absorption spectrum and the constituents of the synthesized material.

3. Results and Discussion

Optical characterization

The UV-visible absorption spectra of CdS nanostructured particles were recorded using a spectrophotometer (Model: PVE UNICAM SPE-400UV/VIS) in the range 400 – 750 nm (figure 1). It is observed that there is high absorption of visible light within wavelength 400 and 600 nm. The spectra were analyzed by plotting $(\alpha h\nu)^2$ vs $h\nu$ with respect to the relation

$$\alpha = \frac{A(h\nu - E_g)^n}{h\nu}, \quad \dots\dots\dots\dots\dots\dots\dots\dots..(1)$$

where α is absorption coefficient, A is a constant and n the exponent that depends upon the quantum selection rule (n may have values ½, 2, 3/2 and 3 corresponding to the allowed direct, allowed indirect, forbidden direct and forbidden indirect transition respectively). A straight line is obtained (figure 2) by extrapolating a straight portion of the graph. The intercept of the straight line on hν axis corresponds to the optical band gap (E_g) which is obtained to be 2.0 eV.

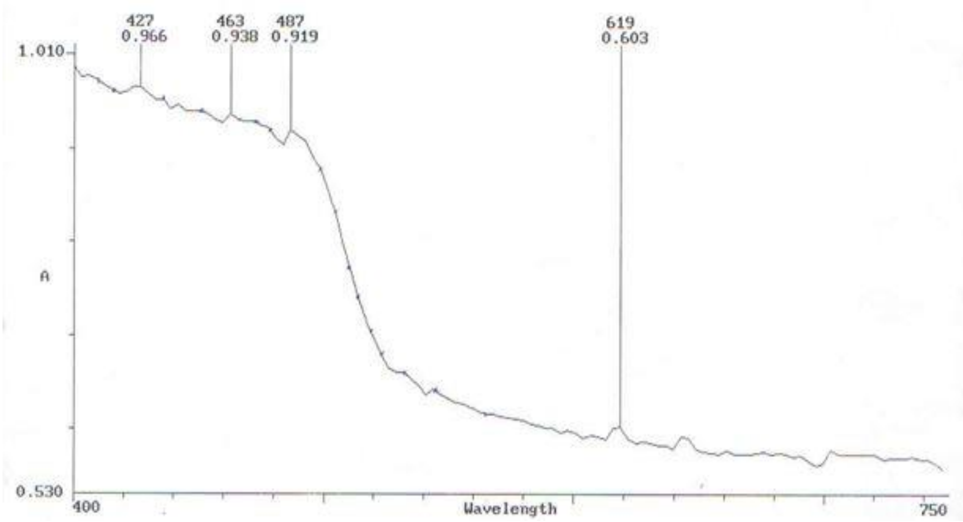

Figure 1: Absorbance Spectral of Cadmium Sulphide (CdS) Nanoparticles

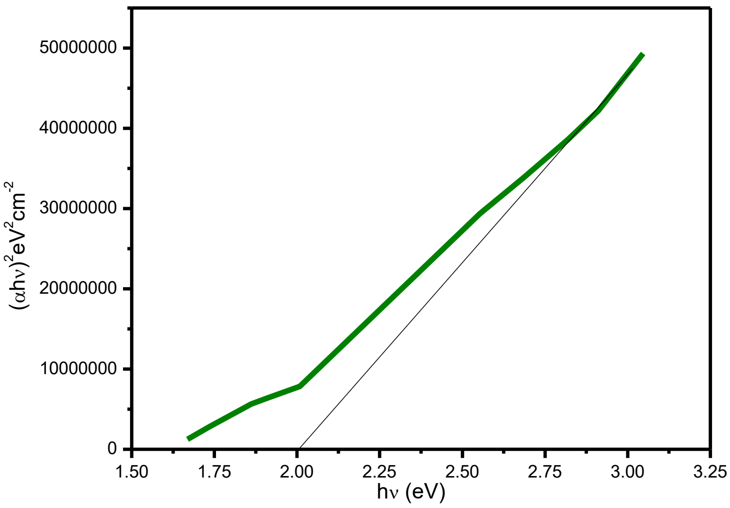

Figure 2: Determination of direct optical band gap of CdS nanoparticles.

Structural characterization

The nanostructured particles were subjected to phase analysis by employing X-ray diffractometer (Model: Thermo ARL) that was equipped with CuKα – 1 as radiation source (λ = 1.5406 Å). Figure 3 shows the X-ray diffractometer pattern of CdS nanoparticles obtained at annealing temperature 425^{o}C and reveal formation of CdS with orthorhombic crystal structure. The peaks at 2ϴ angle 25.11, 26.94, 27.86 and 28.63^0 corresponding to diffraction from planes (040), (313), (420) and (412) which are very close to the expected values from JCPDS 41 – 1049 for CdS. Utilizing the X-ray diffraction data and Scherrer's equation (2) (Kulkarni et al., 2001), the average grain size of cadmium sulphide nanoparticle was computed.

$$g = \frac{K\lambda}{\beta cos\theta} \quad \dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots (2)$$

Where k = 0.94 is the shape factor, λ = wavelength of X-ray used 1.5406 Å,

β = Full Width at half Maximum (FWHM) in radians, θ = Bragg angle of the diffraction peak in radians and $\boldsymbol{g}$ = average grain size.

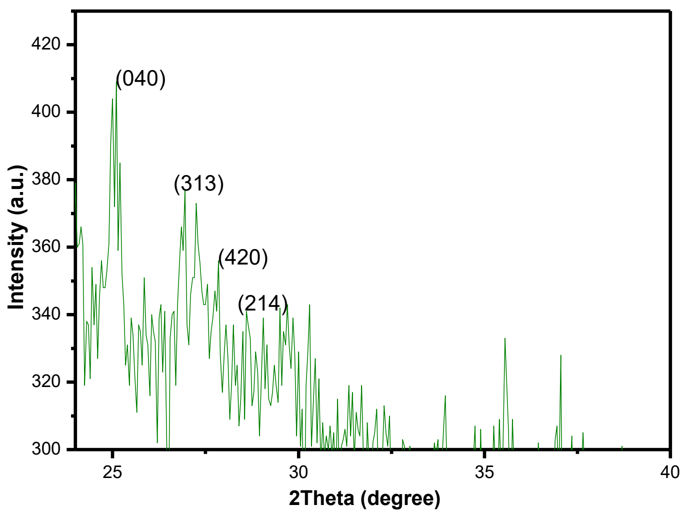

Figure 3: XRD spectrum of CdS thin films annealed at 425 ºC

The average grain size of CdS was calculated to be 42.49 nm, which confirmed that the CdS synthesized is in nanoscale. The 2ϴ peaks for CdS, even though with background noise, shows crystallinity of the nanostructured particles, achieved by annealing process.

Morphological study of the material

Scanning Electron Microscope (SEM) (Philips Model: 515SEM) was used to examine the surface morphology of cadmium sulphide nanostructure particles and the result are shown in figure 4. SEM analysis reveals formation of particles with grain agglomerates. This might be induced during the crystal growth itself. The particles adopt irregular morphology with different sizes.

The scanning electron microscope (SEM) is a type of electron microscope that reveals the sample surface by scanning it with a high-energy beam of electrons in a faster scan pattern (Jamali et al., 2007). The electrons interact with the atoms sample produce signals that contain information about the sample's surface topography, composition and other properties such as electrical conductivity (Barglik-Chory et al., 2003).

The types of signals produced by an SEM include secondary electrons, Back Scattered Electrons (BSE), characteristic x-rays, light (cathodoluminescence), specimen current and transmitted electrons. These types of signal all require specialized detectors that are not usually all present on a single machine (Marandi et al., 2006).

The SEM micrograph reveals the particle surface and looks highly porous and interconnected network of honeycomb. The honeycomb network of CdS nanostructured films may provide novel platforms for photovoltaic, sensor and other device applications.

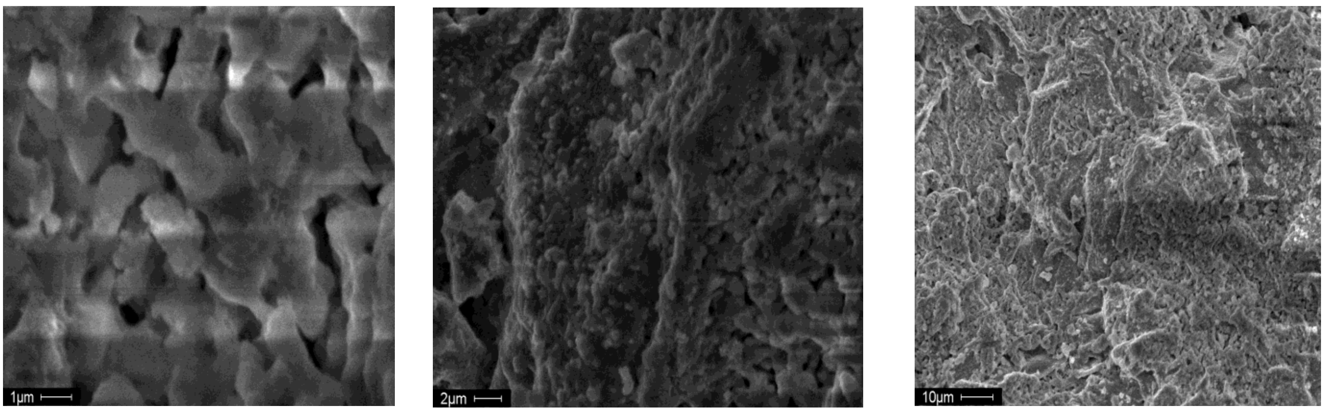

Figure 4: SEM images of CdS_np at different magnifications: Scale bars are 1, 2 and 10 μm.

Fourier Transform Infrared (FTIR) of CdS films

FTIR is technique used to obtain information about the chemical bonding in a material. In this work it is used to identify the elemental constituents of the CdS thin film. FTIR analysis was performed using (SHIMADZU Model: 8400S FTIR). Double transmission mode was used because it offers highest sensitivity. The beam makes a double pass through the sample before reaching the detector. Figure 5 shows the FTIR spectra of CdS particles. The broad absorption band in the region 3000 to 3600 cm^{-1} and the small band around 1600 to 1650 cm^{-1} are due to the OH stretching vibration of water molecules. The peaks around 2000 and 2200 cm^{-1} are attributed to the CN stretching band of cadmium cyanamide or thiocyanate, which result from the chemical reaction of thiourea and ammonia.

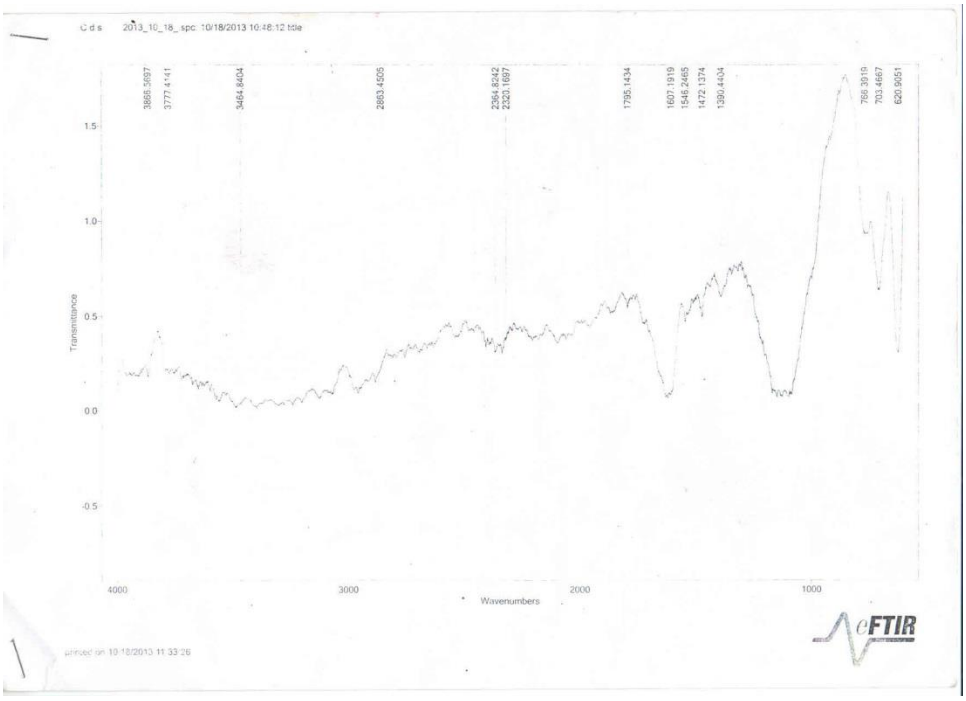

Figure 5: FTIR spectra of CdS Nanoparticles

4. Conclusion

CdS nanoparticles were synthesized by simple one step wet chemical synthesis. The synthesized CdS was characterized by X-ray diffraction (XRD), UV-Visible spectrophotometer, Infra-red(IR) and Scanning electron microscopy (SEM). From XRD analysis, it is understood that the nanoparticles were small in nature with an average grain size of 42.49 nm. SEM analysis showed the particles were highly agglomerated. The SEM micrograph reveals the nanoparticles surface looks highly porous and interconnected network of honeycomb. The honeycomb network of CdS nanostructured films may provide novel platforms for photovoltaic, sensor and other device applications. The optical band gap was calculated to be 2.0 eV. FTIR was used to obtain information about the chemical bonding in a material and in this work it is used to identify the elemental constituents of the synthesized material CdS.

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