

## Research Article

## Synthesis and Characterization of CdSe Nanoparticle in Aqueous Phase

Priyadharsini N<sup>1</sup>, Sangeetha S<sup>2</sup>, Mangalam R<sup>3</sup>, Vairam S<sup>2</sup>, Thamilselvan M<sup>4</sup><sup>1</sup>Dept. of Physics, GCT, Cbe-13,<sup>2</sup>Dept. of Chemistry, GCT, Cbe-13,<sup>3</sup>Dept. of Physics, SriGuru institute of technology, Cbe.<sup>4</sup>Dept of Physics, TPGIT, Vellore-02.**Abstract**

By employing selenium dioxide as source, in the present work it has been demonstrated that CdSe nanoparticles can also be prepared conveniently from aqueous solution through wet chemical method. The obtained products were characterized by UV-visible spectroscopy, Powder X-ray diffraction (PXRD), FTIR and Scanning electron microscopy (SEM). This route is very facile, inexpensive and less hazardous and ensures

almost complete yield of the precursors. XRD analysis shows that the CdSe NPs possess cubic zinc-blend structure with diameters in the range of 30-35 nm. UV-Visible absorption spectra and SEM exhibits that the NPs were monodispersed and are relatively uniform.

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**Keywords** XRD, Cubic zinc blend, SEM.**Introduction**

Nanostructured materials have attracted a great range of attention in the last few years for their unique characteristics that cannot be obtained from conventional macroscopic materials. Materials belonging to inorganic, organic and their composite categories can be obtained in the nanometer size range. Small dimensions of materials show novel chemical, physical and biological properties. New behavior at nanoscale arises due to order of magnitude reduction in size. An extremely active and prolific field in nanomaterials is finding new ways to control size and morphology of the nanoparticles, since the properties and applications of the nanoparticles are largely dependent on them.

The most evident manifestation of properties is the optical light emission in the blue-red spectral region characterized by a blue shift at smaller crystallite dimensions [1]. Such properties are used to construct semiconducting nanostructures suitable for several kinds of applications, from anti reflecting coatings [2] to biomolecular detection and light emitting devices [3]. In the past decade, II-VI semiconductor nanoparticles attracted much attention because of their size-dependent (and thus tunable) photo- and electro-luminescence properties and promising applications in optoelectronics. Among the family of II-VI semiconductors, ZnS [4], CdS, ZnO [5], CdSe [6], are the foremost candidates because of their favorable electronic and optical properties. In particular CdSe NP's is an important II-VI semiconductor having a wide optical band gap, making it a very attractive material for optical applications, especially in nanocrystalline form. CdSe can have two different crystal structures cubic zinc blende and hexagonal wurtzite.

To date, two elementary routes have been reported for the synthesis of CdSe NPs. One is the most conventional organic approach and other one is aqueous synthesis. There are several methods involving many variables for the synthesis of CdSe QDs and are based on highly toxic Cd and Se ingredients. There are reports on synthesis of CdSe nanoparticles involving costly and hazardous techniques with or without surfactant/stabilizing reagents [7]. Recently a general room temperature method has been developed with facile aqueous solution-phase strategy to grow nanostructured metal chalcogenides which is of low cost and less hazardous [8].

We here report a straight-forward, simple wet chemical technique for the synthesis of CdSe nanoparticles. Less hazardous aqueous solution process for the preparation of cubic zinc-blend CdSe nanoparticles in hydrazine hydrate using cadmium nitrate as a precursor is described. The present study tries to satisfy all of these requirements to the large-scale production of CdSe nanoparticles.

## Experimental procedure

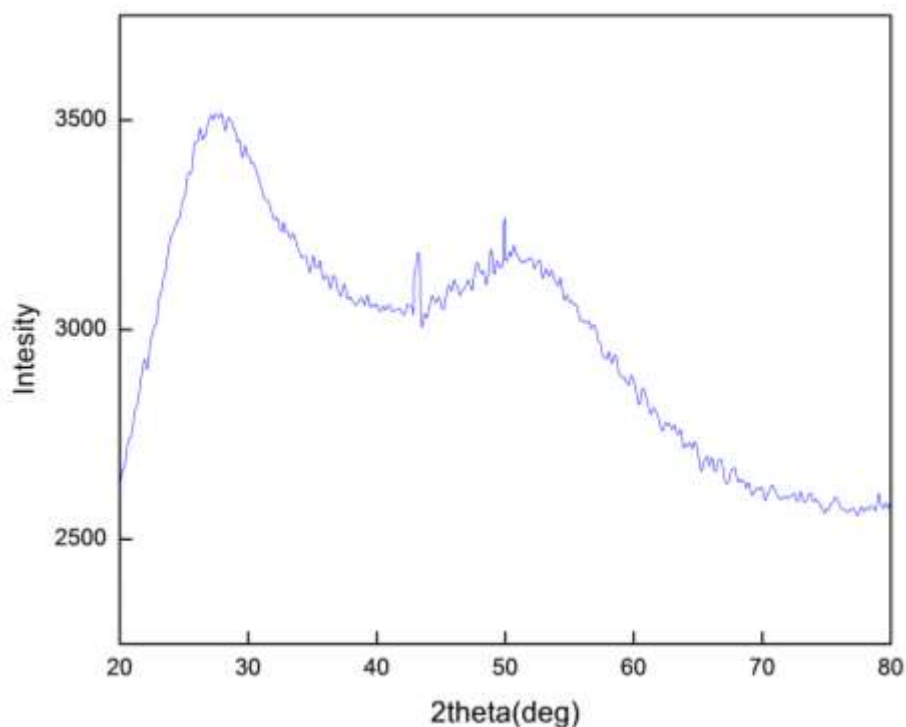
### *Synthesis of CdSe nanoparticles*

For the typical synthesis of CdSe, highly pure CdNO<sub>3</sub> powder (99.9%) , Selenium dioxide (99.999%) and Hydrazine hydrate purchased and used without further purification. In this synthesis process, CdNO<sub>3</sub> (2 mmol) and selenium dioxide (2.0 mmol) was dissolved deionized water, and hydrazine hydrate in the volume ratio of 1 1 2 respectively in a 200 ml capacity glass beaker. Then, the solution was refluxed under vigorous stirring for 6 hrs. The cherry red precipitate was collected and washed with dry ethanol and hot distilled water several times, then dried in room temperature for 12 h.

### *Characterizations of CdSe nanoparticles*

The X-ray diffraction pattern of as synthesized, freshly dried CdSe powder was recorded by XPERT-PRO diffractometer equipped with Cu K $\alpha$  radiation (operating at 45 kV, 30 mA) with wavelength 1.54Å. Scanning electron microscopy (SEM) was applied to investigate the size and morphology, which was carried out under JEM-3010 with an operating voltage 200 kV. UV-Visible spectra was recorded by UV-Vis1033 Spectrophotometer in the spectral range between 200 and 800 nm using a spectral bandwidth of 1nm absorption, experiments were performed at room temperature. Fourier transform infrared spectroscopy (FTIR) analysis has done using Jasco-5300 spectrometer at room temperature with the sample milled in KBr.

## Results and Discussion



**Figure 1** XRD spectrum of CdSe Nanoparticle

## Structural study

X-ray diffraction pattern of the as prepared CdSe sample is shown in **Figure 1**. The XRD measurements of CdSe nanoparticles shows that the position of diffracted peaks match well with the standard powder diffraction data ( $a = 4.299 \text{ \AA}$ ). From the XRD pattern, three typical distinct features of the zinc-blended structure can be distinguished the one at  $2\theta = 27.3^\circ$  is due to the (111) reflection and the other two appearing at  $2\theta = 43.2^\circ$  and  $49.8^\circ$  result from the (220) and (311) reflections (JCPDS Standards file No. 19-0191), respectively. Moreover, no typical diffraction of the wurtzite lattice structure can be found, which further confirms the cubic zinc-blended structure. In comparison, the zinc-blende structure is the more stable form at lower temperature, whereas the wurtzite structure is preferred to Figure 1 form at higher temperature (above  $2008^\circ\text{C}$ ). The main reason for the formation of the zinc-blende structure is that the reaction occurred at a comparative low temperature ( $1008^\circ\text{C}$ ).

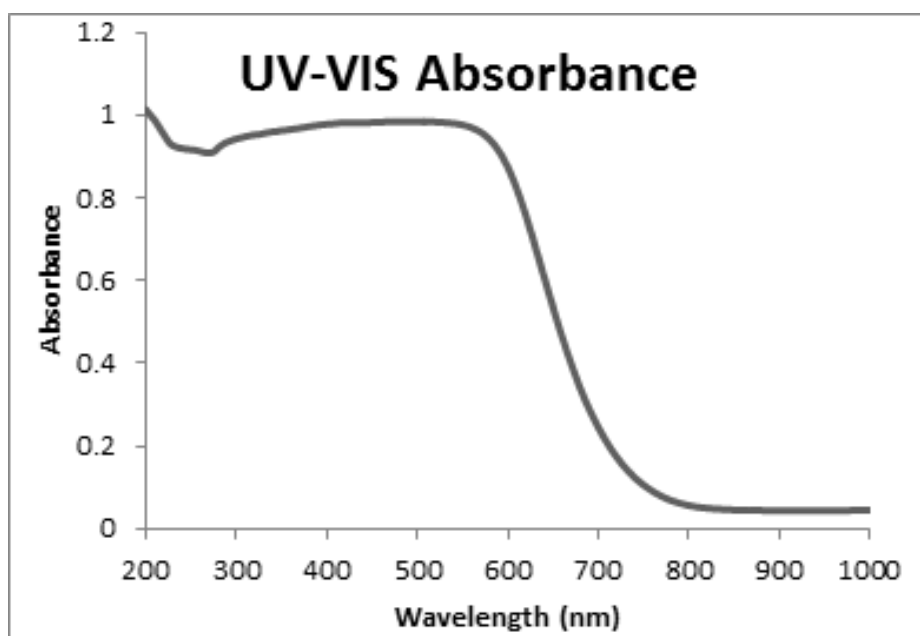
The size of the nanocrystallites was estimated using the Debye-Scherrer formula

$$D = \frac{0.94 \lambda}{\beta \cos \theta} \quad (1)$$

Where,  $\beta$  is the full-width-at-half maximum (FWHM) in radians of the diffraction peak,  $\lambda$  ( $1.5418 \text{ \AA}$ ) is the wavelength of X-ray radiation, and  $\theta$  is the angle of diffraction. Inherent stress inside a nanoparticle may be the cause behind to broadening of the XRD peaks. From the Debye-Scherrer formula the crystallite size determined from the major peak centered at  $27.3 \text{ deg}$  was estimated to be about  $36 \text{ nm}$ .

## UV-VIS spectroscopy Analysis

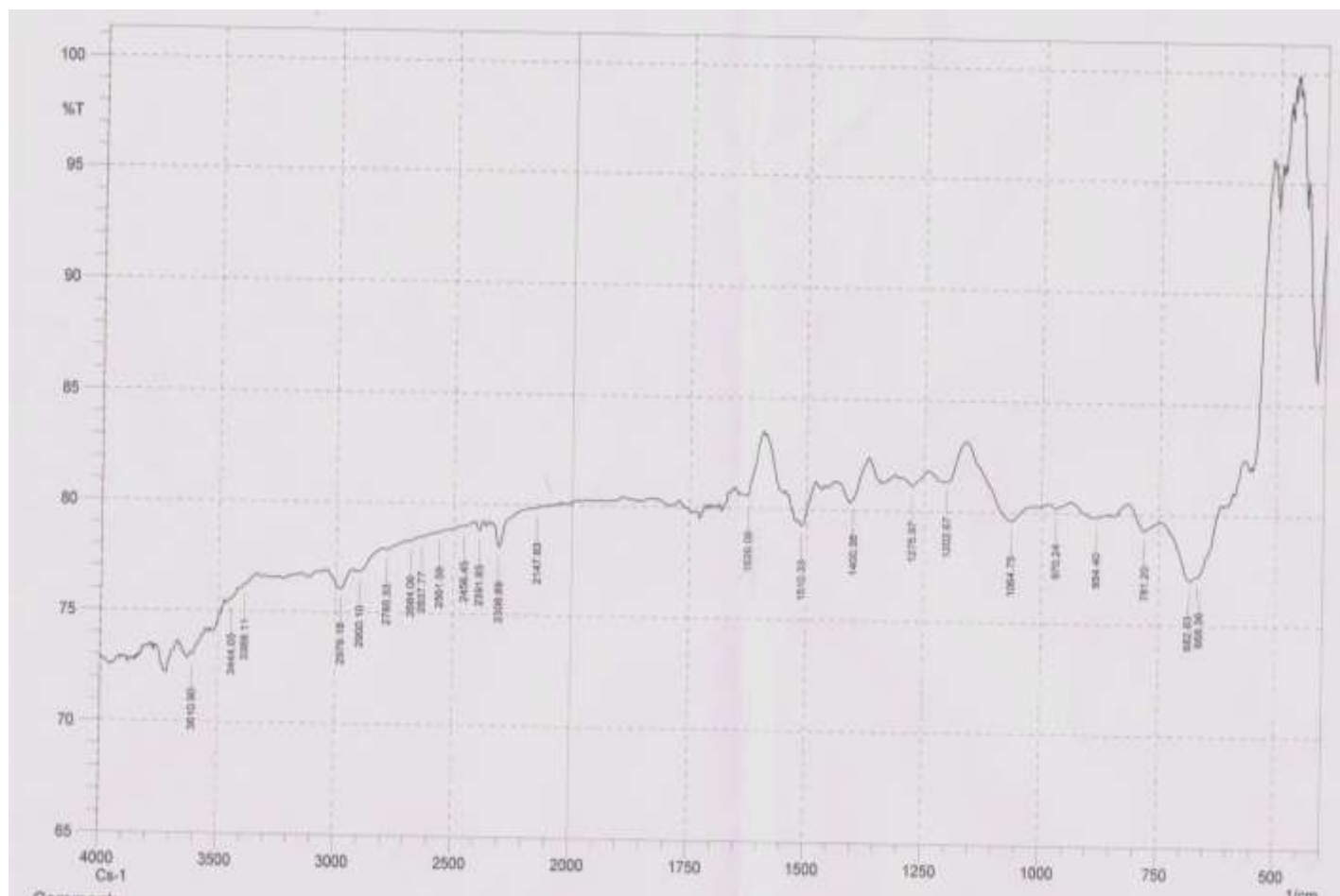
The recorded absorbance spectrum (range  $200 \text{ nm}$  to  $800 \text{ nm}$ ) of CdSe NPs is shown in Figure 2. The band gap calculated from the absorption edge is  $2.23 \text{ eV}$ . This has been blue shifted from the bulk band gap of  $1.732 \text{ eV}$  due to quantum confinement effect in the nanostructure. UV-Vis absorption spectra show excitonic peaks at  $556 \text{ nm}$ . For bulk CdSe, absorption peak is expected at  $\sim 716 \text{ nm}$ . Thus there is a strong blueshift in the absorption spectra and this indicates that particles must be smaller than bulk. In CdSe nanoparticles, the highest occupied molecular orbital (HOMO) originates from Se  $4p$  atomic orbital, and the lowest unoccupied molecular orbital (LUMO) from Cd  $5s$  atomic orbital. These electronic states are strongly dependent on the size (the degree of confinement).



**Figure 2** UV-Vis spectrum of CdSe Nanoparticle

## FTIR Analysis

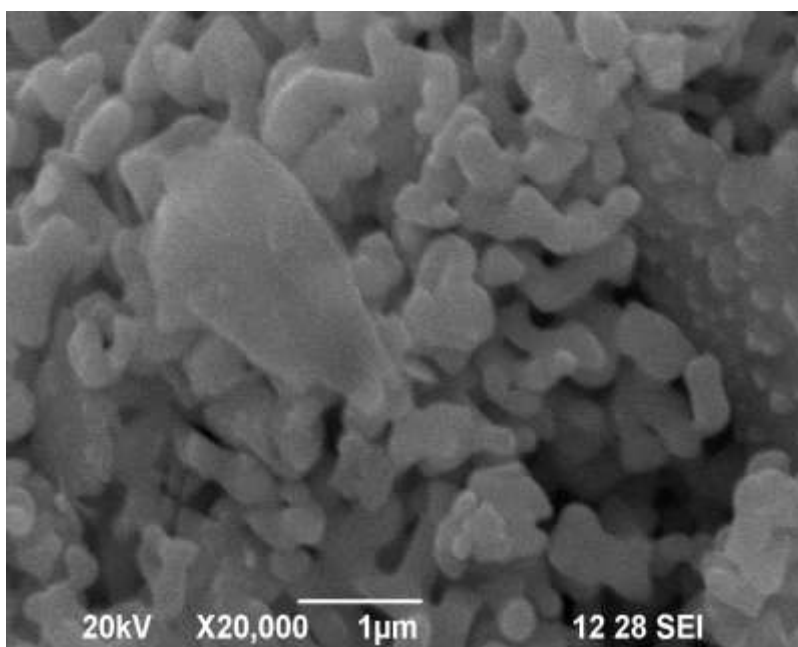
The fig 3 shows the FT-IR spectrum of as-prepared CdSe NPs. Initially, the sample was washed with absolute ethanol and then dried in a vacuum. The IR absorption of the obtained CdSe powder with KBr was obtained. From the **fig**, it can be seen that the broad peak at  $3448\text{ cm}^{-1}$  is assigned to  $-\text{OH}$  stretching intra molecular hydrogen bonds due to the small quantity of  $\text{H}_2\text{O}$  on the sample. N-H stretching vibration peak is observed at  $2882\text{ cm}^{-1}$  due to the presence of hydrazine hydrate in our sample. The peak observed at  $1602\text{ cm}^{-1}$  is assigned to OH of water absorbed from the molecular precursors. C-N stretching vibration peak is positioned at  $1142\text{ cm}^{-1}$  is due to the interaction of hydrazine hydrate **Figure 3** regular and periodic structure of molecular precursors.



**Figure 3** FTIR spectrum of CdSe nanoparticle

## Morphological study

SEM image of the synthesized CdSe nanoparticles is shown in Figure 4. The particles growth is not uniform, but mostly they have elongated shape. There are bigger particles have cross-sectional diameter of almost 70 nm and smaller elongated particles have less than 35 nm diameter.



**Figure 4** SEM analysis of CdSe nanoparticles

## Conclusion

A pure nanoparticle of CdSe has been synthesized at room temperature through wet chemical method. The XRD spectra gives average particle crystallite size in the range of 30-35 nm and also it shows the Zinc blende structure of particle. The SEM analysis confirms the particle size. The technique makes the synthesis of aqueous soluble CdSe is convenient, economic and safe.

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