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Effect of reducing agent in the formation of CdSe nanoparticles by chemical reduction route

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ABSTRACT

CdSe nanoparticles were synthesized using a simple chemical reduction route at room temperature. Nanoparticle size was controlled by the amount of reducing agent and was characterized by TEM and TED. With increased amounts of sodium borohydride as a reducing agent, the size of the nanoparticles decreased. Size of the nanoparticles varies between 5 and 12 nm with a size dispersion of 1.5 nm. The grown sample was ultra-sonicated in ethanol. The dispersed sample was characterized structurally, optically and electrically. The long-duration photoconductive decay at room temperature shows exponential variation under weak illumination.

1. Introduction

Nanoscience is a rapidly emerging field of science. The synthesis and control of materials in nanometer dimensions can lead to access to new material properties and device characteristics in unprecedented ways [1,2]. Semiconductor nanoparticles, which exhibit properties different from bulk materials, are a new class of materials that hold considerable promise for numerous applications in the field of electronics and photonics [3,4]. Nanoscale modification of the molecular design and morphology of such particles provides a powerful approach toward control of their electrical and optical properties [5–8]. Among the colloidal nanocrystals, CdSe (generally Gr-II to Gr-VI) is studied because of the efficiency of its synthesis, the high quality of the resulting sample, and the fact that the optical gap lies in the visible range. Also it is an important semiconducting material with unique electrical properties, which makes it a promising material in the field of opto-electronic devices such as Light Emitting Diodes, Photovoltaic Cells, Solar Cells, Photo Detectors, High Density Magnetic Information Storage, Biosensors, etc. [9,10]. There are various methods [11,12] of the preparation of CdSe nanoparticles. Some of the above mentioned methods have some drawbacks. Used precursors are unstable, are an environmental hazard, and require very high temperatures [13,14]. These methods are not cost effective either. Hence a simple chemical reduction route has been preferred. The grown sample is characterized structurally, optically and electrically by varying the amount of reducing agent. The obtained particle sizes are within the range of 5–12 nm and the dispersion of size is 1.5 nm.

2. Experimental section

Anhydrous $CdCl_2$ (532 mg), selenium powder (208 mg) and sodium borohydride (200, 300, 400 and 500 mg) have been taken to prepare different samples. Ethylenediamine has been used as a capping agent. Sodium borohydride has been taken to initiate the reaction at room temperature and its ratio has been changed for different samples (CdSe-1, CdSe-2, CdSe-3 and CdSe-4) to establish control over the particle size. In order to prepare different samples, the amounts of $CdCl_2$, Se and $NaBH_4$ were taken in the ratios of 1:1:2; 1:1:3; 1:1:4 and 1:1:5. The stirring was continued for 3 h at a particular speed at 30 °C. As for TEM and TED measurements, the as-prepared CdSe nanoparticles have been dispersed in ethanol by ultrasonification. A small drop of dispersed CdSe nanoparticle has been taken on a thin carbon film supported on the copper grid and kept for some time for drying. The Transmission Electron Micrograph of the as-prepared samples has been taken using a JEOL-JEM-200 transmission electron microscope operating at 200 kV. SAED pattern and EDX analyses of the said nanoparticle were also performed. Optical absorption measurements of the dispersed samples have been studied in the range of 500 nm–800 nm using a Shimadzu Pharmaspec 1700 UV–vis spectrophotometer possessing a spectral resolution of 1 nm. Photoluminescence spectra of the same sample have been obtained using a Hitachi F-7000 FL Spectrophotometer.

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A thin film of the CdSe nanoparticles has been grown from the dispersed sample. The glass substrate was dipped into the dispersed solution at least for 6 h. Uniformly thin film of CdSe nanoparticles has been deposited on the glass substrate. Gold is used as an ohmic contact. *I–V* characteristics are found to be linear. The photoelectrical characteristics have been studied using a Kiethly electro-meter-6514. From the long photoconductive decay the relaxation time has been measured. Intensity of light falling on the sample is measured by a luxmeter and is found to be 30 lux.

3. Results and discussion

A TEM image of the bright field of CdSe nanoparticles and its selected area diffraction pattern are shown in Fig. 1(a), (b), (c) and (d). Particle size is determined to be approximately 5–12 nm for different samples. A clear hexagonal phase of the as-prepared CdSe nanoparticles is revealed in the TED pattern. The interplaner spacing (d) is determined from the SAED pattern. The determined d values are 3.828, 3.141 and 2.213 Å for first, second and third rings, respectively. The calculated *d* values match well with JCPDS file values of 3.720, 3.290 and 2.151 Å, which correspond to the (1 0 0), (1 0 1) and (1 1 0) planes, respectively [15]. The EDX analysis is also performed on these nanoparticles and the weight percentage is shown in the table. Initially as the reducing agent is increased percentage of cadmium increases. However at a higher reducing agent ratio the selenium percentage dominates over that of cadmium.

Fig. 2 displays the variation in optical absorbance with wavelength of the as-prepared nanoparticle. Optical absorption coefficient has been calculated in the wavelength region of 500–800 nm. The band gap of the as- prepared nanoparticles is determined from the relation

$$(\alpha h\nu) = C(h\nu - \Delta E_g)^{1/2} \quad (1)$$

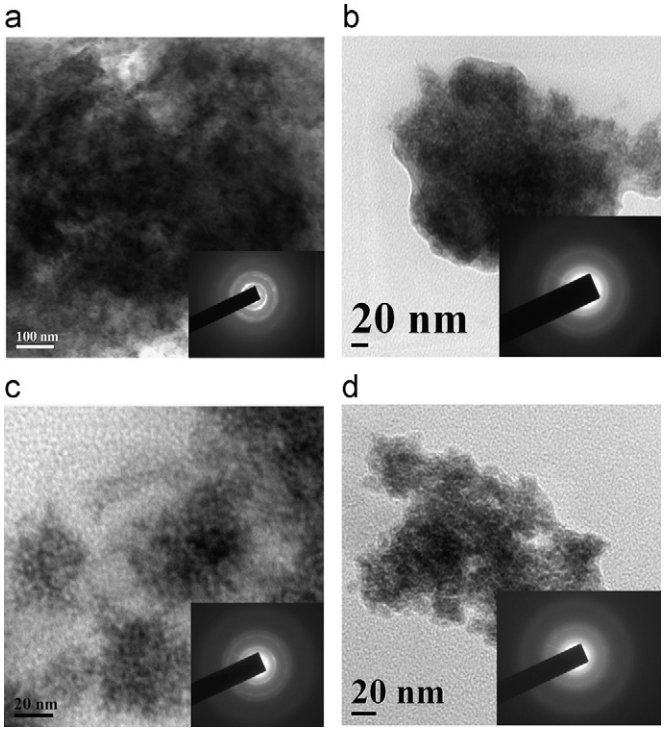

Fig. 1. TEM image and SAED pattern (at inset) of (a) CdSe-1, (b) CdSe-2, (c) CdSe-3 and (d) CdSe-4.

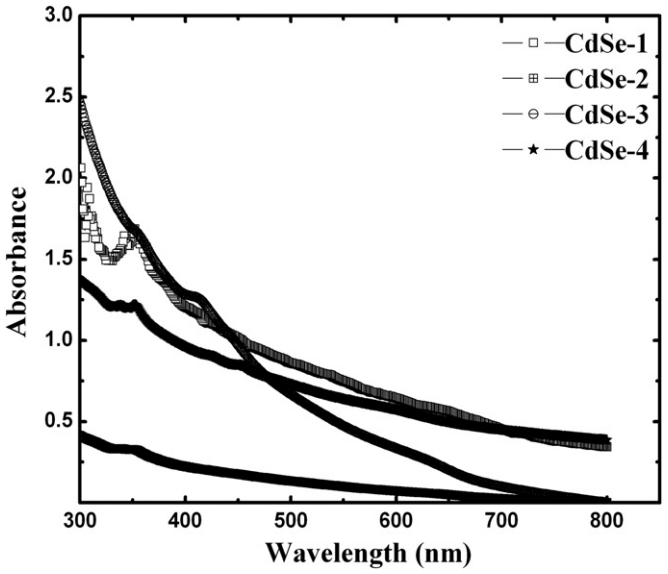

Fig. 2. The optical absorption spectra of as-prepared CdSe nanoparticles.

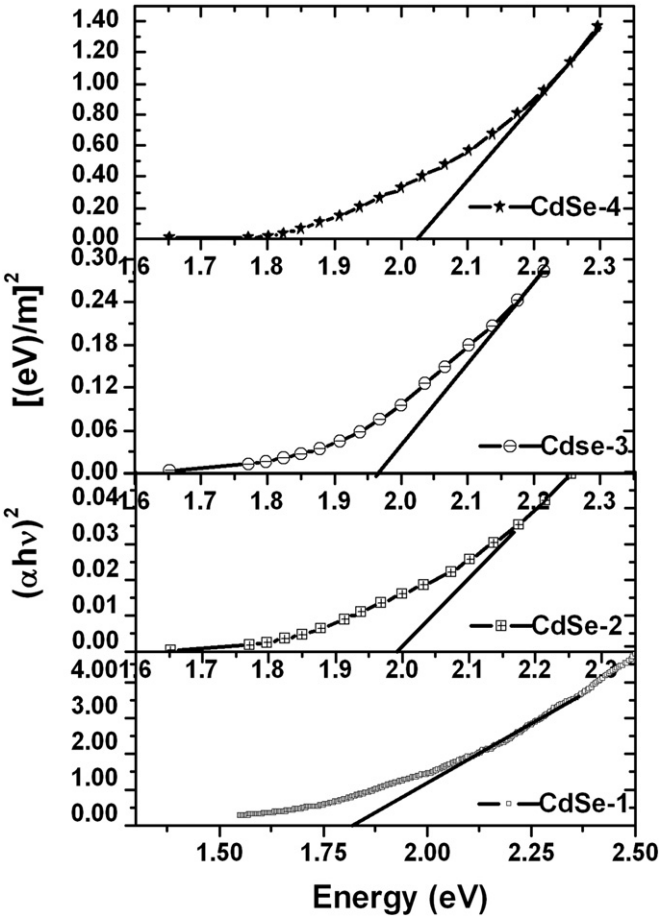

Fig. 3. Band gap determination of as-prepared CdSe nanoparticles.

where *C* is a constant, $\Delta E_g = 1.82$, 1.92, 1.96, and 2.02 eV for the as-prepared samples, as shown in Fig. 3, whereas the bulk band gap is 1.74 eV.The band gap is determined with a precision of 0.002 eV. It confirms that the absorption peak is shifted toward a higher energy than the bulk band edge with the increased ratio of the reducing agent. This is due to the quantum confinement effect.