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Temperature Induced Structural Transition of CdSe Nanoparticles to CdSe Nano-rings

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ABSTRACT

In the present work a simple and cost effective chemical reduction route was followed to synthesize the CdSe nanostructures by varying growth temperatures. The prime aim of this work is to find the effect of temperature on structural and optical properties of CdSe nanocrystalline material. The samples grown with different temperatures were characterized structurally using TEM, TED and EDX. The surface morphologies of the samples were studied using FESEM. Photoluminescence properties and photo absorption properties were also studied for a better understanding of the size confinement effect of the CdSe nanostructures.

Keywords: Nanoparticles, Nanorings, Structural Properties, Surface Morphology, Bandgap

1. Introduction

Since last two decades, rapid growth of nanoscience and nanotechnology are noticed due to their numerous applications. The nanomaterials have drawn great interest of the researchers because that can be utilized in various fields for their potential physical and chemical properties which are unable to achieve at their bulk state [1]. In addition, the electronic and optical properties of the materials at the nano dimension (typically 1 – 100 nm) are the key factors that decide the performance of devices. The invention opens a new vision of study on nanomaterials of different structures viz. nanoparticles, nanotubes, nanowires, nanobelts, nanorings, nanoflex, and nanorods [16-21]. Quantum dots are belonged to zero dimensional materials. All the above said structures of the nanocrystalline gain keen attention among the researchers owing to their significant, enhanced, and altered properties [22-26]. Influence of size on the physical and chemical properties of the materials has taken as serious issue of research since changes in few nanometers in size can have significant impact on the properties of materials [2-4]. The naturally occurred functionalities of nanomaterials have been realized for some time. In nature, the impact of design on the nano scale is realized and it developed some peculiar properties and applications for nanomaterials [5-8]. Elements in various groups of periodic table are combined and generated compound semiconductors. Particularly II-VI, III-V and IV-VI groups binary compound semiconductor have drawn much attraction among the researchers because of their appreciable physical and chemical properties. Significantly, II-VI compound semiconductors such as ZnO, ZnS, ZnSe, ZnTe, CdO, CdS, CdSe, and CdTe and their nano-structural states are now widely used in various semiconducting devices [9-10]. II–VI compound semiconductor nanostructures have attracted great attention due to their fascinating and tunable optical and electrical properties [14] based on quantum confinement effect, such as their tunable bandgaps which can be varied from visible (2.42 eV) to near infrared (IR) range (1.74 eV) [15], and their excellent optical properties such as good photoconduction, fast response times [11-13], and provide them with a wide range of potential applications in biomedicine [27–28]. The present study was focused to synthesize the CdSe nanostructure material in different growth temperatures. The morphological study was done using field emission scanning electron microscopy (FESEM). The structural and optical characterizations were done using transmission electron microscopy (TEM), x-ray diffraction (XRD), UV-VIS absorption spectra and photoluminescence (PL) technique respectively.

2. Experimental

Sodium borohydride ($NaBH_4$) (500 mg), anhydrous cadmium chloride ($CdCl_2$) (532 mg) and selenium powder (Se) (208 mg) were taken as reactants for the chemical synthesis process. Ethylenediamine was used here as a capping agent for the experiment and $NaBH_4$ was taken to initiate the reaction at room temperature (30 ^{0}C). The stirring time was fixed for 3 hours with a magnetic stirrer rotation speed of 850 rpm. CdSe nanostructures were synthesized at 15 ^{0}C, 30 ^{0}C and 55 ^{0}C growth temperatures. The powder x-ray diffraction (XRD) pattern of CdSe samples were recorded by x-ray diffractometer (miniflex II, desktop-x-ray diffractometer) using Cu-kα radiation. For TEM and TED measurements, the grown samples were dispersed in ethanol by ultrasonification. A small drop of dispersed CdSe samples were taken on three separate thin carbon films supported on the copper grids and kept for some time for drying. The transmission electron micrograph (TEM) of the as-prepared samples was taken using a JEOL-JEM- 200 transmission electron microscope operating at 200 kV. SAED pattern and EDX analyses of the said nanostructured material were also performed. Optical absorption measurements of the dispersed samples were studied using a Shimadzu Pharmaspec 1700 UV–VIS. Photoluminescence spectra of the same samples were obtained using a Hitachi F-7000 FL Spectrophotometer. Field emission scanning electron microscopy (FESEM) of CdSe nanostructured material was also done using Agilent 5500 for its morphological characterization.

3. Results and discussion

3.1. Structural characterization

The structures and surface morphologies of the CdSe nanostructures grown at different synthesis temperatures were characterized using TEM and FESEM. The Figure 1 shows the TEM and SAED patterns of CdSe material synthesized at different temperatures. At 15 ^{0}C growth temperature, spherical shaped nanoparticles were observed. The particles were seemed to be agglomerated at low synthesis temperature. The average particle size estimated from the TEM image was 10-15 nm. Few particles were also tried to form bigger particles of average diameter 20 nm. This phenomenon supports the Ostwald ripening where two small particles in the vicinity, have a natural tendency to coalesce to form a bigger particle. As the growth temperature increased to 30 ^{0}C, the particles were trying to get separated, thus clear and distinct spherical granules were observed. The average particle size estimated at 30 ^{0}C growth temperature was 5-8 nm. When the growth temperature was further increased to 55 ^{0}C, nano-ring shaped structure with average estimated diameter of 69-75 nm was observed from its corresponding TEM image. The SAED patterns of the CdSe nanostructures revealed the polycrystalline nature

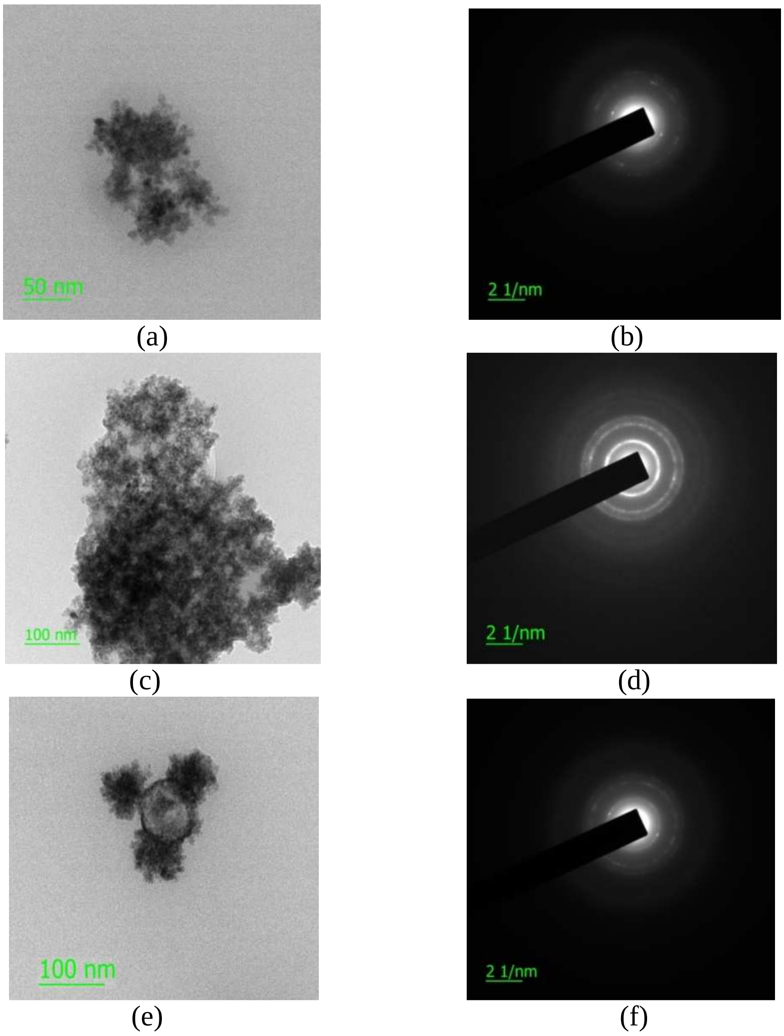


Figure 1: The TEM images and corresponding SAED patterns of the CdSe nanostructures synthesized at: (i) 15 ^{0}C (a & b), (ii) 30 ^{0}C (c & d), and (iii) 55 ^{0}C (e & f).

of the samples. At low temperature synthesis process generally the mobility of the particles was low hence the particles showed the natural tendency to keep their positions. But with the increase in temperature, mobility of the particles increased and hence they tried to self-assemble to achieve a more thermodynamically stable configuration, and as a result of that nano-ring shaped structure was observed. The FESEM images and EDX spectra of the CdSe nanostructure material grown at different temperatures are shown in

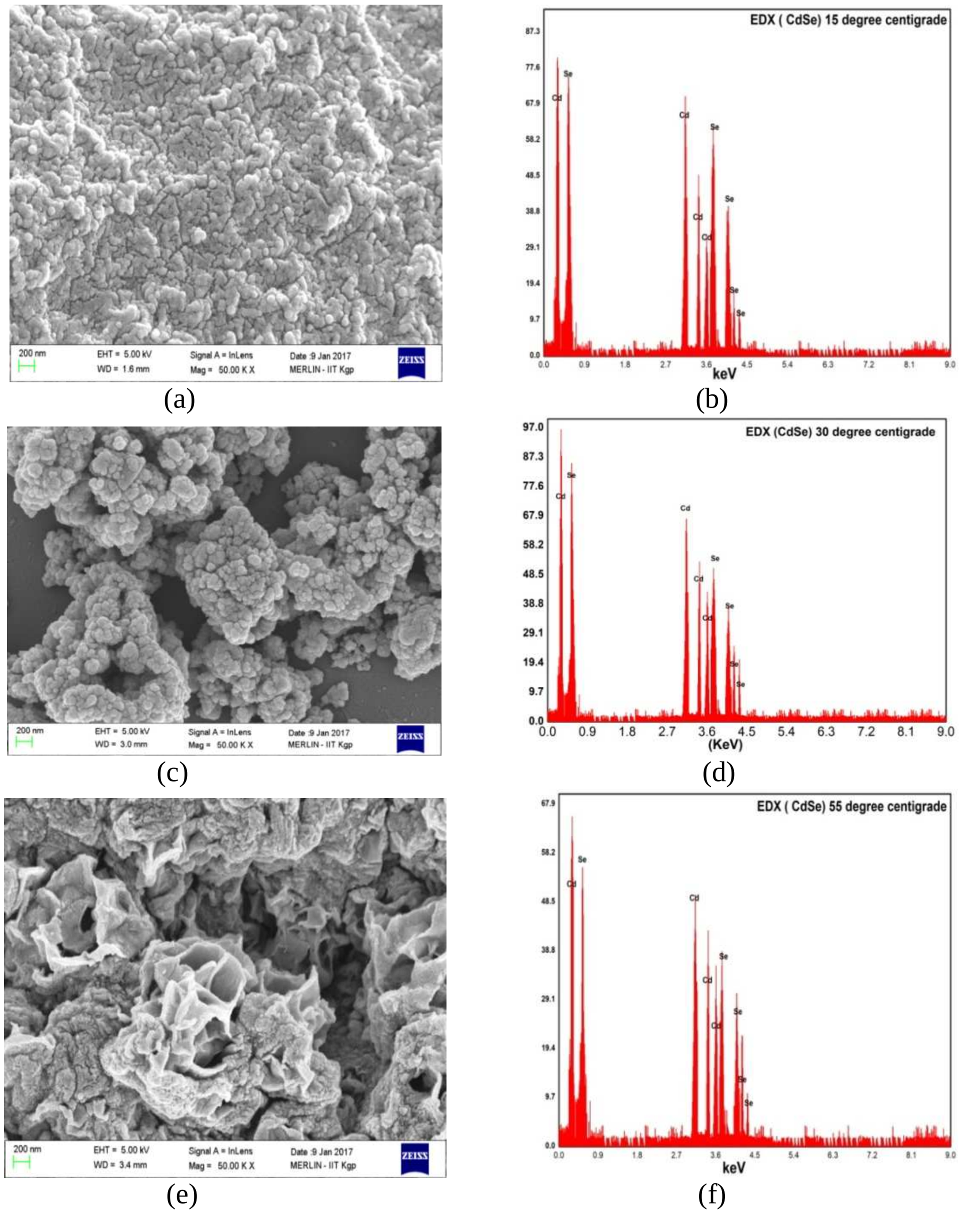
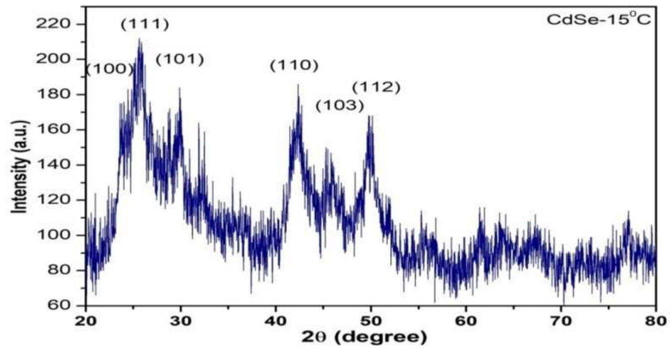


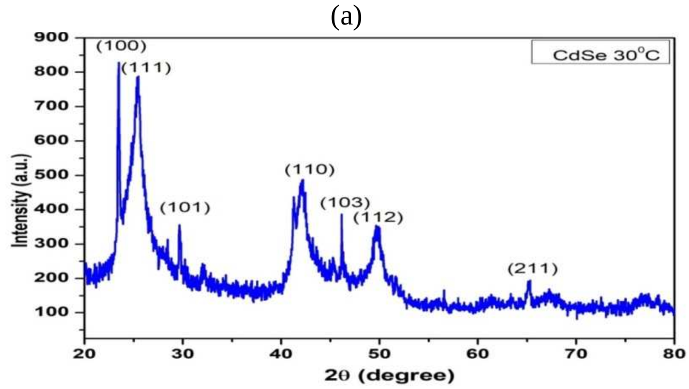
Figure 2: The FESEM images and EDX spectra of the CdSe nanostructures synthesized at: (i) 15 ^{0}C (a & b), (ii) 30 ^{0}C (c & d), and (iii) 55 ^{0}C (e & f).

The surface morphology of the as prepared CdSe nanostructure was also observed through its FESEM image. The spherical shaped particles were observed when the samples was grown at 15 ^{0}C. The particles were also observed in a little agglomerated condition which supported the corresponding TEM image. At 30 ^{0}C growth temperature a distinct spherical shaped nanoparticles were observed from its FESEM image, whereas at

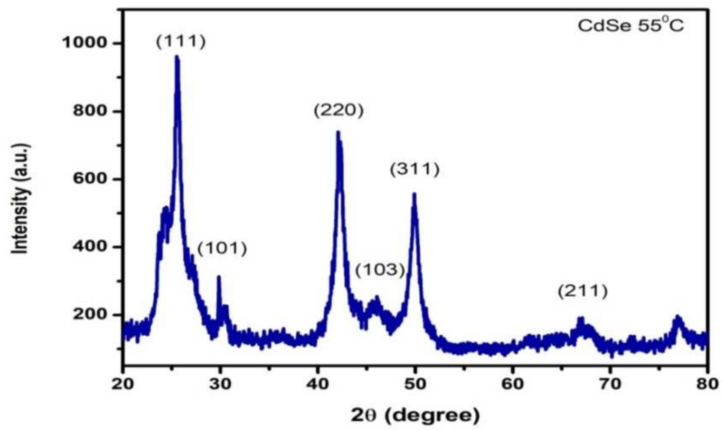
55 ^{0}C the nano-ring was observed. The atomic percentage of Cd and Se calculated from EDX analyses was nearly 1:1 in the CdSe nanostructures. X-ray diffraction of the CdSe nanostructures synthesized with different growth temperatures are shown in Figure 3.



(a)



(b)



(c)

Figure 3: XRD patterns of the CdSe nanostructures synthesized: (a) 15 ^{0}C, (b) 30 ^{0}C, and (c) 55 ^{0}C growth temperatures.

The Bragg's angles and the corresponding peaks were matched with the JCPDS data. The crystal planes corresponding to the particular XRD peaks were (100), (111), (101), (110), (103), (112), (211) for the Bragg's angles 23.72^0, 25.55^0, 28.76^0, 42.4^0, 47.04^0, 50.28^0, 67.36^0 respectively. The phase of the nanocrystalline CdSe was mostly hexagonal.

3.2. Optical characterization

The photoluminescence spectra of the CdSe nanostructures synthesized at different growth temperatures were taken in the wavelength range of 500-750 nm and are shown in Figure 4. The PL peaks for CdSe nanostructures produced having growth temperatures 15 ^{0}C, 30 ^{0}C & 55 ^{0}C, were observed at 650 nm, 610 nm and 540 nm respectively. Figure 5 shows the optical spectra of the CdSe nanostructures prepared at different temperatures.

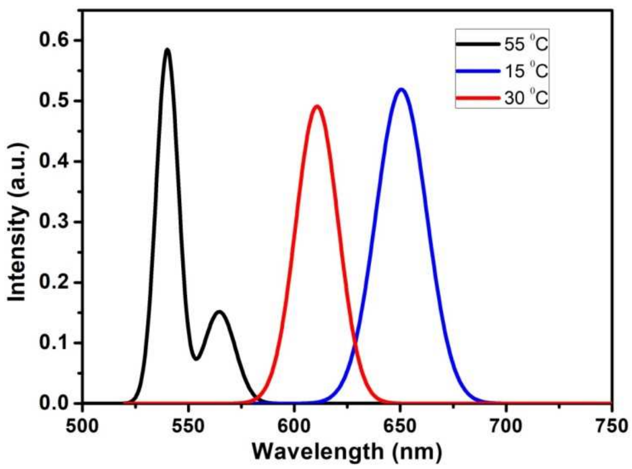


Figure 4: PL spectra of CdSe nanostructure samples synthesized at different growth temperatures.

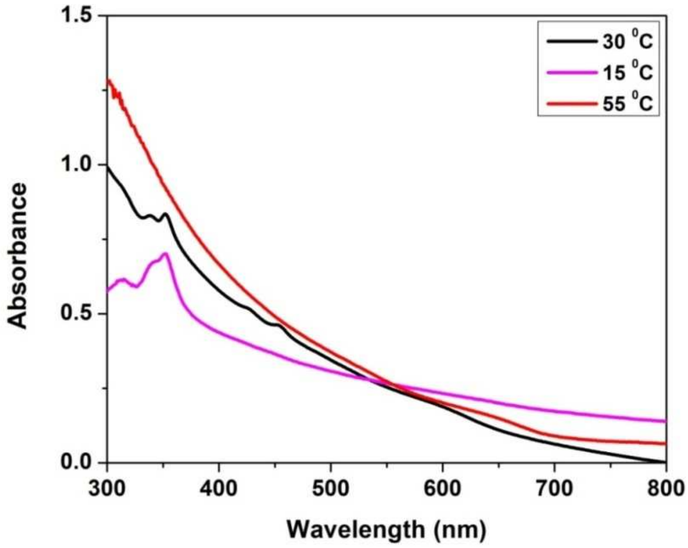


Figure 5: The optical absorption spectra of CdSe nanostructures synthesized at different growth temperatures.

The corresponding bandgap calculation plots are shown in Figure 6. The calculated bandgaps for the CdSe nanostructures grown at 15 ^{0}C, 30 ^{0}C and 55 ^{0}C were 1.89 eV, 2.02 eV and 2.26 eV respectively. The increased bandgap of the material with respect to increased synthesis temperature reflected a decrease in particle size. A summarization table of different parameters calculated for the samples are given in Table 1.

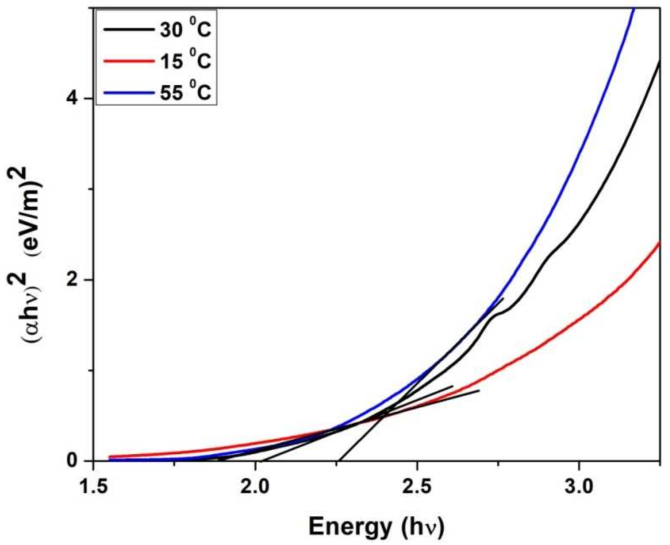


Figure 6: The band gap determination plots of CdSe nanoparticles synthesized at different growth temperatures.

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Growth temperature of CdSe nanostructure (^{0}C)	Shape change of CdSe nanostructure	Average size of the CdSe nanostructure	Bandgap (eV)
15	Nanoparticles	10-15 nm	1.89
30	Nanoparticles	5-8 nm	2.02
55	Nano-ring & nanoparticles	Diameter 69-75 nm & particle size 6-8 nm	2.52

Table 1: The shape, size and bandgap of CdSe nanostructures synthesized at different growth temperatures

4. Conclusion

Different CdSe nanostructures were synthesized in a cost effective way by varying the growth temperature only. Little agglomerated tendency was observed at low temperature because of lower mobility of the particles. The average particle size decreased with the increase of temperature. At 55 ^{0}C nano-ring shaped CdSe nanoparticles were produced which is probably due to the natural tendency of the particle to achieve the more thermodynamically stable structure. The TEM and FESEM pictures supported the formation of different structures with the variation of growth temperature. The shift in PL peak towards the higher energy region and corresponding increased in bandgap of the CdSe nanostructures supported an improved quantum confinement effect with the increase of growth temperature.

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