Available online at www.derpharmachemica.com

Scholars Research Library

Der Pharma Chemica, 2013, 5(5):1-4
(http://derpharmachemica.com/archive.html)

ISSN 0975-413X
CODEN (USA): PCHHAX

Synthesis and characterization of CdS quantum dots by reverse micelles method

Shri Prasad S. and Madhavan J.*

Department of Physics, Loyola College, Chennai

ABSTRACT

Quantum dots (QDs) of direct band gap semiconductor cadmium sulfide (CdS) were synthesized using aqueous $CdCl_2$ and Na_2S by microemulsion method at 303 K. The cationic surfactant cetylpyridinium chloride (CPC) and co-surfactant 1-pentanol were used in water in heptane emulsion to form reverse micelles. The broad X-ray diffraction pattern and clear lattice fringes in the high-resolution transmission electron microscope (HRTEM) images indicate cubic phase of the CdS nanocrystals. The zero dimensional nature of the synthesized particles was observed in the field emission scanning electron microscope (FESEM) image. The average size of the obtained nanoparticles was 4.8 nm. Optical properties revealed that the change of band gap energy from its bulk value of 2.4 to 2.8 eV due to the typical quantum size effects. Blue shift was observed in the photoluminescence (PL) spectrum.

Keywords: nanoparticles, nanocrystals, quantum dots, cadmium sulfide, cetylpyridinium chloride, reverse micelle, micro emulsion, QDs, CdS and CPC.

INTRODUCTION

The synthesis and characterization of semiconductor QDs of size less than 10 nm have been extensively studied in the past three decades because of their physical, chemical, and electronic properties change dramatically due to quantum confinement effects when their sizes become comparable to the Bohr exciton radius [1–2]. Among various kinds of II–VI compound semiconductors, CdS is widely studied due to the band-gap energy (2.4 eV) existing in the visible region [3], which is suitable for photovoltaic cells [4] and light-emitting diodes [5]. Well known QDs synthetic routes are hot-injection [6], solvothermal, hydrothermal [7], hydrogel [8], microwave [9], electrochemical [10] and reverse micelle [11]. The reverse micelle method is a wet synthetic technique for obtaining nanocrystals with narrow size distribution and good mono-dispersity [12]. This technique does not require any special instruments or extreme conditions. Reverse micelle synthesis of CdS quantum dots by the surfactant CTAB was reported by Ian Harvey J et.al. [13]. In this work, the capping agent CPC was first time employed with the co-surfactant 1-pentanol in water/heptane emulsion to synthesis CdS QDs.

MATERIALS AND METHODS

In this synthesis $CdCl_2$ (GR, Merck), Na_2S (Merck), 1-Pentanol (GR, Merck), heptane (GR, Merck), methanol (GR, Merck) and CPC (AR, Sigma-Aldrich) were used without further purification. The 18.2 ohm cm (Millipore Direct-Q3 UV) purified water was used for preparation and washing in all reactions.

Characterization

The prepared CdS nanopowder was characterized by a X-ray Diffractometer RAYOS-X with monochromatic $CuK_{\propto}$ (λ=1.54060 Å) radiation. The UV-Vis absorption spectrum of the sample was recorded in the range 200-800 nm, employing a double beam Varian Cary 5E spectrometer. The photo luminance study was done on the CdS

nanoparticles using Jobin Yvong flurolog-3-11 spectroflurometer. FESEM image was obtained from F E I Quanta FEG 200 High Resolution Scanning Electron Microscope. HRTEM image was taken using JEOL 3010.

Synthesis

10 g of CPC was mixed in 50 ml of 1-pentanol for 10 minute using magnetic stirrer. The solution was again stirred in 200 ml heptane for 20 minutes. 10 ml aqueous $CdCl_2$ (0.5 g) was added drop wise in the above solution to form micro emulsion. Similarly micro emulsion for 10 ml aqueous Na_2S (.485 g) was prepared. Na_2S added micro emulsion was added drop wise in the $CdCl_2$ added micro emulsion within 10 minutes. 50 ml methanol was added and left for some time. It was observed that the nano particles were floating on the solution which was separated by a separating funnel and washed five times by water to remove the surfactant and water soluble mater and finally washed it by methanol five times to remove other organic matter. The residue was dried in open air for one hour and kept inside the desicater with $CaCl_2$ for 12 hours. The obtained powder was annealed at 373 K for 3 hours.

RESULTS AND DISCUSSION

The powder XRD pattern of the CdS QDs. in the Fig. 1 shows broad peaks indicating the small sizes of the QDs. The XRD peaks at 26.825°, 43.875° and 51.925° correspond to the crystal planes (1 1 1), (2 2 0) and (3 1 1) of the cubic CdS phase (JCPDS 10-0454) respectively. The average size of the QDs calculated from (2 2 0) reflection by the Scherrer formula [14], [15] was about 4.8 nm.

The UV–visible absorption spectrum in the Fig. 2 shows the first absorption peak was absorbed at 480 nm. Photoluminescence (PL) spectrum was observed for excitation wavelength 480 nm and exhibits a blue luminescence spectrum as shown in the Fig. 3. The photoluminescence maximum was observed at 441 nm which is equivalent to the band gap energy of 2.81 eV .The quantum confinement effect is the reason for the blue shift in the photoluminescence maximum than the bulk CdS. The photoluminescence in the direct band gap semiconductor is due to the recombination of excitons [16]. The FESEM image in the Fig.4 shows the zero dimensional particle nature of the CdS QDs. The EDAX spectrum in the Fig. 5 shows the elemental composition of the synthesized CdS QDs. The average particle size was determined as 4.8 nm from the HRTEM image in the Fig.5. The regular and clear lattice fringe patterns in the HRTEM image is the evidence for the crystalline nature of the synthesized QDs.

The reverse micelles are formed in the water/heptane emulsion by the surfactant CPC and the co-surfactant 1-pentanol. The reverse micelles encapsulated the aqueous $CdCl_2$ and Na_2S separately in the separate emulsions. During mixing, the micelles are colliding causing the fusion of a micelle containing Cd^{2+} and another micelle containing S^{2-} which results in the formation of CdS entrapped in a micelle. This controlled release of precursors prevents rapid precipitation thus controlling the size of the particles formed and preventing coagulation [13].

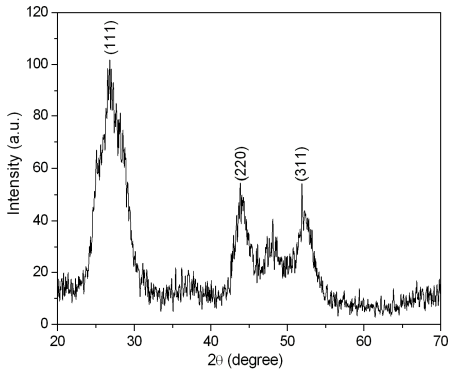

Fig.1 X-ray diffraction pattern of CdS QDs

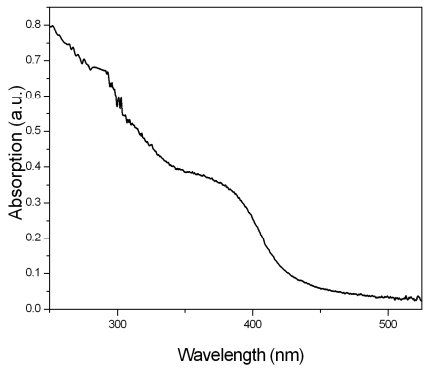

Fig.2 UV-Vis Absorption spectrum of CdS QDs

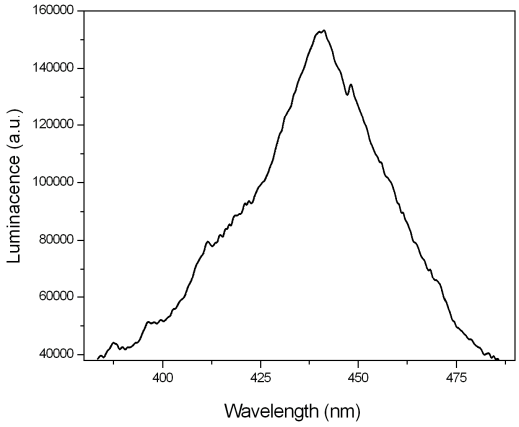

Fig.3 Photoluminescence spectra of CdS QDs for the excitation wave length 380 nm

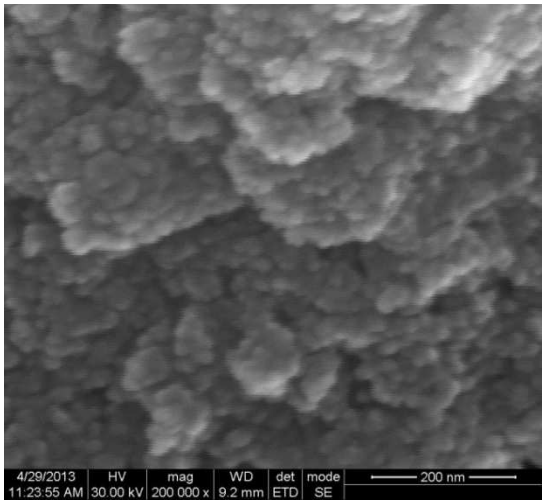

Fig.4 FESEM image of CdS QDs

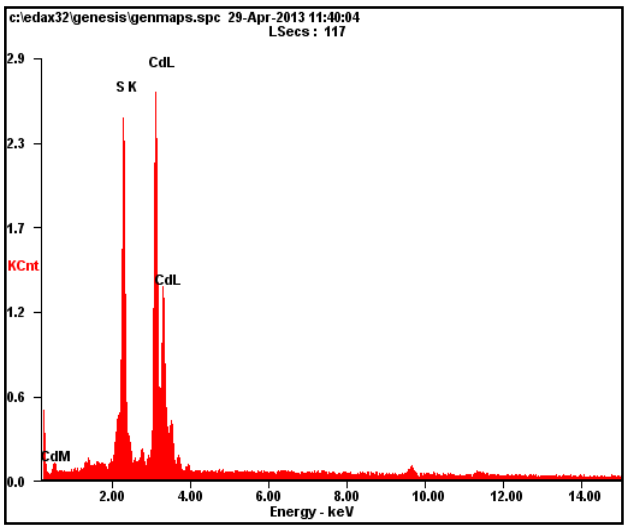

Fig.5 EDAX image of CdS QDs

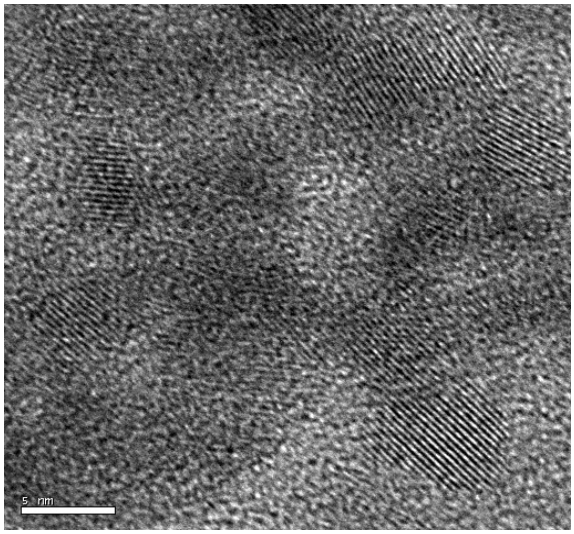

Fig.6 HRTEM image of CdS QDs

CONCLUSION

The direct band gap semiconductor quantum dots of cadmium sulfide have been successfully synthesized in the room temperature by CPC reverse micelles. The CdS nanocrystals formation mechanism was discussed. The crystal structure, surface morphology, elemental composition and particle size were studied from the XRD, FESEM, EDAX and HRTEM images. Optical properties were studied through UV-Vis and PL spectra and its band gap energy was determined.

REFERENCES

[1] W. W. Yu, L. Qu, W. Guo, X. Peng, *Chem. Mater.*, **2003**, 15, 2845.
[2] Y. Wang, N. Herron, *Phys. Rev. B*, **1990**, 42, 7253 .
[3] X. Fan, M. L. Zhang, I. Shafiq, W. J. Zhang, C. S. Lee, S. T. Lee, *Cryst. Growth Des.*, **2009**, 9, 1375.
[4] P. V. Kamat, *J. Phys. Chem. C*, **2008**, 112, 18737.
[5] J. L. Zhao, J. A. Bardecker, A. M. Munro, M. S. Liu, Y. H. Niu, I. K. Ding, J. D. Luo, B. Q. Chen, A. K. Y. Jen, D. Ginger, *Nano Lett.*, **2006**, 6, 463.
[6] C. B. Murray, D. T. Norris, M. G. Bawendi, *J. Am. Chem. Soc.*, **1993**, 115, 8706.
[7] S. Q. Sun, T. Li, *Cryst. Growth Des.*, **2007**, 7, 2367.
[8] Nurettin Sahinera, Kivanc Selb, Kadem Merald, Yavuz Onganerd, Sultan Butuna, Ozgur Ozaya, Coskun Silanb, *Colloids and Surfaces A: Physicochem Eng. Aspects*, **2011**, 389, 6.
[9] E. Caponetti, D. C. Martino, M. Leone, L. Pedone, M. L. Saladino, V. Vetri, *J. Colloid Interface Sci.*, **2006**, 304, 413.

[10] Y. Liang, C. Zhen, D. Zou, D. Xu, *J. Am. Chem. Soc.*, **2004**, 126, 16338.
[11] M. L. Curri, A. Agostiano, F. Mavelli, M. Della Monica, *Mater. Sci. Eng. C*, **2002**, 22, 423.
[12] Saim Emin, Norihito Sogoshi, Seiichiro Nakabayashi, Masumi Villeneuve, Ceco Dushkin, *Journal of Photochemistry and Photobiology A: Chemistry*, **2009**, 207, 173.
[13] Ian Harvey J Arellano, Joey Mangadlao, Iris Bea Ramiro, Kiall Francis Suazo, *Materials Letters*, **2010** , 64, 785.
[14] P. Scherrer, *Göttinger Nachrichten Gesell*, **1918**, 2, 98.
[15] A. L. Patterson, *Phys. Rev.*, **1939**, 56 (10), 978.
[16] W. M. de Azevedo, F. D. Menezes, *Journal of Luminescence*, **2012**, 132, 1740.