



SONOCHEMICAL METHOD FOR SYNTHESIS AND CHARACTERIZATION OF CDS NANO PARTICLES IN AQUEOUS MEDIUM

S.R.Patil*¹, Vijay K. Suryawanshi² and A. P. Manake^{3*}

¹Department of chemistry, MGSM'S A.S.C. College, Chopda, M.S.India-425107.
(srpatil_001@rediffmail.com)

²Department of chemistry, R.C.Patel Institute of Technology, Shirpur,M.S.India.

³Department of Chemistry, Pratap College, Amalner. Dist-Jalgaon. M.S. India. Pin-425401, India.(apmanake@gmail.com).

ABSTRACT

In Sonochemical methods the researchers have intention on the study of the effects of sound waves and sound properties on chemical reactions or systems. The purpose of this research was to synthesize fast and facile method for the synthesis of CdS nanoparticles. For the present investigation we used Cadmium Chloride and Sodium sulphide in aqueous medium. The resultant product is investigated by using X-ray diffraction technique (XRD), UV spectroscopy and Scanning electron microscopy (SEM). The analysis of X-ray diffraction data confirms the average grain size as 23.12nm. During the Course of reaction the increase in the temperature was occurred because of absorption of ultrasound waves was controlled conveniently by ice bath.

Keywords: Sonochemistry, CdS nanoparticles, U.V., XRD, SEM.

Introduction

Now a day's nanocrystals of the transition metals and the leading group elements have attracted much interest due to their unique thermoelectric, semiconducting and optical properties. Semiconductors of sulfides have already found applications as sensors or laser materials, optical filters, solar cells and in many other devices¹. Among these materials, CdS is particularly interesting due to its high photosensitivity², which makes it an excellent n-type window material in hetero-junction solar cells³. Using n-type CdS, solar cells of different efficiencies were fabricated⁴. However, a

further improvement in the efficiency should be achievable by employing nanocrystalline CdS. It is well known that crystals with dimensions in the nanometer range show characteristics that are substantially different from the characteristics of bulk materials. In nanocrystalline CdS, an effective increase in the band-gap is observed due to quantum-confinement effects.

Many approaches have been suggested for the synthesis of group II–VI semiconductors, including the well-known solid-state reaction⁵, gas-phase reactions between the elements or its compounds and gaseous H₂S/H₂Se⁶, or the pyrolysis of single-source precursors⁷. All these methods require high temperatures which make the size control difficult) and the use of toxic precursors. A direct combination of elements in amine has been reported⁸; however, the obtained products were amorphous and had to be crystallized at elevated temperatures. The group of Gedanken et al. used microwave-assisted methods for the preparation of metal selenides^{9,10}. Recent reports include the bacterial biosynthesis of CdS nanocrystals. Chemical reactions such as oxidation, reduction, dissolution and decomposition, which have then been exploited to prepare nanoparticles^{11,12}. Ultrasonic waves have, as a result, been extensively used for producing novel materials with unusual properties. They can induce the formation of particles with a remarkably smaller size, which usually show novel physical properties¹³. The interest results from the special properties of materials in the nanoscale rule, such as a large surface-to-volume ratio and increased surface

activity, as compared to that of the bulk material. In our laboratory, the sonochemical method has been successfully used in the synthesis of nanoparticles of CdS.

The first synthesis of CdS nanoparticles by ultrasonic irradiation was reported by Wang et al.^{2,14,15}. The method used the reaction of a mixed solution of cadmium chloride and sodium thiosulfate in isopropyl alcohol in an Argon atmosphere. A sonochemical approach to the synthesis of CdS nanoparticles using Cadmium acetate and elemental S in dimethyl sulfoxide under a H₂/ Ar atmosphere was also reported¹. Recently, Behboudnia and Khanbabaee reported on the synthesis of CdS nanoparticles by the sonication of an ethylenediamine solution of 1-decanthiol, cadmium acetate and elemental S¹⁶ and the sonochemical preparation of CdSe nanoparticles from cadmium acetate, tartaric acid, freshly prepared sodium selenosulfate and thiophenol¹⁷. However, to the best of our knowledge, the sonochemical synthesis of CdS from aqueous solutions has not been reported so far. A direct, simple and general method, avoiding the necessity of using inert atmospheres and non-aqueous systems, seems to be required. In this paper we present a new, simple method to prepare CdS nanoparticles from aqueous solutions in the ambient atmosphere.

2. Experimental

2.1 Materials

CdCl₂, CdO₂, Na₂S/S elemental sulphur were obtained from SRL and Sigma Aldrich. All the chemicals were used as received without further

purifications. All solutions were prepared in milimoles.

2.2 Synthesis of CdS

For the preparation of CdS nanoparticles, a cationic precursor solution was prepared by dissolving 2.58 gm (2 mmol) of cadmium oxide in 30mL of water at room temperature. Then we prepared the anionic precursor solution by dissolving 1.56 g (2 mmol) of Na₂S in 30mL of water. The two solutions were mixed in a flat-bottomed beaker and irradiated with high-intensity. After 40 min of sonication (1sec on and 2 sec off) the mixture is allowed to stirred for 3 hours. The product thus obtained is filtered and washed twice with distilled water and once with absolute ethanol. Finally, the brown colored product was dried for 2 h at 150 LC followed by calcination at 450 LC in muffle furnace for 4 to 5 hours.

3. Results and Discussion

Characterization was performed via X-ray powder diffraction (XRD) and Scanning electron microscopy (SEM). Fig. 1 shows the XRD spectra of typical nanoparticles of CdS prepared by the sonochemical method after 40 min of sonication. The peaks can be classified as the 111, 200 and 222 reflections of cubic CdS (JCPDS-411049). The broadening of the peaks indicate that the particles are on the nanometer scale. The average size of the nanoparticles was calculated using the Debye–Scherrer formula:

$$D = \frac{0.9 \lambda}{\beta \cos \phi}$$

Table 1- Average Size of Nanoparticles

2 θ	β	θ	θ in rad	β in rad	cosθ	0.9*0.15406 (nm)	Grain size (D) nm
17.7552	0.5531	8.87761	0.15494	0.00965	0.98802	0.138654	14.5368341
3	2	5	4	4			
17.2260	0.2842	8.61304	0.15032	0.00496	0.98872	0.138654	28.26603838
8	6	6	6	1	2		
30.348	0.3053	15.174	0.26483	0.00533	0.96513	0.138654	26.95595082
	6	6	6	5			
33.039	0.1817	16.5195	0.28832	0.00317	0.95872	0.138654	45.58685943
	7			2	3		
38.3501	0.3530	19.1750	0.33466	0.00616	0.94451	0.138654	23.8256524
	2	5	8	1	9		
49.7781	0.8902	24.8890	0.43439	0.01553	0.90712	0.138654	9.837418734
6	4	8	6	8	4		
55.3330	0.3989	27.6665	0.48287	0.00696	0.88566	0.138654	22.48422596
5	4	3	2	3	5		

58.3192	0.5605	29.1596	0.50893	0.00978	0.87326	0.138654	16.22996683
9	2	5	2	3	5		
65.9737	0.4418	32.9868	0.57572	0.00771	0.83879	0.138654	21.43502946
1	5	6	9	2	5		
69.3166	0.4378	34.6583	0.60490	0.00764	0.82255	0.138654	22.05633741
3	8	2	2	2	8		
Avg. grain size (D)							23.12143135

Here, λ is the wavelength of the X-ray radiation, β is the full width at half maximum (FWHM) of the corresponding peak and θ is the dif-fraction angle.

The value obtained for CdS is 23-60 nm. The size and morphology of the as-prepared CdS

nanoparticles were assessed with Scanning electron microscopy (SEM). The SEM image of the CdS (Fig.2) shows that the particles are mainly cubic, but strongly agglomerated.

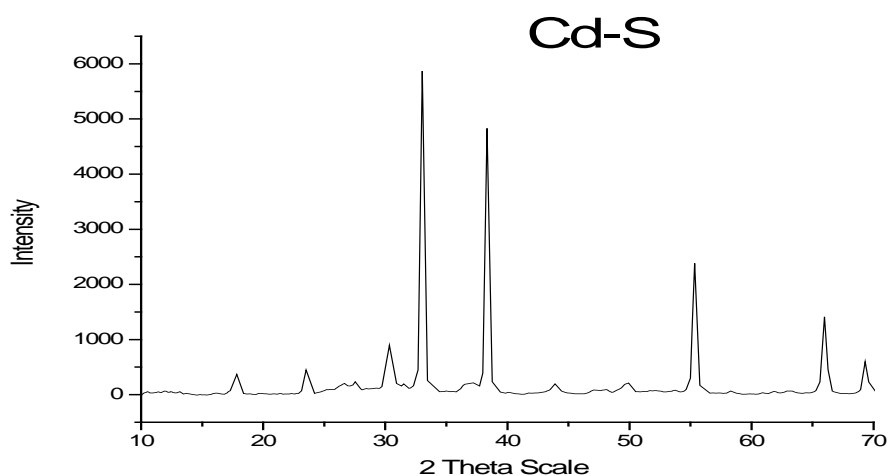


Fig. 1- X-ray diffraction spectra of CdS nanoparticles synthesized by ultrasonic irradiation for 40 min.

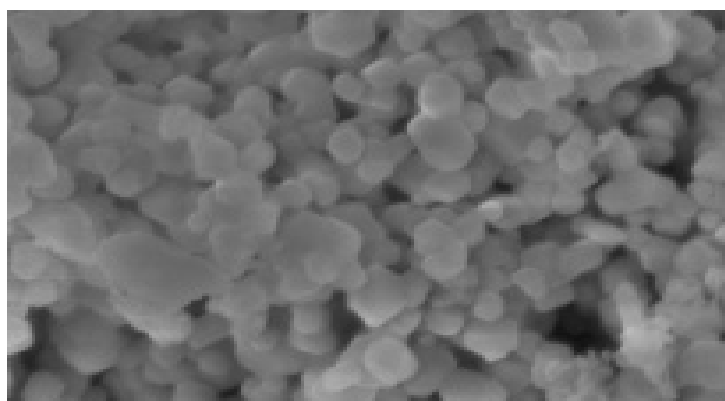


Fig. 2- SEM image of as-prepared CdS nanoparticles.

4. Conclusion

CdS nanoparticles have been successfully synthesized via a novel sonochemical route. The preparation method is relatively simple in comparison to other reported methods, avoids the use of an inert atmosphere, and uses less hazardous precursors and environmental friendly aqueous solvents. The average size of nano crystallites was about 23 nm for the CdS. The products were characterized with powder diffraction analyses and thermal analyses including characterization of the intermediates. The crystal structure changed from cubic. SEM observations indicated that the particles were agglomerated, but confirmed their cubic structure and uniform composition. The obtained nanoparticles are expected to be applicable in modern optoelectronic devices and solar cells. Further investigations may lead to the preparation of a variety of metal chalcogenides using similar preparation methods in aqueous systems.

REFERENCES

- [1] Li, H.L., Zhu, Y.C., Chen, S.G., Palchik, O., Xiong, J.P., Koltyipin, Y., Gofer, Y., Gedanken, A. (2003). *J. Solid State Chem*, 172, 102–110.
- [2] Wang, G.Z., Chen, W., Liang, C.H., Wang, Y.W., Meng, G.W., Zhang, L.D. (2001). *Inorg. Chem. Commun*, 4, 208–210.
- [3] Singh, V.P., McClure, J., Lush, G.B., Wang, W., Wang, X., Thompson, G.W. et al. (1999). *Sol. Energy Mater. Sol. Cells*, 59, 145–161.
- [4] Britt, J., Ferekides, C. (1993). *Appl. Phys. Lett.* 62, 2851–2852.
- [5] Coustal, R., *J. Chem. Phys.* (1958). 28, 277–280.
- [6] Metcalf, H.C., Williams, J.E., Caska. (1982). *J.F. Modern Chemistry*, Holt, Reinhart, Winston, New York, p. 54.
- [7] Steigerwald, M.L., Alivisatos, A.P., Gibson, J.M., Harris, T.D., Kortan, R., Muller, A.J., et al. (1988). *J. Am. Chem. Soc*, 110, 3046–3050.
- [8] Henshaw, G., Parkin, I.P., Shaw, G. (1996). *Chem. Commun*, 1095–1096.
- [9] Zhu, J.J., Palchik, O., Chen, S.G., Gedanken, A. (2000). *J. Phys. Chem*, 104, 7344–7347.
- [10] Palchik, O., Kerner, R., Gedanken, A., Weiss, A.M., Slifkin, M.A., Palchik, V. (2001). *J. Mater. Chem*, 11, 874–878.
- [11] Suslick, K.S. (1989). *Sci. Am.* 56, 80–86.
- [12] Suslick, K.S. (1990). *Science*, 247, 1439–1445.
- [13] Gedanken, A. (2004). *Ultrason. Sonochem*, 11, 47–55.
- [14] Wang, G.Z., Wang, Y.W., Chen, W., Liang, C.H., Li, G.H., Zhang, L.D. (2001). *Mater. Lett.* 48, 269–272.
- [15] Wang, G., Wang, Y., Wu, Y., Li, G., Zhang, L. (2003). *J. Mater. Sci. Technol*, 19, 278–280.
- [16] Behboudnia, M., Khanbabaee, B. (2006). *Colloids Surf. A*, 290, 229–232.
- [17] Behboudnia, M., Azizianekalandaragh, Y. (2007). *Mater. Sci. Eng. B*, 138, 65–68.