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Sonochemical Method for the Synthesis and Characterization of Cadmium Compound Nanoparticles in Aqueous Medium

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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 CdX nanoparticles. For the present investigation we used Cadmium Chloride and different metal halide or sulphide compounds in aqueous medium. The resultant product is investigated by using X-ray diffraction technique (XRD), UV spectroscopy and Scanning electron microscopy (SEM). 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: Son-chemistry, CdX nanoparticles, U.V., XRD, SEM.

I. INTRODUCTION

Now a days nanocrystals of the transition metals and the leading group elements have attracted much interest due to their unique thermoelectric, semiconducting and optical properties. Semiconductor of sulfidesand selenides have al-ready found applications as sensors or laser materials, optical filters, solar cells and in many other devices [1]. Among these materials, CdS is particularly interesting due to its high photosensitivity [2], which makes it an excellent n-type window material in hetero-junction solar cells [3]. Using n-type CdS and P-type CdTe, solar cells of different efficiencies were fabricated [4]. 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 ob-served due to quantum-confinement effects.

Many approaches have been suggested for the synthesis of group II–VI semiconductors, including the well-known solid-state reaction [5], gas-phase reactions between the elements or its compounds and gaseous H2S/H2Se [6], or the pyrolysis of single-source precursors [7]. 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 re-ported [8]; 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 properties [13]. The interest results from the special properties ofmaterials 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 reported [1]. Recently, Behboudnia and Khanbabaee reported on the synthesis of CdS nanoparticles by the sonication of an ethylenediamine solution of 1-decanthiol, cad-mium acetate and elemental S [16] and the sonochemical preparation of CdSe nanoparticles from cadmium acetate, tartaric acid, freshly prepared sodium selenosulfate and tiophenol [17]. How-ever, 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.

II. METHODS AND MATERIAL

2.1 Materials

$CdCl_2$, CdO_2, Na_2S/Selemental sulphur and SeO_2 were obtained from SRL and Sigma Aldrich. All the chemicals were used as received without further purifications. All solutions were prepared in mill moles.

2.2 Synthesis

For the preparation of CdS nanoparticles, acationic 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 Na_2S 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) themixture is allowed to stirred for 3 hours. The product thus obtained is filteredand 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.

For the preparation of CdSe nanoparticles, a cationic precursor solution was prepared by dissolving 2.58 gm (2mmol) of cadmium oxide in 30mL of water at room temperature. Then we prepared the anionic precursor solution by dissolving 2.22 g (2 mmol) of SeO_2 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 white colored product was dried for 2 h at 150 LC followed by calcination at 450 LC in muffle furnace for 4 to 5 hours.

III. RESULTS AND DISCUSSION

Characterization was performed via X-ray powder diffraction (XRD) and Scanning electron microscopy (SEM). Figs. 1 and 2 show the XRD spectra of typical nanoparticles of CdS and CdSe 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) and CdSe

(JCPDS-020330) respectively.The broadening of the peaks indicates that the particles are on the nanometer scale. The average size of the nanoparticles was calculated using the Debye–Scherrer formula:

$$D = \frac{0.9\,\lambda}{\beta \cos \emptyset}$$

2 θ	β	θ	θ in rad	β in rad	cosθ	0.9*0.15406 (nm)	Grain size (D) nm
17.75523	0.55312	8.877615	0.154944	0.009654	0.98802	0.138654	14.5368341
17.22608	0.28426	8.61304	0.150326	0.004961	0.988722	0.138654	28.26603838
30.348	0.30536	15.174	0.264836	0.00533	0.965135	0.138654	26.95595082
33.039	0.18177	16.5195	0.28832	0.003172	0.958723	0.138654	45.58685943
38.3501	0.35302	19.17505	0.334668	0.006161	0.944519	0.138654	23.8256524
49.77816	0.89024	24.88908	0.434396	0.015538	0.907124	0.138654	9.837418734
55.33305	0.39894	27.66653	0.482872	0.006963	0.885665	0.138654	22.48422596
58.31929	0.56052	29.15965	0.508932	0.009783	0.873265	0.138654	16.22996683
65.97371	0.44185	32.98686	0.575729	0.007712	0.838795	0.138654	21.43502946
69.31663	0.43788	34.65832	0.604902	0.007642	0.822558	0.138654	22.05633741
						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 and CdSe 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 in Fig. 3 shows that the particles are mainly cubic, and Fig. 4 shows the SEM image of the CdSe are hexagonal but strongly agglomerated.

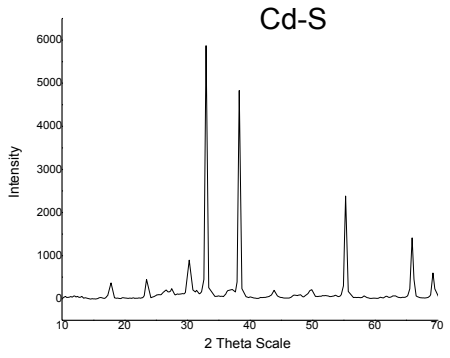

Fig. 1. X-ray diffraction spectra of CdS nanoparticles Synthesized, by ultrasonic irradiation for 40 min.

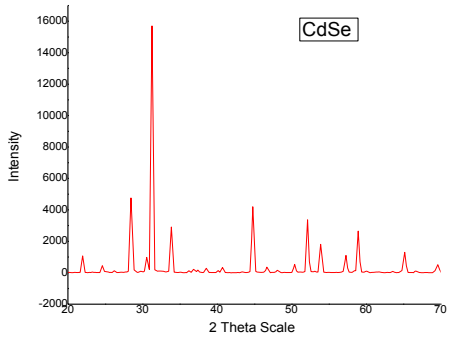

Fig. 2. X-ray diffraction spectra of CdSe nanoparticles Synthesized, by ultrasonic irradiation for 40 min.

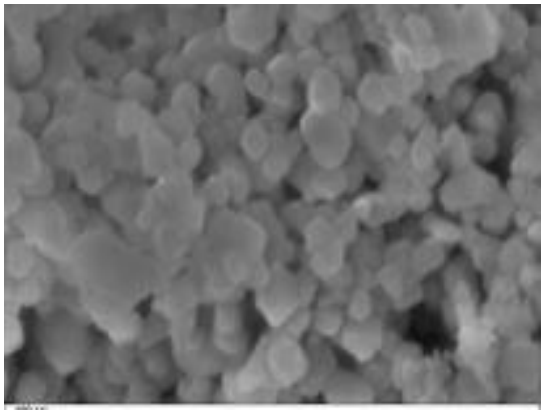

Fig. 3 SEM image of as-prepared CdS nanoparticles

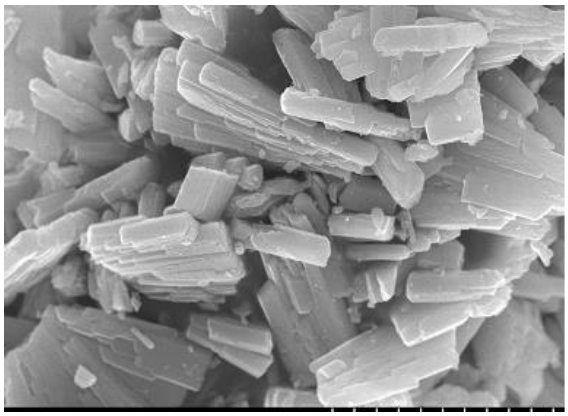

Fig. 4 SEM image of as-prepared CdSe nanoparticles

IV. CONCLUSION

CdS and CdSe 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 nanocrystallites was about 23-60 nm for the CdSand CdSe. 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 con-firmed their cubic and hexagonal structure and uniform composition. Theobtained 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.

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