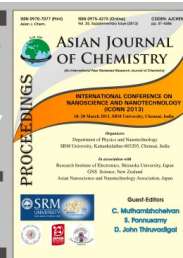




Asian Journal of Chemistry; Vol. 25, Supplementary Issue (2013), S381-S383

ASIAN JOURNAL OF CHEMISTRY

www.asianjournalofchemistry.co.in



Synthesis and Characterization of Lead-Cadmium Selenide Nanoparticles by Polyol Method for Semiconductor based Applications†

P.C. KARTHIKA*, A. RATNAKAR, N. BALASUBRAMANIAM, PARIKSHIT SAHATIYA, KRISHNA TEJA and ARJUN AJIT MOHAN

Department of Physics and Nanotechnology, SRM University, Kattankulathur, Chennai-603 203, India

*Corresponding author: Tel: +91 44 27417831; E-mail: karthim.technano@gmail.com

AJC-12881

Lead-cadmium selenide nanoparticles were synthesized using magnetic stirrer assisted polyol method using cadmium acetate, lead acetate and selenium powder. The obtained resultant product was found to have a cubic structure from the XRD data analysis and UV-visible spectrum, scanning electron microscopic pictures showed that lead and cadmium selenide particles of smaller than 60 and 150 nm showed that the product have a good band gap of 3.1 eV making it a good semiconducting material. While comparing the result with cadmium selenide nanoparticles it was found that lead-cadmium selenide is a little enhanced than the former with respect to semiconducting property and semiconductor based electronics applications.

Key Words: Lead-cadmium selenide, XRD, UV-visible, Bandgap, Semiconducting material.

INTRODUCTION

Semiconductors are those materials that neither fit into good electrical conductors or insulators categories. They don't fit into any 4 structural materials with respect to atomic bonding. Generally they are combination of materials from group III and V or from II and VI or from IV group of periodic table. Silicon and germanium are the two most commonly used semiconductors but a lot of application oriented real time systems are in need of semiconductors that are a little enhanced than the two mentioned earlier. Hence the most recent trend with respect to the semiconducting materials are selecting or choosing or identifying various other elements from periodic table and trying to use them as a possible replacement for the existing semiconducting materials. These new semiconducting materials have great scope and vast applications in many fields.

With this new trend of engineering semiconductor materials, using nanotechnology and nanosciences yields the most sought and favoured nanoparticles. Cadmium selenide nanoparticle is one such semiconducting material. It is a semiconductor obtained by engineering cadmium-II group element and selenium-VI group element. This CdSe nanoparticles has drawn a lot of attention towards it due to its amazing property of possessing a good band gap value and for its optoelectronic properties. CdSe particles of size less than 100 nm exhibit quantum confinement and also they possess size dependent

fluorescence spectrum¹. CdSe nanoparticles have exceptional optical characteristics such as transmission, reflection, absorption and scattering.

Cadmium selenide is a solid hexagonal or cubic crystal. It is a direct band gap *n*-type semiconducting material with a band gap of 1.74 eV at 300 K². When there is a considerable change in increase of annealing temperature, there is a gradual increase in the size of the crystal CdSe nanoparticles and E_g value is found to be 2.3 eV at 573 K of annealing temperature³. Also in many cases the optical absorption edge of CdSe is reported to be approximately 610-690 nm⁴⁻⁶. These make cadmium selenide to be a good semiconductor but in application point of view it can only be used as an *n*-type semiconductor. Hence to enhance the semiconducting property of CdSe addition of lead is preferred. Lead an IV-group element when added with CdSe can result in the combination of lead-cadmium selenide a semiconducting material with 4 electrons in outermost shell. This lead-cadmium selenide like the pure CdSe is not *n*-type but is an intrinsic semiconductor *i.e.*, it can be doped with impurities for various applications.

CdSe nanoparticles had been reported to be successfully synthesized by microwave oven assisted polyol reduction method, aqueous phase approach method⁶, photochemical synthesis process⁷, hydrothermal method⁸, solvothermal method, sol-gel approach, surfactant-assisted approach *etc.* In this work nanoparticles of lead-cadmium selenide was

†International Conference on Nanoscience & Nanotechnology, (ICONN 2013), 18-20 March 2013, SRM University, Kattankulathur, Chennai, India

synthesized by magnetic stirrer assisted polyol method. Stirring basically brings fresh parts of the solvent into contact with the solute and particles are forced to connect and the presence of temperature assists the entire process for effective desired results. Furthermore synthesis is conducted without requiring special and/or sensitive reagents or expensive equipment.

EXPERIMENTAL

Nanoparticles of lead-cadmium selenide was synthesized by polyol method. All reagents used were of high grade and quality. Cadmium acetate, lead acetate and ethylene glycol were purchased from SISCO Research Laboratory Pvt. Ltd., Mumbai, India. While selenium powder was obtained from LOBA Chemie Laboratory Reagents and Fine Chemicals, Mumbai, India.

Synthesis of lead-cadmium selenide: 0.33 g of cadmium acetate and 0.05 g of lead acetate were dissolved in 40 mL of ethylene glycol by gently heating the solution in an oven for few minutes (3-5 min). The solution was rigorously stirred combined with constant heating at 300 °C using a magnetic stirrer. The constant heating and stirring was carried out till a change of colour from the initial dark black to reddish brown took place. Approximately the time took for the colour change to appear was around 50 min. Once the change in colour was noticed the heating temperature was reduced by 100 °C and the stirring was continued. Stirring was carried out till the reddish brown colour of the solution turned reddish in colour. Once the reddish colour was obtained the heating and stirring was halted immediately and the solution was allowed to cool down. The approximate time taken for the colour change from reddish brown to red happened within 20 min. The product obtained was centrifuged once with mother liquid and with acetone (twice or thrice) for 45 min and 15 min respectively. The resulting product was then dried at room temperature overnight.

RESULTS AND DISCUSSION

In order to study the nature of the obtained resultant product XRD, SEM and UV-visible spectroscopy techniques was carried out.

X-ray diffraction studies: Fig. 1a and 1b shows the XRD pattern of the Pb-CdSe sample. From the XRD data-crystal parameters $a(\text{\AA}) = b(\text{\AA}) = c(\text{\AA}) = 4.6990$ and $\alpha(^{\circ}) = \beta(^{\circ}) = \gamma(^{\circ}) = 90^{\circ}$ concluded that the crystal structure of the sample is cubic. The peak lists obtained too is in accordance with the same. The particle size of the synthesized Pb-CdSe sample is determined using the Scherrer's relation $d = (0.9\lambda) / (\beta \cos \theta)$ where β is the full width at half maximum in radians, β is the wavelength of X-rays used and λ is the Bragg's angle. The nanoparticles obtained were of size range from 10-20 nm. In most of the other synthesis methods reported by other scholars it's been found that the reason for the appearance of cubic crystal structure instead of hexagonal structure is due to the usage of ethylene glycol.

The XRD data analysis reveals the amazing results that the obtained product Pb-CdSe is very much similar to CdSe. The random peaks appearing should be due to the presence of lead but some peaks shows that there could be some PbSe

nanoparticles present in the sample. Just in case if the sample is a mixture of PbSe and CdSe the final crystal structure obtained should possess Pb and Cd in opposite equally corners complemented by selenium. The reason for coming to this conclusion is the crystal structure of both PbSe and CdSe nanoparticles. They both form cubic structures. This means the sample contains both *n*-type and *p*-type semiconductor nanoparticles. Hence the conclusion of having obtained a hybrid intrinsic semiconductor material is not ruled out.

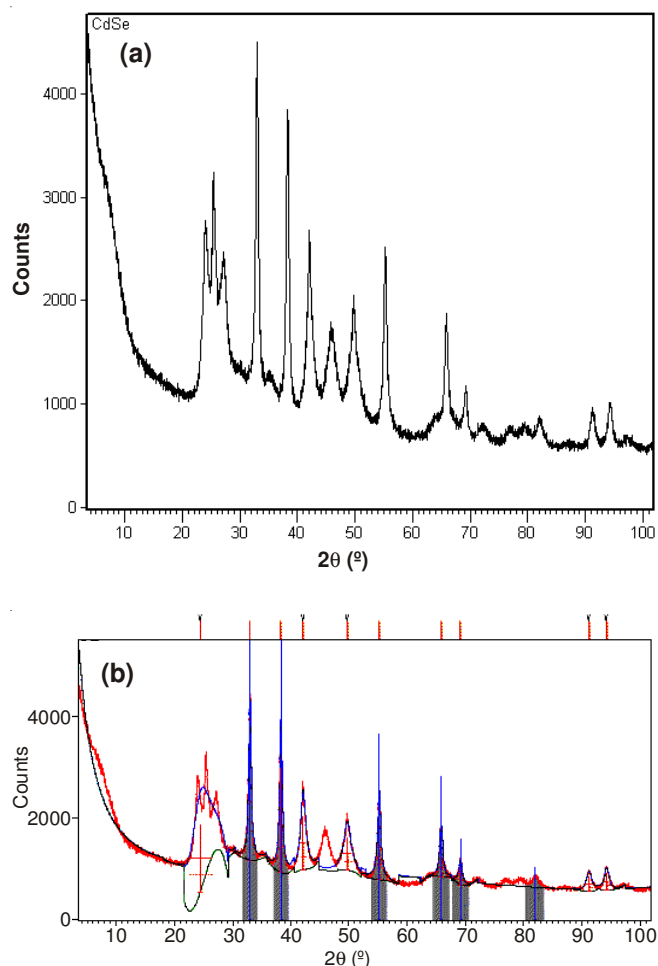


Fig. 1. (a) XRD pattern for prepared CdSe sample; (b) XRD pattern for prepared Pb-CdSe sample

UV-visible spectroscopy studies: UV-visible absorption measurements of solutions of cubic Pb-CdSe in water are shown in Fig. 2. It is noticed that the absorption is in near UV region and it should be due to the electronic transitions. From the graph no prominent peaks were noticed but the absorption shoulders and absorption edge presence at 655 nm, 684 nm, 719 nm are taken seriously because those explains the electronic structure of the material obtained⁴. The absorption shoulder is noticed to be broad which only means that there exist some elongated particles. The peak obtained at 369.50 nm is neglected due to the fact that it's due to the change of filament source used in the instrument. The important conclusion to draw from UV-visible spectroscopy is the peak that we got at 684 nm. The absorbance corresponding to the peak is 0.119. The absorbance of the UV-visible spectroscopy is

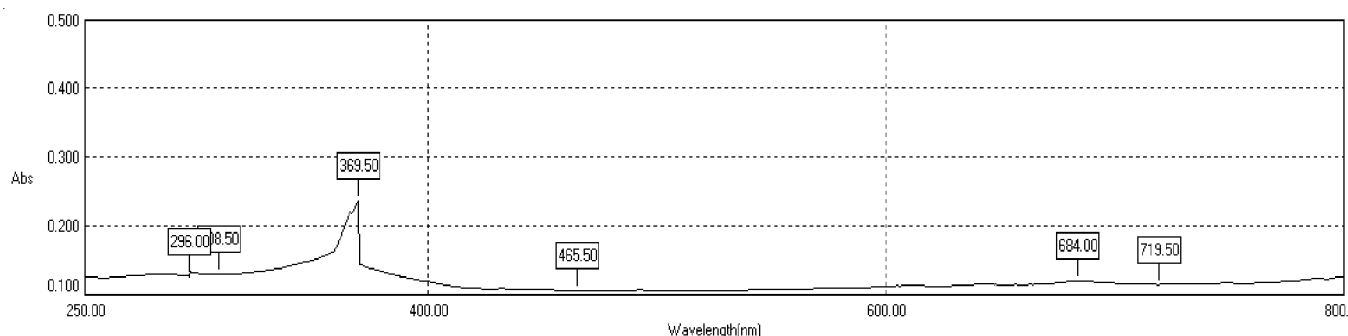


Fig. 2. UV-visible absorption spectrum for the prepared Pb-CdSe sample

related to concentration by means of Beer-Lambert formula which is given by absorbance $A = KcL$, where K is constant, c is constant and L is cell length. It can be seen that, since absorbance is low, concentration is low. The reason for the peak not so dominant at 640 nm is due to low concentration of sample used. The band gap energy corresponding to the absorbed wavelength can be calculated by $E = hc/\text{wavelength}$, which comes out to be 3.1 eV which corresponds to band gap energy of the sample, which is good when comparing with the standards.

Another important conclusion obtained from the analysis results is that the sample satisfies the earlier reported UV-visible results of CdSe nanoparticles by other scholars with the only difference in the bandgap of the material. The band gap of Pb-CdSe is found to be better than CdSe, making it a good replacement for CdSe and other semiconductor nanoparticles.

SEM image of Pb-CdSe nano particles formed during this process were barely visible in the SEM picture due to their small sizes (Fig. 3). Here the average particle sizes are significantly smaller in size. SEM micrographs indicated that the as synthesized nanoparticles were spherical in shape were found in the chemically synthesized powders.

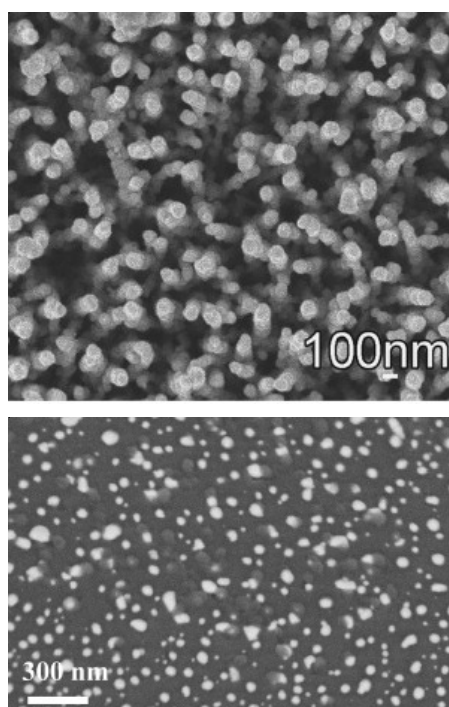


Fig. 3. SEM image Pb-CdSe nano particles

Conclusion

Thus Pb-CdSe nanoparticles were synthesized using magnetic stirrer assisted polyol method. The reported work presents a novel polyol method-based technique for synthesis of water-soluble and bio-tagable Pb-CdSe nanoparticles at room temperature. The crystal structure and the particle size were determined by XRD, SEM and the bandgap of the material was determined by UV-visible spectroscopy studies. This new synthesis method can be considered as an environmentally friendly and inexpensive procedure without need to use expensive equipments for producing other selenide based nanoparticles. Our study may provide a new way for fabrication of Semiconductor based solar cells, electronics application based on Pb-CdSe nanoparticles.

ACKNOWLEDGEMENTS

The authors thank the staff members, scientific officers of physics and nanotechnology for their constant support and encouragement and Department of Physics and Nanotechnology for helping them out for characterization.

REFERENCES

1. D. Ham, K.K. Mishra and K. Rajeshwar, *J. Electrochem. Soc.*, **138**, 100 (1991).
2. R. Jose, V. Thavasi and S. Ramakrishna, *J. Am. Ceram. Soc.*, **92**, 289 (2009).
3. M.K. Nazeeruddin, F.D. Angelis, S. Fantacci, A. Selloni, G. Viscardi, P. Liska, S. Ito, B. Takeru and M. Gratzel, *J. Am. Chem. Soc.*, **127**, 16835 (2005).
4. W.W. Yu, L.H. Qu, W.Z. Guo and X.G. Peng, *Chem. Mater.*, **15**, 2854 (2003).
5. S. Singh, A. Guleria, M.C. Rath, A.K. Singh, S. Adhikari and S.K. Sarkar, *Adv. Mater. Lett.*, In press.
6. D.J. Suh, O.O. Park, H.T. Jung and M.H. Kwon, *Korean. J. Chem. Eng.*, **19**, 529 (2002).
7. D.K. Dwivedi, V. Kumar, M. Dubey and H.P. Pathak, *World J. Sci. Technol.*, **1**, 21 (2011).
8. S.G. Raman, P. Selvarajan, K. Ratnam and Chidambaramhanu, *Adv. Mater. Lett.*, In press..