

Study of optical and morphological properties of lead sulphide nanoparticles with different capping agents

R.Thanal¹, S.Sasikala², V.Jayapriya³, P.Vani⁴, S.Siva⁵

^{1,3} Department of Physics, Gonzaga Arts and Science for Women, Kirishnagiri, India.

² Department of Physics, Kamban College of Arts and Science for Women, Tiruvannamalai, India.

⁴ Department of Physics, VIT university, Chennai, India.

⁵ Department of EEE, Vel Tech Multi tech Dr. Rangarajan Dr.Sakunthala Engineering College, Avadi, India.

Abstract: Study of optical and morphological properties of lead sulphide nanoparticles with different capping agents were prepared by co-precipitation method. The synthesized nanopowder has been characterized by UV-Visible absorption spectroscopy, powder XRD pattern, FTIR and SEM studies. The absorbed SPR peaks for Glycerol and SDS capped PbS nanoparticles were compared and band gap values were calculated. The average particles size and structure of products were calculated by XRD pattern. The present work shows that surfactant plays a significant role on optical and morphological properties of the nanoparticles.

Keywords:

Lead sulphide

Glycerol

SDS (Sodium Dodecyl Sulphate)

SPR (Surface Plasmon Resonance)

1. Introduction

Nowadays semiconducting nanomaterial have gained more appreciable attention in industrial area and scientific techniques due to its considerable properties such as electric, optical and magnetic, which depends on size and band gap of the particles[1-2]. Especially lead sulphide semiconducting nanomaterial have received a great attention because of its significant applications such as solar cell[3], photovoltaic devices[4], electroluminescent devices[5], telecommunications[6] and optical devices[7]. Also bulk lead sulphide has a band gap range of ($E_g = 0.41\text{eV}$) with bohr radius value of 180 \AA and PbS nanoparticles exhibit the effect of quantum confinement by optical absorption. When crystallite size is smaller than bohr radius value, then SPR absorption peak is blue shifted. The size, shape and surfactant have a significant influence on the optical and morphological properties of lead sulphide nanoparticles[8]. Surfactant plays an important role on lead sulphide nanoparticles such as control the particle growth and reduce particles aggregations between them[9]. A variety of methods are used to synthesize lead sulphide nanoparticles such as hydrothermal method[10], sonochemical method[11] and solvothermal method[12].

In this present work, lead sulphide nanoparticles with different capping agents were synthesized by co-precipitation method. The synthesized Glycerol and SDS capped PbS nanoparticles were compared by various characterizations such as Powder X-ray diffraction pattern, FTIR, UV-Visible absorption spectroscopy and SEM studies.

2. Experimental details

2.1. Materials

All of the chemicals used in this work were analytical grade reagents and used without further purification. Lead acetate [$\text{Pb}(\text{CH}_3\text{COO})_2$], sodium dodecyl sulphate (SDS), glycerol and Na_2S were purchased from Merck Company. Deionized water was used to prepare all solutions.

2.2. Preparations of Glycerol and SDS capped PbS nanoparticles

Lead acetate was taken as precursor, sodium dodecyl sulphate (SDS) and glycerol were taken as capping agents and Na_2S was taken as precipitating agent. 1.89 gm of Lead acetate [$\text{Pb}(\text{CH}_3\text{COO})_2$] was dissolved in 50 ml of deionized water and stirred it for one hour. 1gm of Na_2S was dissolved in 50 ml of deionized water. Then sodium sulphide of solution was added into the lead acetate solution drop by drop under continuous stirring until reaches the formation of yellow precipitate. Meanwhile 0.144 gm of sodium dodecyl sulphate (SDS) was dissolved in deionized water and the solution was added to lead sulphide solution and its stirred continuously for 3 hours. The solution was adjusted to maintain the pH range at 4 by adding nitric acid solution in acid medium and color of the solution was changed from yellow to black precipitate. The resulting precipitates after filtration were washed two times with double-distilled water, then with acetone. The samples were dried in an oven at 80°C for 30 min. The same procedure was followed for glycerol capped lead sulfide nanoparticles.

2.3. Characterizations

The powder XRD analysis was carried out by Enraf Nonius CAD4-F diffractometer with the $\text{CuK}\alpha$ ($\lambda=1.540\text{\AA}$) radiation. The Fourier infrared spectra (FTIR) were recorded in the range of 400- 4000 cm^{-1} on a Perkin-Elmer Spectrum II FT-IR spectrometer. UV-Visible absorption spectra were recorded at room temperature using a Varian Carey UV-Visible spectrophotometer from 200 nm to 1200 nm. The Scanning Electron Microscopy studies were carried out by SEM quanta spectrophotometer.

3. Results and discussion

3.1. Structural analysis

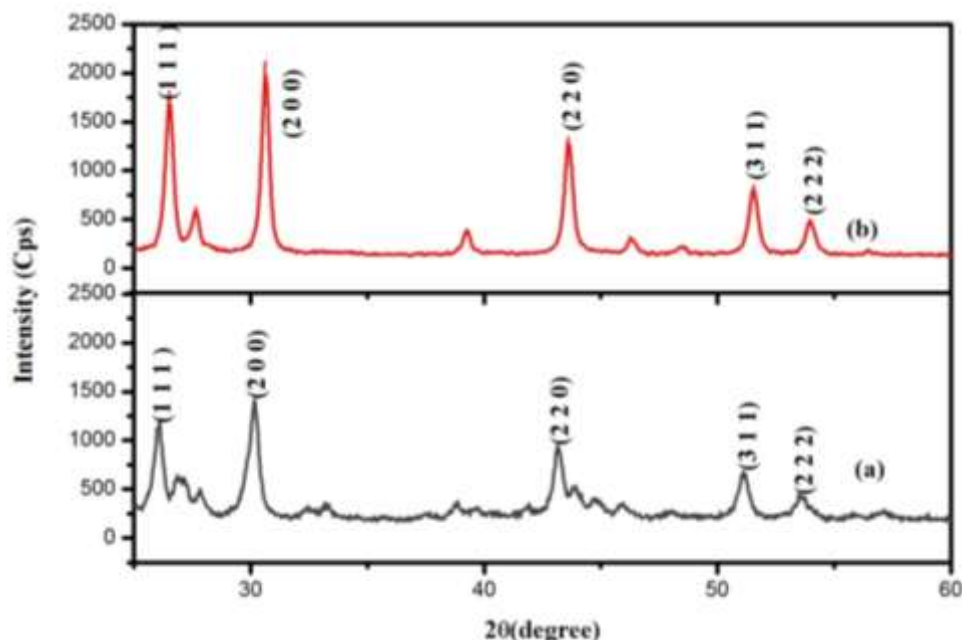


Fig.1. Powder XRD patterns of (a) Glycerol capped PbS and (b) SDS capped PbS

Figure 1. shows XRD pattern of PbS nanoparticles prepared with Glycerol and sodium dodecyl sulphate (SDS) as capping agents respectively. It shows several diffraction peaks at 2θ values of 26.0, 30.68, 43.40, 51.0 and 53.87 which correspond to the Miller index of reflecting planes for (111), (200), (220), (311) and (222) planes are observed indicates the face-centered cubic structure. All the peaks are perfectly indexed with JCPDS (card no.5-592). The strong and sharp diffraction peaks suggest that obtained products are well crystallized. Further, there is no significant change in position of the peaks, which represents that the capping agent only arrests particle growth and does not affect any phase change. The crystallite sizes were calculated using Debye-scherrer's formula.

$$D = k\lambda/\beta\cos\theta$$

Where, d is particle size in nanometer, λ is wavelength of the radiation, k is constant equal to 0.94, β is the full width at maximum intensity and θ is peak position. The value of crystallite size calculated for glycerol capped PbS and SDS capped PbS is 17.9 nm and 20 nm respectively.

3.2. Optical analysis

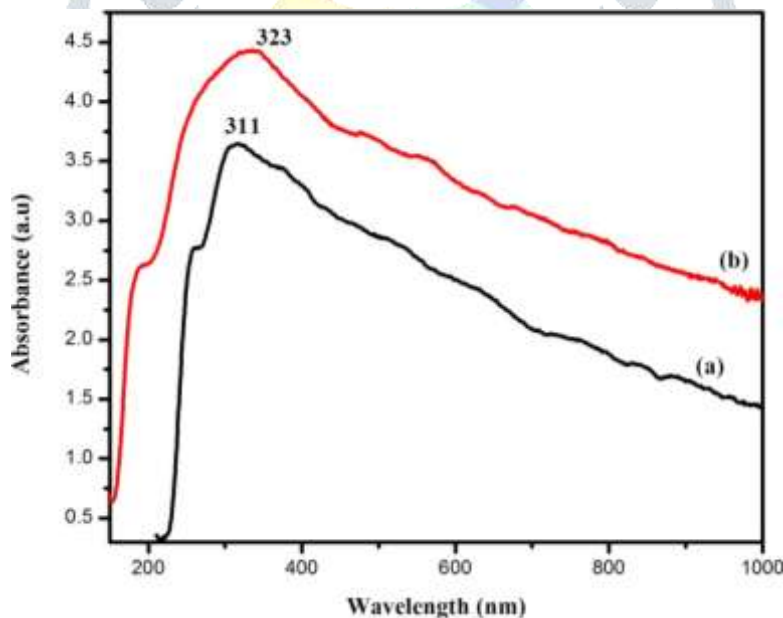


Fig. 2. UV-Vis absorption spectra of (a) Glycerol capped PbS and (b) SDS capped PbS

Figure 2. shows the optical absorption spectra of glycerol capped PbS and SDS capped PbS nanoparticles. The absorbed SPR peaks were found to be maximum at 311nm and 323nm for glycerol capped PbS and SDS capped PbS nanoparticles. SPR peaks indicate that particles are in uniform size and morphological property is well. The band gap energy of PbS nanoparticles are calculated from energy wave equation,

$$E=hc/\lambda.$$

Where h is the planck's constant, c is the velocity of light, and λ is the wavelength of absorbed peak. Band gap energy of the samples corresponds to the absorption edge is found to be 3.9eV for the glycerol capped PbS and 3.8eV for SDS capped PbS nanoparticles.

3.3. Functional group analysis

Figure 3. shows FTIR spectra of Glycerol capped PbS and SDS capped PbS nanoparticles. The presence of functional groups are analyzed by this FTIR studies. The peak at 2109cm^{-1} is assigned to C=C stretching vibration of PbS nanoparticles. The peak absorbed at 1022cm^{-1} is due to C-O stretching mode of vibration[13]. The peak absorbed at 867cm^{-1} confirms that Pb-S compound is successfully formed by co-precipitation method[14]. Then peak appears at 590cm^{-1} and 475cm^{-1} correspond to presence of S-S bond and lead in the samples[6].

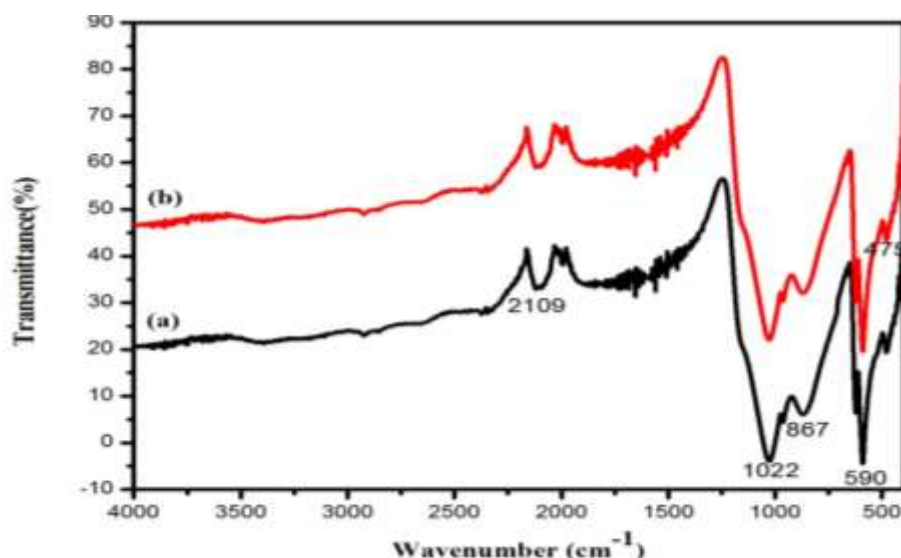


Fig.3. FTIR spectra of (a) Glycerol capped PbS and (b) SDS capped PbS

3.3. Morphological analysis

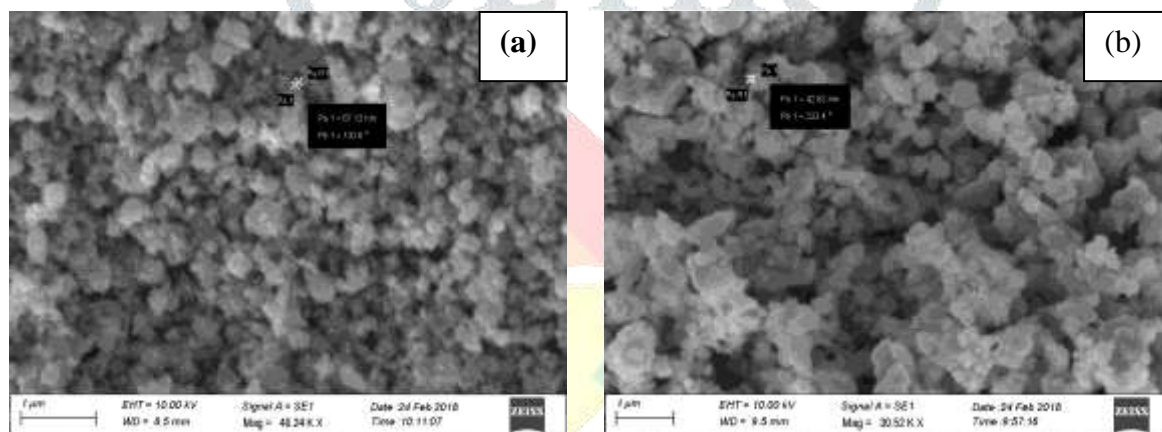


Fig.4 SEM images of (a) Glycerol capped PbS (b) SDS capped PbS

Figure 4. shows SEM images of Glycerol capped PbS and SDS capped PbS nanoparticles. From this image its clear that Glycerol capped PbS nanoparticles are in spherical shape and the particles are in very small size. Then SDS capped PbS nanoparticles are also in spherical shape but few particles are aggregated with one another. The particle size of SDS capped PbS is slightly larger than Glycerol capped PbS and this data has good agreement with XRD pattern.

Conclusions

Glycerol capped PbS and SDS capped PbS were synthesized by co-precipitation method. XRD patterns exhibit face centered cubic structure for both synthesized Glycerol and SDS capped PbS nanoparticles. The crystallite size is found to be around 17.9 nm and 20 nm for lead sulphide nanoparticle with Glycerol and SDS capping agents respectively. The absorbed SPR peaks are maximum at 311 nm for Glycerol capped PbS and 323 nm for SDS capped PbS by UV-Visible absorption spectra. SEM images of prepared samples reveals that the Glycerol and SDS capped PbS particles has spherical in shape. The formation of PbS compound and presence of varies functional groups are confirmed by FTIR studies.

Reference

- [1] D. V. Talapin, S. Haubold, A. L. Rogach, A. Kornowski, M. Haase, H. Weller, *J. Phys. Chem. B* 105 (2001) 2260
- [2] F. E. Kruis, H. Fissan, B. Rellinhaus, *Mater. Sci. Eng. B* 69–70 (2000) 329-334
- [3] Hassan Karami, Mina Ghasemi, Sara Matini, *Int. J. Electrochem. Sci.*, 8 (2013) 11661 – 11679
- [4] M.Rezaul Karim, M.D. Aktaruzzaman, M.Ashrafuzzaman, M.D. Badruzzaman, *Chalcogenide Letters*, 11(2014) 531-539
- [5] Muhammad Hamid Sarwar Wattoo, Abdul Quddos, Abdul Wadood, Muhammad Bilal Khan, Feroza Hamid Wattoo, Syed Ahmad Tirmizi, Karamat Mahmood, *Journal of Saudi Chemical Society* (2012) 16, 257–261
- [6] R.Hepzi Pramila Devaman, M.Archana, K.Maheshwari, D.Susmitha, *International Journal of Engineering Science Invention*, 7(2018)58-65
- [7] Huynh, W.U., Dittmer, J.J., Alivisatos, A.P., *Science*, 295(2002) 2425–2427
- [8] Eychmueller, A. Haesselbarth, L. Katsikas, H. Weller, *Ber. Bunsenges. Phys. Chem.* 95, 79 (1991)

- [9] G. Lelong, S. Bhattacharyya, S.kline, T. Caccia guerra , M.A.Gonzalez, M.L.Saboungi J Phys chem., 112 (2008) 10674-10680
- [10] P.Sakthivel, S.Sharon Tamilselvi, T.Arokiya Mary, Joe Jesudurai, International Journal of Scientific & Engineering Research, 5(2014)152-155
- [11] H.C. Metcalf, J.E. Williams, J.F. Caska, Modern Chemistry, 14 (1982) 54
- [12] F. Li, Q. Qin, J. Wu, Z Li, Journal of Materials Science, 45(2010) 348.
- [13] Diwaker Kumar, Garima Agarwal, Balram Tripathi, Devendra Vyas, Vaibhav Kulshrestha, Journal of Alloys and Compounds 484 (2009) 463-466
- [14] M. Mozafari, F. Moztarzadeh, M. Tahriri, Advances in Applied Ceramics, 110(2011)1.

