ISSN: 2277-9655
(I2OR), Publication Impact Factor: 3.785

IJESRT

INTERNATIONAL JOURNAL OF ENGINEERING SCIENCES & RESEARCH TECHNOLOGY

SYNTHESIS OF LEAD SELENIDE WITH SURFACTANT (SDS) BY HYDROTHERMAL METHOD

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DOI: 10.5281/zenodo.49721

ABSTRACT

Lead Selenide nano cubes were synthesized by using convenient lead soruce i.e., lead acetatate along with Sodium dodecyl sulfate (SDS) as a surfactant by a simple hydrothermal technique. The as prepared nanoparticles were characterized by XRD, FTIR, I-V studies, and SEM with EDAX to exhibit the phase structure, mode of vibration, conductivity, morphology and the purity of the as prepared PbSe nanoparticles. The crystallite size was calculated using Scherrer's formula. The variation in current with the applied voltage was studied and SEM analysis indicates the morphological structure to be cubic.

KEYWORDS: Lead Selenide, Nano cubes, surfactant/ SDS

INTRODUCTION

The vast study on IV-VI semiconductors are of growing interest due to their narrow bandgap (0.28eV) and its unique physical properties [1]. Since the mobility of the charge carriers of Lead Selenide nano particles is so high [2], PbSe nanoparticles are suitable materials for solar cell [3, 4], photodetector [5], and infrared emitter [6] applications. It has a Bohr exciton radius (46 nm) [7], so that the optical properties can be tuned based on the application's need by controlling the size of the particle. Several solution based methods were carried out to synthesize PbSe nanoparticles such as solvothermal [8], sonochemical [9], photochemical [10], pH induced precipitation [11, 12], microwave-assisted [13–15], continuous flow [16], and hot injection methods [17, 18]. Different types of surfactants (Cationic, anionic, non-ionic, Zwitterionic) were used to control size of a nanoparticle and its distribution [19]. In this paper synthesis of PbSe nanoparticles with the anionic surfactant SDS (Sodium dodecyl sulfate) was adopted and their characterization studies were reported.

EXPERIMENTAL

To synthesize PbSe with SDS, the organic precursor of analytical grade of lead acetate [PbAc], Sodium hydroxide [NaOH], and Sodium dodecyl sulfate [SDS] were obtained from Merck. Sodium Selenide[Na_2SeO_3] and hydrazine hydrate[N_2H_4] acquired from Lobo, Mumbai were used as a precursor and reducing agent respectively. In a typical synthesis 0.6 mole of NaOH was mixed in 100ml of double distilled water under continuous stirring. Simultaneously 0.1 mole of Na_2SeO_3 and 0.1mole of PbAc and 20ml of hydrazine hydrate were added step by step to the above mixture. The SDS was added to the above solution. The as-prepared solution was transferred into a Teflon lined autoclave which was maintained at 200^0C for 20hrs. Next the autoclave was allowed to cool to room temperature naturally. The solution was filtered and the precipitate was washed with double distilled water and ethanol repeatedly in order to remove the impurities. Finally it was vaccum dried at 200^0C for 6hrs. The as-synthesized samples were characterized by XRD, FTIR, SEM, EDAX and conductivity studies.

RESULTS AND DISCUSSION

Fig 1 shows the X-ray diffraction (XRD) pattern of the PbSe nanoparticles with the surfactant SDS. It can be seen that all the peaks are in good agreement with the lead selenide cubic phase of JCPDS #78-1903. The diffraction peaks at 25.17, 29.154, 41.70 and 49.33 can be indexed to the hkl planes (111), (200), (220) and (311) which are the

characteristic peaks of the crystal planes of Lead Selenide.

The lattice constants were thereby calculated and are tabulated below.

	JCPDS#78-1903	Calculated Data
Structure	Cubic	Cubic
a=b =c (Å)	6.121	6.134±0.012

Table 1: Lattice parameters of PbSe with SDS

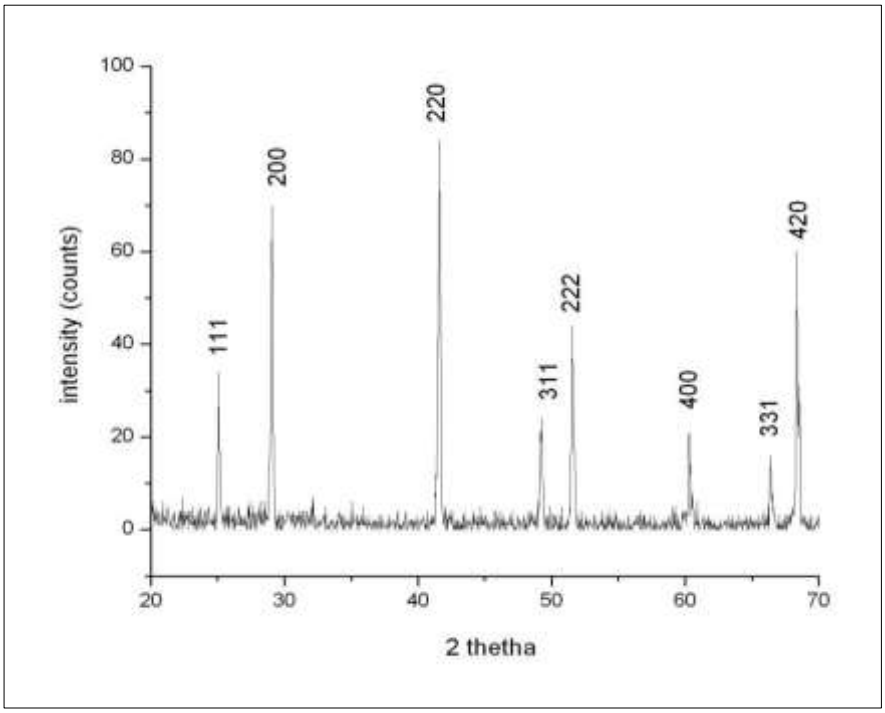

Figure 1: XRD pattern of PbSe with SDS

The average crystallite size of the synthesized sample can be calculated using the Scherrer's formula

$$d = \frac{k\lambda}{\beta \cos\theta}$$

Where
λ is the wavelength of the copper Kα line (1.546 Å)
θ is the diffraction angle
β is the full width at half maximum value
d is the average crystallite size

The average crystallite size of PbSe with SDS 48.3 nm.

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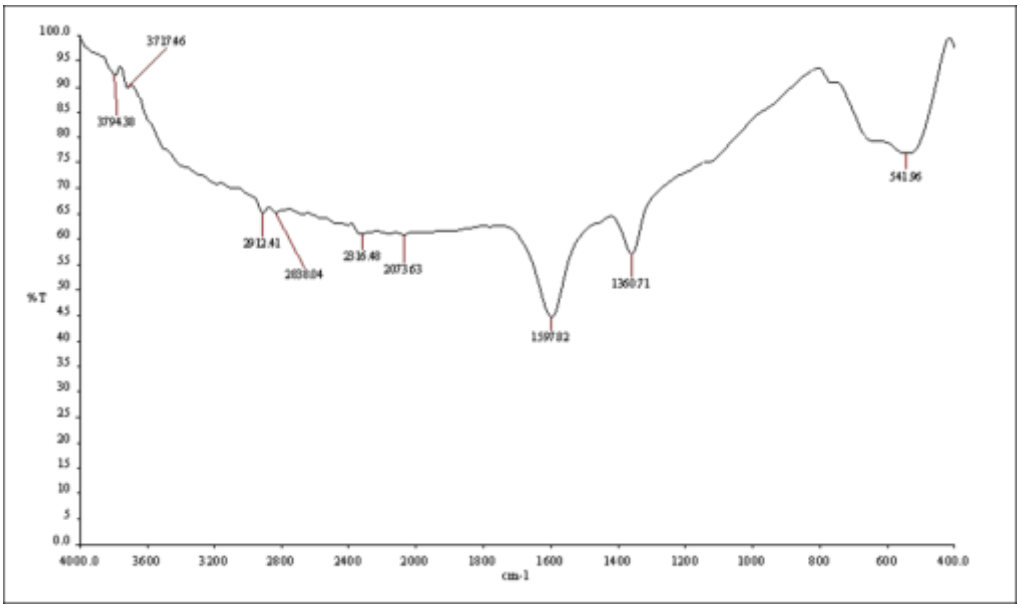

Figure 2: FTIR spectra for Lead Selenide with SDS

The FTIR spectrum of lead selenide synthesized with the surfactant SDS is given in Fig 2. The peak at 1360.71 cm^{-1} can be attributed to the OH bending because of the water molecule and the peak at 541.96 cm^{-1} may be due to the presence of the metal. Other peaks due to the presence of organic elements are also observed. Presence of N–H bending transmission band with the peak at 1597.82 cm^{-1} arises from the use of hydrazine hydrate in the synthesis reaction. The other peaks which are present may be due to other modes of vibration present in the sample.

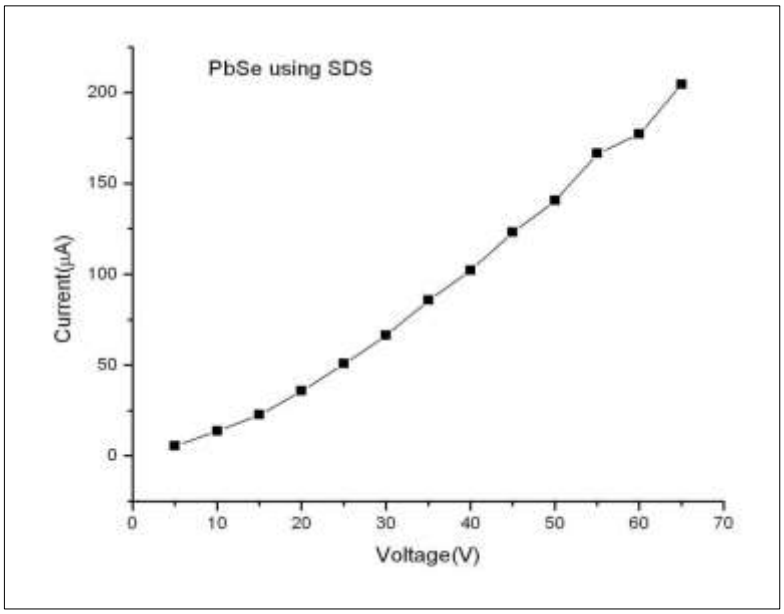

Figure 3: I-V characteristics for Lead Selenide with SDS

The electrical properties of the sample was studied by taking the current–voltage (I–V) measurements of SDS coated PbSe structures and which was recorded using a Keithley electrometer. The I–V characteristic curves are Ohmic i.e

the current increases as the voltage increases. (Fig 3)

SEM results of the synthesized PbSe reveal clusters of cubic shaped structures (Fig 4). Most of PbSe nano-cubes are of uniform size and shows a glazed and flat surface along with regular shape. From the literature survey it's observed that the lead selenide nano particles will preferably grow in a cubic shape because of their cubic symmetry when there are no constraints present [21]. But in the higher magnification PbSe nano-cubes appears like a grain particle. The EDAX analysis reveals the elemental composition of the sample to be 74.73% of Pb and 25.27% of Se. There are no traces of any impurity in the as-prepared sample (Fig 5).

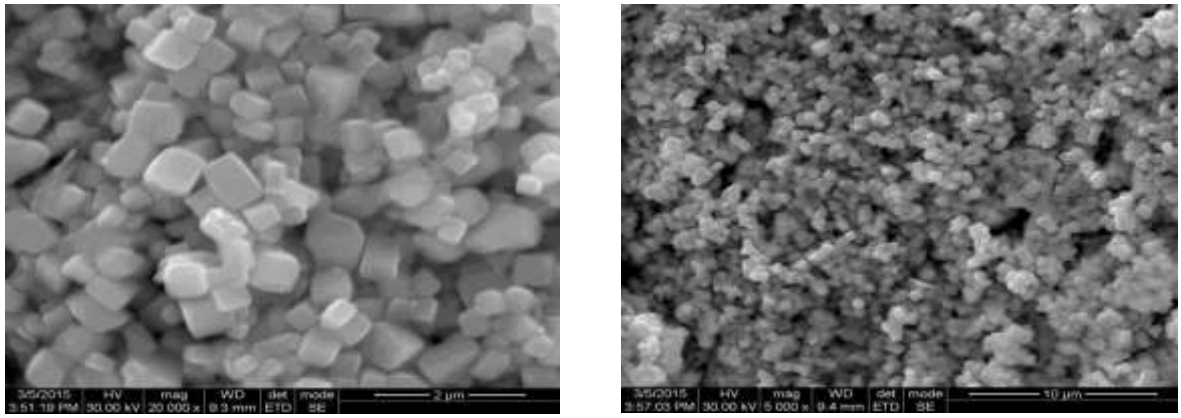

Figure 4: SEM micrographs of Lead Selenide with SDS

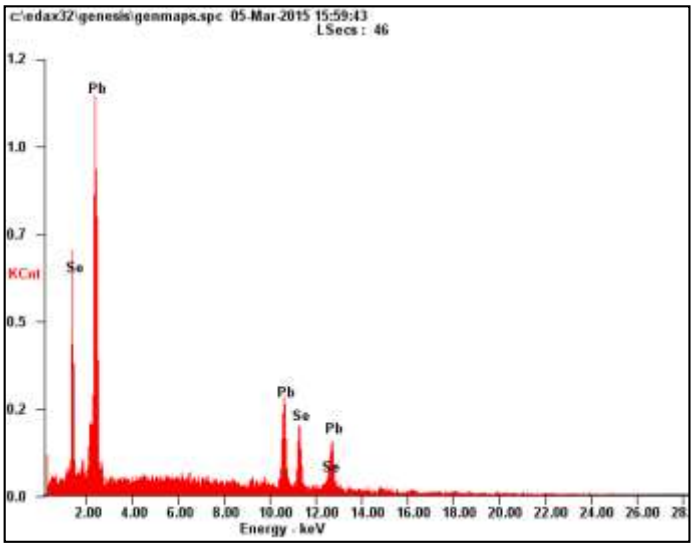

Figure 5: EDAX spectrum of Lead Selenide with SDS

CONCLUSION

A simple hydrothermal technique was adopted to synthesize lead selenide nanocubes with the surfactant SDS. The XRD results revealed that PbSe exists in the cubic structure and the crystallite size was calculated. By the FTIR studies the presence of various modes of vibration of a molecule was observed. The result of I-V studies help to understand the Ohmic behavior of the as prepared sample. The SEM images show the cubic shape with regular morphology of the PbSe. The EDAX spectrum confirms composition of element and shows that there are no other

impurities present in the PbSe nanoparticles.

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