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Synthesis and Characterization of Lead II Sulphide Nanoparticles

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Abstract: *Lead II Sulphide nanoparticles were synthesized via chemical co-precipitation method from lead (II) nitrate and sodium sulphide. The formed nanoparticle is characterized by powder x-ray diffraction, scanning electron microscopy, ultra-violet spectroscopy and fourier transform infrared spectroscopy, confirmed the preferential growth of lead II sulphide nanoparticles that width is 28 nm. The SEM image shows the synthesized lead II sulphide nanoparticles with spherical morphology. The FTIR spectrum is used to study the stretching and bending frequencies of molecular functional groups in the sample. From UV spectrum, the band gap of lead II sulphide nanoparticles is found to be 3.5 eV.*

Keywords *- XRD, SEM, FTIR, UV*

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I. Introduction

Semiconductor nanoparticles have good optical and electronic properties, and there has been much interest in the synthesis and characterization of sulphide nanoparticles. Lead sulphide is an important IV-VI group semiconductor, which has attracted much attention because of its special small direct-band gap (0.41 eV) and large excitonic Bohr radius of 18 nm [1]. It has high dielectric constant and very high carrier mobility. Hence PbS nanoparticles (NPs) show strong quantum size effects for relatively large size [2]. Lead (II) sulphide (PbS) has attracted much attention for its well-known narrow band gap semiconducting features. PbS is chosen for this work because it is commercially available and can also be synthesized easily. A variety of chemical and physical methods were developed to prepare PbS. In the present work we have prepared PbS nanoparticles by chemical Co-precipitation method. The lead sulphide nanoparticles have wide applications in many fields such as solar cells, solar absorbers, photographs, lasers, LED devices, telecommunications, detectors, optical switches, optical amplification, and also as gas- sensing agents in the solid - state sensors [1]. Lead sulphide has several industrial applications. It is used in infrared detectors, transistors, photoconductive cells, high temperature lubricants and for glazing earthenware. It also is used as a catalyst in petroleum refining for removal of mercaptans from petroleum distillates.

II. Materials And Methods

Nanoparticles of lead II sulphide were prepared by chemical co precipitation method by adding lead (II) nitrate and sodium sulphide. Precise amounts of reagents taking into account their purity were weighed and dissolved separately in distilled water into 0.1M concentration. After obtaining a homogeneous solution, the reagents were mixed using magnetic stirring. The precipitate was separated from the reaction mixture and washed several times with distilled water and ethanol. The wet precipitate was dried and thoroughly ground using agate mortar to obtain the samples in the form of fine powder [3].

III. Tests Conducted

X-ray diffraction is an ideal technique for the determination of crystallite size of the powder samples. The basic principle for such a determination involves precise quantification of the broadening of the peaks. XRD line broadening method of particle size estimation was chosen in this investigation for determining the crystallite size of the powder sample. The morphology of the powder samples was studied by the scanning electron microscope (SEM) analysis. The infra red spectroscopic (IR) studies of lead II sulphide nanoparticles were made by using 'SHIMADZU' FTIR 8400S model spectrometer through KBr method. The UV spectrum was taken in the absorbance mode in the wavelength range from 200 to 800 nm.

IV. Results And Discussion

XRD Studies

XRD – Particle Size Calculation

The XRD patterns of the prepared samples of lead II sulphide nanoparticles are shown in figure.1. XRD studies reveal that the samples are nano sized and crystalline. The fine particle nature of the samples is reflected in the X-ray line broadening. The size of the synthesized lead II sulphide nanoparticles are calculated using Scherrer equation

$$\mathbf{D = 0.9\ \lambda / \beta\ cos\theta} \qquad (1)$$

where λ represents wavelength of X rays, β represents half width at full maximum and θ is the diffraction angle [4]. The average grain size of the particles is found to be 28 nm. The XRD pattern of lead II sulphide nanoparticles is shown in fig.1.

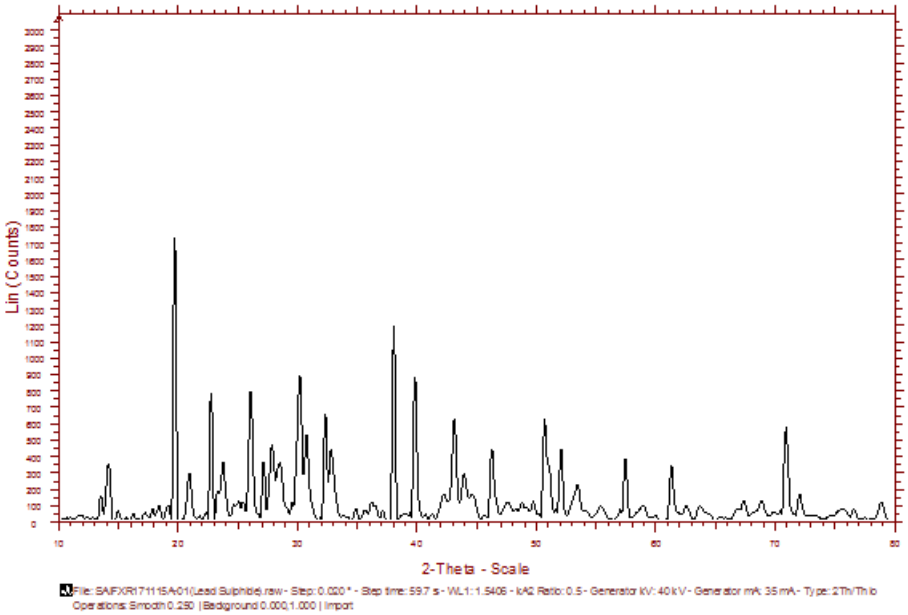

Fig .1. XRD pattern of lead II sulphide nanoparticles.

A good agreement between the Experimental diffraction angle [2θ] and Standard diffraction angle [2θ] of specimen is confirming standard of the specimen. Many peaks at 2θ values of Lead sulphide is observed and tabulated in table.1 and compared with the standard powder diffraction card of Joint Committee on Powder Diffraction Standards (JCPDS), Lead II sulphide file No. 65-0157. The d-spacing values of experimental is also confirming to the standard values.

Table.1. Experimental and standard diffraction angles of lead II sulphide nanoparticles.

Experimental		**Standard – JCPDS 65-0157**	
Diffraction angle (2θ in degrees)	**D spacing (Å)**	**Diffraction angle (2θ in degrees)**	**D spacing (Å)**
26.016	3.42275	25.987	3.4260
30.143	2.76162	30.095	2.9670
43.122	2.09626	43.081	2.0979
70.992	1.32659	70.976	1.3268

XRD – Dislocation Density

The dislocation density is defined as the length of dislocation lines per unit volume of the crystal. In materials science, a dislocation is a crystallographic defect, or irregularity, within a crystal structure. The presence of dislocations strongly influences many of the properties of materials. The movement of a dislocation is impeded by other dislocations present in the sample. Thus, a larger dislocation density implies a larger hardness. The X-ray line profile analysis has been used to determine the dislocation density. The dislocation density can be calculated from equation

$$\delta = \frac{1}{D^2}$$

Where δ is dislocation density and D is the crystallite size. Results of the dislocation density calculated from the formula is given in table.2. The number of unit cell is calculated from equation

$$n = \pi\,(4/3) \times (D/2)^3 \times (1/V)$$

Where D is the crystallite size and V is the cell volume of the sample.

Table .2. Dislocation Density and Number of Unit Cell from XRD of lead II sulphide nanoparticles.

2θ (deg)	Particle Size D (nm)	Dislocation Density (m^2) x10^{15} $\delta = 1 / D^2$	Number of Unit Cell X10^5
19.628	27.51910145	1.32048E+15	0.521962
20.853	21.42833154	2.17783E+15	0.246434
23.692	19.42090161	2.65131E+15	0.183461
26.016	24.12503288	1.71816E+15	0.351674
27.101	32.04895856	9.73581E+14	0.824476
30.143	22.66656307	1.94638E+15	0.291671
32.287	36.1180577	7.66569E+14	1.180074
38.037	28.01159852	1.27445E+15	0.55049
39.819	25.99920966	1.47938E+15	0.440166
43.122	23.861341	1.75635E+15	0.340268
46.278	26.02271894	1.47671E+15	0.441361
50.721	18.74545762	2.84582E+15	0.164977
52.093	32.27145304	9.60203E+14	0.841766
57.502	30.00395093	1.11082E+15	0.676506
61.365	35.2559389	8.04517E+14	1.097572
70.992	26.73057608	1.39953E+15	0.478367

It is observed from these tabulated details, and from figure.2, figure.3 and figure.4, dislocation density is indirectly proportional to particle size and number of unit cells. Dislocation density increases while both particle size and number of unit cell decreases.

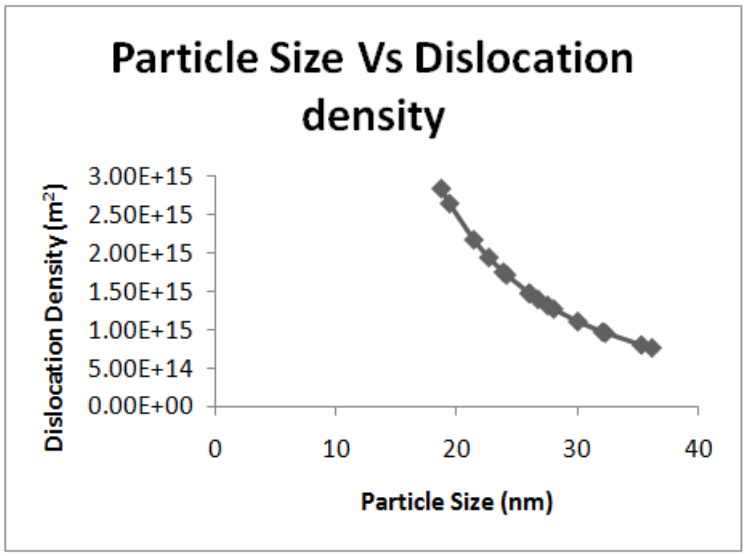

Fig.2. Particle size Vs Dislocation density curve of lead II sulphide nanoparticles.

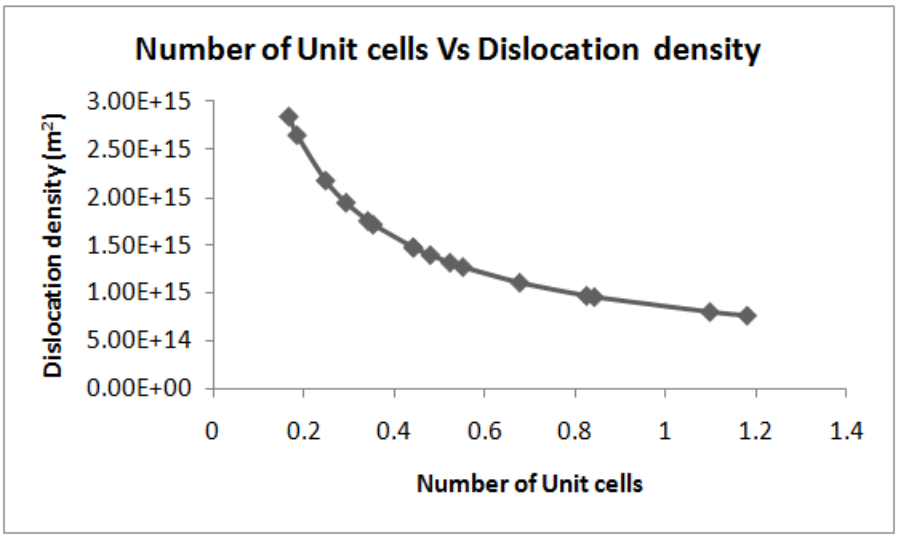

Fig.3. Number of Unit cells Vs Dislocation density curve of lead II sulphide nanoparticles.

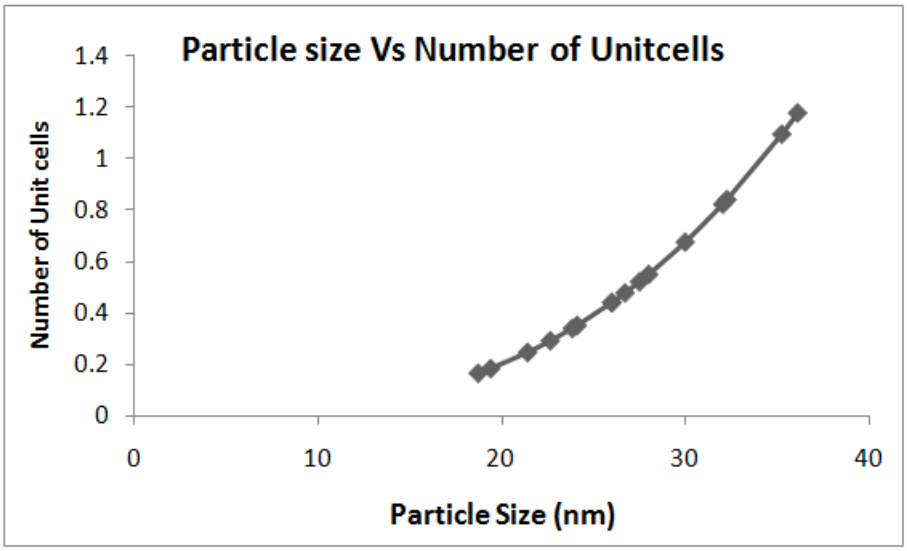

Figure.4. Particle Size Vs Number of Unit cells curve of lead II sulphide nanoparticles.

XRD – Morphology Index

A XRD morphology index (MI) is calculated from FWHM of XRD data using the relation

$$M.I = \frac{FWHM_h}{FWHM_h + FWHM_p}$$

Where M.I. is morphology index, $FWHM_h$ is highest FWHM value obtained from peaks and $FWHM_p$ is value of particular peak's FWHM for which M.I. is to be calculated. The relation between morphology index and particle size is shown in table .3.

Table .3. Relation between Morphology Index and Particle sizefor lead II sulphide nanoparticles.

FWHM (β) radians	Particle Size(D) nm	Morphology Index (unitless)
0.005111	27.51910145	0.499989
0.006577	21.42833154	0.437303
0.007292	19.42090161	0.412085
0.005896	24.12503288	0.464331
0.004448	32.04895856	0.534661
0.006332	22.66656307	0.446636
0.003995	36.1180577	0.561292

0.005233	28.01159852	0.494087
0.005669	25.99920966	0.474099
0.006245	23.861341	0.450066
0.005792	26.02271894	0.468789
0.008181	18.74545762	0.384504
0.00478	32.27145304	0.516744
0.005268	30.00395093	0.492426
0.00457	35.2559389	0.527917
0.006367	26.73057608	0.445278

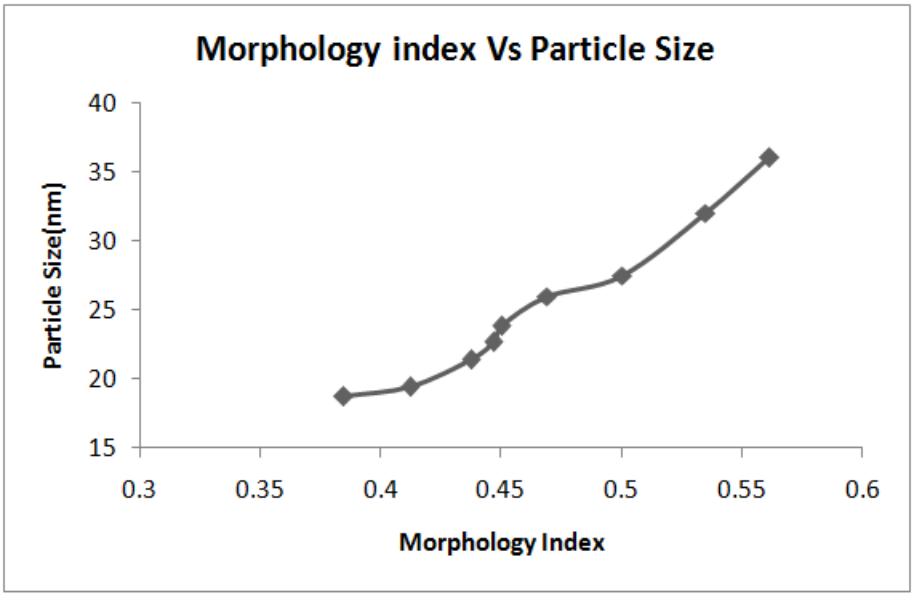

Fig .5. Morphology Index of lead II sulphide nanoparticles.

It is observed that MI has direct relationship with particle size and the results are shown in Figure .5.

XRD – Unit Cell Parameters

Unit cell parameters values calculated from XRD are enumerated in table .4.

Table .4. XRD Parameters of Lead II Sulphide Nanoparticles.

Parameters	Values
Structure	Face Centered
Space group	Fm3m [225]
Symmetry of lattice	Cubic
Particle size	30.8 nm
Lattice parameters	a=5.934;b=---;c=---
Vol.unit cell(V)	208.95
Density (ρ)	7.606
Dislocation Density	1.32048×10^{15}
Mass	239.26amu

V. Sem Studies

Scanning electron microscopy was used to analyze the morphology and size of the synthesized lead II sulphide nanoparticles. Figure.6, shows the SEM images of the lead II sulphide nanoparticles at various magnifications. The SEM images of lead II sulphide nanoparticles show nanoparticles with spherical shape. In this case the particles sizes are slightly increased and is also observed that the particles are distributed with agglomeration.

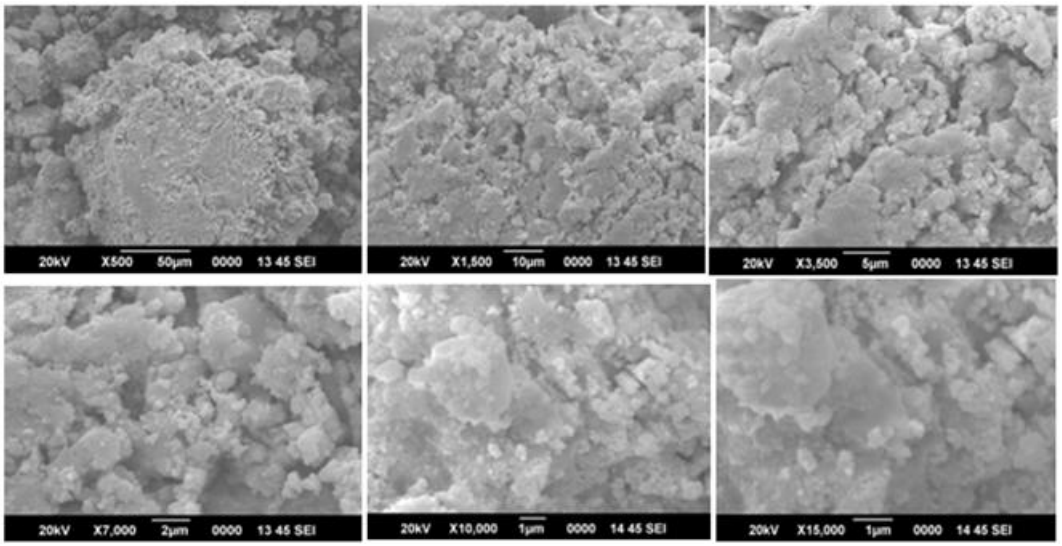

Fig.6. SEM images of Lead Sulphide Nanoparticles at various magnifications.

VI. Ftir Studies

The FTIR spectrum of the lead II sulphide sample is shown in the figure.7.The FTIR spectrum for lead II sulphide nanoparticles show peak at 3448.65 cm^{-1} corresponds to the free O-H group [5] and the peaks at 1764.40 cm^{-1} and 1624.57 cm^{-1} are due to the presence of hydroxyl group of water. The peaks at 879.30 cm^{-1} and 829.49 cm^{-1} indicates the Pb-S band [6]. The peak at 624.79 cm^{-1} is due to S-S bond and the peak at 477.29 cm^{-1} is due to the presence of Lead.

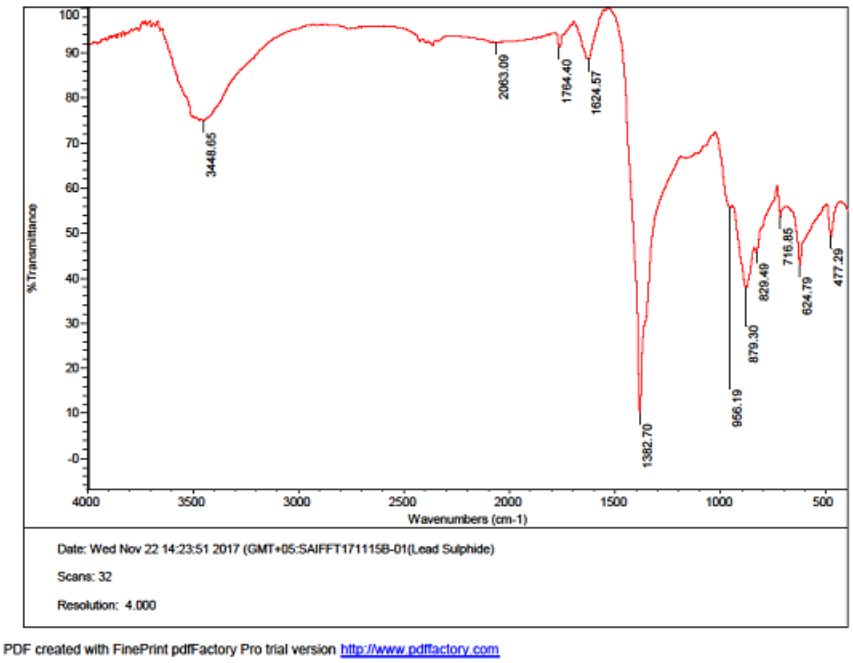

Fig.7. FTIR spectra of lead II sulphide nanoparticles.

VII. Uv Studies

The band gap of the prepared sample lead II sulphide was determined by using UV visible studies. Figure.8 shows the UV-Visible absorption spectra for lead II sulphide nanoparticles and the maximum absorption is at 275 nm wavelength. Figure.9 shows the graph to find the band gap of lead II sulphide nanoparticles. From the graph, the optical band gap of lead II sulphide is 3.5 eV.

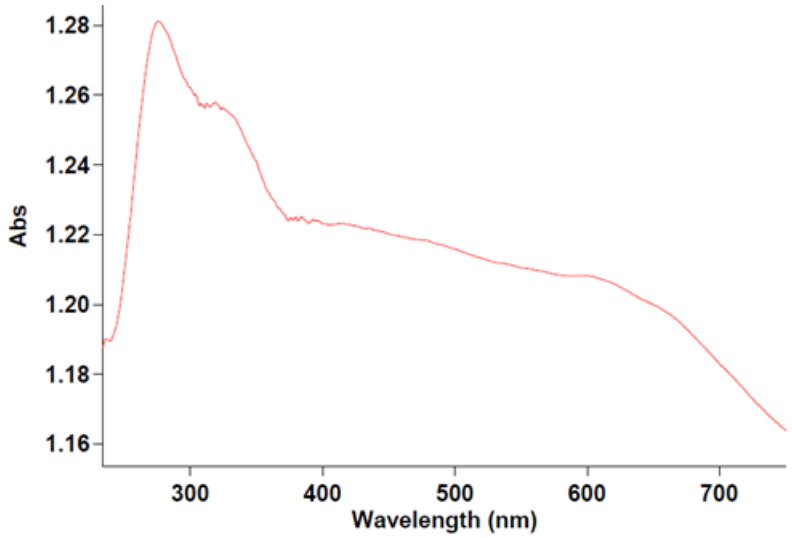

Fig.8. UV-Visible absorption spectra of lead II sulphide nanoparticles.

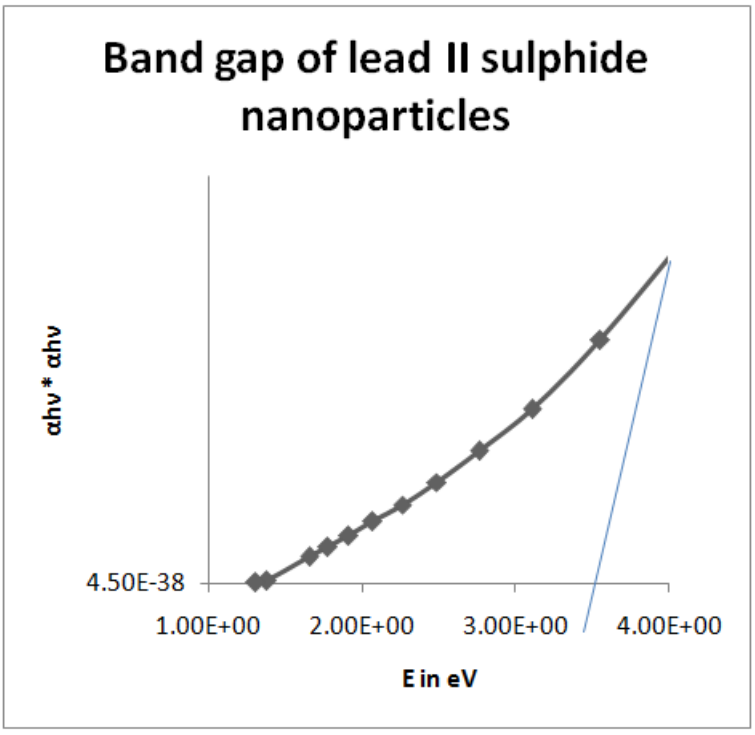

Fig.9. Graph to find the band gap of lead II sulphide nanoparticles.

VIII. Conclusions

The **l**ead II sulphide nanoparticles have been prepared by chemical co-precipitation method. XRD analysis suggests that the average particle size is in the nano range (28nm). The SEM picture reveals the nanoparticles with spherical morphology. From the FTIR spectrum, the stretching and bending frequencies of the molecular functional groups in the sample are studied. From the UV spectra, the band gap was found.

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