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Website: www.aarf.asia Email : editor@aarf.asia , editoraarf@gmail.com

SYNTHESIS AND CHARACTERIZATION OF LEAD II IODIDE NANOPARTICLES

R. Hepzi Pramila Devamani[1] , A. Akila[2] & M.Indhumathi[2]

[1.] Assistant Professor, Department of Physics, V.V.Vanniaperumal College for Women,

[2.] M.Sc Students, Department of Physics, V.V.Vanniaperumal College for Women,

Virudhunagar.

ABSTRACT

Lead II Iodide nanoparticles were synthesized via chemical co-precipitation method from lead (II) nitrate and sodium iodide. 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 iodide nanoparticles that width is 30.8 nm. The SEM image shows the synthesized lead II iodide show well crystallized particles 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 iodide nanoparticles is found to be 3.5eV.

KEYWORDS: XRD, SEM, FTIR, UV.

INTRODUCTION

Nanotechnology is concerned with synthesis of nanoparticles of various size, shape and chemical composition and their potential use. Nanomaterials have created high interest in recent years by virtue of their unusual mechanical, electrical, optical and magnetic properties. Lead nanostructures are attractive materials for its applications such as lead batteries and catalysis. Lead II iodide is used for recording optical images; for making gold spangles and mosaic gold for decorative purposes; in photographic emulsions; in mercury-vapor lamps; in asbestos brake linings; in far-infrared filters; in thermal batteries; in printing and recording papers; and in aerosols for cloud seeding. This paper deals with easy, simple, fast and low

cost synthesis of lead II iodide nanoparticles by chemical co-precipitation method and its characterizations.

MATERIALS AND METHODS

Nanoparticles of lead II iodide were prepared by chemical co precipitation method by adding lead (II) nitrate and sodium iodide. 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 [1].

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 iodide 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.

RESULTS AND DISCUSSION

XRD STUDIES

XRD – PARTICLE SIZE CALCULATION

The XRD patterns of the prepared samples of lead II iodide 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 iodide 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[2]. The average grain size of the particles is found to be 30.8 nm. The XRD pattern of lead II iodide nanoparticles is shown in figure 1.

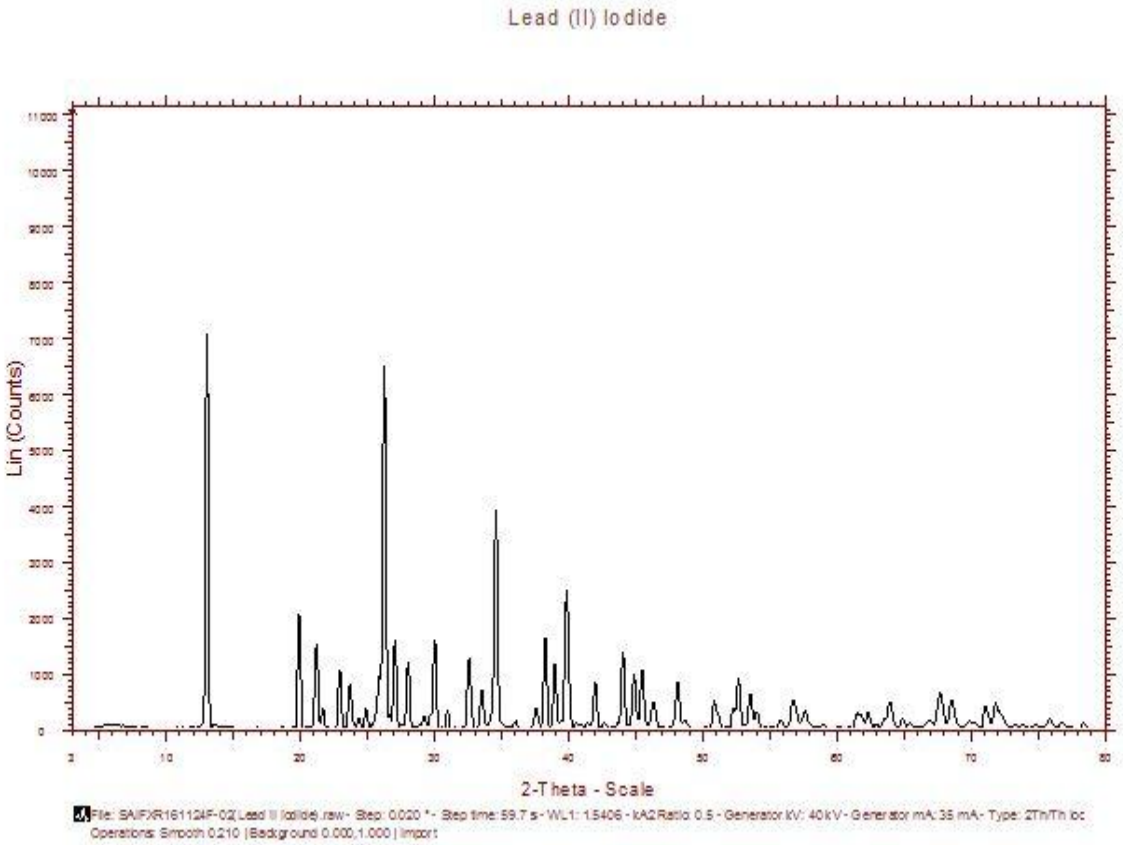

Figure .1. XRD pattern of lead II iodide nanoparticles.

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 iodide 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 iodide file No. 89-1974. The d-spacing values of experimental is also confirming to the standard values.

Experimental		Standard – JCPDS 89-1974	
Diffraction angle (2θ in degrees)	D spacing (Å)	Diffraction angle (2θ in degrees)	D spacing (Å)
12.945	6.833	12.674	6.979

19.063	4.652	19.060	4.653
22.87	3.885	22.791	4.653
24.837	3.582	24.624	3.613
25.492	3.491	25.568	3.481
26.193	3.399	26.278	3.388
28.353	3.145	28.741	3.104
32.538	2.749	32.135	2.783
34.543	2.594	34.829	2.573
35.982	2.494	35.391	2.534
38.611	2.329	38.674	2.326
39.414	2.284	39.519	2.278
41.381	2.18	41.665	2.119
43.273	2.089	43.907	2.060
46.984	1.932	46.96	1.933
47.169	1.925	47.119	1.927
47.578	1.909	47.663	1.906
49.395	1.843	49.593	1.837
52.305	1.747	52.399	1.744
58.571	1.574	58.462	1.577

64.931	1.435	64.732	1.439
66.82	1.398	66.774	1.399
66.929	1.396	66.991	1.395
67.495	1.386	67.569	1.385
73.149	1.293	73.149	1.293

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

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.

2θ (deg)	Particle Size	Dislocation Density (m^2) x10^{15}	Number of Unit Cell

	D (nm)	$\delta = 1 / D^2$	$X10^5$
12.947	30.79	1.054	3.648
19.826	30.56	1.071	3.568
26.193	29.11	1.180	3.083
34.543	28.08	1.268	2.767
39.824	24.22	1.705	1.796

Table .2. Dislocation Density and Number of Unit Cell

from XRD of lead II iodide nanoparticles.

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.

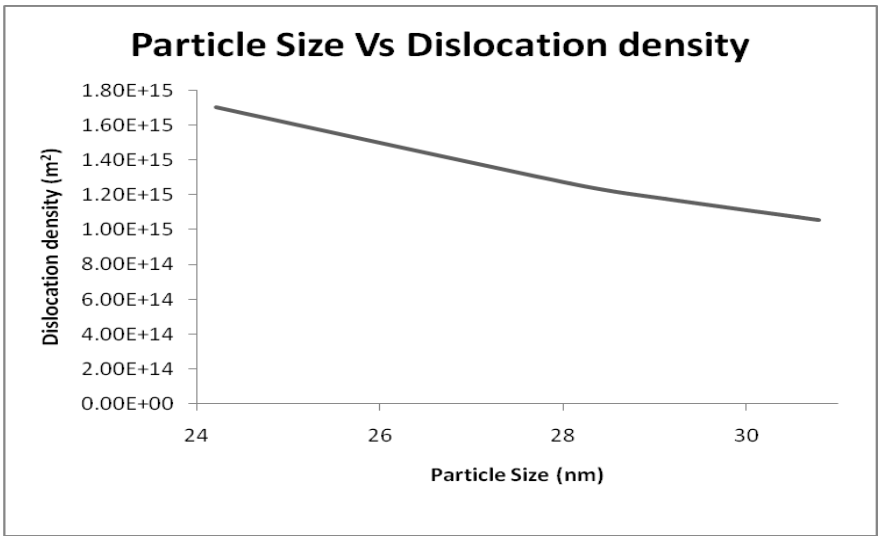

Figure.2. Particle size Vs Dislocation density curve of lead II iodide nanoparticles.

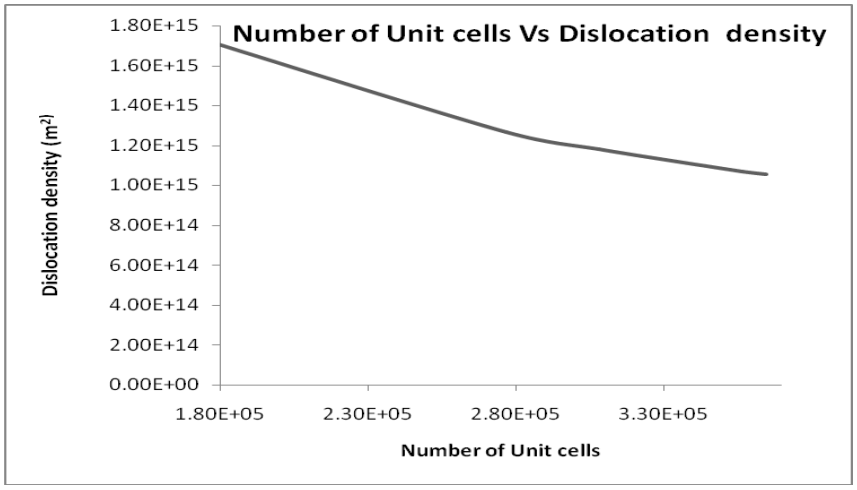

Figure.3. Number of Unit cells Vs Dislocation density curve of lead II iodide nanoparticles.

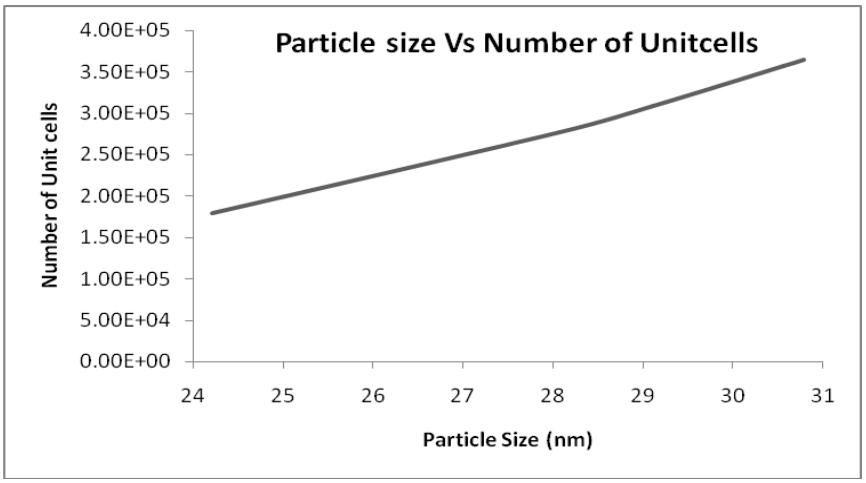

Figure.4. Particle Size Vs Number of Unit cells curve of lead II iodide nanoparticles.

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.

FWHM (β) radians	Particle Size(D) nm	Morphology Index (unitless)
0.004506	30.79	0.5
0.004535	30.56	0.4984
0.004268	29.11	0.4362
0.004936	28.08	0.4772
0.005722	24.22	0.4405

Table .3. Relation between Morphology Index and Particle size for lead II iodide nanoparticles.

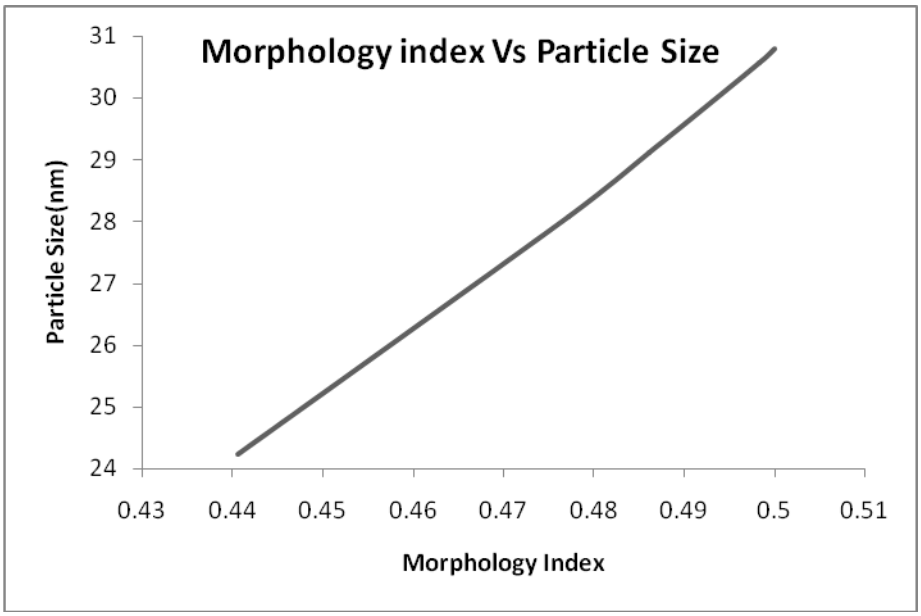

Figure .5. Morphology Index of lead II iodide nanoparticles.

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.

Parameters	Values
Structure	Rhombohedral
Space group	R3m (166)
Symmetry of lattice	Rhomb-centered
Particle size	30.8 nm
Lattice parameters	a=4.557;b=---;c=125.62
Vol.unit cell(V)	2259.2
Density (ρ)	6.099
Dislocation Density	$1.054x10^{15}$
Mass	461.01amu

Table .4. XRD parameters of lead II iodide nanoparticles.

SEM STUDIES

Scanning electron microscopy was used to analyze the morphology and size of the synthesized lead II iodide nanoparticles. Figure.6, Figure.7, Figure.8 and Figure.9 show the SEM images of the lead II iodide nanoparticles at various magnifications. The SEM images of lead II iodide nanoparticles show well crystallized particles with spherical shape. In this case the particles sizes are slightly increased and is also observed that the particles are distributed with agglomeration.

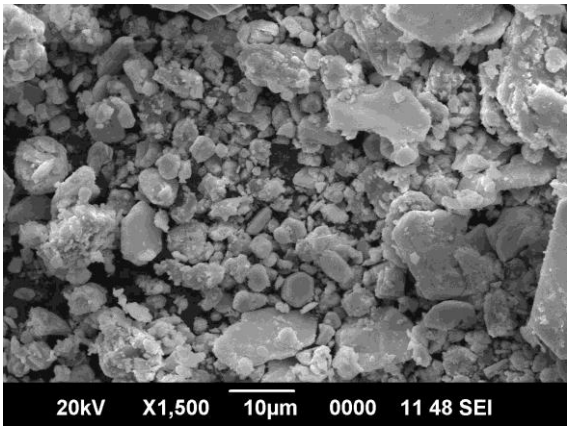

Figure.6. SEM image at 1500 magnifications.

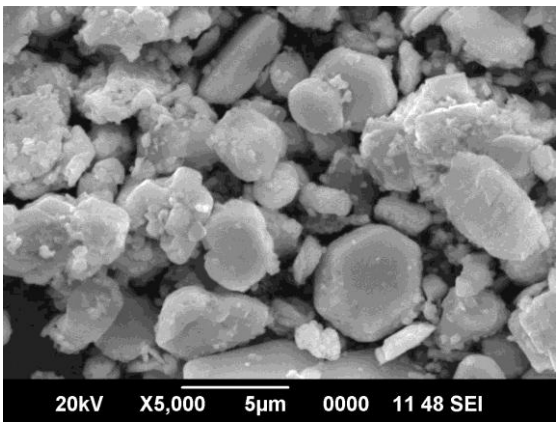

Figure.7. SEM image at 5000 magnifications.

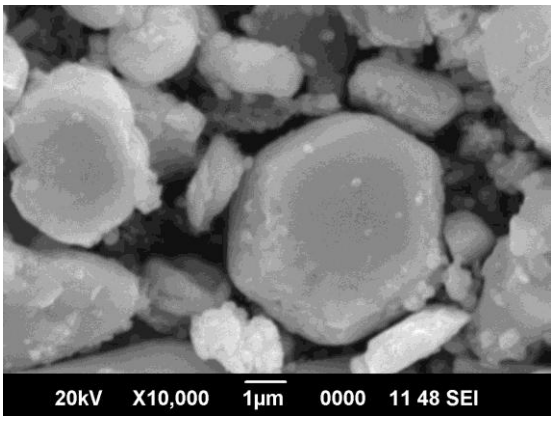

Figure.8. SEM image at 10000 magnifications.

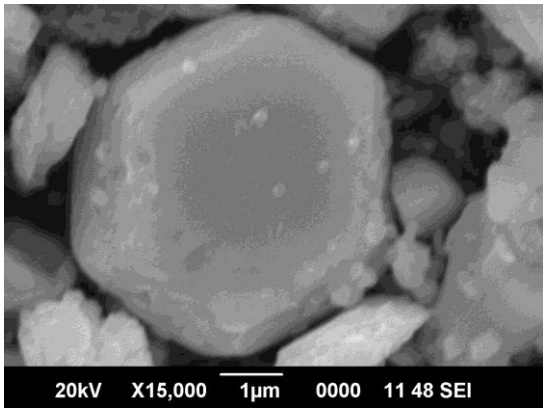

Figure.9. SEM image at 15000 magnifications.

FTIR STUDIES

The FTIR spectrum of the lead II iodide sample is shown in the figure.10.The FTIR spectrum for lead II iodide nanoparticles show peak at 3434.82 corresponds to the free O-H group [3] and the peak at 1159.66 cm^{-1} is due to the presence of lead [4] and the peaks at 714.42 cm^{-1} and 593.26 cm^{-1} are due to Pb-I bond.

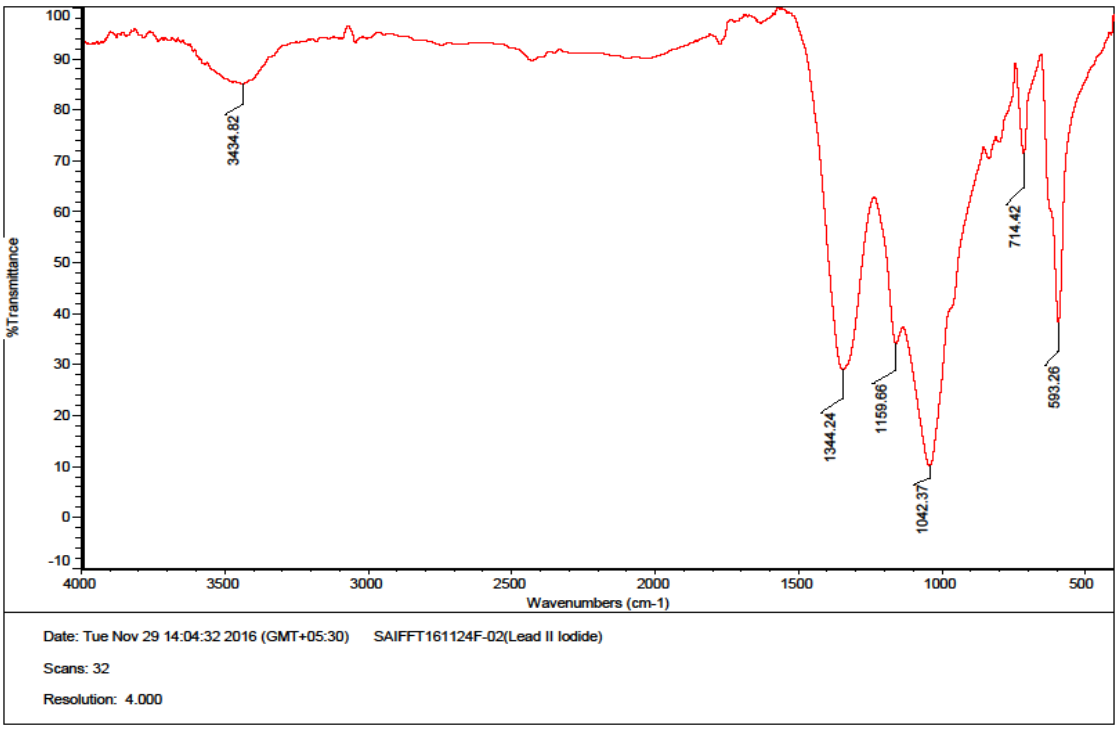

Figure.10. FTIR spectra of lead II iodide nanoparticles.

UV STUDIES

The band gap of the prepared sample lead II iodide was determined by using UV visible studies.. Figure.11 shows the graph to find the band gap of lead II iodide nanoparticles. From the graph, the optical band gap of lead II iodide is 3.5 eV.

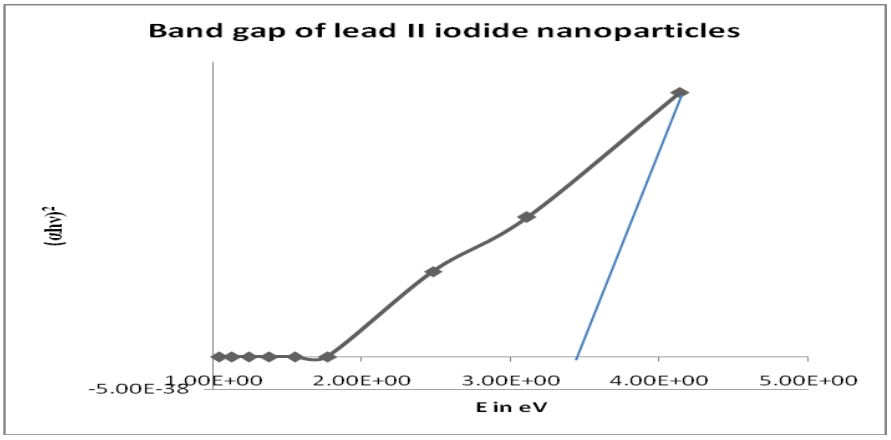

Figure.11. Graph to find the band gap of lead II iodide nanoparticles.

CONCLUSIONS

The **l**ead II iodide nanoparticles have been prepared by chemical co-precipitation method. XRD analysis suggests that the average particle size is in the nano range (30.8nm). The SEM picture reveals the well crystallized particles 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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