INTERNATIONAL JOURNAL OF CURRENT RESEARCH IN CHEMISTRY AND PHARMACEUTICAL SCIENCES

(p-ISSN: 2348-5213: e-ISSN: 2348-5221)
www.ijcrcps.com

Research Article

SYNTHESIS AND CHARACTERIZATION OF LEAD CHLORIDE NANOPARTICLES

R HEPZI PRAMILA DEVAMANI[1*], S.ALAGURANI[2] AND S.KANIMOZHI[3]

[1]Post Graduate Department of physics, V.V. Vanniaperumal College for Women, Virudhunagar, Tamil Nadu, India
[2&3] M.Sc Students, Post Graduate Department of physics, V.V. Vanniaperumal College for Women, Virudhunagar.Tamil Nadu, India

Corresponding Author: hepzi.justin2001@gmail.com

Abstract

Lead chloride nanoparticles were synthesized via chemical co-precipitation method from lead nitrate and sodium chloride. Structural and compositional properties were characterized by XRD, SEM, FTIR and UV spectroscopy. X-ray diffraction (XRD) confirmed the preferential growth of lead chloride nanoparticles that width is 80.03nm. The SEM image shows the synthesized lead chloride show well crystallized particles with spherical like 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 chloride nanoparticles is found.

Keywords: XRD, SEM, FTIR, UV.

Introduction

Nanoparticles have one dimension that measures 100 nanometers or less. Nanoparticles have a greater surface area per weight than larger particles which causes them to be more reactive to some other molecules. Nanoparticle research is currently an area of intense scientific interest due to a wide variety of potential applications in biomedical, optical and electronic fields. Nanoparticles are of great scientific interest as they are effectively a bridge between bulk materials and atomic or molecular structures. A bulk material should have constant physical properties regardless of its size, but at the nano-scale size-dependent properties are often observed. Thus, the properties of materials change as their size approaches the nanoscale and as the percentage of atoms at the surface of a material becomes significant. For bulk materials larger than one micrometer (or micron), the percentage of atoms at the surface is insignificant in relation to the number of atoms in the bulk of the material. The interesting and sometimes unexpected properties of nanoparticles are therefore largely due to the large surface area of the material, which dominates the contributions made by the small bulk of the material [1].

Lead chloride is used in making many basic chlorides, such as Pattison's lead white, Turner's Patent Yellow, and Verona Yellow, used as pigments. Also, it is used as a flux for galvanizing steel; as a flame retardant in nylon wire coatings; as a cathode for seawater batteries; to remove H2S and ozone from effluent gases; as a sterilization indicator; as a polymerization catalyst for alphaolefins; and as a co-catalyst in manufacturing acrylonitrile.

This paper is discussing about easy, simple and low cost preparation i.e. chemical co precipitation of Lead chloride nanoparticles and its characterizations – XRD, SEM, FTIR and UV.

Materials and Methods

Nano particles of Lead chloride were prepared by chemical co-precipitation method by adding lead nitrate

and sodium chloride. 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.

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. XRD study of the powder samples was carried out at Alagappa University, Karaikudi. The morphology of the powder samples was studied by the scanning electron microscope (SEM) analysis taken at STIC Cochin. The infra red spectroscopic (IR) studies of lead chloride 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 chloride are shown in fig.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 chloride nanoparticles are calculated using Scherrer equation

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

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 80.03nm. The peak list in the XRD pattern is given in table-1.

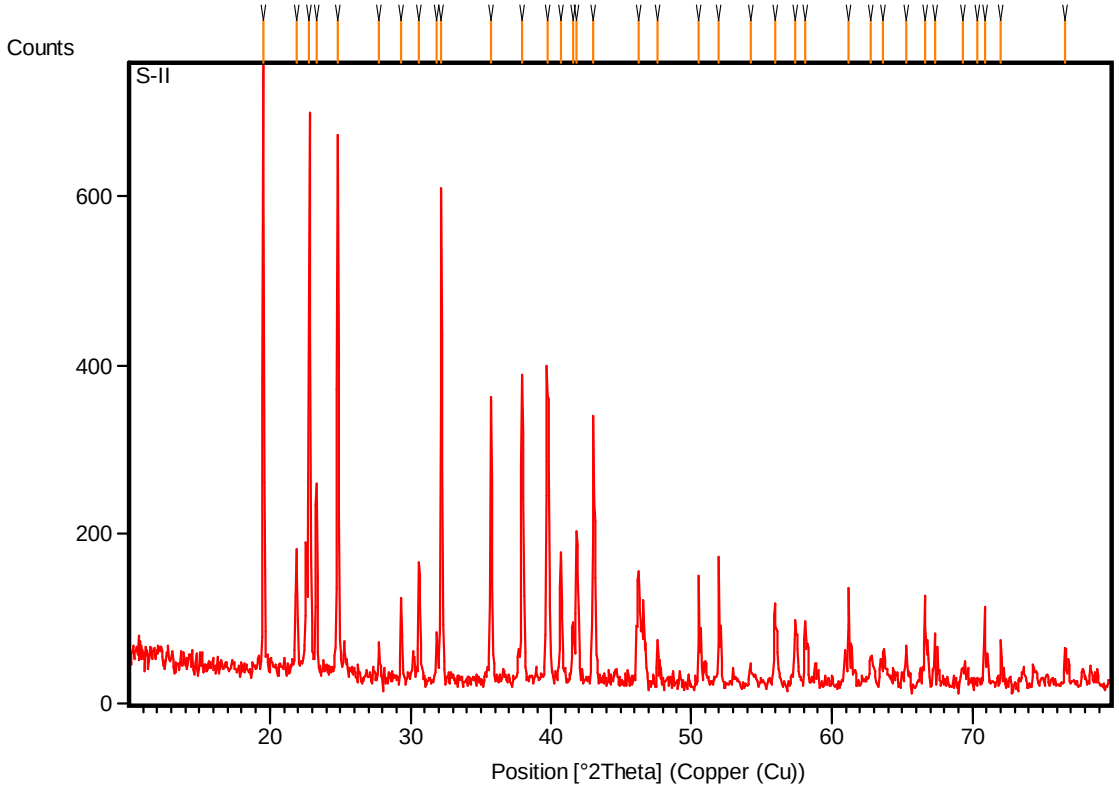

Fig.1 XRD pattern of lead chloride nanoparticles.

Pos.[°2Th]	Height[cts]	FWHM[° 2Th]	d-spacing [A °]	Rel.Int[%]
19.527	592	0.101	4.54231	100
23.290	180	0.10	3.81622	30.32
24.813	505	0.10	3.58534	85.26
27.75	24	0.10	3.21192	4.02
31.848	55	0.07	2.80762	9.26
32.167	545	0.094	2.78050	92.08
37.920	355	0.11	2.37085	59.99
39.705	323	0.17	2.26826	54.54
40.656	42	0.10	2.21734	24.01
41.597	69	0.1	2.16934	11.66
43.072	298	0.11	2.09842	50.37
47.640	51	0.07	1.90731	8.59
51.978	145	0.07	1.75787	24.49
54.244	36	0.07	1.68967	6.03
57.406	76	0.14	1.60389	1287
58.128	74	0.12	1.58567	12.45
61.215	111	0.09	1.51289	18.69
62.79	25	0.2	1.47873	4.22
63.66	22	0.4	1.46050	3.65
65.29	35	0.14	1.42798	5.9
66.634	95	0.11	1.40240	16.04
67.331	54	0.09	1.38957	9.18
69.34	14	0.3	1.35410	2.43
70.873	81	0.1	1.32856	13.64
72.036	39	0.1	1.30995	6.64

Table-1. Intensity of XRD peaks.

A good agreement between the Experimental diffraction angle [2θ] and Standard diffraction angle [2θ] of specimen is confirming standard of the specimen. Twenty five peaks at 2θ values of lead chloride is observed and tabulated in table-2 and compared with the standard powder diffraction card of Joint Committee on Powder Diffraction Standards (JCPDS), lead chloride file No. 86-1130. The d-spacing values of experimental is also confirming to the standard values.

Experimental		Standard – JCPDS 86-1130	
Diffraction angle (2θ in degrees)	**D spacing (Å)**	**Diffraction angle (2θ in degrees)**	**D spacing (Å)**
20.39	4.54231	20.4	4.35
23.290	3.81622	23.097	3.8476
24.813	3.58534	24.097	3.6902
27.75	3.21192	27.550	3.2351
31.848	2.80762	31.965	2.7976
32.167	2.78050	32.905	2.7198
37.920	2.37085	37.400	2.4025
39.705	2.26826	39.648	2.2714
40.656	2.21734	40.432	2.2291
41.597	2.16934	41.484	2.1750
43.072	2.09842	43.555	2.0762
47.640	1.90731	47.207	1.9238
51.978	1.75787	51.047	1.7877
54.244	1.68967	54.296	1.6881
57.406	1.60389	57.074	1.6124

58.128	1.58567	58.063	1.5873
61.215	1.51289	61.186	1.5135
62.79	1.47873	62.645	1.4817
63.66	1.46050	63.523	1.4633
65.29	1.42798	65.724	1.4196
66.634	1.40240	66.827	1.3988
67.331	1.38957	69.437	1.3524
69.34	1.35410	70.254	1.3387
70.873	1.32856	70.938	1.3275
72.036	1.30995	72.165	1.3079

Table.2. Experimental and standard diffraction angles of lead chloride specimen.

XRD - Expected 2θ Positions

The value of d (the interplanar spacing between the atoms) is calculated using Bragg's Law:
2d sin θ = n λ

$$d = \frac{\lambda}{2 \sin \theta} \quad (n = 1)$$

Wavelength λ = 1.5418 Å for Cu Kα

The expected 2θ positions of all the peaks in the diffraction pattern and the interplanar Spacing d for each peak is calculated using following formula and the details are shown in table-3.

$$\frac{1}{d2} = \frac{h2}{a2} + \frac{k2}{b2} + \frac{l2}{c2}$$

Bragg's Law is used to determine the 2θ value: The expected 2θ and d values are close with the experimental 2θ and d values [2].

hkl	2θ(deg)		D((Å)	
	Experiment	Expected	Experiment	Expected
0 2 0	20.39	20.4	4.35	4.35
0 1 1	23.086	23.097	3.8490	3.8476
1 2 0	24.082	24.097	3.6911	3.6902
2 1 0	27.532	27.550	3.2359	3.2351
1 2 1	31.943	31.965	2.7984	2.7976
2 2 0	32.882	32.905	2.7206	2.7198
0 3 1	37.298	37.400	2.4028	2.4025
1 3 1	39.628	39.648	2.2716	2.2714
2 3 0	40.411	40.432	2.2294	2.2291
0 4 0	41.467	41.484	2.175	2.175
1 4 0	43.541	43.555	2.0761	2.0762
0 2 2	47.180	47.207	1.9241	1.9238
2 1 2	51.021	51.047	1.7879	1.7877
1 5 0	54.276	54.296	1.6881	1.6881
4 0 1	56.966	57.074	1.6146	1.6124
3 4 0	58.022	58.022	1.5877	1.5873
4 2 1	61.148	61.186	1.5138	1.5135
3 2 2	62.616	62.645	1.4818	1.4817
2 5 1	63.494	63.523	1.4634	1.4633
1 6 0	65.695	65.724	1.4196	1.4196
2 4 2	66.793	66.827	1.3989	1.3988
4 0 2	69.405	69.437	1.3525	1.3524
2 6 0	70.219	70.254	1.3388	1.3387
1 5 2	70.962	70.938	1.3266	1.3275
2 1 3	72.165	72.165	1.3079	1.3079

Table-3. The Lattice plane and the lattice spacing from d from XRD

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 also be calculated from

$$\delta = \frac{1}{D^2} \quad (2)$$

Where δ is dislocation density and D is the crystallite size. Results of the dislocation density calculated from both the formulas are given in table-4. The number of unit cell is calculated from

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

Where D is the crystallite size and V is the cell volume of the sample [2].

2θ (deg)	Particle Size D (nm)	Dislocation Density (m^2) $\delta = 1 / D^2$ $X10^{14}$	Number of Unit Cells $X10^5$
19.527	80.03	1.5613	10.312
23.290	81.33	1.5118	10.822
24.813	81.56	1.5033	10.914
27.75	82.05	1.4854	11.11
31.848	118.34	7.1406	33.34
32.167	88.19	1.2858	13.79
37.920	76.57	1.7056	9.03
39.705	49.81	4.0306	2.51
40.656	84.94	1.3860	12.33
41.597	85.21	1.3773	12.45
43.072	77.85	1.6499	9.49
47.640	124.39	6.4629	38.72
51.978	126.59	6.2402	40.81
54.244	127.86	6.1169	42.05
57.406	64.87	2.3764	5.49
58.128	75.94	1.7340	8.81
61.215	102.83	9.4572	21.87
62.79	46.66	4.5931	2.04
63.66	23.44	1.8201	0.26
65.29	67.57	2.1902	6.21
66.634	86.66	1.3316	13.09
67.331	106.34	8.8431	24.19
69.34	39.81	6.3098	1.27
70.873	97.76	1.0464	15.0
72.036	98.49	1.0309	19.21

Table-4. Dislocation Density and Number of Unit Cells from XRD.

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

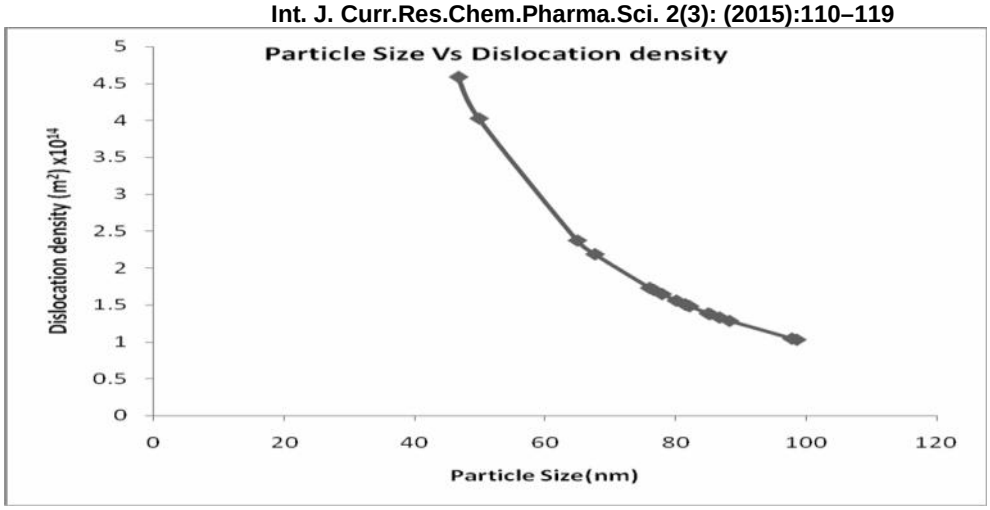

Fig.2 Particle size Vs Dislocation density

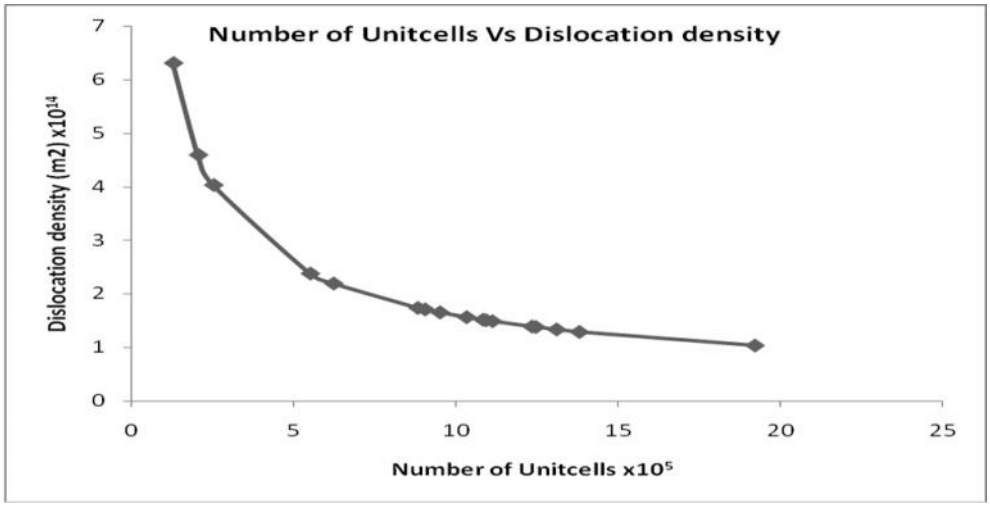

Fig.3 Number of Unit cells Vs Dislocation density.

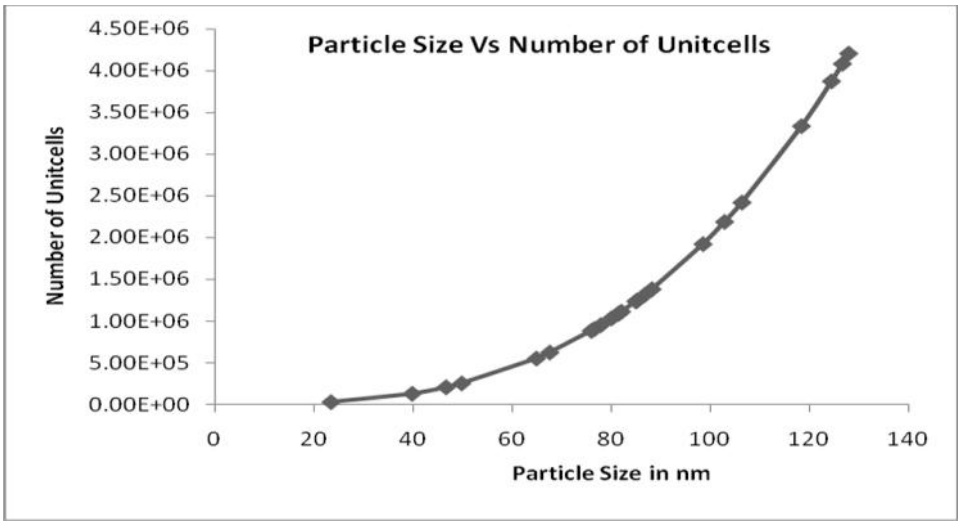

Fig.4 Particle Size Vs Number of Unitcells.

XRD – Morphology Index

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

$$M.I = \frac{FWHM_h}{FWHM_h + FWHM_p} \quad (5)$$

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-5.

FWHM (β) radians	Particle Size(D) nm	Morphology Index (unitless)
0.00176	80.03	0.5
0.00174	81.33	0.5022
0.00174	81.56	0.5022
0.00174	82.05	0.5022
0.00122	118.34	0.5904
0.00164	88.19	0.5177
0.00192	76.57	0.4784
0.00297	49.81	0.3724
0.00174	84.94	0.5022
0.00174	85.21	0.5022
0.00192	77.85	0.3724
0.00122	124.39	0.5904
0.00122	126.59	0.5904
0.00122	127.86	0.5904
0.00244	64.87	0.4188
0.00209	75.94	0.4567
0.00157	102.83	0.5285
0.00349	46.66	0.3353
0.00698	23.44	0.2014
0.00244	67.57	0.4188
0.00192	86.66	0.4784
0.00157	106.34	0.5285
0.00523	39.81	0.2517
0.00174	97.76	0.5022
0.00174	98.49	0.5022

Table-5. Relation between Morphology Index and Particle size.

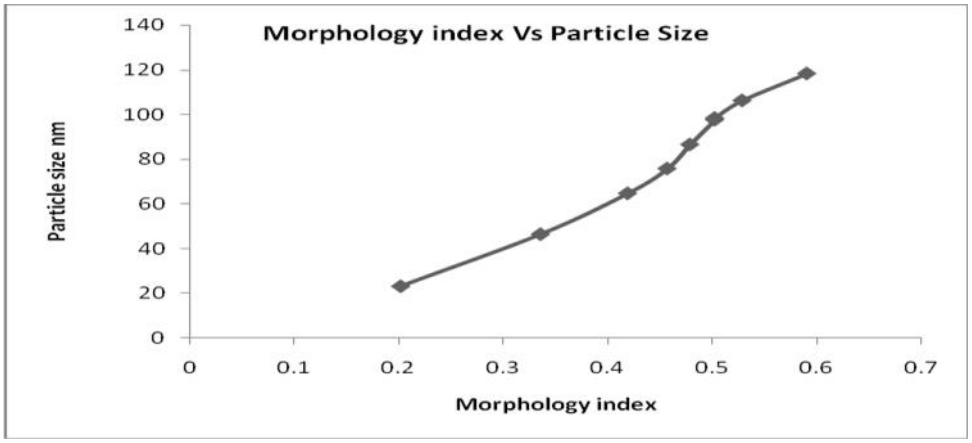

Fig.5 Morphology index of lead chloride nanoparticles.

It is observed that MI has direct relationship with particle size [2] and the results are shown in Fig.5.

XRD – Unit Cell Parameters

Unit cell parameters values calculated from XRD are enumerated in table-6.

Parameters	Values
Structure	Primitive
Space group	Pnam(62)
Symmetry of lattice	Orthorhombic
Particle size	80.03 nm
Lattice parameters	a = 6.97;b = 8.70;c = 4.29
Vol.unit cell(V)	260.14
Density (ρ)	7.101
Dislocation Density	$1.5613x10^{14}$
Mass	278.11amu

Table-6. XRD parameters of lead chloride nanoparticles.

SEM studies

Scanning electron microscopy was used to analyze the morphology and size of the synthesized lead chloride nanoparticles. Fig.6, Fig.7, and Fig.8 show the SEM images of the lead chloride nanoparticles at various magnifications. The SEM images of lead chloride 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.

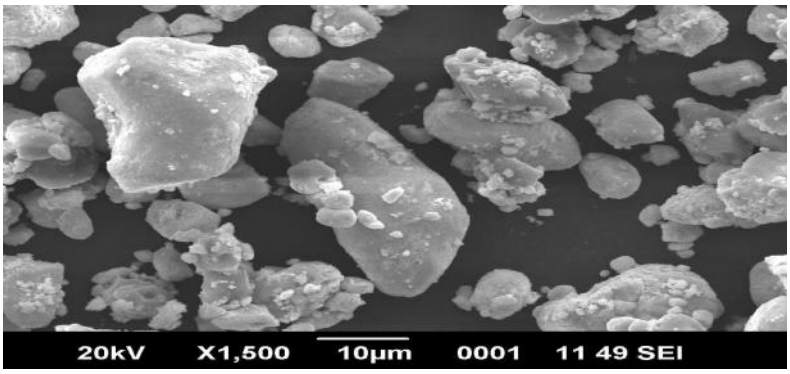

Fig.6 SEM image at 1500 magnifications

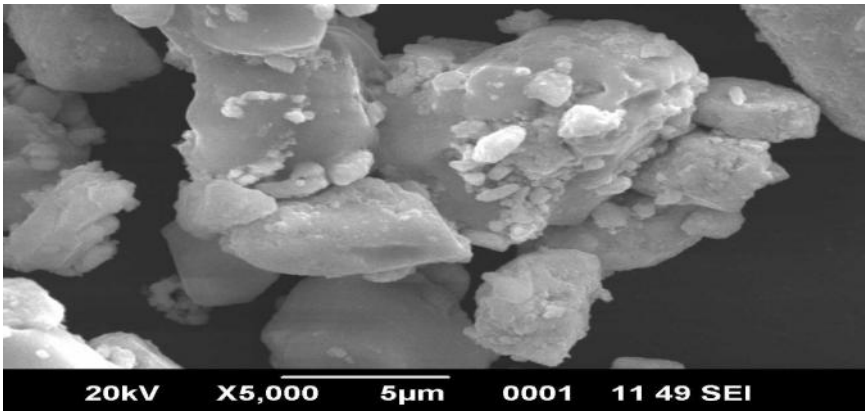

Fig.7 SEM image at 5000 magnifications

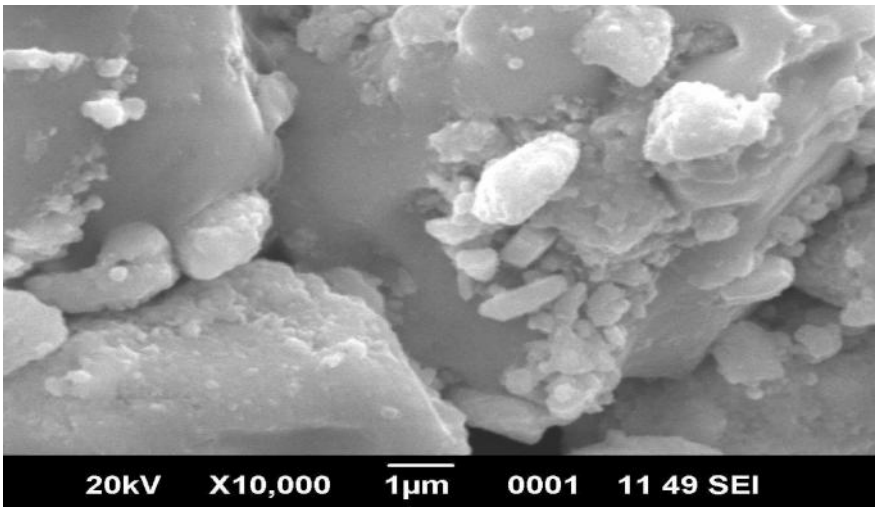

Fig.8 SEM image at 10000 magnifications

FTIR Studies

The FTIR spectrum of the lead chloride sample is shown in the fig.9.The FTIR spectrum for lead chloride shows a strong peak at 3450.65cm^{-1} corresponding to O-H group [1] and the peak at 1680.00, 1649.14 cm^{-1} , 1562.34cm-1, 1543.05 cm-1 and 1516.05 cm-1 are due to bending mode of the hydroxyl group of water molecules [3]. The spectrum also show peaks at 744.52 cm-1 and 673.16 cm-1 indicating chloride and the peak at 451.34cm-1 is due to the presence of lead [2].

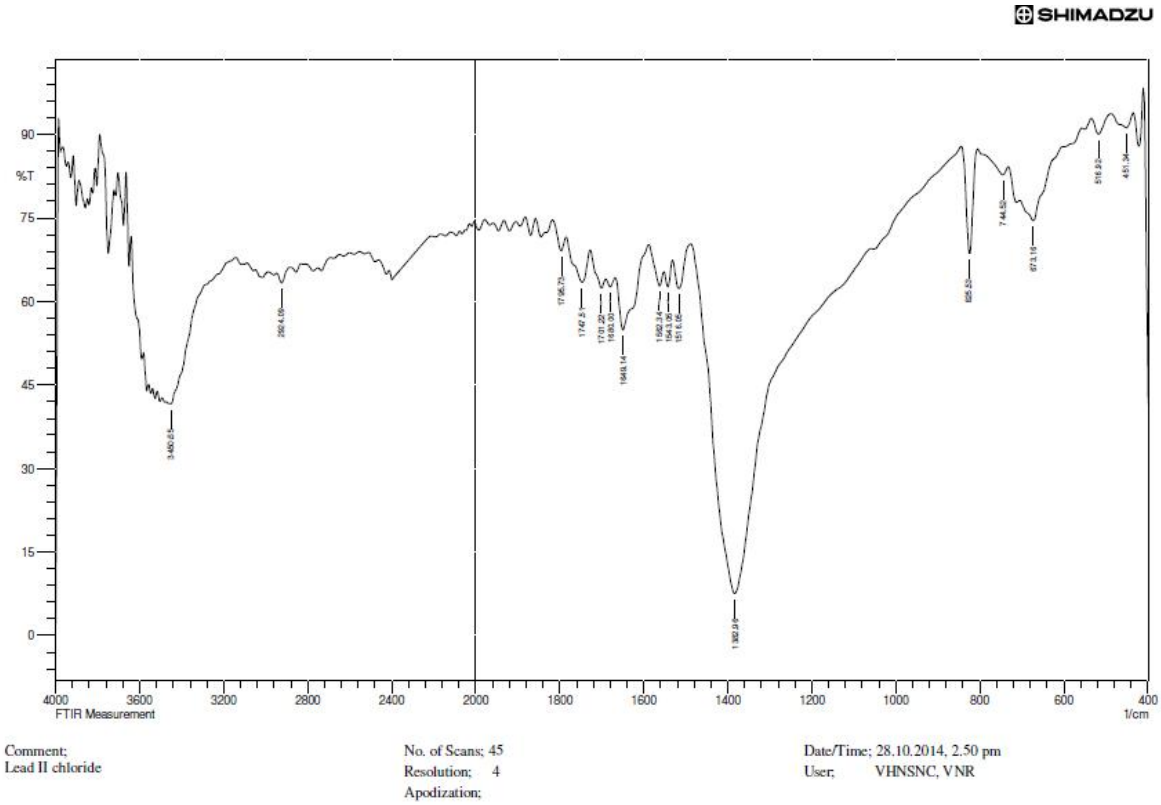

Fig.9 FTIR spectra of lead chloride nanoparticles.

UV Studies

The band gap of the prepared sample lead chloride was determined by using UV visible studies. From the UV spectrum the optical band gap of lead chloride is 3.5eV. Fig.10 shows the graph to find the band gap of lead chloride

.

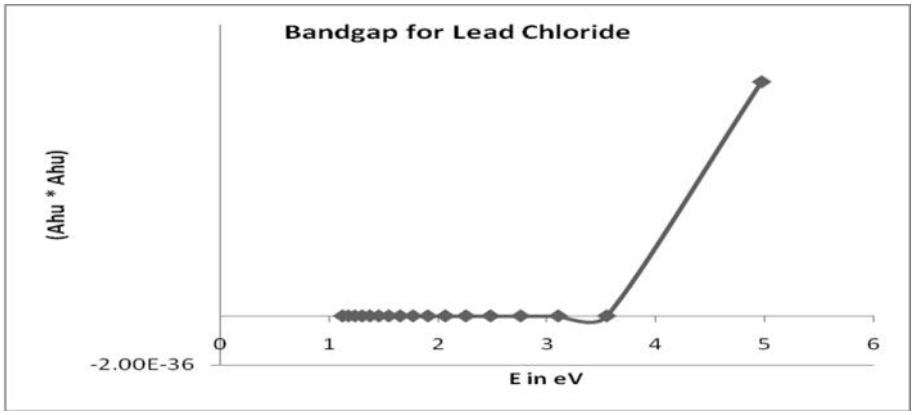

Fig.10 Graph to find the band gap of lead chloride nanoparticles

Conclusion

The lead chloride nanoparticles have been prepared by chemical co-precipitation method. XRD analysis suggests that the average particle size is in the nano range (80.03nm). 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.

References

1.R.Hepzi Pramila Devamani,M. Jansi Rani, 2014, “Synthesis and characterization of Lead Chromate nanoparticles”, Int. J. of Sci. Research, 3(4): 398-402.
2.M.Alagar and T.Theivasanthi, 2011,”Nano sized copper particles by electrolytic synthesis and characterizations”, Int. J. Phy. Sci. 6(15): 3662-3671.
3.R.Hepzi Pramila Devamani and M.Alagar, 2013, “Synthesis and characterization of Iron (II) Chromate nanoparticles”, Elixir NanoTechnology, 61, 16842-16844.