iJRASET
International Journal For Research in Applied Science and Engineering Technology

INTERNATIONAL JOURNAL FOR RESEARCH

IN APPLIED SCIENCE & ENGINEERING TECHNOLOGY

Volume: 2 **Issue:** VIII **Month of publication:** August 2014

DOI:

www.ijraset.com

Call: 08813907089 | E-mail ID: ijraset@gmail.com

ISSN: 2321-9653

INTERNATIONAL JOURNAL FOR RESEARCH IN APPLIED SCIENCE AND ENGINEERING TECHNOLOGY (IJRASET)

Synthesis, morphology and structural characterization of lead hydroxide nano particles

P.B.Taunk[1]*, R. Das[2], D.P.Bisen[3]

[1]*Department of Physics, Govt. Digvijay Autonomous PG College, Rajnandgaon, Chhattisgarh, India*

[2]*Department of Physics, Bhilai Institute of Technology, Durg, Chhattisgarh, India*

[3]*Department of Physics, Pt. Ravi Shankar Shula University, Raipur, Chhattisgarh, India*

Abstrac:tNano structured, crystalline lead hydroxide powder were synthesized by mixing lead nitrate (0.04M) and sodium hydroxide (0.08M) using chemical route method. 0.001M molar concentrations of TEA (Tri ethanolamine) in aqueous solution used to the growing reaction solution. The powder samples are annealed at 80^0 C. The experimental results indicate a successful growth of lead hydroxide in solid form which is rarely observed. EDX, FESEM, XRD, TEM and UV were performed to characterize the morphology, growth and the nature of crystalinity of the samples. The plate like morphology of lead hydroxide has been examined by SEM and transmission electron microscopy. The FTIR spectrum is used to study the stretching and bending frequencies of molecular groups in the sample. The absorption spectra of the sample are recorded in the UV range. From the analysis of absorption spectra, lead (II) hydroxide is found to have a direct band gap of 4.54eV. The average particle size was found 31nm.

Key words ***Lead hydroxide, Chemical route method, nano particles, XRD, SEM, TEM***

I. INTRODUCTION

Lead hydroxides also have wide application such as staining [1, 2] and absorbent [3]. It is also used in making porous glass; electrical-insulating paper; electrolytes in sealed nickel-cadmium batteries; recovery of uranium from sea water and as a catalyst for oxidation of cyclododecanol [4].However, it is doubtful if the simple Lead hydroxide $Pb(OH)_2$ is stable in solid state so the morphology of lead hydroxide powders has rarely been reported[5] .Some researchers have studied the crystal structure of lead hydroxide. Previous studies have shown that the $Pb(OH)_2$ powder can be formed in solution at low sodium hydroxide concentration. Sole and Yoff observed plate like morphology of lead hydroxide crystal [6] whereas Cheng et al observed the rod like/plate like morphology [7].

Nano structures have been prepared by different methods like combustion method, sol gel, oxide-assisted growth, solution-liquid growth in organic solvent and chemical bath deposition method [8-11]. In this paper, we have tried to report plate like morphology of lead hydroxide nano powder prepared by chemical route method by using complexing agent(TEA), because of its simplicity, convenience and low cost. In this paper, we report the observation of surface morphology and the result of optical characterization of lead hydroxide nano powder.

II. EXPERIMENTAL PROCEDURE

A. Powder preparation 0.04M aqueous solution of lead nitrate mixed with 20 ml of 0.08M aqueous solution of sodium hydroxide (All AR grade 99.9% pure). 8ml of 0.001M aqueous

ISSN: 2321-9653

solution of TEA added after in growing reaction solutions. Keep the reaction solution for 18-24 hours. The powder was washed many times (more than 8 times) with distilled water, filtered and dried in sunlight. After this again it is annealed in hot air oven at 80^{o} C.The color of powder is white.

In the above case of lead (II) salt reacting with alkali without adding TEA, color of the powder was observed as very light yellowish (hydrated oxide) [5].TEA is alkali and complexing agent and also restrained lead hydroxide [7,12,13].

Reaction formula is shown as [7] follow:

$$Pb^{2+}+2OH \rightarrow Pb(OH)_2 \rightarrow PbO+H_2O$$

$$Pb(OH)_2 \xrightarrow{\text{Containing-TEA (Dehydration restricted)}} PbO+H_2O$$

B. Tests conducted

Energy Dispersive X-ray (EDX), Field Emission Scanning Electron Microscopy (FESEM), XRD and Transmission Electron Microscopy (TEM) were employed to characterize the sample. Energy Dispersive X-ray analysis [EDX] was used for element analysis of the powder**.** FESEM was used for morphological characterization of sample. The surface morphology of the white precipitate was determined by Field Emission Scanning Electron Microscope (FESEM) JSM-7600F. The structural parameters of the powder were determined using X-Ray Diffraction technique. The XRD patterns were recorded with Bruker D8 Advanced X-Ray Diffractometer using a Cu Ka radiation source (λ ± 1.54056 Å). The X-rays were detected using a fast counting detector based on silicon strip technology (Bruker Lynx Eye detector). Particle diameter and surface morphology of white powder were determined by Transmission Electron Microscope using Philips CM -200. Absorption spectrum was measured by UV Spectrophotometer (Varian).

III RESULT AND DISCUSSION

A. EDX analysis

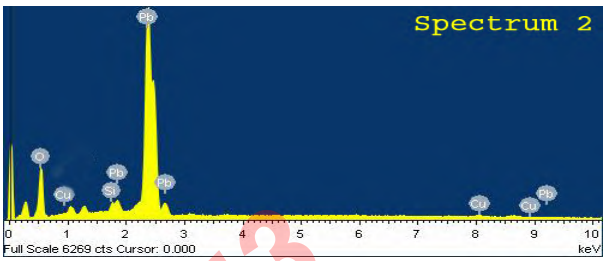


Figure 1 Spectrum 2 shows EDX of the white powder with 8ml of 0.001M aqueous solution of TEA

EDX analysis was done to analyze the composition of powder. Spectrum 2 shows EDX of the white powder with 0.001M aqueous solutions of TEA. Pb, O, Cu, and Si peaks are present in the spectrum. The Cu and other peaks come from Copper grid that used to support samples for EDX characterization. The Pb and O peaks originate from the precipitate. Thus, the probability is that the powder was lead oxide, but there is an obvious inconsistency between the white color of the powder and color of any other observed lead oxide. So this experiments exhibits that it is not lead oxide, instead it could have been lead hydroxide or basic lead hydroxide because of its white color. However, a very noteworthy point observed is that no carbon peak is found in the EDX spectrum. Thus, it could not be basic lead carbonate; it is often encountered in reactions where lead hydroxide is the anticipated product because lead carbonate can be formed when lead hydroxide is exposed to the carbon dioxide in air [6].

B. SEM Studies

Scanning electron microscopy [SEM] is a convenient technique to study the morphology of the nano structures. Fig 2 showed that white powder posses plate like morphology.

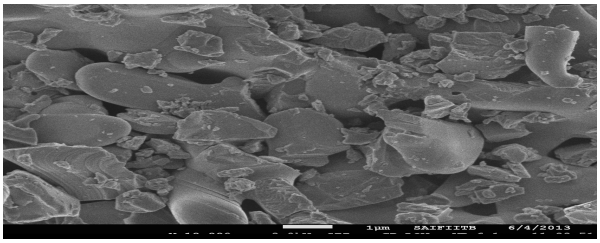


Fig 2 shown morphology of white powder

ISSN: 2321-9653

C. XRD Studies

To identify the crystal structure of white powder, we performed XRD analysis. Fig. 3 shows XRD pattern of the white powder. In the range of $20<2\theta<80^0$C, most of the diffraction peaks can be assigned to lead hydroxide according to report by cheng. The diffraction peaks are assigned using JCPDS card No.11-270 [7]. X-ray diffraction pattern of $Pb(OH)_2$ confirms proper phase formation and crystalline nature of the samples. The highest intensity peak (110) plane of $Pb(OH)_2$with other smaller intensity peaks (101,102,110,112,201,202,203,204,205,206,207and 221) are observed. Inter planer spacing of other peaks marked by the sign * are matched with lead hydroxide file No.00-003-0607. The crystal size of the powder sample is calculated using the Debye Scherer's formula: $D=\lambda k/\beta \cos\theta$.

XRD results match with reported results [5, 7]. The structure of the prepared lead hydroxide is hexagonal with a=5.28 Å, c=12.84 Å and c/a=2.43. The observed spacing data from XRD are listed in the Table 1. The calculated spacing data are extracted using the formula of the calculated spacing data are extracted using the formula of crystal plane distance d for hexagonal crystals,

$$\frac{1}{d^2} = \frac{4}{3} * \frac{h^2+hk+k^2}{a^2} + \frac{l^2}{c^2}$$

Where h, k and l are Miller Indices of particular plane

This experiment display that spacing data are very well in agreement with the calculated data. The values obtained of relative diffraction peaks and inter planer spacing of $Pb(OH)_2$ for 0.001 M concentration aqueous solution of TEA used, are shown in Table 1. Average particle size is obtained 31 nm.

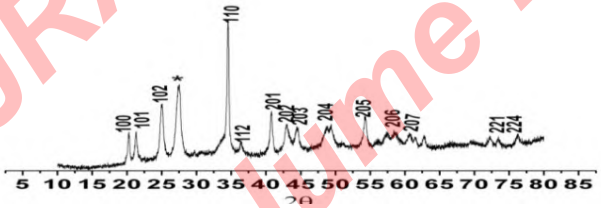


Fig 3 XRD patterns of the white powder annealed in hot air oven at 463°C (8ml of 0.001M aqueous solution of TEA added after in reaction solution).

Table 1

Miller index and crystal plane distance and partical size of lead hydroxide white powder

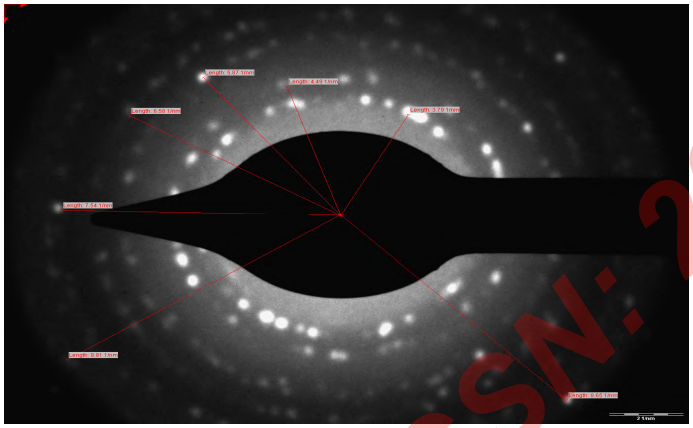
Line	Assigned Indices (hkl)	Calculated spacing	spacing Observed
1	100	4.51	4.48
	101	4.30	4.24
2	102	3.58	3.56
3	*	3.23	3.24
4	110	2.64	2.62
5	112	2.42	2.44
6	201	2.25	2.21
7	202	2.15	2.10
8	203	2.02	2.04
9	204	1.86	1.85
10	205	1.71	1.70
11	206	1.56	1.58
12	207	1.43	1.43
13	221	1.31	1.31

D. TEM Studies.

The TEM micrographs of the lead hydroxide powder along with the selected area electron diffraction SAED pattern shown in fig 4. The crystalline nature of the material is clear from the SAED

ISSN: 2321-9653

pattern (Fig 4a). The image also clearly shows that crystals present in the powder were highly agglomerated. The selected area electron diffraction pattern (SAED) shows the bright spots indicating the high crystalline nature of the material. Transmission electron microscopy (TEM) is used to determine actual size of particle. TEM analysis shows the nano crystalline behavior of the prepared powder with random particle size distribution in the range of 5.7nm to 70.5 nm which is good agreement with XRD data (average particle size 31nm) (Fig 4 b). The prepared material shows the existence of plate with spherical particles.



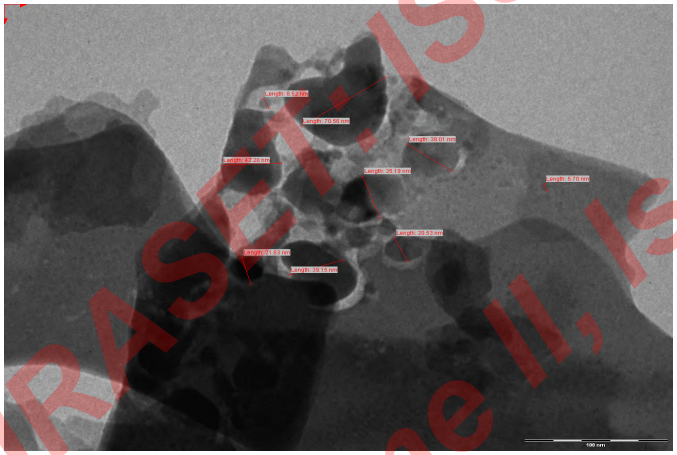


Fig 4 a and b shown TEM images of lead hydroxide nano powder.

E. FTIR Studies

The FTIR spectrum of the Lead (II) hydroxide sample is shown in the fig.5. The FTIR spectrum for Lead II hydroxide shows a strong peak at 3529cm-1 corresponding to the O-H stretching[14]. Another strong and sharp peak with a maximum of 1430 cm^{-1} shows due to H-OH stretching. and the peak at 690cm^{-1} indicates the presence of Lead [14,15],

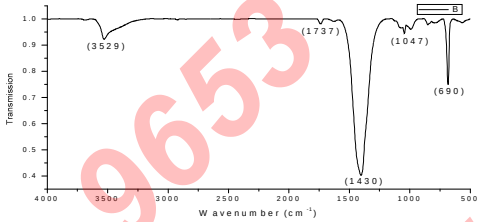


Fig 5 The FTIR spectrum for Lead (II) hydroxide

F. UV Studies

The band gap of the prepared sample Lead (II) hydroxide was determined by using UV visible

Studies. It has been noticed that the optical band gap of Lead (II) hydroxide is 4.54eV. Fig .6 shows the graph to find the band gap of Lead (II) hydroxide.

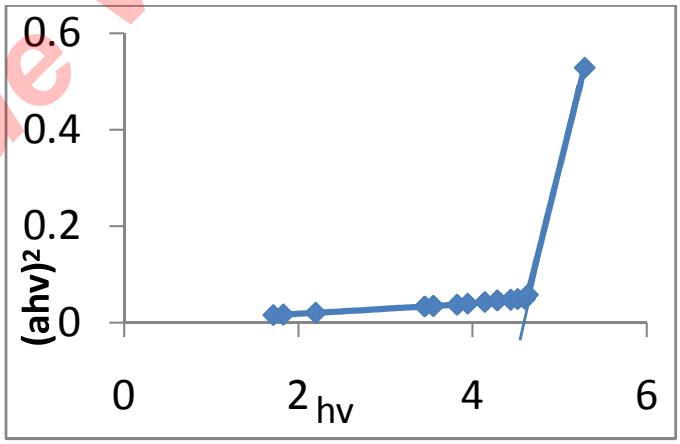


Fig 6: Band gap of Lead (II) hydroxide

IV CONCLUSIONS

This study is focused on the synthesis and structural characterization of lead hydroxide nano particles. Presence of

ISSN: 2321-9653

TEA in the reaction solution results in the solid form of lead hydroxide nano particles. No carbon peak is found in EDX spectrum. The nano particle has a hexagonal crystalline structure. XRD study reveals better crystallization of the powder. The particle size obtained 31 nm Band gap of nano particle obtained 4.54eV.

REFERENCES

[1] D.G Feldman. Method of stain thin sections with Lead hydroxide for precipitate J. Cell Biol. 15, 592. 1962

[2] T. C Norman. Stain Technol. A method of stain thin sections with Lead hydroxide.39,50 1964

[3] W. A Roach Lead Hydroxide as a Selective Absorbent for Certain Organic Constituents of Plants Annals of Botany 22 ,127 1958

[4] I.R Hepzi. . D Pramila, M., J Alagar Synthesis and characterization of Lead (II) hydroxide Nanoparticles .Applied Sciences and Engineering Research, 1, Issue 3, . 2012.

[5] G Todd. and E Parry. Character of Lead Hydroxide and Basic Lead Carbonate Nature 202 ,3861964

[6] M. J. Sole and A. D Yoffe. Decomposition of single crystals of lead hydroxide, in the electron microscope 198, 1262 1963

[7] X. P Cheng W.L Zou.. M.S Song, Y G Cao., Su, Q Yang,.. X.M Lu, F..X Zhang. Shape –Controlled synthesis and related growth mechanism of lead hydroxide Nano rods by solution –phase method Chin. Phys. Lett. 27, No. 5, 057302,2010

[8] M.M Biggs. , O.M Ntwaeaborwa ,. J.J Terblans Swart. H.C Characterization and luminance properties of SiO2: ZnS:Mn2+ and ZnS:Mn2+ nano phosphors synthesized by sol–ge l method Elevier Physica B 404 . 4470–4475 ,2009

[9] C Cruz-.R Bernal., S.E Burruel- M H, Grijalva-.,F.M.Barboza. Thermo luminescence properties of new ZnO nanophosphors exposed to beta irradiation. Optical Materials 27 , 1235–1239 ,2005

[10] B Liu and H C Zeng, 2003 hydrothermal synthesis of ZnO nanorods in the diameter regime of 50 nm *J. Am. Chem.Soc.* **125** 4430

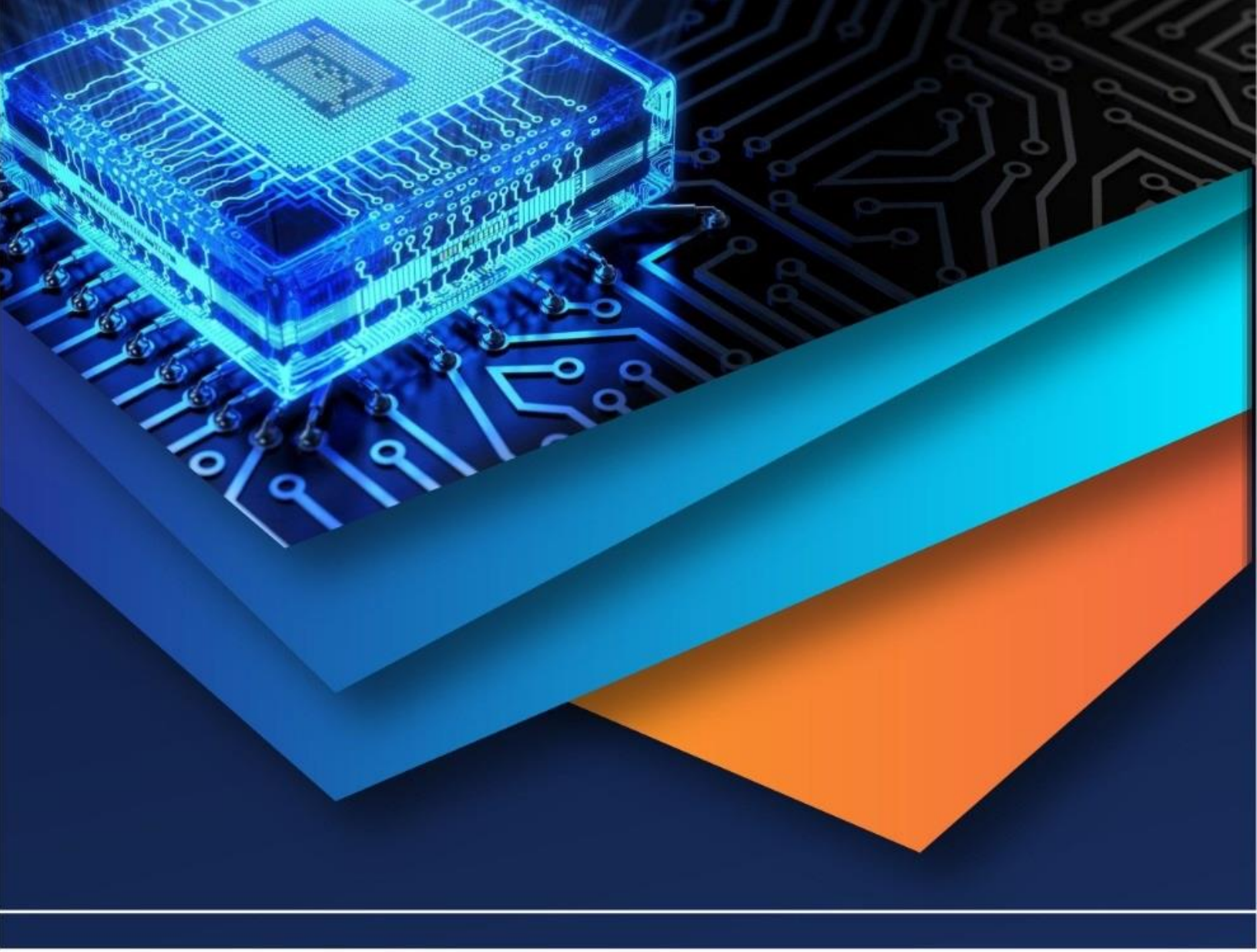
[11] A M Morales and C M Lieber A. laser ablation method for the synthesis of crystalline semiconductor nanowires *Science* **279** 2081998

[12] D.D.O. Eye, A. Ekpunobi. C .E Okeke Optical properties of lead oxide thin films prepared by chemical bath deposition technique's Academic Open internet. 14(2006)

[13] D.D.O Eye. Annealing effect of lead oxide thin films prepared by chemical bath deposition technique The pacific journal of science and technology,7, NO.2(2006)

[14] Pharmacy 510 Lecture 8 Predicting IR Absorption Frequencies.

[15] C.R Gautam ,D Kumar and Omprakash ,IR study of Pb – Sr titanate borosilicate glasses. Bulletin materials Science, 33(2), 145-148. 2010



10.22214/IJRASET

45.98

IMPACT FACTOR: 7.129

IMPACT FACTOR: 7.429

INTERNATIONAL JOURNAL FOR RESEARCH

IN APPLIED SCIENCE & ENGINEERING TECHNOLOGY