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One Step Colloidal Route for the Preparation of Lead Sulfide Nanoparticles Using Thiol

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***Abstract* - Lead sulfide (PbS) nanoparticles were prepared in an aqueous solution using 2-Mercaptoethanol as a capping agent by simple colloidal route. The structural, optical, morphological and compositional analysis were carried out by using X-ray diffraction (XRD), Optical absorption spectroscopy, Transmission electron microscopy (TEM) and Energy dispersive X-ray analysis (EDAX) respectively. Effect of concentration of 2-Mercaptoethanol on particle size was investigated. In the absence of capping agent the yield of PbS particles was maximum. XRD shows the formation of PbS nanoparticles with cubic structure. Absorption spectra reveals the blue shift with increase in the concentration of 2-Mercaptoethanol due to decrease in particle size. EDAX study confirms the formation of stoichiometric PbS nanoaprticles for all concentration of 2-Mercaptoethanol. The average particle size of PbS nanoparticles synthesized in presence of 0.5 wt% 2-Mercaptoethanol observed from TEM image is 15 nm. SAED pattern shows all diffraction rings are indexed to cubic structure of PbS. The interplaner distance 'd' observed from HRTEM image is equal to 1.3612 A^0 which corresponds to (420) plane.**

***Keywords* – Lead sulfide nanoparticles, TEM, 2-Mercaptoethanol**

I. INTRODUCTION

Semiconductor nanoparticles attracted considerable attention due to their applications in various areas like optoelectronics, photocatalysis, and solar cells [1-3]. Lead sulfide (PbS) is an important binary semiconductor material due to its small direct band gap energy (0.41 eV, for bulk) and a large exciton Bohr radius (18 nm). PbS nanoparticles are useful in electroluminescent devices such as light emitting diodes and optical switch devices due to its optical property [4]. Various techniques like spray pyrolysis, thermal decomposition, molecular beam epitaxy, chemical vapor deposition, laser ablation etc. have been used in the preparation of nanostructures. Chemical synthesis is one of the most useful techniques for the preparation of nanoparticels [5]. Up to now, numerous chemical methods such as sol-gel technique, hydrothermal technique and microwave irradiation have been developed to synthesize PbS nanoparticles [6]. However, high temperature or long reaction time was usually involved in these methods. In this work we use simple one step colloidal method to prepare PbS nanoparticles using 2-Mercaptoethanol as a capping agent. Effect of capping agent concentration on particle size was extensively studied. Optical and morphological measurements have been carried out on PbS nanoparticles to investigate quantum size effects.

II. EXPERIMENTAL

Lead acetate and sodium sulfide are used as lead and sulfur source, respectively. 2-Meracptoethanol was used as a capping agent. In the experimental part Lead acetate of concentration 0.7 mM was dissolved in 30 ml of deionized water. pH of the resultant solution was adjusted to 11 by adding Triethylamine. Different amount of 2-Mercaptoethanol were added into the solution (0 wt%, 0.1 wt%, 0,5 wt% and 1 wt%). Finaly sodium sulfide of concentration 1.7 mM was added into the solution. The synthesis was carried out at low temperature for duration 20 min. The resultant solution was vigorously stirred during the synthesis of nanoparticles. The color of the final solution changed from transparent to dark brown or black depending upon the concentration of 2-Mercaptoethanol. Finally, the product was centrifuged for several times and characterized by using various characterization techniques. Structural measurements were carried out by X-ray diffraction (XRD), using a Bruker D8 advance diffractometer. Optical absorption measurements were carried by JASCO UV-Vis-NIR spectrophotometer. Transmission electron microscopy (TEM) images were recorded using TECNAI G^2 20U-Twin microscope with operating voltage 200 kV. EDAX was recorded using a JEOL JSM-6360A microscope with operating voltage 20 kV.

III. RESULTS AND DISCUSSION

Fig 1 shows XRD pattern of PbS powder synthesized in the absence of 2-Mercaptoethanol. All diffraction peaks (112),

(200), (220), (311), (222), (400), (331), (420), and (422) are exactly matches to standerd JCPDF data, which confirms the cubic structure of PbS. Crystallite size of PbS nanoparticles was calculated by using Scherrer's formula as given below,

$$D = \frac{K\lambda}{\beta \cos\theta} \qquad (1)$$

where, D = grain size in Å, λ = wavelength of incident radiation in Å, β = full width at half maximum (in radians), θ = Bragg angle (in degrees), K = 0.9 (For spherical shape of particles),The average crystallites size 30 nm in the solution was calculated for PbS nanoparticels without 2-Mercaptoethanol.

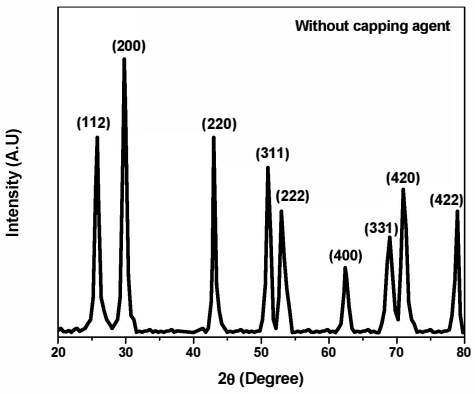

Fig 1 XRD pattern of PbS powder without 2-Mercaptoethanol

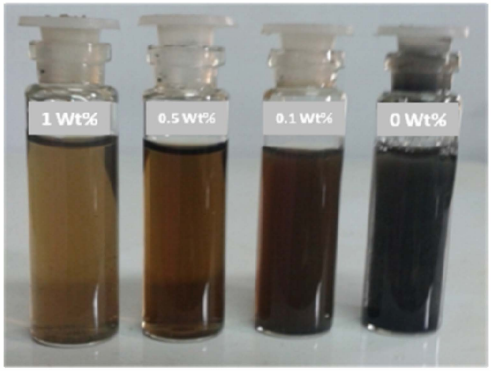

Fig 2 Photo of PbS nanoparticles capped with different concentration of 2-Mercaptoethanol

Black colored of PbS colloidal solution is observed in the absence of 2-Mercaptoethanol, while the color of colloidal solution become dark brown and pale brown with systematic addition of 2-Mercaptoethanol, which could be due to decrease in particle size. The actual photograph of the colloidal solution of PbS nanoparticles is as shown in figure 2. Fig. 3 shows the UV-Vis absorption spectra of the PbS nanoparticles. Inset shows absorption spectra of PbS nanoparticels without 2-Mercaptoethanol. For uncapped PbS nanoparticles the absorption edge appear at 1220 nm. The addition of 2-Mercaptoethanol results the blue shift from 1021 nm towards 998 nm which reveals the decrease in particle size due to quantum size effect. Figure 4 shows the plot of band gap

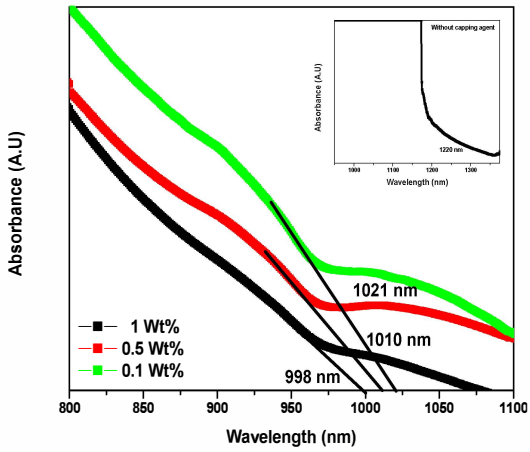

Fig 3 UV-VIS absorption spectra of PbS nanoparticles with various concentration of 2-Mercaptoethanol (Inset shows absorption spectra of uncapped PbS)

determined from absorption spectra Vs concentration of 2-Mercaptoethanol. The band gap of PbS is systematically increases with increasing the concentration of 2-Mercaptoethanol in the bath due to quantum size effect. Band gap observed for uncapped and capped PbS nanoparticles was 1.01 eV and 1.24 eV respectively.

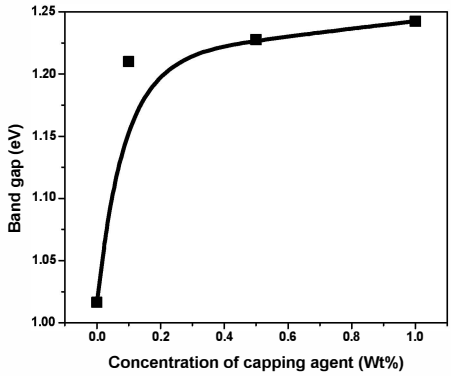

Fig 4 Plot of Band gap Vs concentration of 2-Mercaptoethanol

Table 1 shows EDAX of PbS nanoparticles synthesized with different concentration of 2-Mercaptoethanol.The atomic percentage concentration of sulfur increases with increase in concentration of 2-Mercaptoethanol in the bath it may be due to presence of sulfur in thiol group. Stoichiometric nanoparticles are formed for all concentration of 2-Mercaptoethanol.

TABLE 1 A SUMMARY OF ATOMIC PERCENTAGE CONCENTRATION PB AND S IN PBS NANOPARTICLES OBTAINED BY EDAX ANALYSIS

Concentration of 2-Mercaptoethanol	Atomic concentration (%)	
	Pb	S
0 wt%	51.71	48.22
0.1 wt%	50.36	49.64
0.5 wt%	49.96	50.04
1 wt%	46.28	53.72

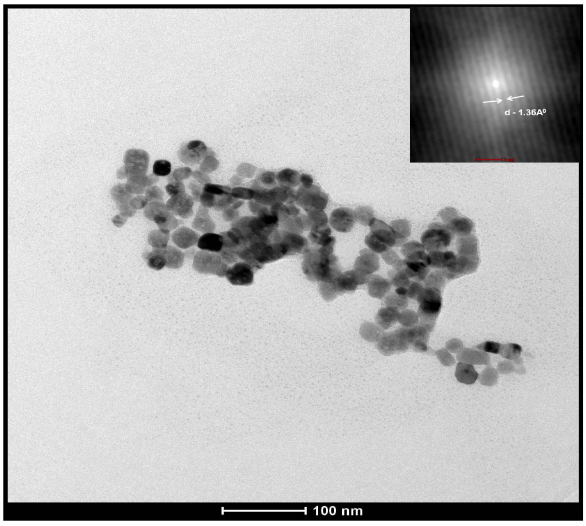

Fig 5 TEM image of PbS nanoparticles capped with 0.5wt % of 2-mercaptoethanol (Inset shows HRTEM image)

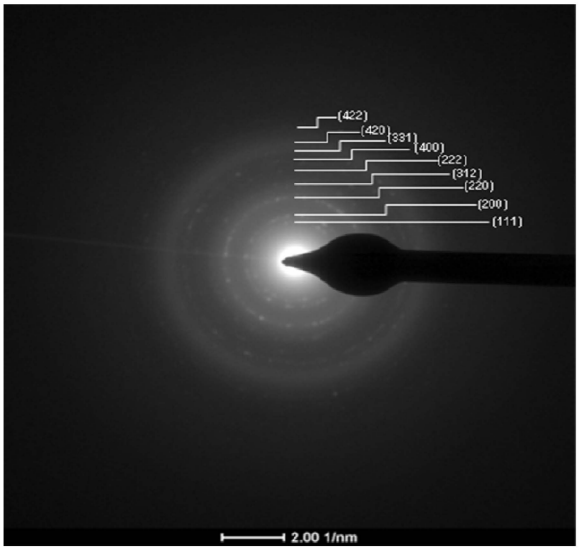

Fig 6 SAED pattern of PbS nanoparticles capped with 0.5wt % of 2-mercaptoethanol

Fig 5 shows TEM image of PbS nanoparticles capped with 0.5 wt% of 2-Mercaptoethanol. TEM shows uniform granular morphology of PbS nanoparticles of average 15 nm particle size. Inset shows the High Resolution TEM (HRTEM) image of PbS nanoparticles. The observed 'd' value is around 1.3612 A^0 which is close to the standard 'd' value 1.3607 A^0 for (420) plane. Fig 6 shows selected area electron diffraction (SAED) pattern of PbS nanoparticles capped with 0.5 wt% 2-Mercaptoethanol. All the diffraction rings are indexed to cubic structure of PbS.

IV. CONCLUSION

PbS nanoparticles were successfully prepared using simple colloidal route without and with 2-Mercaptoethanol. Particle size of PbS nanoparticle depends on concentration of 2-Mercaptoethanol. XRD confirms the formation of Lead sulfide with cubic structure. Absorption spectra shows the blue shift in absorption edge by increasing the 2-Mercaptoethanol which reveals the decrease in particle size of PbS nanoparticles due to quantum size effect. EDAX shows with increase in 2-Mercaptoethanol concentration the sulfur content in PbS increases. Near stoichiometric PbS nanoparticle were synthesized for all concentration of 2-Mercaptoethanol. TEM micrograph confirms the size of PbS nanoparticles is 15-20 nm with capping agent. All diffraction rings observed from SAED pattern are indexed to cubic structure of PbS.

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