Study of PbS Nanoparticles Synthesized by Chemical Route

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Abstract: PbS nanoparticles (Nps) was synthesized by chemical route at room temperature. Lead acetate and thiourea was used for source of Pb^{+2} and S^{-2} ions respectively. The elemental composition of synthesized nanoparticles was evaluating using energy dispersive analysis of X-ray (EDAX). The EDAX spectra show the peaks of Pb and S elements only. The X-ray diffraction (XRD) pattern shows the cubic PbS phase. The broadening of the X-ray diffraction peaks indicating the nanocrystalline nature of the synthesized sample. The crystallite size determines using the Scherrer formula was came out to be 6.07 nm. The crystallite size and residual strain evaluated using Hall-Williamson relation was came out to be 4.02 nm and -4.6 x 10^{-3} respectively. The obtained results are discussed in details.

1. INTRODUCTION:

Different morphologies at nano level can play significant roles to tune the properties of the materials. They include nanoparticles, nanowhishkers, nanowires and nanoribbons [1]. PbS in nano scale with different morphologies has been synthesized by diverse methods such as decomposition of a single source precursor [2], facile synthesis [3], solvothermal [4], hydrothermal [5] etc methods. Typically the physical properties of PbS can alter by the size effect. The size controlled PbS NPs can be potentially used in preparing solar cells, optical switches, photographs, solid-state laser, solar absorbers, lasers, LED devices, telecommunications, electro-luminescent devices, detectors, optical amplification such as light emitting diodes [6-8]. The PbS at nano scale was synthesized by simple chemical route and comprehensively characterized. The synthesis procedure, elemental and structural analysis of as-synthesized PbS nanoparticles were deliberated in details.

2. EXPERIMENTAL DETAILS:

PbS nanoparticles were synthesized by simple chemical route at room temperature. In the typical synthesis the clear solution of 20 ml of 0.052 M lead acetate was prepared in double distilled water. The another clear prepared solution of 20 ml of 0.24 M thiourea in double distilled water was mixed into the first solution and then continuously stirring the solution for 30 min. using magnetic stirrer at room temperature (28 ^{o}C). Then adding the 10ml of 14M aqueous ammonia into the mixed solution to increase the pH of the final solution. After the pH of the solution reach at 10 then the solution was kept at room temperature for 30 min. at rest in which the solution turn into dark black from clear transparent that indicating the formation of PbS. The particles from the beaker were filtered out and given a multiple wash with double-distilled water and absolute methanol to remove impurity. After that they were dried in an oven at 45 °C for 2 h to get nanoparticles yield.

Reaction mechanism

The chemical reaction for the synthesis of PbS nanoparticles is given below,

$$Pb(CH_3C00)_2 \longrightarrow Pb^{2+} + 2CH_3COO^- \quad (1)$$
$$NH_2\text{- } CS \text{ - } NH_2 + OH^- \longrightarrow CH_2N_2 + H_2O + SH^- \quad (2)$$
$$SH^- + OH^- \longrightarrow S^{2-} + H_2O \quad (3)$$
$$Pb^{2+} + S^{2-} \longrightarrow PbS \quad (4)$$

3. CHARACTERIZATION:

The elemental stoichiometric analysis of the as-synthesized PbS nanoparticles samples was done by energy dispersive analysis of X-rays (EDAX) attached to JEOL JSM 5600 scanning electron microscope. The XRD pattern was recorded on a Philips Xpert MPD X-ray diffractometer using graphite monochromotized CuK_α radiation (λ = 1.5405 Å). The scanning rate of 0.20^o s^{-1} was applied to record the pattern in the 2θ range of 3^o - 99.96^o.

4. RESULT AND DISCUSSION:

The energy dispersive analysis of X-ray of the synthesized PbS nanoparticles is shows in fig. 1. The spectrum clearly shows the presence of two elemental peaks of Pb and S only. No other elemental peaks from impurities were observed which indicating the synthesized material was of PbS only. The Pb-to-S atomic percentages ratio came out to be 0.99 for PbS nanoparticles from EDAX. The ratio state that the synthesized PbS nanoparticles are lead deficient.

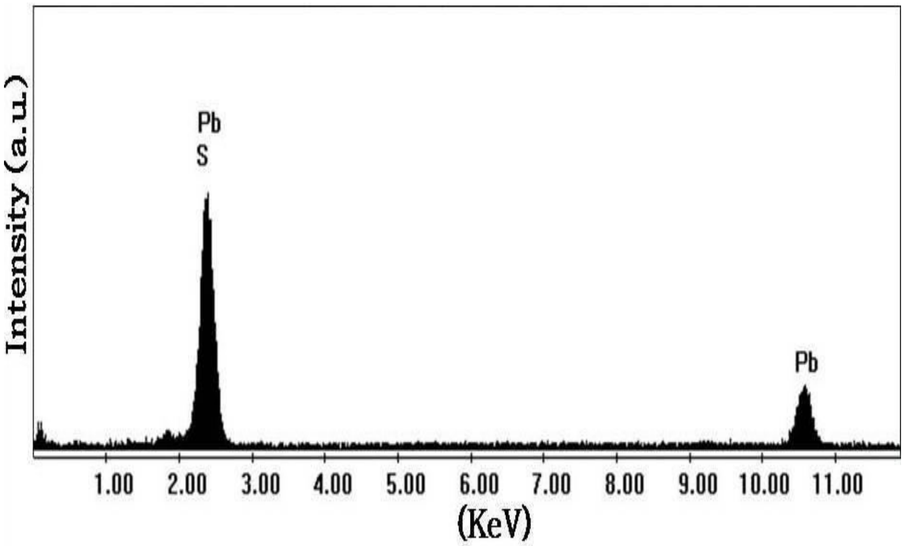

Fig. 1. EDAX spectrum of synthesized PbS nanoparticles.

The XRD pattern of the as-synthesized PbS nanoparticles is shows in fig. 2. All the diffraction peaks can be indexed as the single cubic phase of PbS only having lattice parameter a=5.936 Å in good agreement with the standard data (JCPDS 05-0592). The XRD did not show any other phases like lead disulphide or lead oxide. The broadening of peaks in the XRD pattern indicates the nanocrystalline nature of the sample.

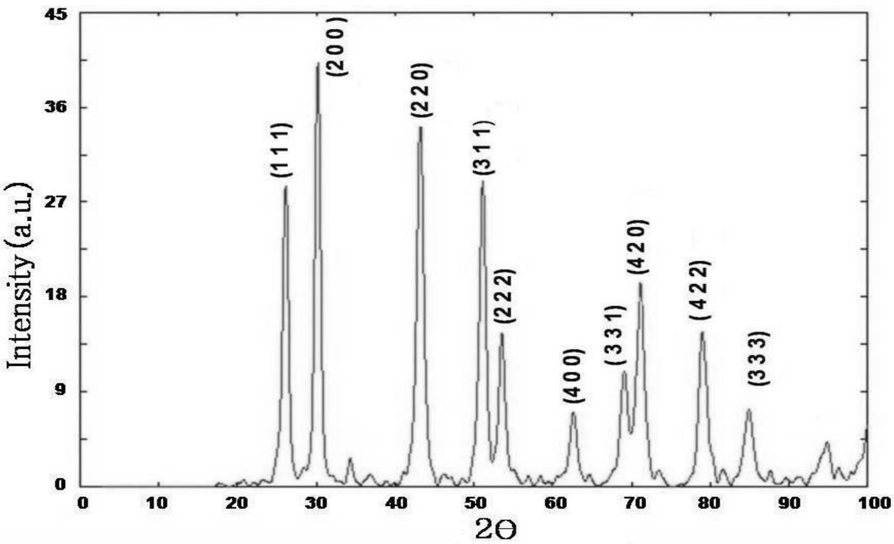

Fig. 2. X-ray diffraction pattern of synthesized PbS nanoparticles.

The crystallite size of the as-synthesized PbS nanoparticles were evaluate using the Debye-Scherrer formula [9] for different X-ray diffraction given as;

$$L = K\lambda/\beta\cos\theta \quad (5)$$

Where K is a shape factor (i.e. 0.9 for spherical shape particles), λ is the wavelength of X-ray (1.5405 Å), β is the angular line width at half maximum intensity and θ is the Bragg angle in degree. The average crystallite size estimated from the XRD analysis using Scherrer's formula came out to be ~6.07 nm.

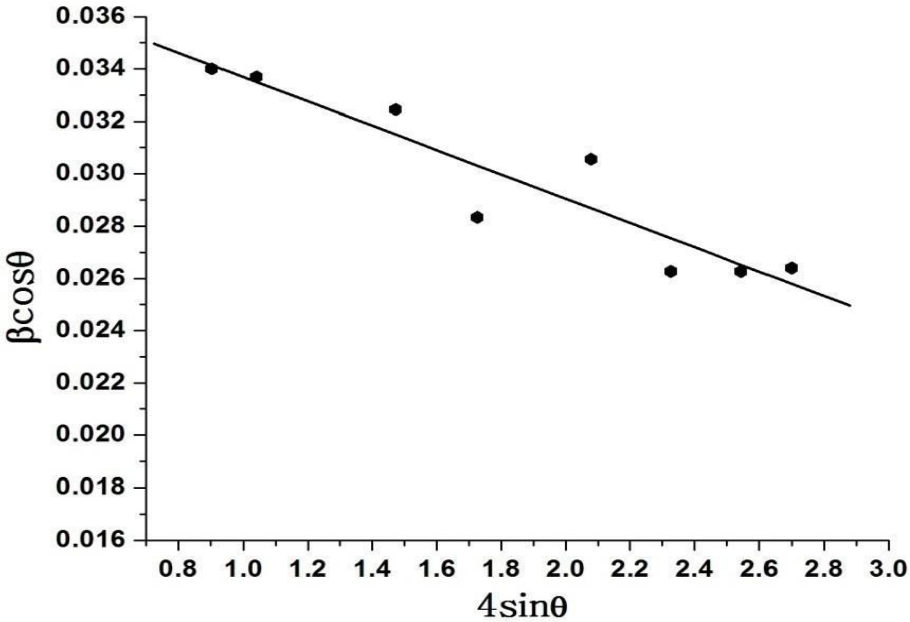

Fig. 3. Hall-Williamson (HW) plot for as synthesized PbS nanoparticles.

The crystallite size and the micro strain in the as-synthesized PbS nanoparticles were estimated by Hall-Williamson relation [9].

$$\beta\cos\theta/\lambda = K/L + 4\varepsilon\sin\theta/\lambda \quad (6)$$

The Hall-Williamson equation incorporates the Scherrer's formula of crystallite size and the micro strain terms. Here β is full width at half maximum (FWHMs) of the diffraction peaks. The relation is expressed as a linear combination of the contributions from the strain (ε) and crystallite size (*L*). The determinate residual strain is -4.6×10^{-3} and average crystallite size 4.02 nm using Hall-Williamson plot for the synthesized PbS nanoparticles. The negative value of the residual strain for the PbS nanoparticles indicates it to be the compressive strain.

5. CONCLUSION:

The PbS nanoparticles were successfully synthesized by simple chemical route at room temperature. EDAX analysis shows the presence of Pb and S elements only. The second peak of Pb element in EDAX spectra is stating the emitted radiation from Pb is K_β. The X-ray diffraction pattern shows the broadening of the diffraction peak that indicating the formation of the nano scale. The diffraction peaks are indexed with cubic PbS only no other phase or impurity elements detect. The crystallite size evaluated using scherrer's and Hall-Williamson relation was come out to be 6.07 nm and 4.02 nm respectively. The produced residual strain during the chemical reaction was -4.6×10^{-3} which is compressive. The phase pure chemically synthesized PbS with nanoparticles morphology was widely used in sized tunable optoelectronic devices.

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