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Effects of thiourea concentrations on the structural, morphological and optical properties of lead sulphied thin films prepared by solution growth method

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ABSTRACT: Chemical bath deposition (CBD) route employed to deposit lead sulphied (PbS) thin films at a bath temperature of 60 ^{0}C for 40 minutes. The influence of applying different amount of thiourea concentration on the optical, morphological, structural properties of the deposited PbS films were studied. The prepared thin films were uniform, smooth and well adherent. The synthesized films were preferentially orientation along the (111) plane with face centered cubic crystal structure. The XRD studies showed that the lattice strain and dislocation densities of the synthesized PbS films decreased with increasing thiourea concentrations. The surface morphology study confirmed that as the thiourea molar concentrations increased the grains became enhanced. The EDS maps of PbS thin films revealed that the Pb and S elements were homogenously distributed on the surface of the thin films. The FTIR analysis of PbS nanocrystals revealed the presence of Pb-S vibration of Pb-S bonds observed around 613 cm^{-1}. The optical band gap estimated from UV-Vis analyses was found in the range of 0.82 eV to 0.98 eV.

Keywords/phrases: Band gap energy, CBD method, Characterization, FTIR, PbS, thin film

INTRODUCTION

Binary Metal- sulphide semiconductors have been widely studied to assess their potential applications for diverse optoelectronic uses due to their exclusive properties (Deuk Ho Yeon, 2014). Among binary metal sulphides, PbS is one of the most studied semiconductor materials because of its potential applications in nonlinear optical devices (Choudhury N, et al., 2011). PbS is a IV-VI group semiconductor material with a narrow direct band gap of 0.41 eV at room temperature and an optical absorption coefficient of >10^5 cm^{-1} in visible light (Karabulut M, et al., 2014; Choudhury N, et al., 2009; Sadovnikov Si, et al., 2013; M. Karabulut, et al.,2014). Depending on the crystalline sizes the optical band gap of PbS nanocrystalline films found either red shift or blue shift from the bulk value. Due to its functional nature, PbS has been extensively used in many application areas like IR sensor, photography, solar absorber and reflector (Qadria SB, et al., 2003). Furthermore, the nature of PbS possessing multiple carrier generations makes it an appropriate entrant for next generation solar cell industry (E. Veena, et al., 2017). So far, several chemical methods used to synthesize PbS thin films such as spray pyrolysis (S. RaviShankar, et al., 2015), sonochemical (Shu Fe Wang, et al., 2006), SILAR (K.C. Preetha, et al., 2012), spin-coating (Patel J, et al., 2014), chemical bath deposition (CBD) (Deuk Ho Yeon, 2014) etc. Among these deposition methods CBD is one of widely used and most suitable technique to prepare well adherent and uniform thin films with better reproducibility (Fekadu, et al., 2002). In CBD technique the physical and chemical properties of PbS nanocrystalline can be control by optimizing the deposition parameters like bath pH, cation and anion precursor concentrations, deposition temperature and time.

The nature of the reactants influences the whole physical and chemical properties of the deposited thin film. By changing the composition of the reactive solution, competition between the processes of homogeneous and heterogeneous nucleation could be altered to favor thin film

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growth (Hodes G, 2002). Several works revealed that complexing agents, bath pH, deposition temperature (Fekadu, et al., 2018; S. Seghaier, et al., 2006) had a significant influence on the properties of PbS thin films. However, only few works dealing with the influence of sulfur source on PbS thin film. Thiourea (NH_2CSNH_2) is an organic sulfur compound known for its wide range of chemical and physical properties. It has a melting point of 171°C and is highly soluble in water, alcohols, and polar solvents. Due to its strong nucleophilicity and ability to act as a reducing agent, thiourea is used in various applications, such as in the synthesis of pharmaceuticals, as a stabilizer in photographic solutions, and in the preparation of metal sulfide thin films. Additionally, thiourea has been studied in the context of its potential in optoelectronic devices and solar cell applications due to its favorable interaction with metal ions. S. Ravi Shankar et al. (2015) were studied the influence of precursor molar concentrations on the optical, electrical, structural and morphological properties of PbS thin films deposited by using spray pyrolysis technique. Their results justified that varying S: Pb molar ratio had considerable effects on the optical band gap of the prepared thin films. Moreover, they observed a strong blue shift for films with S:Pb molar ratio greater than 0.025:0.025. L. Beddek et al. (2016) used CBD method and reported sulfide precursor concentration and lead source effect on PbS thin film properties. As they used the combination of lead acetate and thiourea they found a better crystallinity than the combination of lead nitrate and thiourea when the thiourea concentration was increased.

The aim of this research work is to study the influence of thiourea concentrations on the optical, morphological, structural properties of PbS thin films prepared by the solution growth method. The prepared samples were characterized by variety of characterization techniques and all the results are revealed that thiourea concentration has a significant effect on the physical properties of PbS thin films.

Experiments and Characterization techniques

Materials and Methods

Substrate cleanliness has direct impact on the uniformity and adherence of the thin film. The microscope glass substrates were degreased in nitric acid about 24 hrs. Then washed trolley by water and soap then after dried at normal condition before being used for the deposition. The following procedures were followed to prepare the PbS thin films: 5 ml of 0.06 M lead nitrate, 10 ml of sodium hydroxide and 7 ml of 0.10 M thiourea was added into a 100 ml beaker sequential. Ammonium hydroxide solution (NH_4OH) was added drop wise to adjust the bath pH around 12, then appropriate amount of distilled water was added in the chemical bath to make the total volume 75 ml. In the present work sodium hydroxide used as a complexing agent and the bath temperature was fixed at 60 ^{0}C. Three more samples were synthesized by changing the thiourea concentrations from 0.15 to 0.25 M as per step of 0.05 M following the above procedure. After 40 minutes the substrates were taken out of the chemical bath and rinsed by distilled water and dried in air. The synthesized thin films were smooth and specula reflective.

Characterization techniques

The as-prepared films were characterized for optical, structural, elemental and morphological properties. Bruker D8 diffractometer operating at 40 mA and 40 KV with Cu$K\alpha$ radiation (λ = 1.5406 Å) used for structural analysis. The surface morphology and composition of the samples were studied by using energy dispersive X-ray analysis (EDX) equipped by a high-resolution JED-2300 scanning electron microscope (SEM) operating with an accelerating voltage 20 KV. The optical absorption of the samples was measured by using a Perkin Elmer Lambda 950 UV–Vis spectrometer. The FTIR spectroscopy study was conducted by Perkin Elmer Spectrophotometer and the samples were scanned from 450 cm^{-1} to 4000 cm^{-1}.

Reaction Mechanism

The CBD method is based on the sequential reaction of anions and cations at the substrate surface. The film growth is achieved through the complex-decomposition process according to the following reactions (Beddek, et al.,2016; U. A. Ubale, et al., 2007)

$$Pb(NO_3)_2 + 2NaOH \rightarrow Pb(OH)_2 + 2\,NaNO_3$$

$$Pb(OH)_2 + 4NaOH \rightarrow Na_4Pb(OH)_6$$

$$Na_4Pb(OH)_6 \rightarrow 4Na + HPbO^{2-} + 3OH^- + H_2O$$

$$SC(NH_2)_2 + OH^- \rightarrow CH_2N_2 + H_2O + HS^-$$

$$HPbO_2 + SH^- \rightarrow PbS + 2OH^-$$

Initially the reaction solution was colorless and changed to brownish black after heating around 12 minutes which is an indication of initiation of chemical reaction. At the end of the deposition time the substrate covered on both sides with gray color PbS thin film.

RESULTS AND DISCUSSION

Structural Analysis

Fig. 1 represented the XRD diffraction patterns of PbS thin film for various thiourea molar concentrations. The measurements were carried out to investigate effect of applying different molar concentrations of thiourea on the microstructural parameters of PbS thin films. The diffraction pattern obtained experimentally was compared to the standard. From Fig.1 it is observed that the intensity and the positions of the diffraction pattern of the samples were well matched with the standard JCPDS code No: 00-005-0592 and indexed as face centered cubic crystal structure of PbS. The synthesized films were preferentially orientation along the (111) plane and the diffraction peaks are corresponding to reflections from the (111), (200), (220), (311), (222), (400), (331) planes. The sharp futures of the peaks in the X-ray diffraction patterns are revealed the good crystallinity of the deposited thin films and also confirmed good stoichiometric nature of the prepared material.

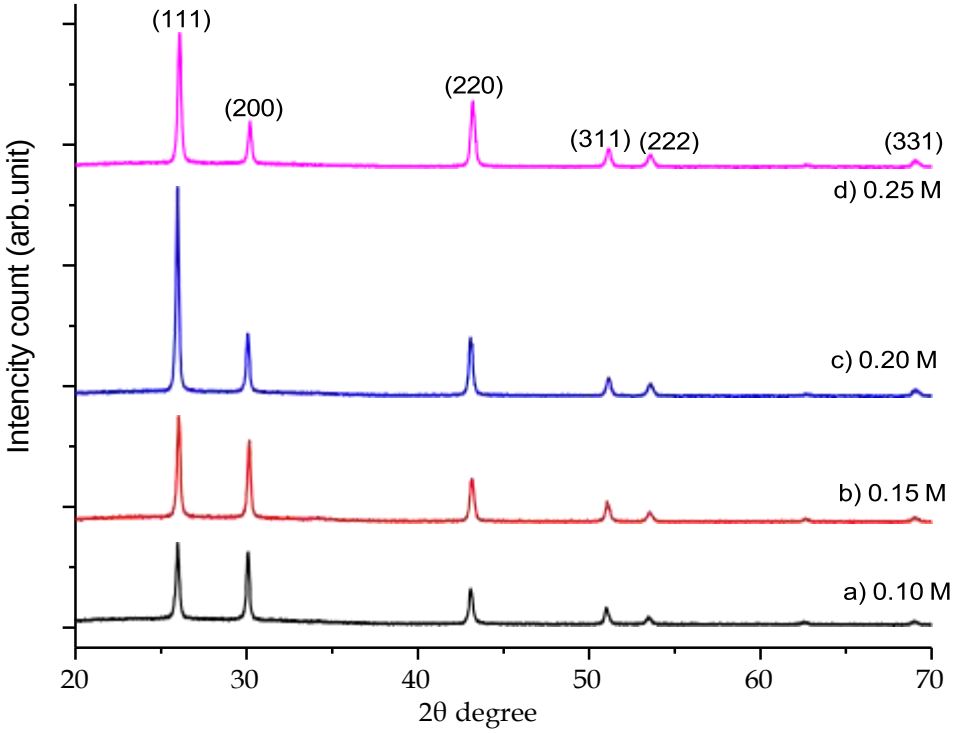

Figure 1: The XRD patterns of PbS thin films prepared at different thiourea molar concentrations

Impurity diffraction pecks from PbO or PbS_2 were not observed, indicating that the as-deposited PbS thin films are pure (Patel J, et al., 2014). The XRD study further established that the intensity of the prefer $D = \frac{0.9\lambda}{\beta Cos\theta}$ on increased with the thiourea molar cor

The increase in XRD intensity up to 0.2 M thiourea can be attributed to the enhancement in the crystallinity and the size of PbS nanoparticles, which is likely due to the presence of thiourea acting as a stabilizing agent, promoting the growth of larger, more ordered crystalline structures. Thiourea could facilitate the formation of well-defined PbS crystals by controlling nucleation rates during the synthesis process. However, the observed decrease in XRD intensity at 0.25 M thiourea could be due to an excess of thiourea leading to a saturation point where it begins to act as a capping agent, hindering further crystal growth and potentially leading to smaller, less crystalline structures. The lattice parameter a (Å) for the FCC structure is mathematically related to the d-space by the following equation:

$$a_{hkl} = d_{hkl}\sqrt{h^2 + k^2 + l^2} \quad (1)$$

Miller indices and the d-space values can be obtained from the X-ray diffraction spectrum; hence the lattice parameter a_{hkl} can be easily calculated from the above mathematical relation. Lattice constant a_{hkl} calculated from (111) plane and the values are listed Table-1. The estimated values of the lattice constant showed that slight deviation from its standard value of 5.9362 Å which is evidence of the presence of strain. Our experimental results show that varying thiourea concentration affects the lattice parameter due to changes in film stoichiometry and sulfur availability during deposition. Higher thiourea concentrations lead to increased sulfur content, which can cause variations of the PbS lattice due to the incorporation of sulfur atoms into the crystal structure. Eq. 2 (Debye-Scherrer relation) used to estimate the crystallite size of the samples.

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The variables in Eq. 2 is defined in Ref. (Fekadu, et al., 2017). The XRD analyses revealed that crystallite size of PbS thin films were found to vary from 37.46 nm to 44.85 nm with increasing thiourea precursor concentrations. Similar observation was reported by L. Beddek et al. (2016). There are different causes for the origin of strain in the thin films. Mathematical the lattice strain (ε) was calculated by using the relation in Eq.3:

$$\varepsilon = \frac{\beta Cos\theta}{4} \quad (3)$$

The dislocation density (σ), defined as the length of dislocation lines per unit volume, has been estimated using the following equation:

$$\sigma = \frac{1}{D^2} \quad (4)$$

The microstructural parameters (strain and dislocation density) were calculated from the most intense peak of the X-ray diffraction pattern for each PbS thin films deposited at different thiourea molar concentrations. From Table 1, it can be observed that the dislocation density and strain decreased with increasing thiourea molar concentrations. Since dislocation density and strain are the manifestation of dislocation network in the films, the decrease in dislocation density indicates the formation of high-quality films (T. Tohidi, et al., 2015). As the dislocation density is the measure of the defects in the crystalline structure, the smaller value of σ obtained for the film deposited at 0.25 M of thiourea mola concentration, suggests the film has a comparatively higher degree of crystallinity. The calculated structural parameters and the energy band gaps are tabulated in Table 1.

Table 1: Microstructural values and optical band gap energies of PbS films

S^{2-} conc.	2θ	hkl plane	Standard Å	Observed Å	FWHM (β)	Eg (eV)	Dislocation density(σ)lines/ m^2	Strain (ε) lines^{-2} m^{-4}
0.10 M	26.116	111		5.939	0.2275	0.98	7.13×10^{-4}	9.66×10^{-4}
0.15 M	26.070	111		5.931	0.2243	0.91	6.93×10^{-4}	9.53×10^{-4}
0.20 M	26.017	111	5.9362	5.945	0.2212	0.86	6.88×10^{-4}	9.49×10^{-4}
0.25 M	26.001	111		5.938	0.1900	0.82	8.07×10^{-4}	4.97×10^{-4}

Fig. 2 represents the variations of crystalline size with respect to sulfur molar concentrations. From the figure it is clearly notice that as the thiourea molar concentrations increased the crystal size enhanced this may be due to the availability of sufficient amounts of S^{2-} ions in the chemical bath to form a lot of PbS nucleation on the substrate which gradually grow to big crystals.

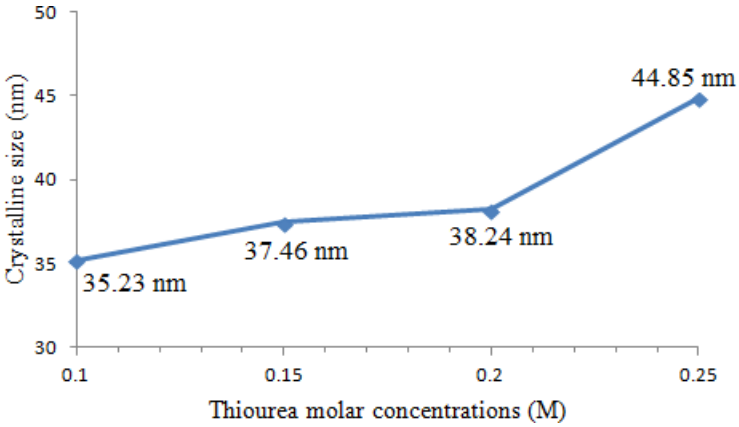

Figure 2: Plot of crystallite size (nm) versus thiourea molar concentrations (M).

Compositional and Morphological Analyses

The EDX analysis was applied to confirm the composition of the deposited films for different thiourea molar concentrations. Fig. 3 represents the EDX spectra of PbS thin films deposited from 0.10 M and 0.25 M thiourea concentrations. The measurement was performed at different locations in the same sample, resulting in the identification of similar chemical compositions, which confirmed the fairly uniform composition of nanocrystals and presences of the expected elements Pb and S. The pecks from calcium, silicon, oxygen and sodium elements comes from the glass substrate used to deposited the thin films. The peak intensities from the impurity elements become decreased as thiourea molar concentrations increased. This may be due to thicker film formed for the higher thiourea concentration, thus, decreased the signals emanating from the substrate (Fekadu, et al., 2015).

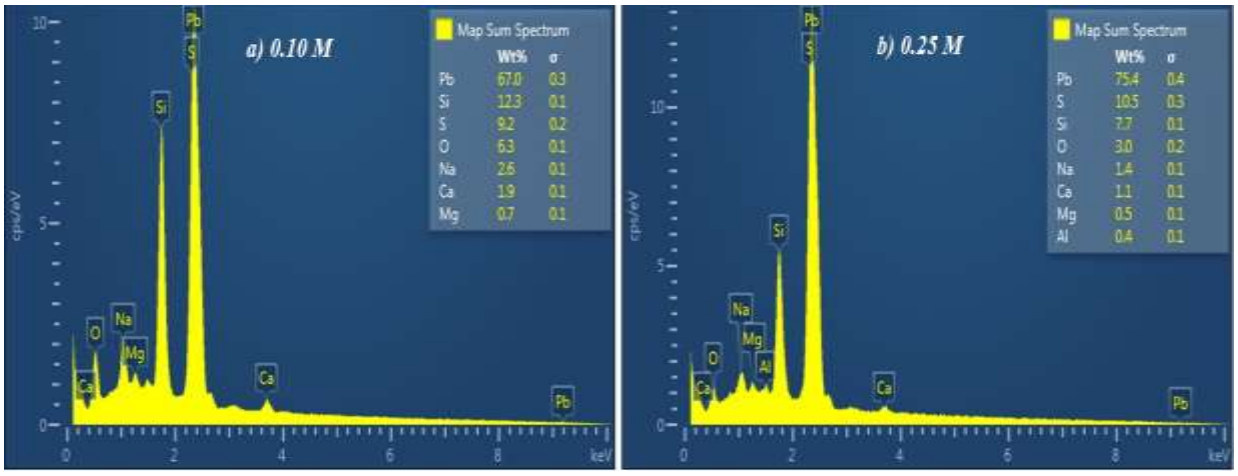

Figure 3: The PbS EDX spectra for different thiourea molar concentrations.

To confirm the compositional distributions of the constituent elements of the samples, the EDX maps of PbS films deposited from 0.10 M and 0.25 M are shown in Figs. 4. It is clear that the Pb and S elements were homogenously distributed on the surface.

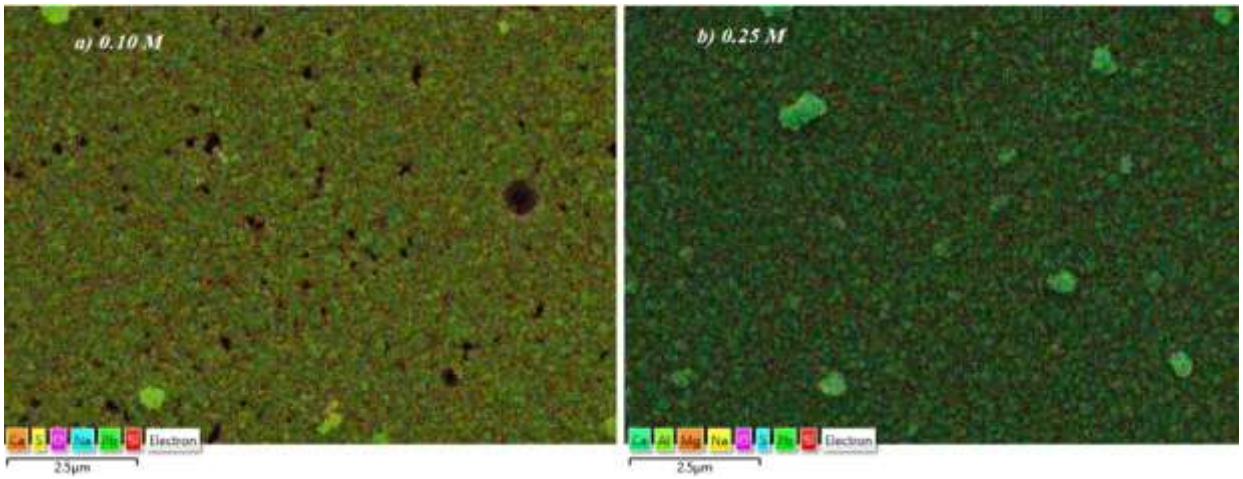

Figure 4: The PbS EDS legend images for different thiourea molar concentrations.

Fig. 5 shows the SEM images of PbS thin films deposited at 0.10 M and 0.25 M thiourea molar concentrations at a magnification of 10KX. The machine was operated at a working distant of 10 nm. The representative thin film SEM images show uniform and well adherent surface morphology over the entire glass substrate without visible defects. Some over growth was observed on both samples; this may be due to colloidal particles formed in the solution and adsorbed on the surface of the films during the growth process. The surface morphology study confirmed that as the thiourea molar concentrations increased the grain became enhanced which is consistent with the XRD results.

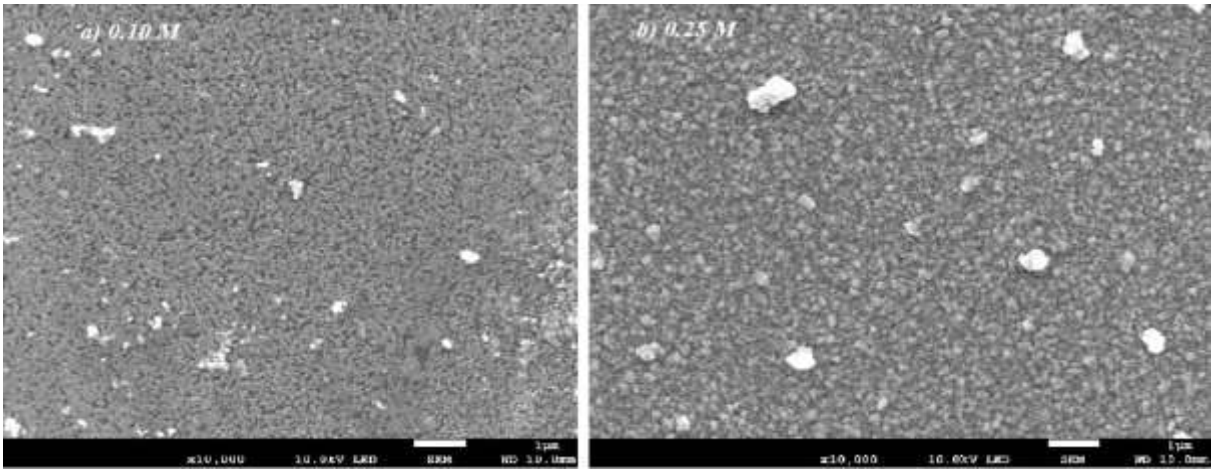

Figure 5: The PbS SEM images for various thiourea molar concentrations.

UV-Vis analysis

The optical band gap of the thin films was estimated from absorbance data by using the Stern relation (Eq.5):

$$A = \frac{(k[h\upsilon - E_g])^{n/2}}{h\upsilon} \quad (5)$$

The constants in Eq 5 defined in Ref. [20]. PbS is a direct band transition hence, n was taken as one. The linear nature of the plot of versus shown in Fig. 6 verifies the presence of a direct transition. The optical band gap energy was estimated by extrapolating the linear portion of $(Ah\upsilon)^2$ versus $h\upsilon$ to the energy axis at $(Ah\upsilon)^2 = 0$.

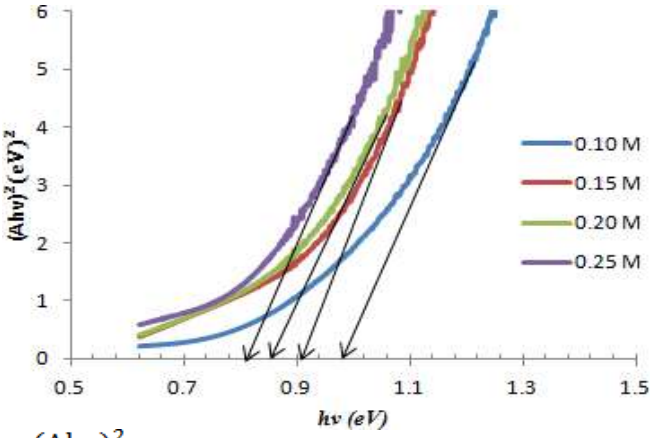

Figure 6. The plot of $(Ahv)^2$ versus the photon energy for various thiourea concentrations.

This intercept gives the value of the optical band gap and the calculated values from Fig.6 are presented in Table-1. From the Fig.6, it is observed that the optical band gap decreased from 0.98 to 0.82 eV as the molar concentration of thiourea increased from 0.10M M to 0.25 M. This reduction of the optical band gap is consistent with theoretically shown band gap dependence on crystal size (N. Choudhury, et al., 2008). As the concentration of the dopant (or impurity) increases, the material's electronic structure is altered. Specifically, introducing more dopants or defects can introduce new energy levels within the band gap. These levels can act as intermediate states that facilitate the transition of electrons between the conduction and valence bands, reducing the energy required for electron excitation. This effect is particularly notable in semiconductors and nanomaterials, where increasing the dopant concentration can lead to the formation of impurity states or localized states that effectively reduce the band gap. The estimated band gaps in the present study were slightly higher compared to the bulk value (~ 0.41 eV), this may be due to the change of dislocation density, crystallite size and microstrain of the samples with thiourea concentrations (Fekadu, et al., 2016).

FTIR Analysis

FTIR measurements were conducted on all samples within the range of 400 cm^{-1} to 4000 cm^{-1}, and the spectral features were consistent across different thiourea molar concentrations. Therefore, the FTIR spectrum of the 0.15 M thiourea concentration is selected for discussion and is displayed in Fig. 7.

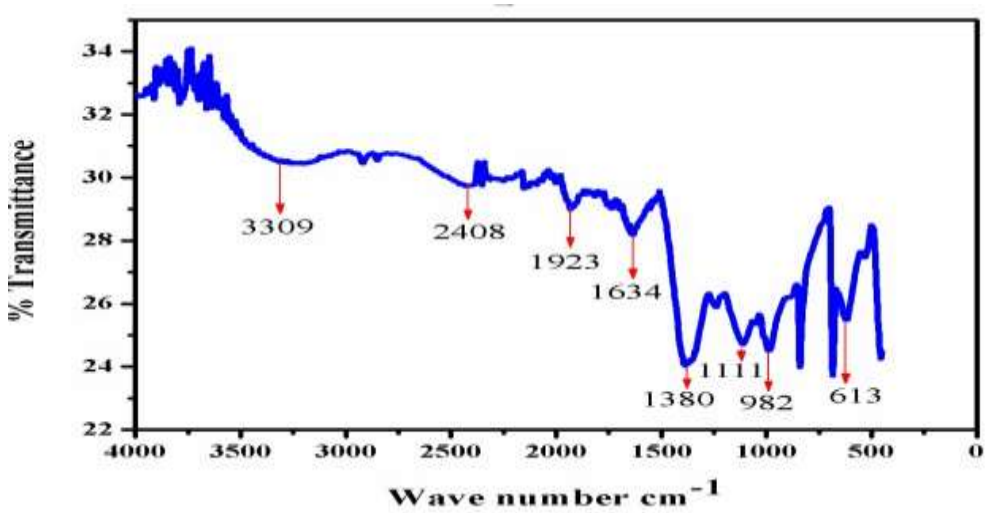

Figure 7: Representative FTIR spectra of PbS thin film for 0.15 M thiourea concentration.

The characteristic peak observed at 1380 cm^{-1} is assigned to asymmetric bending vibrations of C–H (Mohammadreza Khan Mohammadi, et al., 2010) and the peak sulfate ion (SO_4^{2-}) absorbance band was identified at 1111 cm^{-1} wave number (Charity Coury, et al., 2008). The pecks in the range of 880 to 750 cm^{-1} assigned by OH stretching vibration of carboxylic groups (Gang Cao, et al., 2018). The band sited in the range of 1912 to 1925 cm^{-1} was related to one of the fundamental frequencies of COO^- bonds (O. Portillo-Moreno, et al., 2016). A strong =C-H bending is observed in the range of 675 - 1000 cm^{-1} and also the frequency of metal– sulfur vibration bonds (M–S) can be seen 613 cm-1 (Fatemeh Behoudnia, et al., 2012). From the FTIR results it was also noticed that as the thickness of the films were improved the transmittance was decreased.

CONCLUSION

In this study, PbS thin films were successfully synthesized on glass substrates using the Chemical Bath Deposition (CBD) method with precursor solutions containing varying molar concentrations of thiourea. The results demonstrated that the thiourea molar concentration significantly influenced the structural, optical, and morphological properties of the films. X-ray diffraction (XRD) analysis showed that the crystallite size increased from 37.46 nm to 44.85 nm as the thiourea concentration was varied from 0.10 M to 0.25 M. This increase in crystallite size was accompanied by a decrease in dislocation density and lattice strain. The structural analysis confirmed that the films exhibited a face-centered cubic crystal structure. Scanning electron microscopy (SEM) images revealed that the films were uniform, compact, and well-adhered to the substrate. Energy-dispersive X-ray (EDX) analysis confirmed the presence of lead (Pb) and sulfur (S) elements. Additionally, optical absorption measurements indicated that the PbS thin films exhibited direct bandgap transitions, with the bandgap energy decreasing from 0.98 eV to 0.82 eV as the thiourea molar concentration increased from 0.10 M to 0.25 M.

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