

Optical and Structural Studies of Tin Disulfide (SnS₂) Synthesized by Facile Hydrothermal Method

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Abstract: Transition metal dichalcogenides (TMDCs) have found diverse applications for intrinsic semiconducting and tunable electronic properties mediated via the van der Waals interaction between the adjacent chalcogen planes. Therefore, the large-scale production of TMDCs without compromising the material properties is the major challenge at the present moment. We follow a simple, cost-effective, and environmentally friendly hydrothermal technique for synthesizing tin disulfide (SnS₂). The optical properties are investigated using UV-visible, photoluminescence (PL), Raman, and FTIR spectroscopy. The UV-visible absorption spectra of pure SnS₂ show a broad absorption peak located at 340 nm, and the corresponding optical band gap energies are estimated to be 2.14 and 4.02 eV, respectively. The PL spectra display a sharp emission peak at 561 nm due to the radiative recombination of bound excitons for the excitation wavelength of 340 nm. FTIR is used to observe the existence of functional groups in the material. The absorption peak observes at 625 cm⁻¹ corresponds to the vibration of the Sn-S bond. From Raman spectra, a sharp peak appears at 314 cm⁻¹ corresponding to the A_{1g} mode of pure SnS₂, which occurs due to the out-of-plane stretching vibration of sulfur atoms in the SnS₂ material. The XRD is used to identify structural phases. The sharp diffraction peaks at 2 θ = 15.17° and 28.55° corresponds to (001) and (100) planes, respectively, that suggest the hexagonal phase of pure SnS₂. Interplanar spacing is estimated using Bragg's law, and the value is found to be 5.83 Å. The average crystallite size is estimated to be 28 nm from the Williamson-Hall plot, which is comparable with the crystallite size calculated from Scherrer's formula.

Keywords: tin disulfide; hydrothermal; band-gap; Tauc's plot.

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1. Introduction

The layered semiconducting materials transition metal dichalcogenides (TMDCs) are well-known for their fascinating electronic and optoelectronic characteristics, sizeable and tunable band-gap, and high in-plane carrier mobility [1,2]. Therefore, understanding the optical and electronic properties of TMDCs originated from their structural confinement is crucial from a technological and device application perspective [3-5]. Among the different TMDCs, tin disulfide (SnS₂) has drawn promising interest because of its superb electronic and optical properties [6-10]. The SnS₂ is a layered n-type semiconducting material having a band gap in the range between 2-3 eV [1,11], which makes it a promising candidate for various exploitations such as solar cells, optoelectronic devices, and lithium cells, sodium ion storage, sensors, nano semiconductor devices, etc. [12-24]. The band-gap energy of the nanomaterials

depends on the number of layers. Hence, the electronic properties can be tuned easily through precise control. The structure of SnS₂ consists of tin cations sandwiched between two layers of hexagonal closed pack (hcp) sulfur anions through weak van der Waals force between the layers; however, the strong force is present in the layer through in-plane covalent bonding [25]. The tin cations are coordinated octahedrally with six nearest neighbor sulfur anions. Different synthesis techniques are followed to synthesize SnS₂ material, such as solvothermal, hydrothermal, laser ablation methods, sonochemical [26-32], etc. Among these methods, the hydrothermal method is preferable because it is simpler, less harmful, environmentally benign, cost-effective, and large yield. SnS₂ nanomaterial is prepared in this article by a simple hydrothermal method, and the optical and structural properties are investigated and reported here.

2. Materials and Methods

Stannic chloride pentahydrate (SnCl₄·5H₂O) (Loba Chemie Pvt. Ltd.), DI water, thioacetamide (C₂H₅NS) (TAA) (Loba Chemie Pvt. Ltd), and ethanol were used as received for the synthesis of SnS₂ without any further purification. Tin disulfide (SnS₂) was synthesized by following the hydrothermal method as follows. In a typical synthesis (schematically shown in figure 1), 1.72 g of stannic chloride pentahydrate (SnCl₄·5H₂O) and 0.98g of thioacetamide (TAA) were added in 38 mL of DI water. It was then stirred for 5 hours on a magnetic stirrer at room temperature to get the clear homogeneous solution. The resulting solution was then put in a 50 mL Teflon-lined stainless steel autoclave. The autoclave was then heated to 180 °C and maintained for 12 hours. After heating, the autoclave was kept for cooling down naturally. The precipitate was then centrifuged with DI water, followed by ethanol three times each. After drying in a hot air oven at 80 °C for 6 hours, the final yellow product was obtained. The obtained product was then characterized through different optical and structural characterization techniques such as X-ray Diffractometer (XRD) (Xpert³ MRD XL, Panalytical), UV Vis NIR Spectrophotometer (Agilent, Model: Cary 5000), Photoluminescence spectroscopy (Fluoromax-4C Spectrofluorometer, Horiba), Raman Spectroscopy (Make: Horiba Jobin Vyon, LabRam HR), FTIR (Bruker ALPHA II compact spectrophotometer).

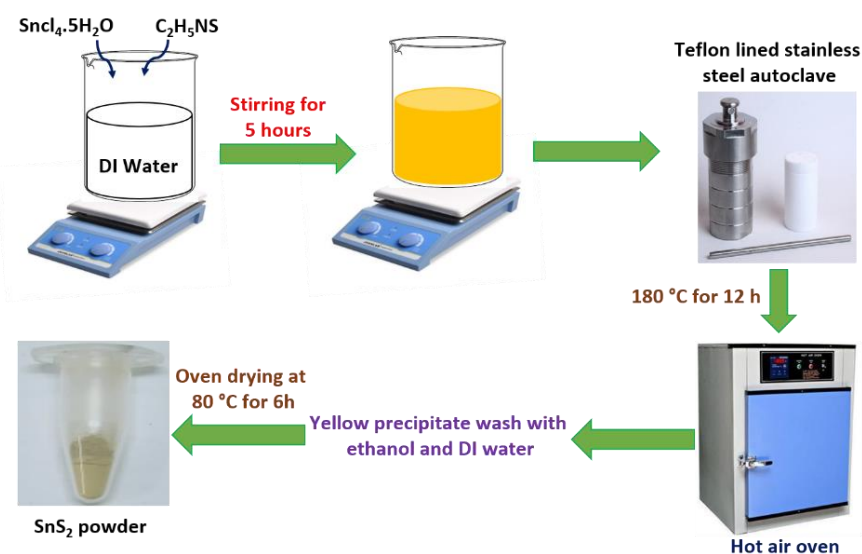


Figure 1. The schematic diagram for the synthesis of SnS₂.

In the synthesis procedure stannic chloride dissociates into Sn^{4+} ions and then reacts with H_2S that is produced during the hydrolysis process of thioacetamide to form SnS_2 ($\text{CH}_3\text{CSNH}_2 + 2\text{H}_2\text{O} = \text{CH}_3\text{CO}_2\text{NH}_4 + \text{H}_2\text{S}$; $2\text{H}_2\text{S} + \text{Sn}^{4+} = \text{SnS}_2 + 4\text{H}^+$).

3. Results and Discussion

3.1. Optical studies.

3.1.1. UV-visible DRS spectroscopy.

The UV-vis diffuse reflectance (DRS) spectra of SnS_2 are performed in the wavelength band of 200-800 nm, as displayed in figure 2(a). A broad hump appears at 340 nm, and also other three peaks are observed at 235, 295, and 395 nm, respectively, after deconvolution [33-35]. The optical band gap energies are estimated using Tauc's plot, and the values are found to be 2.14 and 4.02 eV, respectively, as shown in figure 2(b) using the formula $(\alpha h\nu)^n = A (h\nu - E_g)$ (where the absorption coefficient is represented by α , h corresponds to Plank constant, ν represents frequency, n corresponds to the type of transition, E_g represents band-gap, A is a constant).

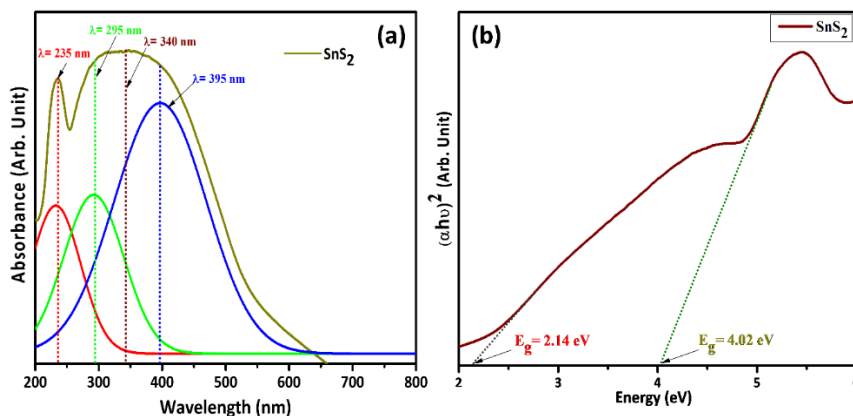


Figure 2. (a) UV-vis DRS spectroscopy of SnS_2 ; (b) Tauc's plot of SnS_2 .

3.1.2. Photoluminescence (PL) spectroscopy.

The PL spectrum of SnS_2 is performed for the excitation wavelength 340 nm ($\lambda_{\text{ex}} = 340$ nm), as shown in figure 3. It is related to the emission of photons and the recombination of electron-hole. The PL spectrum shows a sharp emission peak at 561 nm, possibly because of the radiative recombination of bound excitons [36-39].

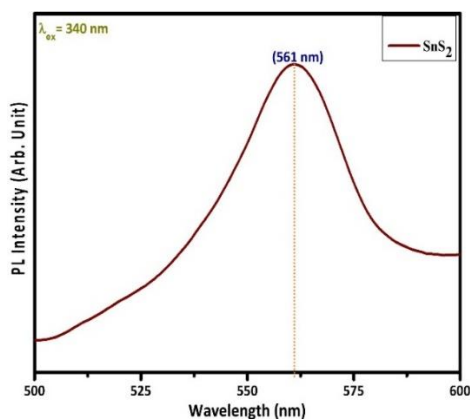


Figure 3. PL spectroscopy of SnS_2 .

3.1.3. FTIR spectroscopy.

FTIR analysis of SnS₂ is performed in the wavenumber region of 4000-500 cm⁻¹, as shown in figure 4. It is performed to examine various functional groups in the material. The peak observed at 3410 cm⁻¹ represents the vibration of the O-H bond. A peak at 625 cm⁻¹ represents the Sn-S stretching vibration. The peak observed at 1711 cm⁻¹ represents the existence of the carboxylic group [40-43].

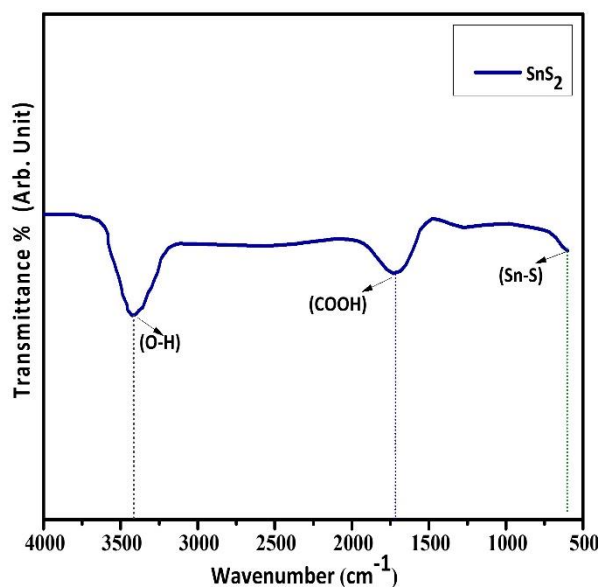


Figure 4. FTIR spectra of SnS₂.

3.1.4. Raman spectroscopy.

Raman spectra of SnS₂ nanocrystal are displayed in figure 5. The peak at 314 cm⁻¹ represents A_{1g} mode of SnS₂, which occurs due to the out-of-plane vibration of sulfur atoms that signifies the presence of SnS₂ [44]. Another small peak observed at 206 cm⁻¹ represents E_g mode, which is very weak and occurs due to plane stretching vibration [44,45].

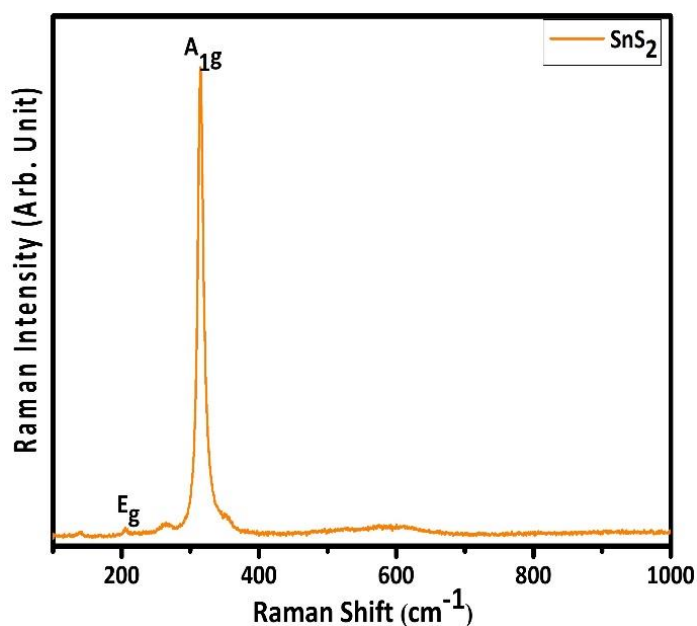


Figure 5. Raman spectra of SnS₂.

3.2. Structural analysis of SnS₂.

3.2.1. X-ray Diffraction (XRD).

The structural phase and interplanar spacing of SnS₂ are calculated using XRD analysis as shown in figure 6. It is performed in the 2θ range of 5° to 80° with CuK_α source with a wavelength of radiation λ = 1.54Å. Different peaks are observed at 15.17°, 28.52°, 30.42°, 32.41°, 42.13°, 46.31°, 50.29°, 52.66°, 55.15°, 58.78°, 59.77°, 60.91°, 63.14°, 67.59°, 70.63°, and 77.71° which corresponds to (001), (100), (002), (101), (102), (003), (110), (111), (103), (200), (112), (201), (004), (202), (113), (203) planes, respectively [40,42,46-48]. These diffraction peaks confirm the hexagonal crystal structure of the material. Bragg's law $2d \sin\theta = n\lambda$ is used to determine the interplanar spacing of the material (where, n=1, d, and θ represent interplanar spacing and Bragg angle, respectively, and λ is the wavelength of X-ray) and is found to be 5.83 Å. Scherrer's formula is used to determine average crystallite size, $D = \frac{k\lambda}{\beta \cos\theta}$ (where λ and β represent the X-ray wavelength and full-width half maxima, respectively. k is Scherrer constant (0.90), and θ is peak position in radian) and is found to be 28 nm. From W-H plot, the strain of the material is calculated, which is equal to -0.00011 (Negative value represents the compressive strain in the material), and also the average crystallite size is estimated to be 28 nm which is comparable with the crystallite size (27.68 nm) that is calculated from Scherrer's formula.

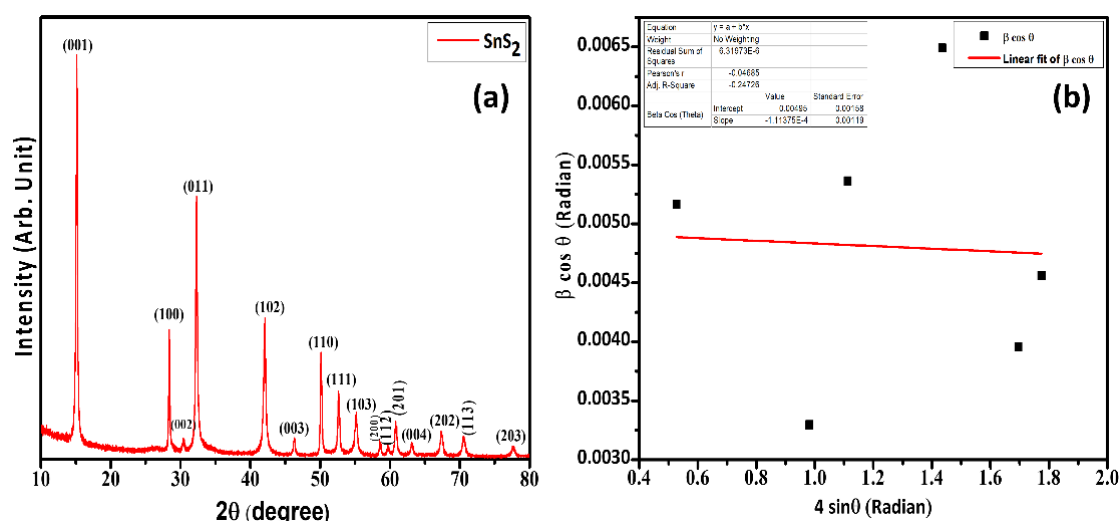


Figure 6. (a) XRD pattern of SnS₂; (b) Williamson-Hall (W-H) plot of SnS₂.

4. Conclusions

In this article, we follow a simple, environmentally benign, and cost-effective hydrothermal method to prepare SnS₂. The optical and structural investigations have been done for the synthesized material. Raman spectra depict the A_{1g} mode of vibration that occurs due to of plane vibration of sulfur atoms. PL emission spectra and UV-visible absorption spectra reveal electrons' emission and excitonic absorption in the material. An absorption peak near 625 cm⁻¹ represents the existence of Sn-S vibration. The presence of different diffraction peaks suggests the hexagonal crystal structure of the material. Realization of better optical and structural behavior of the material can have found many exploitations in electronic and optoelectronic devices in coming years.

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Conflicts of Interest

The authors declare no conflict of interest.

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