

Synthesis and study of the structural and optical properties of SnS and SnO₂/SnS thin films prepared by spray pyrolysis

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Abstract

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This study aims to synthesize and characterize the structural and optical properties of tin sulphide (SnS) and tin oxide/tin sulphide (SnO₂/SnS) bilayer thin films by spray pyrolysis technique from aqueous solutions. X-ray diffraction revealed polycrystalline structure of the SnS films of mixed orthorhombic and zincblende phases, while the deposition of the upper SnO₂ layer introduced additional crystalline SnO₂ phases. Field emission scanning electron microscopy (FE-SEM) indicates a uniform porous structure of SnS, as well as significant variations in surface morphology after the deposition of the upper layer. Photoluminescence spectroscopy (PL) revealed band-to-band transitions and defect-related emissions; an additional emission peak at 420 nm, attributed to SnO₂ band-to-band transitions, appeared and intensified with increasing SnO₂ thickness. UV-visible absorbance spectroscopy indicated an overall increase in absorbance spectra and slight variations in the optical bandgap induced by the incorporation of oxide states in the SnS matrix. These findings highlight the ability to modify the structural and optical properties of SnO₂/SnS heterostructures, indicating their potential use in photocatalytic applications.

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1. Introduction

Stannous sulfide (SnS) is an IV–VI has a band gap close to the optimum value for solar absorbers in a photovoltaic devices [1]. SnS is a p-type semiconductor, and is preferred for use in many applications due to its low cost and non-toxic nature. It can crystallizes in Zincblende and orthorhombic structures [2]. SnS thin films have significant interest due to their unique optical, electrical, and structural properties. Various deposition routes have been used to deposit SnS thin films including simple and complex techniques, each with distinct advantages and limitations [3]. Among these techniques, spray pyrolysis stands out as a simple method involving the spraying of a precursor solution onto a heated substrate [4]. This technique is particularly useful for large-surface deposition. It was employed for different fields of applications [5]. SnS thin films are used in optoelectronic device applications such as photodetectors [6], light-emitting diodes (LEDs) [6], and as an absorber layer in solar cells due to their favorable band gap, high absorption coefficient, and superior charge carrier mobility [7]. In addition, SnS thin films exhibit good sensitivity to many gases, making them suitable for gas sensing applications, including detection of toxic gases in factories and monitoring of indoor and outdoor air quality [8]. Furthermore, SnS thin films have been applied in many studies in photocatalytic technology [9], such as water decomposition for hydrogen generation [10], and analysis of organic pollutants in wastewater treatment processes [11].

Wang et al. (2020) [12] utilized a SnS-SnO₂ composite for detecting mercury in the atmosphere. The sensor exhibited high response of 14 mg/m³ of mercury, and a high selectivity towards various harmful gases. Jana et al. (2022) [13] fabricated a SnS-SnO₂ heterostructure by

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oxidization the surface of SnS thin films. The result demonstrated a p-n junction with improved rectifying properties under light exposure. The prepared layers developing treatment process for the removal of toxic Cr(VI) from contaminated water. It reduces approximately 95% of Cr(VI) to Cr(III) within 100 minutes under visible light irradiation. Josephine et al. [14] investigated SnS/SnO films for photovoltaic applications using the SILAR approach. The crystallite size and structure vary concentration. The 0.1 M concentration of tin ions showing rods structures. The electrical conductivities of the deposited SnS/SnO material increased proportionally with thickness. Aparna et al. (2023) [9] employed the SLAR method, a chemical approach, to produce SnS thin films. The deposition process resulted in smooth, homogenous, and uniform films having an orthorhombic structure.

The aim of this study is to synthesize, and characterize the structural and optical properties of SnS thin films, as well as SnO₂/SnS thin films, utilizing the spray pyrolysis technique from aqueous solutions. Structural characterization will involve analyzing the crystalline nature of the films and assessing the impact of the upper layer of SnO₂. Optical analysis will also be conducted to study the optical properties of the films. The study aims to explore the possible use of the deposited thin films in photocatalytic applications. While previous studies have explored SnS/SnO₂ heterostructures using methods such as SILAR and surface oxidation, limited work has been conducted using the spray pyrolysis technique for controlled layering of SnO₂ over SnS. This study introduces a simple route to fabricate SnO₂/SnS thin films via spray pyrolysis, aiming to understand how the structural and optical properties evolve with the incorporation of varying SnO₂ thicknesses. The novelty lies in correlating the morphological and photoluminescent changes induced by the SnO₂ overlayer with enhanced light absorption and emission behavior, which are critical for optimizing photocatalytic performance.

2. Experimental Work

SnS thin films were deposited by spray pyrolysis onto corning glass at 400 °C substrate temperature using aqueous solutions of metal salts. The substrates were ultrasonically cleaned in methanol and distilled water for 15 minutes each, followed by drying in blowing nitrogen. Tin chloride (SnCl₂·2H₂O) and sodium thiosulfate (Na₂S₂O₃·5H₂O) from Sigma Aldrich were used as sources for Sn and S, respectively. Each precursor was dissolved separately in 100 ml distilled water to form 0.1 M solutions, which were subsequently combined to produce the final solution. Additional 0.25 ml of concentrated HCl was added to the solution. To enhance the properties of the thin film for photolytic applications, upper layers of SnO₂ were deposited at various thicknesses by spraying different volumes (0.5, 1, 1.5, and 2 ml) of SnCl₂·2H₂O solution (0.1 M) onto the pre-prepared SnS thin films, forming n-p nano-heterojunctions between the upper layer and base SnS. These samples were noted as SS1, SS2, SS3, and SS4 respectively. Structural properties, surface characteristics, and optical properties of the as-deposited layers were characterized using X-ray diffraction (Shimadzu XRD 6000), scanning electron microscopy (JSM-7600F JEOL), Fourier transform infrared spectroscopy, and UV-visible absorption spectroscopy (SP-8001 spectrophotometer).

3. Results and Discussion

(Fig. 1) displays the XRD patterns of bare SnS and SnO₂/SnS thin films at varying upper layer thicknesses. The SnS thin film displays polycrystalline structure of mixed Orthorhombic and Zincblende SnS phases. The dominant phase is Zincblende, corresponding to crystallographic data in the JCPDF database with the number 01-071-3678, exhibiting preferred orientation along the (111) crystalline plane at a diffraction angle of 26.6172°. While, the minor phase exhibits an orthorhombic structure, matching the JCPDF number 00-039-0354. In the literature, zinc blende SnS is a metastable phase, while the most stable phase is orthorhombic. The prepared films demonstrate significant potential for application across various domains owing to their distinctive structural and crystalline attributes [15,16]. Following the deposition of the upper layer, an additional phase of tetragonal SnO₂ emerged, characterized by diffraction peaks located at 33.7°, 38.9°, and 51.7°, corresponding to Miller indices of (101), (111), and (310), respectively, according to JCPDF database No. 96-900-9083. An observable decrease in

diffraction peaks corresponding to the SnS structure was noted with increasing upper layer thickness, attributable to the coverage provided by the upper layer. Conversely, the intensity of SnO₂ diffraction peaks increased with its thickness, indicating enhanced crystallinity. This phenomenon can be attributed to the availability of more material for grain growth, leading to larger grains with more ordered crystal structure, thereby contributing to increased crystallinity. Furthermore, more film thickness causes more opportunities for the merge among adjacent grains. The Bragg equation was employed to determine the inter-planar spacing (d_{hkl}) [17].

$$n\lambda = 2d_{hkl} \sin\theta \quad (1)$$

where λ is the wavelength of X-rays, θ is the diffraction angle, and n is the diffraction order. While, Scherrer's formula was utilized to calculate the crystallite size values (D), as detailed in Table 1 [18]:

$$D = \frac{0.9 \lambda}{FWHM \cdot \cos(\theta)} \quad (2)$$

Where FWHM represents the breadth of diffraction line at half intensity. The low crystallite size of the SnS thin film renders it suitable for various applications such as gas sensors and photocatalytic applications.

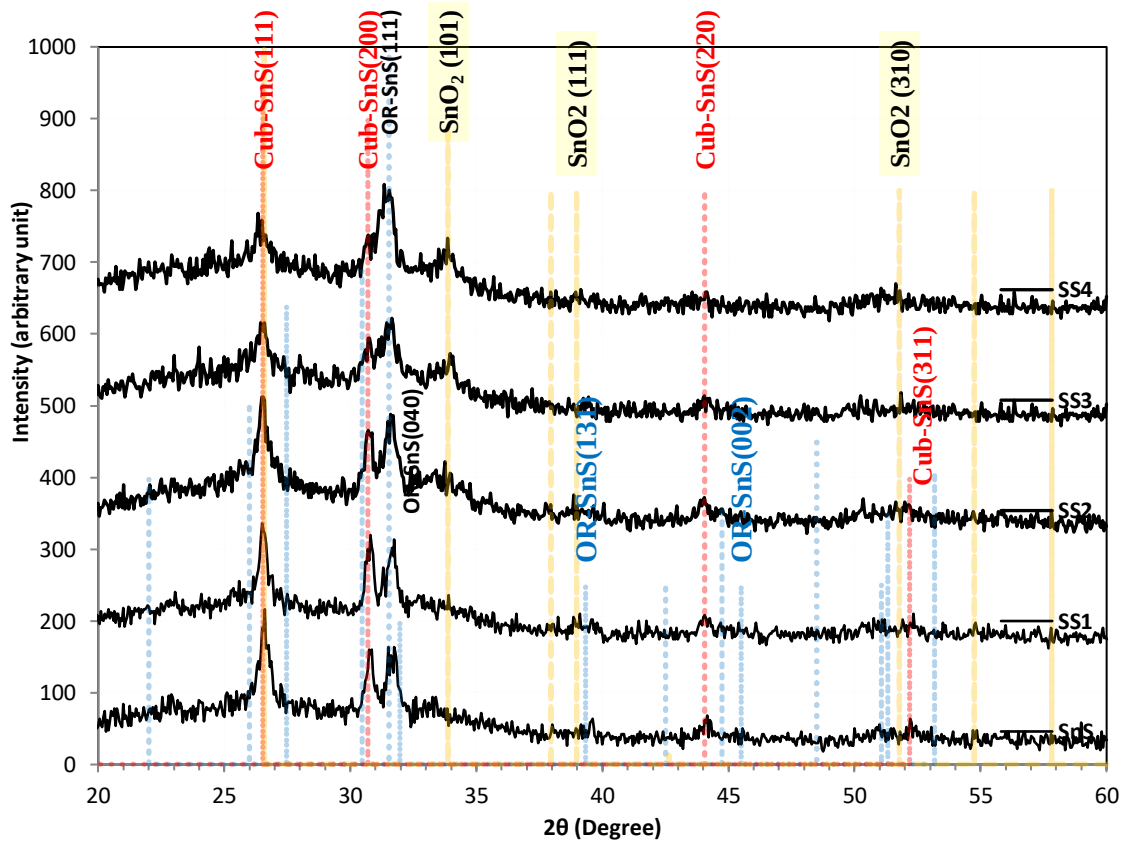


Fig. 1. XRD of bare SnS thin film and decorated with SnO₂ at different thicknesses

The influence of crystallinity through the upper layer thickness variation presents a promising approach for modifying thin films for specific requests [19]. In the field of photocatalysis, the controlled crystallinity can impact optical properties such as bandgap and absorption coefficients, adapting these materials suitable to harvest high amount of solar energy. The Bragg equation was employed to determine the inter-planar spacing (d_{hkl}), while Scherrer's formula was used to calculate the crystal size values (D), as detailed in Table 1. Figure 2 presents the FE-SEM images at two magnification powers of SnS thin films deposited onto glass substrate. The films surface appeared as interconnected nanostructures, forming a complex porous form with

cavities that result in a large surface area. The thickness of the separating walls that constitute the overall structure is approximately 500 nanometers. This porous structure holds significant implications for photocatalysis, as it offers large spaces for interactions between the catalyst and target reactants. The presence of pores allowed to penetrate reactants, thus enhancing the catalytic efficiency, ensuring optimal contact between the substance of high surface area with reactants. As a result, the porosity improved the efficiency of the photocatalytic reaction [20].

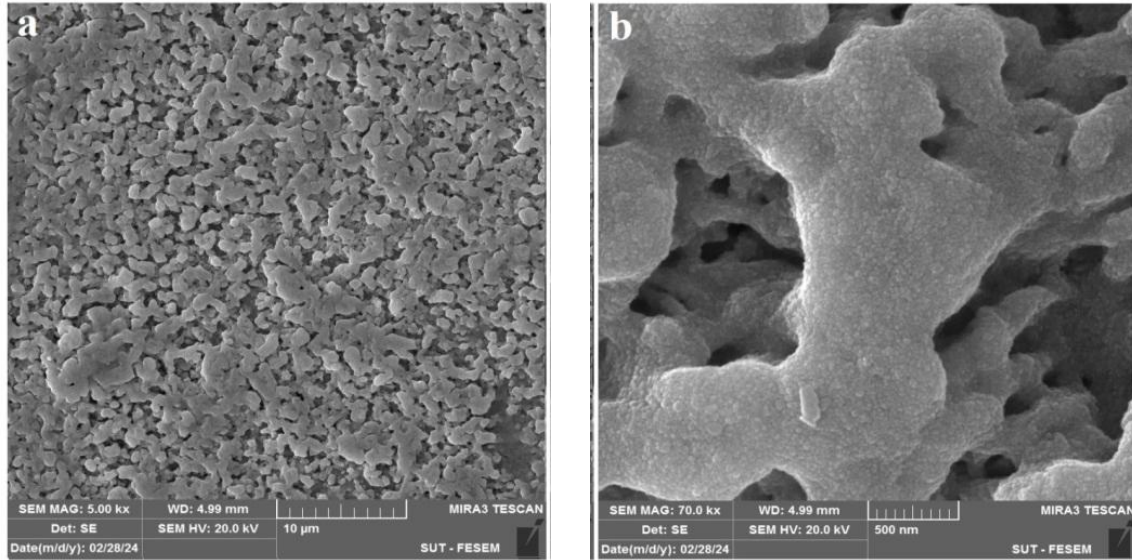


Fig. 2. FE-SEM images of as deposited SnS thin film

Table 1. XRD parameters of bare SnS thin film and decorated with SnO₂ at different thicknesses

Sample	2θ (°)	FWHM (°)	dhkl (Å)	D (nm)	Phase	hkl
SnS	26.6172	0.5090	3.3463	16.0	Cubic SnS	(111)
	27.2959	0.3182	3.2646	25.7	Orth. SnS	(021)
	30.7953	0.3605	2.9011	22.9	Cubic SnS	(200)
	31.5376	0.2757	2.8345	29.9	Orth. SnS	(111)
	31.7497	0.3817	2.8161	21.6	Orth. SnS	(040)
SS1	26.5102	0.3195	3.3595	25.5	Cubic SnS	(111)
	30.7838	0.3795	2.9022	21.7	Cubic SnS	(200)
	31.7624	0.4194	2.8150	19.7	Orth. SnS	(111)
SS2	26.5502	0.4593	3.3546	17.8	Cubic SnS	(111)
	30.7639	0.3594	2.9040	22.9	Cubic SnS	(200)
	31.6226	0.5392	2.8271	15.3	Orth. SnS	(111)
	33.8392	0.4193	2.6468	19.8	Tet. SnO ₂	(101)
SS3	26.5102	0.4393	3.3595	18.6	Cubic SnS	(111)
	30.7439	0.3994	2.9059	20.6	Cubic SnS	(200)
	31.6026	0.5192	2.8288	15.9	Orth. SnS	(111)
	33.9591	0.3994	2.6377	20.8	Tet. SnO ₂	(101)
SS4	26.4304	0.5591	3.3695	14.6	Cubic SnS	(111)
	30.7039	0.3395	2.9096	24.3	Cubic SnS	(200)
	31.4029	0.6191	2.8464	13.3	Orth. SnS	(111)
	33.7793	0.5592	2.6514	14.8	Tet. SnO ₂	(101)

Figure 3 presented the FE-SEM images of SnO₂/SnS thin films at different upper layer thicknesses. The FE-SEM images displayed distinct variation in surface morphologies with varying the upper layer thickness. At thinner upper layer of sample SS1, flake-like structures were observed, distributed irregularly across the surface in random directions. With increasing upper layer thickness, these structures become partially covered the SnS surface in sample SS2

and finally completely covered in samples SS3 and SS4. The surface behaviour converted into spherical nanostructures. These variation in the SnO_2/SnS structure can utilized thin films in various scientific applications. Various shapes of SnO_2 nanostructures have been reported in previous studies, such as flake-like structures [21]. Figure 4 illustrates the EDS spectra of SnS and SnO_2/SnS thin films at various upper layer thicknesses (denoted as SnS, SS1, SS2, SS3, and SS4). Multiple atomic emissions were observed, including Sn, S, and O atoms. Notably, an additional peak corresponding to Au was detected, originating from the prior deposition of a gold layer onto the samples as a preparatory step for subsequent SEM examination. Additionally, an emission peak at 1.7 keV corresponding to Si emission from the glass substrate was observed. The increased intensity of the O emission peak with increasing upper layer thickness indicates greater incorporation of O atoms from the surrounding ambient during the deposition of the upper layer.

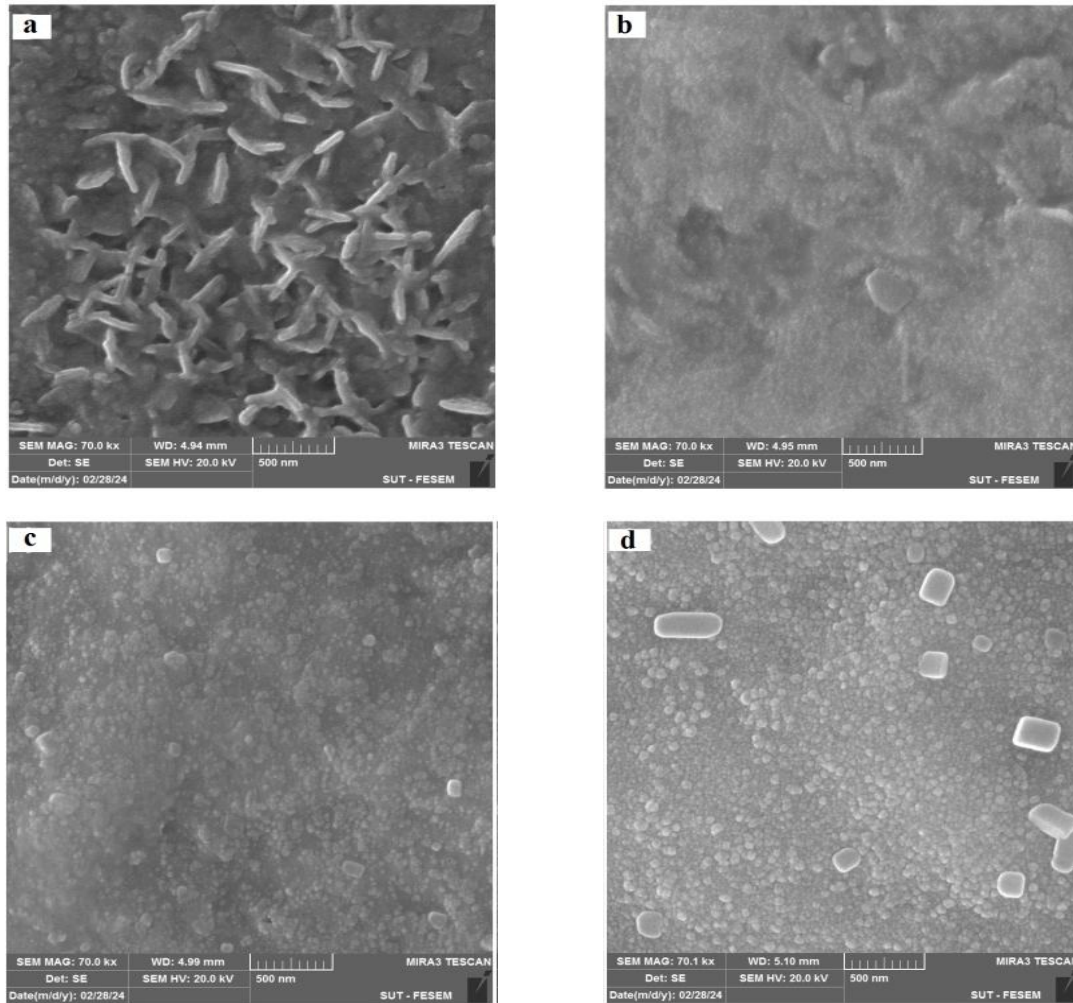


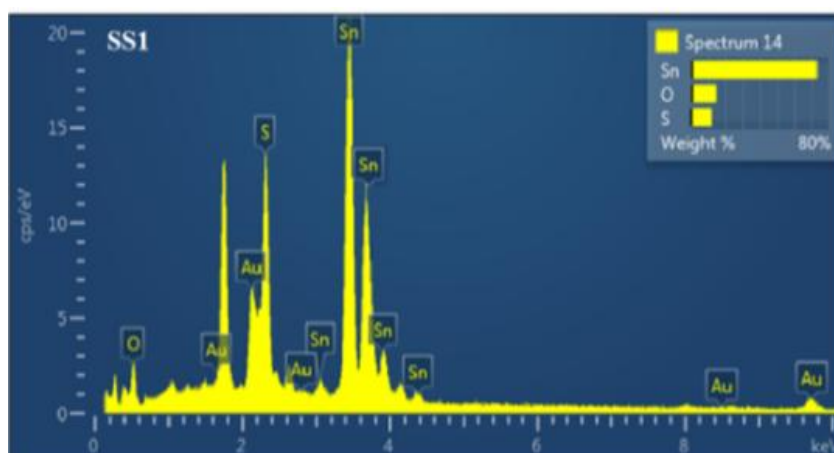
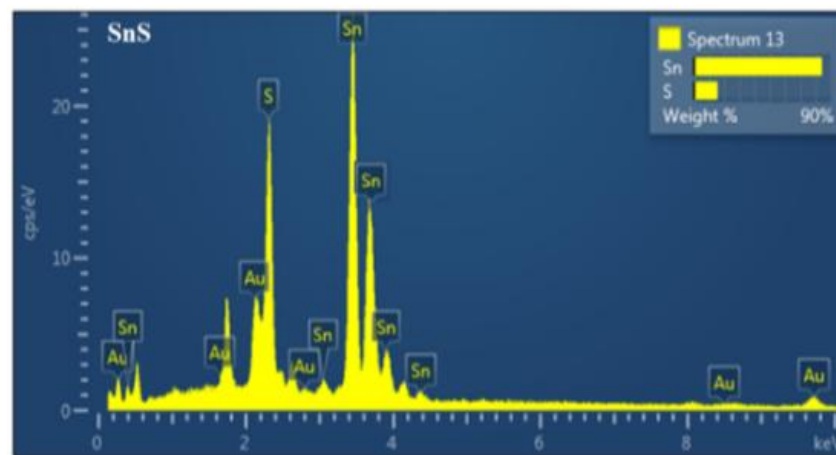
Fig. 3. FE-SEM images of SnO_2/SnS thin films at different upper layer thicknesses

For a comprehensive elemental analysis Table 2 presents the atomic and weight percentages of the constituent elements within the samples. The lower sulfur (S) content compared to its stoichiometric ratio suggests the presence of vacancies or defects within the material. These vacancies could arise from missing atoms or irregularities in the crystal lattice, creating opportunities for unique electronic and surface properties. In the realm of photocatalysis applications, the presence of vacancies or defects can be highly advantageous. Vacancies within the crystal lattice of semiconductors can be as active sites for catalytic reactions. These vacancies can induced electron-hole pairs, and enhancing surface reactivity, making them highly favorable for photocatalytic processes. In particular, these vacancies act as trapping sites for photogenerated carriers, leading to improved charge separation and longer carrier lifetimes. This phenomenon significantly enhances the efficiency of photocatalytic reactions [22].

Table 2. Elemental analysis for SnS and SnO₂/SnS thin films at different upper layer thicknesses

Sample	Element	Wt, %	Atomic, %
SnS	S	15.76	40.91
	Sn	84.24	59.09
SS1	O	14.75	48.3
	S	11.79	19.27
	Sn	73.46	32.43
SS2	O	17.79	54.69
	S	10.05	15.42
	Sn	72.15	29.89
SS3	O	20.31	60.34
	S	7.15	10.6
	Sn	72.54	29.05
SS4	O	24.29	66.38
	S	5.76	7.86
	Sn	69.95	25.76

Figure 5 displays the Fourier-transform infrared (FTIR) spectra of bare SnS thin films and those coated with SnO₂ at different thicknesses (samples SS1, SS2, SS3, and SS4). The FTIR spectrum of SnS exhibits distinct absorption bands. Bands located at 837.87 and 785.43 cm⁻¹ correspond to the Sn-S vibration [23], indicating the formation of the tin sulfide structure. Additional bands are observed at 3404.81 cm⁻¹ for -OH stretching, 2917.12 and 2846.32 cm⁻¹ for CH₂ stretching, and 1650.69 cm⁻¹ for O-H bending [24], which are attributed to adsorbed H₂O and carbonyl groups from the ambient environment.



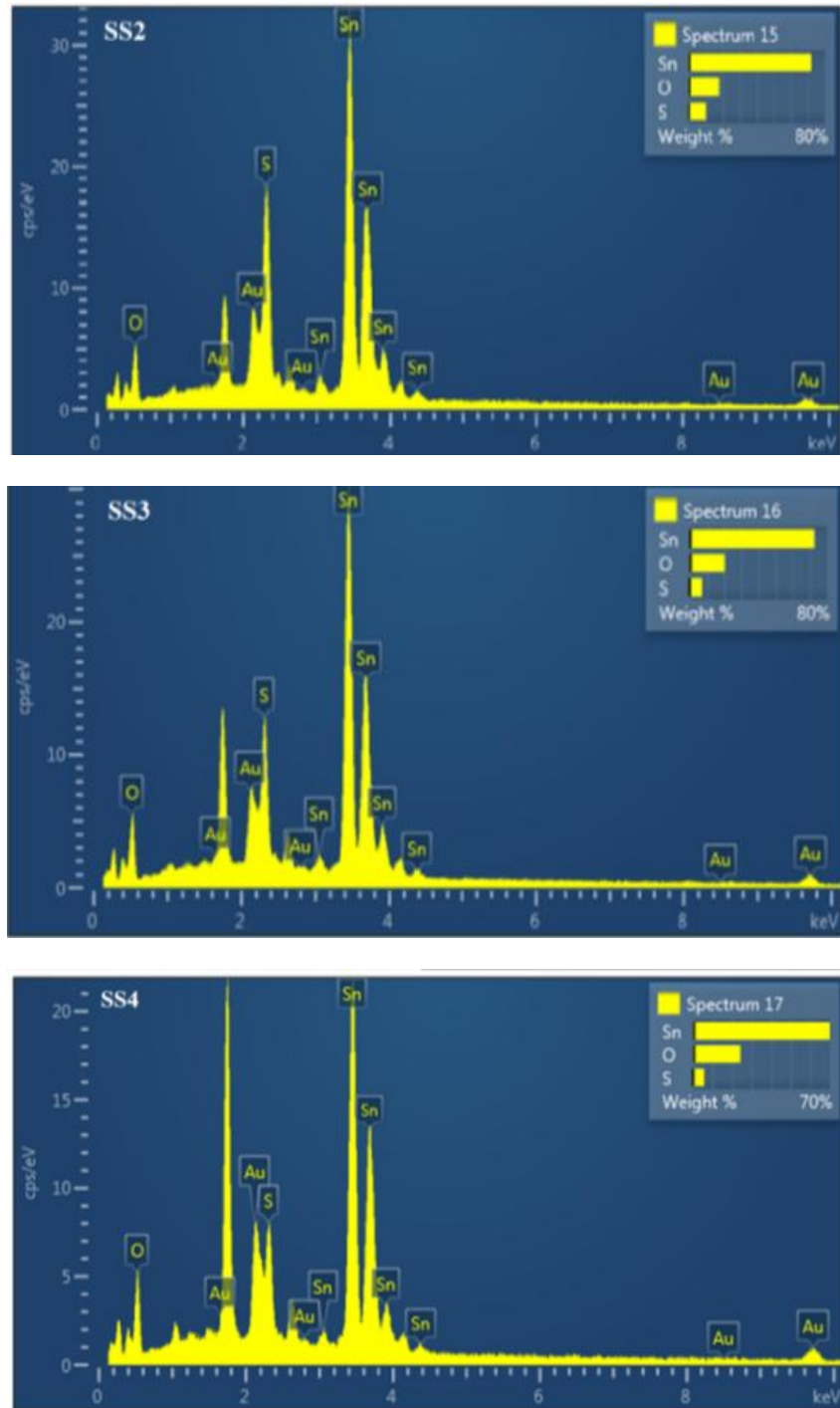


Fig. 4. EDS spectra for SnS and SnO₂/SnS thin films at different upper layer thicknesses

Furthermore, bands at 1286.23, 1215.44, and 1155.13 cm⁻¹ correspond to the SO₂ structure [25], suggesting the presence of some sulfur in oxide form rather than being fully incorporated into the SnS structure. Upon deposition of the SnO₂ upper layer, additional broad bands at 607.14 and 517.99 cm⁻¹ appeared [26], indicating existence in the SnO₂ structure within the FTIR spectra. Additionally, a broad band appeared at 1042.39 cm⁻¹ corresponding to a transition phase of tin hydroxide (Sn-OH) [27]. Some variations are observed of the upper layer, with some peaks reducing in intensity or slightly shifting. These variations may be attributed to changes in the band energies of certain bands due to interactions with oxygen during the deposition process.

Figure 6 illustrates the absorption spectra and determination of optical bandgap of bare SnS thin films and those coated with SnO₂ at varying thicknesses (samples SS1, SS2, SS3, and SS4). The absorption spectra demonstrate a diminishing trend as the wavelength increases, with an absorption edge observed around 700 nm. The gradual decline in absorption near the absorption edge suggests the crystallinity of the deposited films, attributed to the presence of lattice defects generating tail states near the band edges. Increasing the thickness of the upper layer results in enhanced optical absorption [28]. The coupling of SnO₂ with SnS suggests an improvement in photocatalytic activity, enhancing absorption of visible light in the SnO₂/SnS thin film sample. Our experimental findings align with the previous report by Huang [29]. The optical energy gap (E_{g}^{opt}) calculated by Tauc relation altered between 2 to 2.25 eV. Following the deposition of the upper layer of SnO₂ some charge carriers transported from n to p-type layer across the junction, forming depletion layers and causing band bending. Band offset may arise depend on the position of the conduction and valence bands for the two materials, leading to some variations in energy band gaps [30].

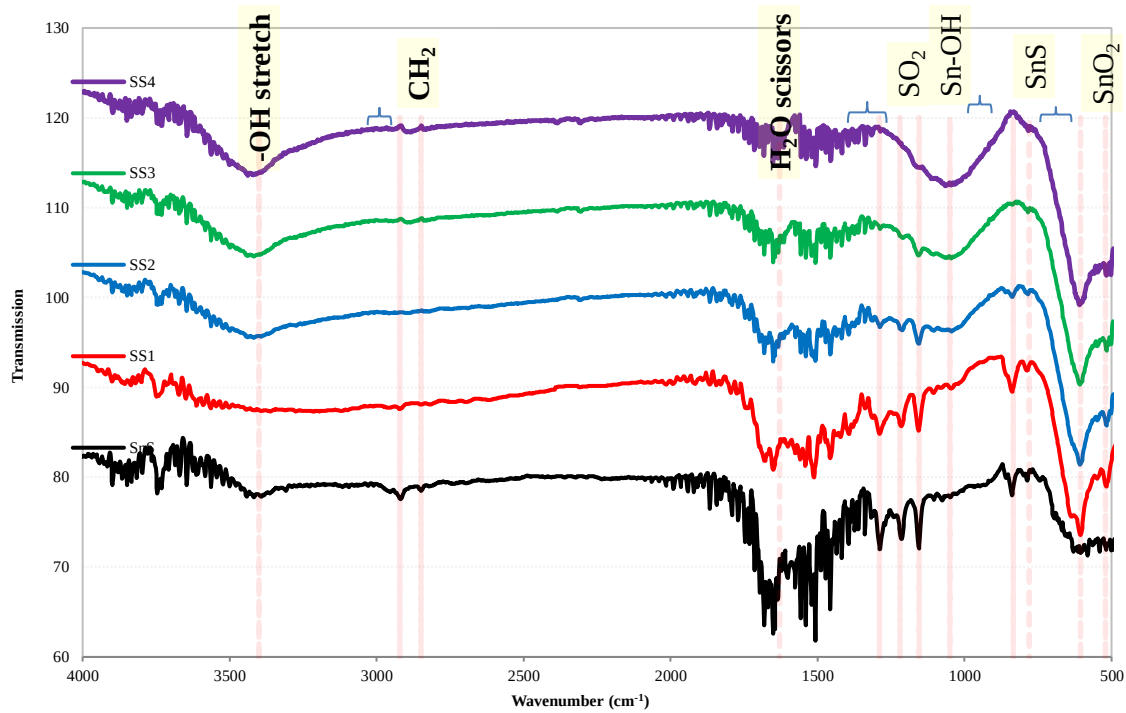


Fig. 5. FTIR patterns of SnS thin film and decorated with SnO₂ at different thicknesses

Figure 7 depicts the photoluminescence spectra of bare SnS thin films and those coated with SnO₂ at different thicknesses (samples SS1, SS2, SS3, and SS4) at room temperature using an excitation wavelength of $\lambda_{exc} = 350$ nm. In the SnS sample, two distinct peaks were observed: a broadband peak at approximately 485 nm (2.55 eV), attributed to yellow emission associated with S vacancies and lattice imperfections in the SnS structure [31], and a green emission peak at 550 nm (2.25 eV), likely associated with band-to-band transitions [32]. In contrast, the photoluminescence spectrum of SnS coated with an upper layer of SnO₂ thin films exhibited an additional emission peak at 420 nm (2.95 eV), which increased in intensity with increasing upper layer thickness. This peak corresponds to the band-to-band transition for the SnO₂ structure. The increase in the emission band intensity at 550 nm with increasing SnO₂ thickness for samples SS1, SS2, SS3, and SS4 suggests an increase in lattice defects [33]. The additional peak at 420 nm after deposition of the upper layer of SnO₂ indicates the formation of tin oxide nanoparticles on the sample surface. This band intensifies with increasing sprayed material, indicating the creation of more oxidation states on the sample. There were nearly no or only small variations observed in the SnS bands after the deposition of the upper layer of SnO₂. The observed changes in the photoluminescence spectra, particularly after the deposition of the upper layer of SnO₂, have significant implications for potential applications and benefits.

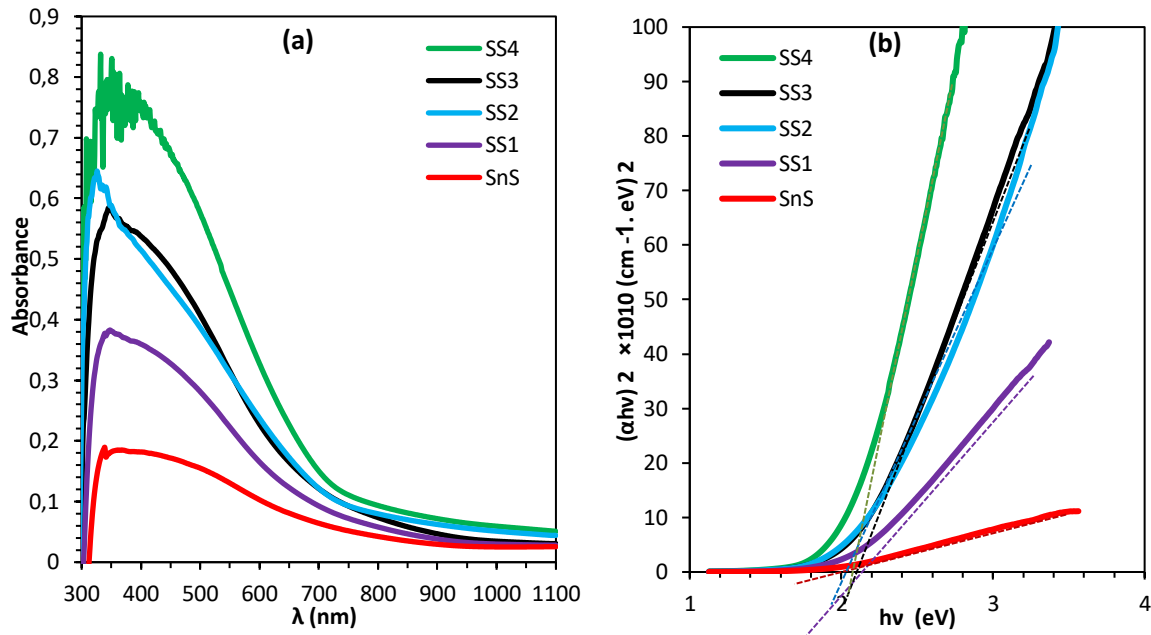


Fig. 6. UV-VIS absorbance of SnS thin film and decorated with SnO₂ at different thicknesses

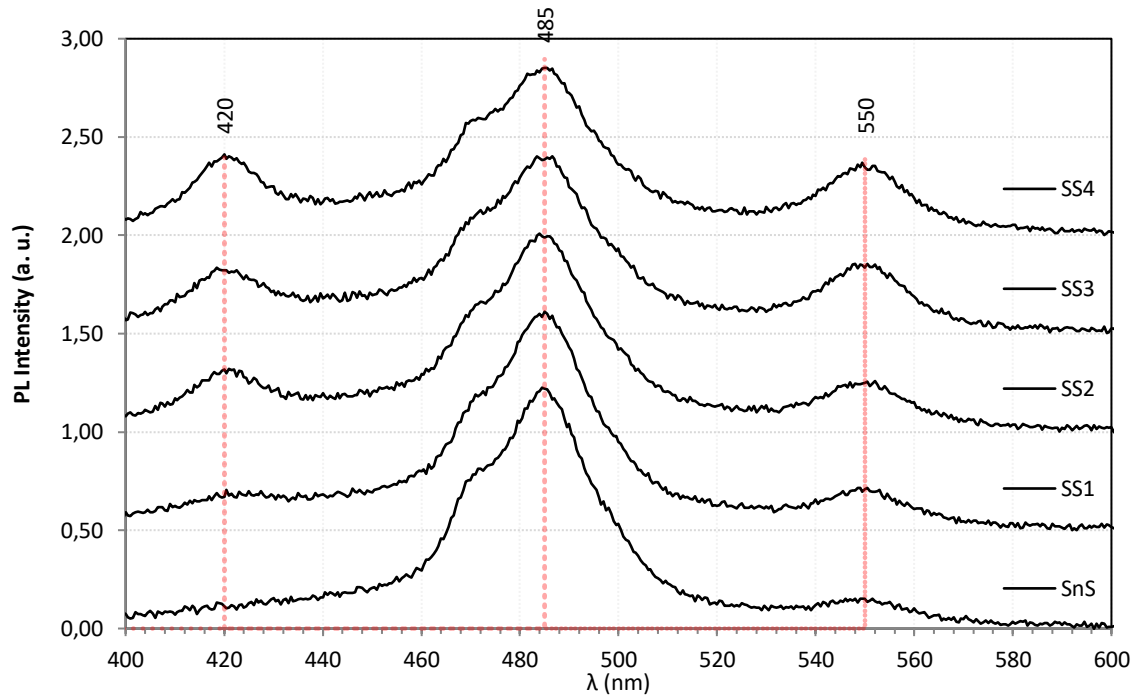


Fig. 7. PL patterns of SnS thin film and decorated with SnO₂ at different thicknesses

4. Conclusions

SnS and dual-layered SnO₂:SnS thin films were successfully deposited using the simple and cost-effective spray pyrolysis technique. Structural analysis confirmed the crystalline nature of the SnS films, with additional peaks indicating the incorporation of SnO₂. FE-SEM and revealed uniform distribution and porous structure of SnS, with significant variations observed after the deposition of the upper SnO₂ layer. These morphological changes indicate improved surface area and interface quality, which are critical for enhancing catalytic activity and sensor performance. Photoluminescence spectroscopy unveiled distinctive emission peaks associated with different processes, and the emergence of an additional SnO₂ emission peak indicate the formation of surface tin oxide nanoparticles. UV-visible spectroscopy demonstrated increased

absorbance spectra and slight variations in the optical bandgap upon deposition the upper layer of SnO₂. The incorporation of SnO₂ as an upper layer modify the surface properties. The observed changes in PL spectra, suggest potential enhancements in gas sensing capabilities. The deposition of the upper layer of SnO₂ offers several benefits. It introduces additional oxidation states and lattice defects, which can enhance the surface reactivity. In addition, the formation of heterojunctions between SnS and SnO₂ can facilitate charge separation and improve the overall device performance in optoelectronic applications. The potential applications of these bilayered SnO₂/SnS films extend beyond photocatalysis to include optoelectronic devices and environmental sensing, owing to their tunable characteristics. The findings demonstrate that the integration of SnO₂ as a functional top layer not only alters the microstructural and optical features of SnS but also opens new avenues for designing efficient heterojunction-based devices in clean energy and sensing technologies.

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