

INVESTIGATION OF CRYSTALLOGRAPHIC STRUCTURE AND FUNCTIONAL GROUP OF SILAR-DEPOSITED THALLIUM CUPROUS SULPHIDE THIN FILMS

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Abstract

This study synthesized and characterized thallium cuprous sulphide (TCS) thin films. TCS thin films were prepared using successive ionic layer adsorption and reaction (SILAR) technique from the aqueous solution of thallium chloride (TlCl), copper (II) chloride dihydrate (CuCl₂·2H₂O), thiourea, and trisodiumcitrate (TSC), while ammonia (NH₃), triethanolamine (TEA) and ethylenediaminetetraacetate (EDTA) were used as pH adjuster and complexing agents respectively. A detailed characterization of the TCS thin film was investigated by X-ray diffractometer (XRD) and Fourier transform infrared spectroscopy (FTIR). XRD results showed prominent peaks around (130), (110), (200), (211) orientation planes corresponding to 23.18°, 26.72°, 37.92° and 51.64°. Other lesser peaks appeared around (101), (310), (301) for 33.88°, 61.82°, 65.84° lattice planes respectively which revealed the growth of a crystalline (carlinite) thallium sulphide (Tl₂S) in the fabricated films. FTIR results revealed further that the deposited TCS films can be attached to the Ti-OH lattice vibrations mainly by their OH groups and that C-OH group is undominant, which implies the presence of impurity in the deposited thin film.

Keywords: Crystallography; Amorphous; FTIR; SILAR; Vibration

Introduction

The investigation of thallium cuprous sulphide (TCS) thin films is scientifically justified by the growing demand for new chalcogenide materials with tunable structural and optoelectronic properties for applications in photovoltaics, sensors, and thermoelectric devices (Odunaike *et al.*, 2021; Aparna *et al.*, 2023). Despite the promising semiconducting characteristics of thallium-based chalcogenides, limited research has been directed

toward understanding their crystallographic and chemical bonding features, particularly when synthesized via the successive ionic layer adsorption and reaction (SILAR) method. The structural evolution and bonding configurations of SILAR-deposited TCS films remain poorly understood. Since the crystallographic structure and surface functional groups play critical roles in determining the film's optical, electrical, and

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chemical behaviour, an investigation of these parameters is essential. Therefore, this study seeks to elucidate the crystallographic phases, preferred orientations, and functional group interactions present in TCS thin films fabricated through the SILAR process. The study provides fundamental insights into the relationship between structural order and chemical composition, thereby contributing to the optimization of TCS thin films for potential optoelectronic and energy-related applications. The SILAR technique offers several notable advantages such as cost-effectiveness, capable of depositing films on almost any type of substrate and over large surface areas (Adeniji *et al.*, 2020). The method ensures efficient material utilization and allows precise control of both the deposition parameters and film thickness. Additionally, it is a simple, user-friendly, highly appealing, and environmentally sustainable process (Adeniji *et al.*, 2020; Fowodu *et al.*, 2024; Olayiwola *et al.*, 2024; Soonmin, 2022)

Experimental Details

Preparation of substrates

In this study, soda-lime glass (SLG) and fluorine-doped tin oxide (FTO) substrates were used for the deposition of the thallium cuprous sulphide (TCS) thin films. The glass substrates were first degreased in concentrated HNO_3 for 48 hours, washed with detergent, rinsed in distilled water and dried in air to remove impurities and provide nucleation centres for the growth of the films purposely to yield highly adhesive and uniformly deposited films on the substrates' surface.

Preparation of precursors

A four-beaker system solutions were obtained by separately preparing 18 ml of 0.02 M TiCl_4 , 4 ml of 0.1 M of $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$, 2 ml of trisodiumcitrate, 2 ml of NH_3 , 2 ml TEA, and 3 ml EDTA and placed them in a 50 ml beaker (Beaker I). The solution was then gently stirred with a 78HW-1 Jinotech magnetic stirrer at room temperature to obtain a homogeneity. Then 30 ml each of distilled water was poured into two different 50 ml beakers (Beakers II and IV). Finally, 20 ml of 1 M thiourea was poured into another 50 ml beaker (Beaker III). The bath pH with temperature of the solution was measured with a Hanna-pH meter. The solutions in the beakers I and III served as cationic and anionic precursors respectively. The same precursors were prepared but at 0.3 M of $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ (Cu).

Deposition of the Thin Films

TCS thin films were deposited on glass substrates when immersed into the precursors using SILAR technique. Each substrate was dipped in each solution one after the other at a constant time interval. The substrate was kept vertically in each beaker using a substrate holder to prevent it from slanting in the beaker. Changes in colour of the solution was observed during the preparation and deposition at large. The deposition was done at $(50 \pm 5.0)^\circ\text{C}$ temperature for 120 seconds dip time per cycle. One SILAR cycle consisted of four steps: adsorption of thallium and copper species for 30 seconds, rinsing with distilled water for 30 seconds to remove excess adsorbed or loosely bounded thallium and copper species, reaction with thiourea precursor solution for 30 seconds to form stable TCS, and rinsing with distilled water for 30 seconds to remove excess or unreacted species of TCS. By repeating such deposition cycles 35, 50 and 65 times, TCS thin films were obtained respectively. After deposition, the substrates were removed and allowed to dry in the air.

Characterization of the grown Films

The crystallographic structure of the grown films was analyzed with X-ray diffractometer, while the functional groups were characterized using FTIR with the view to finding the optimization level. The SLG samples were labeled as: TA, TB, TC, TD, TE, and TF, while FTO samples were labeled as: T1, T2, T3, T4, T5, and T6.

Table 1. TCS Thin Films samples Description

Substrates	No of cycles	Cu Molarity	Substrates
TA / T1	35	0.1 M	SLG / FTO
TB / T2	50	0.1 M	SLG / FTO
TC / T3	65	0.1 M	SLG / FTO
TD / T4	35	0.3 M	SLG / FTO
TE / T5	50	0.3 M	SLG / FTO
TF / T6	65	0.3 M	SLG / FTO

Results and Discussion

The structural phase purity and degree of crystallinity of TCS thin particles analyzed using X-ray diffraction (XRD) are shown in Figure 1 (a) and (b). In Figure 1(a), T1, T2 and T3 show prominent peaks around (130), (110), (200), (211) orientation planes corresponding to 23.18° , 26.72° , 37.92° and 51.64° . Additionally, minor diffraction peaks were

observed at 2θ values of 33.88° , 61.82° , 65.84° , which correspond to the (101), (310), (301) lattice planes, respectively. This signifies the growth of crystalline (carlinite) thallium sulphide (Ti_2S) in the fabricated films (PDF#29-13444). The lower peaks are attributed to the SnO_2 layer of the FTO substrate (Daraz *et al.*, 2022). Also, samples T1, T2, T3 and T6 have shown similar behaviour with an addition peak at T6 which could be due to higher SILAR cycles at T6 (Sinha *et al.*, 2018). The intense peak at 37.92° indicates a carlinites particles positioned in the (200) plane. The XRD pattern of the SLG deposited samples show a hump shaped with a single peak around 22.42° , depicting amorphous structure with some crystallite order, for the 35, 50 and 65 cycles at both 0.1 and 0.3 M. This affirms that the FTO based substrates show better film visibility in thallium sulphide compound thin films than SLG (Daraz *et al.*, 2022) especially when such compounds undergo Cu doping.

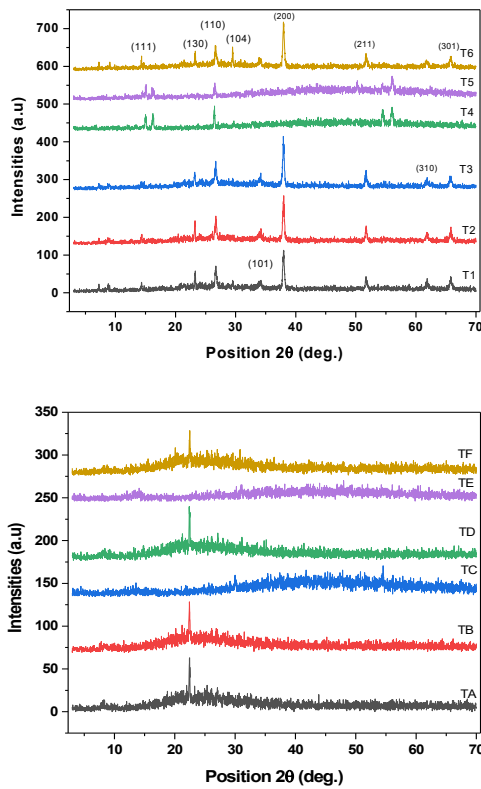


Figure 1. X-ray diffraction patterns of the deposited TCS thin films on (a) FTO Substrates; and (b) SLG Substrates

The Bragg equation relates the angle of diffraction (θ) of X-rays off a crystal lattice to the spacing between the planes of atoms in the crystal (d) and the wavelength of the X-rays (λ):

$$n\lambda = 2d \sin \theta \quad (1)$$

with the angles of diffractions (θ) are 11.59° , 13.14° , 18.96° , 25.82° , 16.94° , 30.91° and 32.92° and their respective full width at half maxima, FWHM (degree) are 0.11, 0.21, 0.07, 0.57, 0.28, 0.23 and 0.28. The average d spacing was calculated using Scherrer equation:

$$d = \frac{k\lambda}{\beta \cos \theta} \quad (2)$$

Where: $k = 0.9$; $\lambda = 0.15406 \text{ (nm)}$
wavelength of the xray sources;
 $\beta = \text{FWHM (radians)}$; and $\theta =$
peaks positions (radians).

The resultant average crystallite size, d for FTO sample is 49.72 nm. The XRD pattern is dominated by intense peak at $2\theta = 37.92^\circ$ corresponding to the crystal plane indexed at (200) as indicated in Figure 1 (a), followed by other lesser peaks. The crystal system of Ti_2S has been reported to be rhombohedral (Radtke and Dickson, 1975). In accordance with the observations of previous researchers, the absorption spectra of a thallium chalcogenides film exhibited a relatively high energy band-gap, which makes it a good window layer to the absorber layer (Fowodu *et al.*, 2024; Adeniji *et al.*, 2021). Figure 1 (a) revealed that the use of FTO substrate could enhance the crystallinity of Ti_2S for a better atomic ordering, enhancing crystallinity and reduces structural defects. Cu act as p-type dopant which increases hole concentration consequently increasing conductivity and carrier mobility.

Thallium copper sulphide belongs to a direct bandgap semiconductor.

Applying Tauc's relationship:

$$(\propto h\nu)^2 = A(h\nu - E_g) \quad (3)$$

Where: E_g is the optical energy bandgap, and A is a constant that depends on the electronic transition probability. From Figure 2, the band gap was extrapolated from the linear region near the onset of the Figure. The estimated values of energy band values decrease from 3.38 eV to 2.2 eV with increase in both SILAR cycle and Cu dopant concentration. Adeniji *et al.* (2021) reported band gap energy value of TIS as 3.9 eV, whereas Taşdemirci (2019) reported band gap energy value of 2.2 to 1.78 eV for CuS. It is observed that the band gap values of the thin films from this study are lower than the earlier reported TIS and within range of the latter for CuS indicating a shift in band gap energy which could be attributed to the doped Cu concentration and the varying SILAR cycles.

Hence, this study revealed that the synthesized TCS thin film material can serve as an absorber layer alternative to CIGS/CdTe materials.

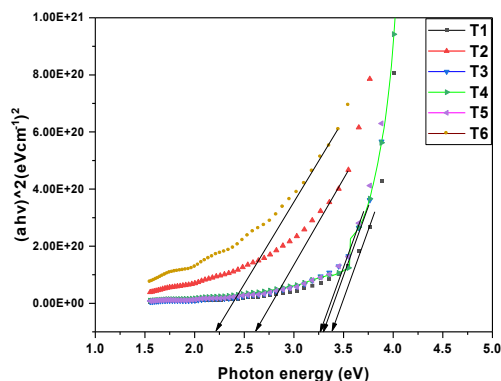


Figure 2. Graph of Bandgap of the deposited TCS thin films at variable SILAR cycle and Cu concentration on FTO

The chemical composition of the film samples was analyzed using Fourier Transform Infrared (FTIR) spectroscopy. From Figure 3, FTIR spectrum of the FTO differs slightly from that of SLG and the variation could be due to differences in elemental composition and bonding environment. The prominent peaks observed are 3306 cm^{-1} , 2130 cm^{-1} , 2859 cm^{-1} , 1718 cm^{-1} , 1595 cm^{-1} , 1319 cm^{-1} . The peak at 3306 cm^{-1} in FTO is attributable to O-H stretching from adsorbed water. The band is more intense in FTO, indicating a higher surface hydroxylation. The band at 2899 cm^{-1} in FTO is assigned to the C-H stretching vibration. In contrast, multiple peaks (2940 , 2903 , 2844 cm^{-1}) were observed in the C-H region of SLG, suggesting the presence of organic residues. The band at 2130 cm^{-1} suggests $\text{C}\equiv\text{C}$, 1718 cm^{-1} , the absorption band at 1595 cm^{-1} in the FT-supported sample is attributed to H-O-H bending vibration of adsorbed water, its higher intensity compared with the 1319 cm^{-1} band in the SLG1 supported sample. This could be because soda lime glass adsorbs less water (Bashiri *et al.*, 2021; Zeribi *et al.*, 2022). In the mid-IR region from 1000 to 1256 cm^{-1} , the envelopes for films on FTO and SLG differ significantly. For FTO, the spectrum shows a broad multi-component band in the range of 1002 – 1256 cm^{-1} , attributed to overlapping contributions from Ti-O/T-OH surface vibrations. In contrast, the SLG-supported films exhibit a single prominent feature near 1256 cm^{-1} , while the intensity in the range of 1000 – 1200 cm^{-1} is considerably lower. This aligns with either a C-O vibration or a shifted Si-O. A distinct absorption at 1319 cm^{-1} , observed only in films on FTO and not

on soda-lime glass (SLG), is attributed to surface or interfacial species. The peak at 872 cm^{-1} is suggesting Ti-O-Ti, and 723 cm^{-1} is assigned to Ti-O in the FTO1. This supports the previous study of Zeribi *et al.* (2022), while the peak at 864 cm^{-1} is Si-O-Si and 756 – 711 cm^{-1} is attributable to Si-O or Si-OH in SLG, which aligns with the results of Mihailova *et al.* (2024).

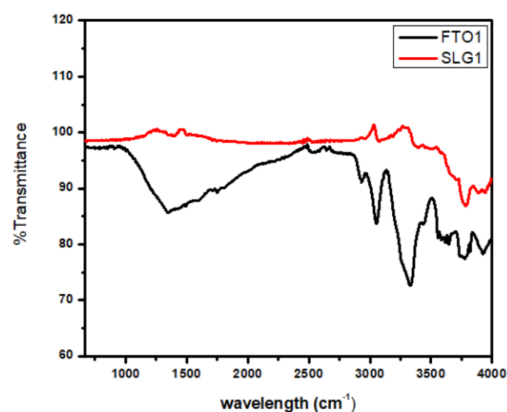


Figure 3. FTIR graphs of SLG and FTO at 10 SILAR

Figure 4 reveals the absorbance band observed between 3280 and 3276 cm^{-1} in both samples which is assigned to the O-H stretching vibration. The band is more prominent in SLG2, indicating a higher surface hydroxylation. Moreover, the peak at 3157 observed in both samples is assigned to the C-H stretching vibration, but it is sharper in SLG than in FTO. Multiple peaks were observed between 2195 and 1923 cm^{-1} in the FTO sample, indicating $\text{C}\equiv\text{C}$, whereas a single peak was observed in SLG2. This may be due to different elemental composition or bonding environment. The absorbance peak at 1722 cm^{-1} in FTO is a characteristic of $\text{C}=\text{O}$, slightly lower in the SLG sample (1718 cm^{-1}). The stretching at 1610 cm^{-1} and 1591 cm^{-1} in both samples are assigned to H-OH stretching vibration, while the peak at 1312 cm^{-1} in FTO2 and 1323 in SLG2 corresponds to C-O, the absorbance peak between 913 and 827 cm^{-1} is attributable to Ti-O-Ti, and 715 cm^{-1} is assigned to Ti-O, similar to a recent finding (Eleutério *et al.*, 2020). The Ti-O stretching band ranged between 900 and 500 cm^{-1} , with a shape and position very similar to that of the present study, was reported in a study of TiO_2 thin films on natural based-cellulose fibers (Zaid *et al.*, 2020), whereas the peak at 916 – 827 cm^{-1} is assigned to Si-O and 723 cm^{-1} is Si-O-Si (Olejníček *et al.*, 2022).

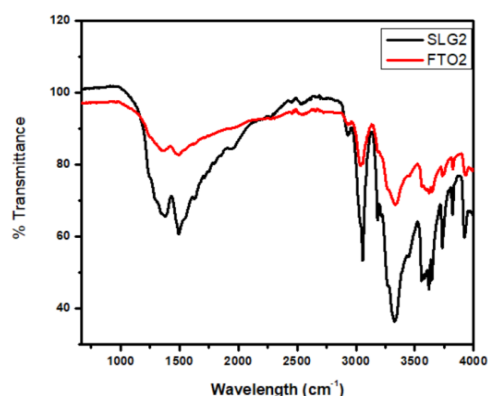


Figure 4. FTIR graphs of SLG and FTO at 20 SILAR

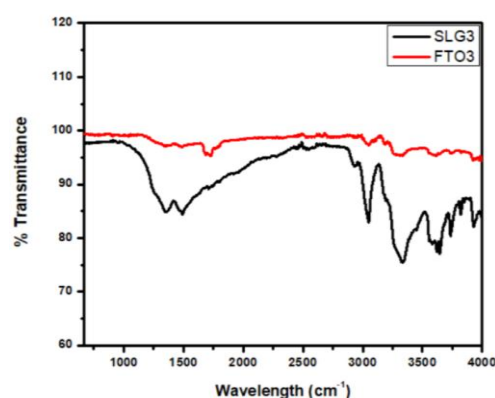


Figure 5. FTIR graphs of SLG and FTO at 30 SILAR

Figure 5 indicates the broad bands appearing at 3570, 3734, 3491 cm^{-1} which are due to O-H stretching vibrations, suggesting the presence of surface hydroxyl groups or adsorbed moisture. A band near 3308 cm^{-1} typically suggests N-H stretch, indicating the presence of an adsorbed amine or amide group. The FTO substrate has a higher surface reactivity compared to the inert SLG (Lukong *et al.*, 2022). Absorbance peaks around 2926-2987 cm^{-1} are assigned to C-H stretching of alkanes. Peaks at 2381, 2348, and 2489 cm^{-1} , respectively, are often associated with $\text{C}\equiv\text{C}$ stretching. The peak at 2124 cm^{-1} could indicate $\text{C}\equiv\text{C}$ stretching (alkyne) or $\text{N}=\text{C}=\text{O}$ group. The peak 1599 cm^{-1} is assigned to H-O-H bending modes of molecular water. Peak at 1457 cm^{-1} indicates C-H bending (alkanes). Peaks at 1032, 1138 cm^{-1} C-O stretching, peak at 909-827 cm^{-1} is attributable to Ti-O-Ti, and 726 cm^{-1} is attributable to Ti-O. A recent study reported similar peaks between 736 and 417 cm^{-1} (Liu *et al.*, 2021). Comparable peaks (3697-3298 cm^{-1}) observed in SGL3 correspond to O-H, 2940 cm^{-1} corresponds to C-H stretching vibration, 1602 cm^{-1} is ascribed to H-O-H, 2120 cm^{-1} is assigned to $\text{C}\equiv\text{C}$, peaks between 1315-1002 cm^{-1} are assigned to C-H, and peaks between 913-719 cm^{-1} are assigned to Si-O-Si and Si-O respectively (Asare *et al.*, 2020). The FTO3 spectrum shows relatively numerous bands across the entire region with significantly suppressed vibrational bands, whereas the SLG samples display comparatively few peaks. This indicates the difference in the chemical and structural nature of the two substrates and their interaction with deposited films and surface species. This confirms the superior optical transparency of FTO, making it more suitable as a substrate for optoelectronic and thin-film applications (Korir *et al.*, 2024).

In Figure 6, both samples display characteristic bands with notable differences. The SLG spectrum exhibited more absorption bands compared to the FTO sample. This can be attributed to the amorphous structure of SLG, which is primarily composed of silica, together with a modifier, in contrast to the more restricted structure. The crystalline order and FTO consist mainly of crystalline titanium oxide doped with fluorine, whose lattice vibrations are fewer and more restricted (Jiang *et al.*, 2020). The crystalline order and semi-metallic nature of FTO also limit the diversity of the absorption band (Korjenic and Raja, 2019). The broad bands observed in the region of 3246-3418 cm^{-1} in both samples correspond to O-H stretching vibrations, though more intense in FTO4, which may arise from surface hydroxyl groups or adsorbed moisture. A weak absorption around 2922-2851 cm^{-1} is assigned to C-H stretching, likely due to traces of organic residues. The absorptions observed at 1002, 1151, 1196, and 1140 cm^{-1} , respectively, in both spectra are attributed to asymmetric C-O stretching vibrations. The FTO4 spectrum shows a broader and more intense profile in this region, which can be linked to structural modifications induced by the conductive oxide coating (Lukong *et al.*, 2022). Furthermore, the absorption bands at 864-700 cm^{-1} are more pronounced in FTO4, corresponding to Ti-O-Ti and Ti-O lattice vibrations, while the peaks at 864-708 cm^{-1} are assigned to Si-O-Si and Si-O, respectively (Jnido *et al.*, 2019; Švagelj *et al.*, 2020; Sorarù *et al.*, 2024).

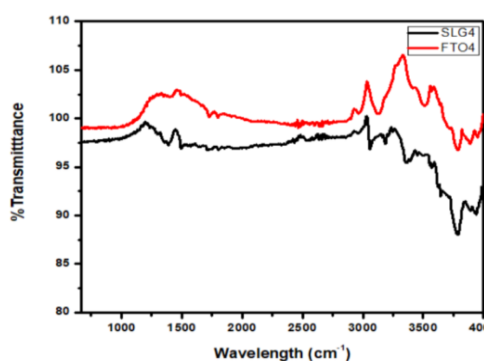


Figure 6. FTIR graphs of SLG and FTO at 40 SILAR

From Figure 7, the absorbance peaks at 3842, 3418, 3347, 3302, 3261 cm^{-1} in both samples assigned to the O-H stretching of surface hydroxyls. The C-H absorption observed between 3153 and 2724 cm^{-1} in both FTO and SLG samples is due to a combination of various chemical and physical effects, including different types of C-H absorption in this region (Aromatic stretches appear between 3100 and 3000 cm^{-1} ; Aldehydic can produce weak bands at 2800-2750 cm^{-1} ; and Aliphatic around 2950-2850 cm^{-1}). The absorbance peak between 2378 and 2340 is assigned to the O=C=O band, corresponding to the atmospheric CO_2 band due to adsorption (Bañuelos *et al.*, 2023). The absorbance at 2217, 2113, 2102, 1960, 1878 cm^{-1} suggests C $\equiv$ C. The bands at 1595 in both SLG and FTO samples indicate H-O-H bending of adsorbed water; this peak is more intense in the SLG sample. The greater intensity observed on the SGL sample aligns with a higher adsorption capacity and stronger ionic interaction of modifier cations within the silicate network. Additionally, the amorphous silicate surface retains more physically adsorbed species compared to the crystalline conductive FTO (Jadhav *et al.*, 2022). The peaks at 1487, 1461, 1457, 1289, 1282, 1250, and 1196 cm^{-1} are assigned to the C-O stretching vibration, and the absorbance peaks at 864, 823, 752, and 708 cm^{-1} are attributable to Ti-O-Ti and Ti-O. The peak at 913 and 823 is assigned to Si-O-Si, and 708 cm^{-1} is attributed to Si-O, which aligns with the report of (Filippatos *et al.*, 2021)

In Figure 8, multiple absorbance peaks were observed from 3716-3291 cm^{-1} in FTO, indicating O-H stretching. Absorbance peaks around 3011-2937 cm^{-1} were assigned to the C-H stretching vibration. The absorbance peak from 1997-1006 cm^{-1} suggests F-Ti-O, equivalent to that of H-O-H bending vibration upshift to 1610 and 1613 cm^{-1} ,

respectively, in SLG and FTO samples, which signifies a change in the hydrogen bonding (Antony and Hyam, 2023). An absorbance peak at 823-663 cm^{-1} is a characteristic peak of Ti-O and Ti-O-Ti bonds comparable to the report of Adeniji *et al.* (2021). More intense peaks were observed in SGL at 3712-3302 cm^{-1} , suggesting O-H, absorbance peak at 2389 and 2840 cm^{-1} was assigned to C-H stretching vibration, absorbance at 1200-1002 cm^{-1} is assigned to C-O stretching vibration, and band at low wave number range between 913 and 670 cm^{-1} was ascribed to the strong stretching vibrations of Si-O-Si and Si-O. In general, the absorption intensity of the peaks of higher precursor concentration samples showed an increase in the absorbance peak than those with lower precursor concentration (Atta *et al.*, 2022).

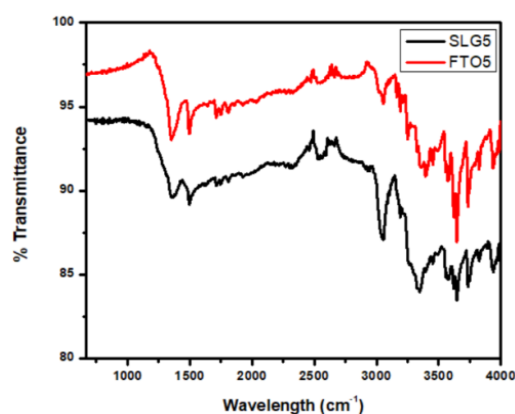


Figure 7. FTIR graphs of SLG and FTO at 50 SILAR

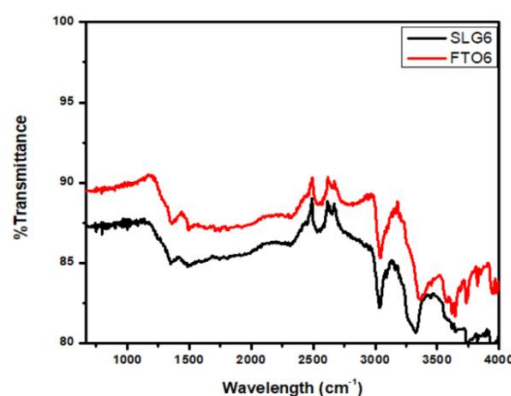


Figure 8. FTIR graphs of SLG and FTO at 60 SILAR

Conclusions

In this study, thallium cuprous sulphide thin films were successfully deposited via SILAR technique from the aqueous solution of thallium chloride (TlCl), copper (II) chloride dihydrate ($\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$), thiourea, and trisodiumcitrate (TSC), ammonia (NH_3), triethanolamine (TEA) and ethylenediaminetetraacetate (EDTA). The XRD results showed that FTO deposited samples of T1, T2, T3 and T6 exhibited similar behaviour with an addition peak at T6 which could be due to higher SILAR cycles at T6, the intense peak revealed a carlinites particles positioned in the (200) plane. And this suggested that the higher the SILAR cycle, the more intense peak observed. Whereas, the XRD pattern of the SLG deposited exhibited a hump shaped with a single peak around 22.42° resulting to amorphous structure with some crystallite order, for the all the SILAR cycles but at different concentration of 0.1 and 0.3 M of Cu dopants. This suggested that the FTO based substrates showed better film visibility in thallium sulphide compound thin films than SLG. Generally, higher precursor concentration samples showed an increase in the absorbance peaks than those with lower precursor concentration; making TCS a suitable material for absorbance layer in the photovoltaic applications.

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Author Contributions

Conceptualization: Q.A.A.; Methodology: T.O.F., S.O.A.; Investigation: Q.A.A.; Data Curation: T.O.F., M.A.S.; Writing – Original Draft: Q.A.A., R.O.O.-S., M.A.S.; Writing – Review & Editing: Q.A.A., S.O.A., K.K.B.; Supervision: Q.A.A. All authors have read and agreed to the published version of the manuscript.

Ethics Statement

Ethical approval was not required for this study because it did not involve human participants, animals, identifiable personal data, or materials requiring institutional ethical review.

Declaration of Generative AI Use

The authors declare that no generative AI tools were used in the preparation of this manuscript.

Conflict of Interest

The authors declare no conflicts of interest related to this work.

Data Availability Statement

The datasets generated and/or analyzed during the current study are available from the corresponding author upon reasonable request.

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