

# Structural and Optical Properties of Silver Sulfide-Reduced Graphene Oxide Nanocomposite

Oyugi Ngure Robert, Tabitha A. Amollo, Kallen Mulilo Nalyanya

**Abstract**—Nanomaterials have attracted significant attention in research because of their exemplary properties, making them suitable for diverse applications. This paper reports the successful synthesis as well as the structural and optical properties of silver sulfide-reduced graphene oxide ( $\text{Ag}_2\text{S-rGO}$ ) nanocomposite. The nanocomposite was synthesized by the chemical reduction method. Scanning electron microscopy (SEM) showed that the reduced graphene oxide (rGO) sheets were intercalated within the  $\text{Ag}_2\text{S}$  nanoparticles during the chemical reduction process. The SEM images also showed that  $\text{Ag}_2\text{S}$  had the shape of nanowires. Further, SEM energy dispersive X-ray (SEM EDX) showed that  $\text{Ag}_2\text{S-rGO}$  is mainly composed of C, Ag, O, and S. X-ray diffraction analysis manifested a high crystallinity for the nanowire-shaped  $\text{Ag}_2\text{S}$  nanoparticles with a d-spacing ranging between 1.0 Å and 5.2 Å. Thermal gravimetric analysis (TGA) showed that rGO enhances the thermal stability of the nanocomposite.  $\text{Ag}_2\text{S-rGO}$  nanocomposite exhibited strong optical absorption in the UV region. The formed nanocomposite is dispersible in polar and non-polar solvents, qualifying it for solution-based device processing. Thus, the surface plasmon resonance effect associated with metallic nanoparticles, strong optical absorption, thermal stability crystallinity and hydrophilicity of the nanocomposite suits it for solar energy conversion applications.

**Keywords**—Silver sulfide, reduced graphene oxide, nanocomposite, structural properties, optical properties.

## I. INTRODUCTION

NANOCOMPOSITES are significantly gaining research interest in the twenty-first century. Unlike traditional composites, nanocomposites possess unique properties and designs [1]. Nanoparticles are tiny materials ranging between 1 and 100 nm [2]. The nanoparticles' nanoscale size and high surface area give them desirable and unique properties. For instance, silver nanoparticles have more advanced chemical, biological, and physical characteristics than their macro-scaled particles [3]. Silver nanoparticles' advanced physical and chemical properties include high thermal and electrical conductivity, enhanced chemical stability, surface-enhanced Raman Scattering, non-linear optical behavior, and improved catalytic activity [3]. Furthermore, the nanoparticles possess other properties, such as toughness, reactivity, and other characteristics that depend on their sizes [2]. This makes the nanoparticles have a wide range of applications.

Silver sulfide nanoparticles ( $\text{Ag}_2\text{S}$  NPs) has properties that largely depend on its size, making them attractive for applications in various fields including photocatalysis and

biomedicine [4]. The nanoparticles of silver sulfide have also attracted optical applications within the visible region through surface plasmon resonance [4]. The  $\text{Ag}_2\text{S}$  NPs have a monoclinic crystal structure, a significant absorption coefficient [5], a narrow band gap (between 0.9 eV to 1.05 eV) [6], good chemical stability, and photoconductivity [4]. The uniqueness of these properties also makes the nanomaterials applicable in various areas, including sensors, food packaging, drug delivery, conductive paints, rechargeable batteries, and active electronic devices [7]. Nanoparticles are also applicable in energy conversion, where they offer surface plasmon resonances which enhance the trapping of light and allow for the tuning of resonant wavelengths [8]. Silver nanowire is one of the nanoparticles that are effective in trapping light through Localized Surface Plasmon Resonance (LSPR) [5]. In a nanocomposite, two nanomaterials are brought together resulting in enhanced properties since they combine the different materials' properties.

Graphene is a monolayer 2D nanomaterial of  $sp^2$ -hybridized carbon atoms densely packed in a honeycomb lattice structure [9]. Graphene has desirable mechanical properties, i.e., a break strength of  $42 \text{ Nm}^{-1}$  [10], an intrinsic tensile strength of 130.5 GPa, and 1.0 TPa Young's modulus [11] and high charge carrier mobility [10]. Graphene oxide (GO) is an oxidized form of graphene having oxygen functional groups that decorate the basal planes of C [12]. GO has hydroxylic and epoxy oxygen functional groups on the basal planes and the carboxylic group on the edges [13]. GO also has hydrophilic properties, making it possible to disperse in water or solvents, as opposed to graphene, which is hydrophobic. It is also possible to tune the sizes of GO variedly between a few nm and  $\mu\text{m}$ , giving them an advantage in wide applications. GO in its reduced form has been applied in electronic devices, biomedical applications, energy storage, supercapacitors, biosensors, catalysts, water purification, and membranes [14]. Removing oxygen functional groups from GO during the reduction process restores the  $sp^2$ -conjugated graphene network, thereby improving electron transport [15]. The reduced GO has sheets like that of graphene and restores the p-conjugated structure [16]. The GO's electrical conductivity is inferior to that of graphene because of the oxygen functional groups. In its reduced form, the rGO becomes electrically conductive and has defect sites [16].

In this study,  $\text{Ag}_2\text{S-rGO}$  nanocomposite was synthesized using chemical reduction method. This method can be carried

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out at room temperature [17]. The advantages of the chemical reduction method are that it can achieve mass production of the nanocomposite [17], it is cost-effective, has a good production rate [18], and allows for the control of the chemical composition [19]. Obtaining homogeneous and stable solutions in synthesizing nanocomposites based on graphene is necessary [20]. Thus, GO was appropriate for synthesizing the nanocomposite since it has oxygen functional groups below and above the basal planes, providing nucleation sites for doping heteroatoms [21]. It is envisaged that the synergistic effect of LSPR of the NPs and the outstanding properties of graphene would suit the nanocomposite for optoelectronic applications such as in solar cells.

## II. EXPERIMENTAL

### A. Materials

The following materials were purchased from Kobian, Kenya: Silver Nitrate ( $\text{AgNO}_3$ ), Polyvinylpyrrolidone (PVP), graphite powder, sodium nitrate, concentrated sulphuric acid, potassium permanganate, hydrogen peroxide (3%), and sodium sulfide. The purity of these materials was 99.95%, 99%, 99.99%, > 99.0%, 99.999%, > 99.0%, 30%, and 99.999%, respectively. Absolute ethanol was also purchased from Kobian, Kenya.

### B. Synthesis

#### 1. Synthesis of Graphene Oxide

Graphene Oxide was synthesized using the modified Hummers method [22]. The process started by mixing 1 g of sodium nitrate, 1 g of graphite powder, and 50 ml of concentrated sulphuric acid and stirring for 30 minutes at 0 °C. 6 g of potassium permanganate was then added to the mixture. The process was followed with further stirring for 3 hours at 35 °C. 200 ml of hydrogen peroxide (3%) was added, stirring the solution for 30 minutes [13]. The obtained solution was washed to a neutral pH with double distilled water. GO films were obtained by drying the solution at 50 °C in a vacuum oven.

#### 2. Synthesis of Silver Sulphide Nanoparticles and $\text{Ag}_2\text{S}$ -rGO Nanocomposite

The synthesis of  $\text{Ag}_2\text{S}$  nanoparticles followed the protocol of [5]. It started with the preparation of various precursor solutions. 1.698 g (0.1M) of silver nitrate hexahydrate ( $\text{Na}_2\text{S}\cdot 6\text{H}_2\text{O}$ ), 3.0 g of polyvinyl pyrrolidone (PVP), and 0.4 g (0.1 M) of sodium sulfide were separately dissolved in 50 mL of double distilled water. The PVP and sodium sulfide solutions were mixed dropwise into silver nitrate solution under stirring with a magnetic stirrer. The solution mixture was then stirred continuously for 3 hours. The precipitate was then centrifuged for 5 minutes at 4500 revolutions per minute. A successive washing with ethanol and double distilled water followed. The residue was then dried in an oven at 80 °C for 3 hours to obtain  $\text{Ag}_2\text{S}$  nanoparticles. For the synthesis of  $\text{Ag}_2\text{S}$ -rGO nanocomposite, GO at varied concentrations of 10 mg, 20 mg, 50 mg, 100 mg, and 150 mg in 10 ml, 20 ml, 50 ml, 100 ml, and 150 ml of double-distilled water, respectively, was injected into

the mixture of sodium sulfide, silver nitrate, and PVP. This was further stirred for 1 hr. The solution mixture obtained was centrifuged and washed with ethanol and double distilled water. The final residue was dried for 8 hours in a vacuum oven at 80 °C.

### C. Characterization

A field emission SEM (SEM: Phenom Pro by Thermofisher) was used to determine the morphological properties of the materials. Electron Dispersive X-ray Spectroscopy (EDX) detector attached to SEM was used for elemental analysis. The structural analysis was done using XRD (MiniFlex 300/600) with  $\text{Cu K}\alpha$  radiation ( $\lambda = 1.5418740 \text{ \AA}$ ). Powder XRD analysis for the determination of the crystallinity and the structure of the  $\text{Ag}_2\text{S}$ -rGO nanocomposite was done with a scanning range of  $2\theta$  from 10° to 90° and a scanning speed of 10 °/min. The essence of the XRD is that it helps determine the material's bulk structure. Fourier transform infrared (FTIR) spectroscopy was used to determine the oxygen functional groups in  $\text{Ag}_2\text{S}$ -rGO nanocomposite using Perkin Elmer spectrum 100 FTIR. A UV-Vis spectrophotometer (UV-1900i by Shimadzu) was used to characterize the optical absorption of the nanomaterials dispersed in ethanol at various wavelengths.

## III. RESULTS AND DISCUSSION

### A. Morphological Properties

Analysis of the morphological properties of the synthesized  $\text{Ag}_2\text{S}$ -rGO nanocomposite was done using SEM, and the obtained images are shown in Figs. 1 and 2. Fig. 1 shows  $\text{Ag}_2\text{S}$  of nanowire shape. The nanowires of  $\text{Ag}_2\text{S}$  possesses unique optoelectronic, electrical, and mechanical properties [23]. Fig. 2 indicates a tightly packed structure of folded rGO. The observed structure resonates with [24] demonstration that rGO possesses a tightly packed interlocking sheet structure. The importance of this structure is that rGO becomes uniformly distributed onto the surface of the  $\text{Ag}_2\text{S}$ , hence obtaining homogeneity in the synthesized nanocomposite. Moreover, research indicates that when GO gets chemically converted to graphene, defects and vacancies are produced in the carbon structure [25]. Consequently, it is possible that crystals of  $\text{Ag}_2\text{S}$  grow around these defects and vacancies as they are inclined to interact with the introduced graphene sheets during the formation of the nanocomposite.

### B. SEM-EDX Analysis

The nanocomposite was subjected to SEM-EDX elemental analysis, and the obtained spectrum is shown in Fig. 3. The essence of EDX analysis is that it reveals the elements present in the samples, as observed in Fig. 3; the elements in the  $\text{Ag}_2\text{S}$ -rGO nanocomposite include C, Ag, O, and S. The C and O peaks in the EDX spectrum indicated the presence of rGO in the nanocomposite. Additionally, only one O peak, with low intensity, was observed, implying the successful reduction of the GO. The distribution of the elemental peaks in the EDX spectrum also suggests that the elements are distributed throughout the rGO matrix and are not confined to limited space [26]. Such a distribution of the elements confirms the

uniformity of the nanocomposite. The purity of the in the EDX spectrum.  
nanocomposite is approved by the non-presence of extra signals

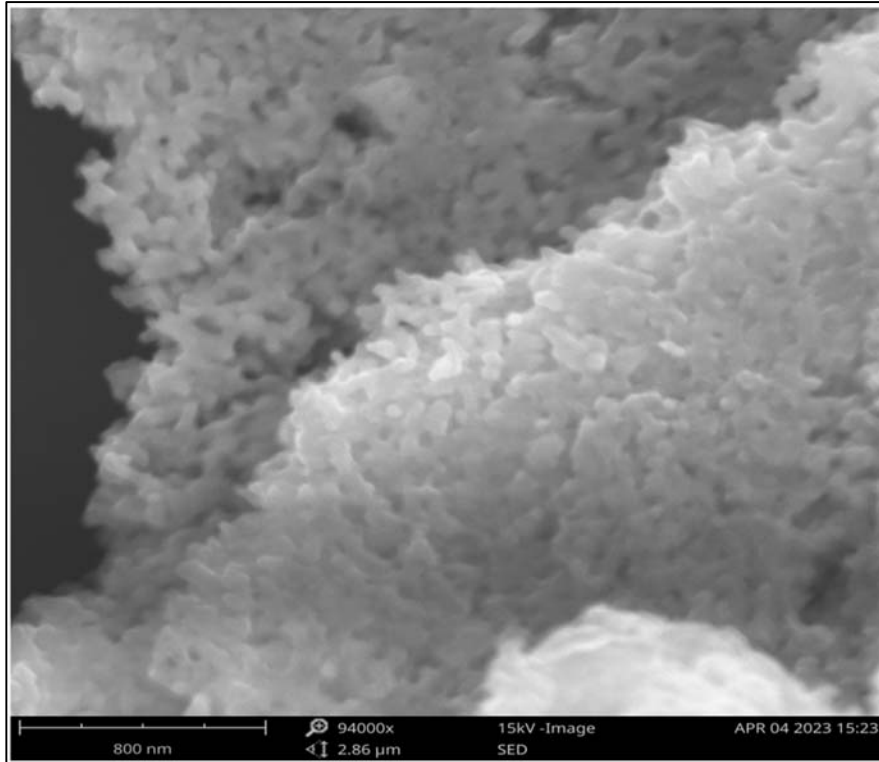


Fig. 1 SEM image of Ag<sub>2</sub>S-rGO with GO concentration of 150 mg

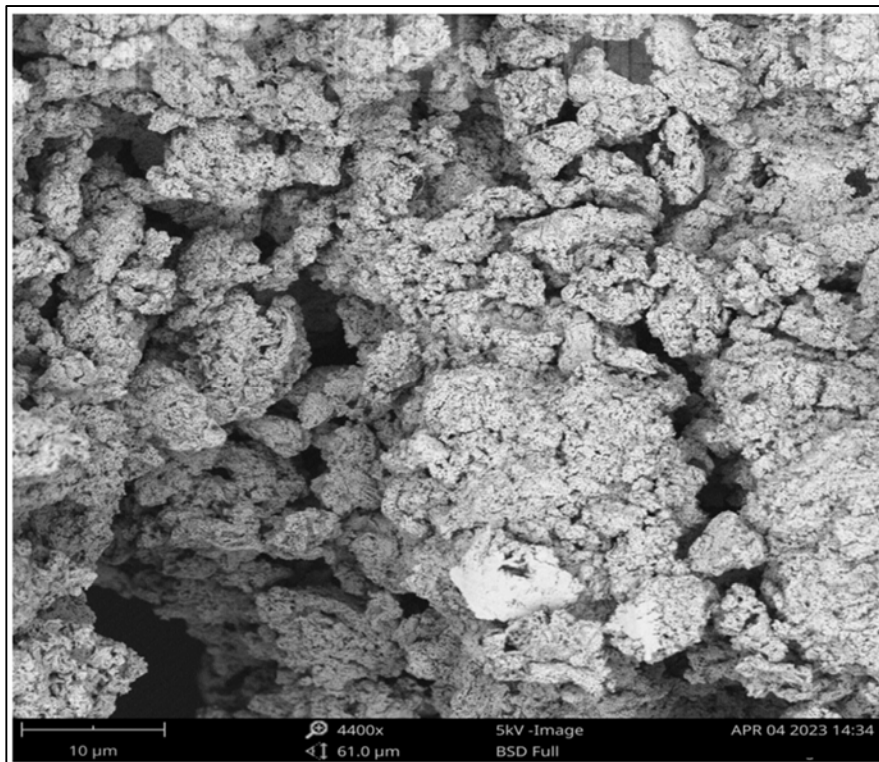


Fig. 2 SEM Images of Ag<sub>2</sub>S-rGO with GO concentration of 10 mg

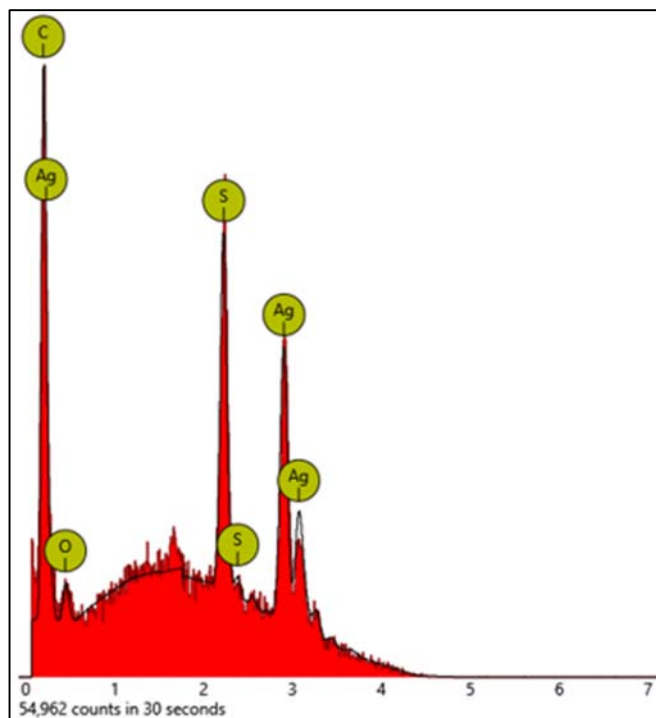


Fig. 3 SEM-EDX Analysis of  $Ag_2S$  -  $rGO$  nanocomposite

### C. Structural Properties

The XRD patterns for the  $Ag_2S$  and  $Ag_2S$ - $rGO$  are shown in Fig. 4. The major diffraction peaks of  $Ag_2S$  were observed at  $2\theta$  values of  $17.2^\circ$ ,  $22.4^\circ$ ,  $24.9^\circ$ ,  $25.9^\circ$ ,  $26.3^\circ$ ,  $28.9^\circ$ ,  $31.5^\circ$ ,  $33.6^\circ$ ,  $34.38^\circ$ ,  $34.7^\circ$ ,  $36.8^\circ$ ,  $37.7^\circ$ ,  $40.7^\circ$ ,  $43.4^\circ$ ,  $46.2^\circ$ ,  $47.8^\circ$ ,  $48.7^\circ$ ,  $53.2^\circ$ ,  $58.3^\circ$ , and  $63.7^\circ$ . According to the Joint Committee on Powder Diffraction Standards (JCPDS) card 96-900-0254, the diffraction peaks shown in Fig. 4 (a) correspond to the (011), (10-1), (110), (11-1), (022), (021), (111), (120), (120), (-121), (112), (013), (-113), (122), (210), (20-2), (113), (220), (221), and (-142) lattice planes of  $Ag_2S$ , respectively [27]. These diffraction peaks indicate a monoclinic acanthite phase of  $Ag_2S$ . The d-spacing of the diffraction lines ranged between 1.0 Å and 5.2 Å, indicating that the  $Ag_2S$  is a nanoparticle.

The diffraction peaks observed in Fig. 4 (b) in the nanocomposite at  $2\theta$  values of  $26.3^\circ$ ,  $44.3^\circ$ ,  $54.2^\circ$ ,  $83.2^\circ$ , and  $85.0^\circ$  are additional to the ones for  $Ag_2S$  in Fig. 4 (a) and correspond to the (002), (101), (400), (112), and (006) crystalline planes of  $rGO$ , respectively (JCPDS-card 01-075-1621). The most substantial peak observed at  $2\theta = 26.3^\circ$  corresponding to the miller index of (002) of carbon with a d-spacing of 3.395 Å coincides with the (022) lattice plane of  $Ag_2S$  nanoparticle, further demonstrating the uniformity of the nanocomposite formed [28]. The lack of diffraction peaks below  $12.0^\circ$ , which is the known diffraction peak for GO, indicates the successful reduction of GO during the chemical reduction process [28], [29].

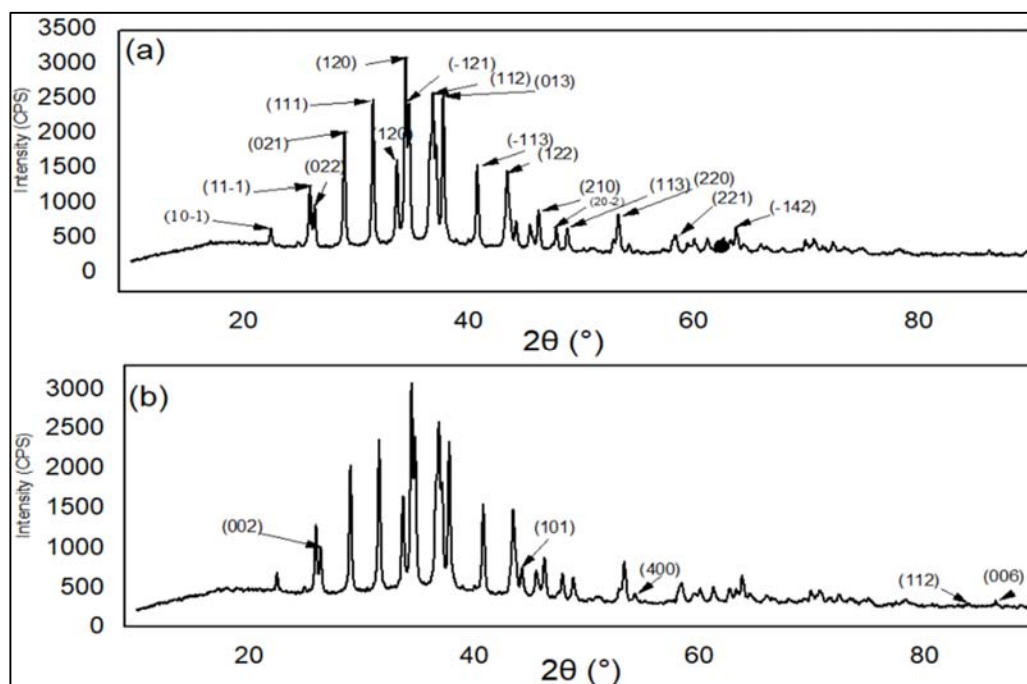


Fig. 4 XRD patterns for (a)  $Ag_2S$  and (b)  $Ag_2S$ - $rGO$  nanocomposite

### D. Thermal Stability

The TGA was used to investigate the thermal stability of  $Ag_2S$  nanoparticles and  $Ag_2S$ - $rGO$  nanocomposite and the results are shown in Fig. 5. The nanoparticles and the

nanocomposite were heated from room temperature to  $1000^\circ C$  in a nitrogen gas atmosphere. The heating rate was set at  $10^\circ C$  per minute. The TGA of  $Ag_2S$  nanoparticles demonstrated a three-phase decomposition behavior, as seen in Fig. 5 (a). The

weight loss was observed in the temperature ranges of 300 °C to 480 °C, 520 °C to 700 °C, and 760 °C to 850 °C.

The slight mass loss of  $Ag_2S$  from 300 °C to 480 °C is attributed to the desorption of the water molecules from the surface of the nanoparticles [30]. When  $Ag_2S$  nanoparticles get heated above the temperature of 175 °C; there is a transition of acanthite to the argentite phases of the  $Ag_2S$  [31]. The region between 227 °C and 527 °C exhibits argentite of  $Ag_2S$  [31]. This is the high weight loss observed from 300 °C to 480 °C. Silver sulphide also exhibits other beta and gamma phases at higher temperatures. For instance, above 160 °C,  $Ag_2S$  exhibits  $\beta$  argentite phase while above 587 °C, it contains  $\gamma - Ag_2S$  phase [31]. After the transformation of  $Ag_2S$  from  $\beta - Ag_2S$  to  $\gamma - Ag_2S$ , at temperatures between 627 °C and 697 °C, the thermal expansion coefficient significantly grows [31]. It is inferred that this increased coefficient of thermal expansion is responsible for the steep weight loss from 520 °C to 700 °C. The last weight loss between 760 °C and 850 °C is associated with the decomposition of the nanoparticles [30].

The first observable weight loss of  $Ag_2S$ -rGO nanocomposite

from the TGA occurred from below 100 °C to 210 °C (Fig. 5 (b)). This weight loss is attributed to decomposition of the oxygenated functional groups in the nanocomposite [28]. It is expected that the graphitic composition of the nanocomposite is all decomposed at 600 °C [28]. Thus, the remaining residue is of  $Ag_2S$ , which continues to decompose until a temperature of 981 °C. It is also observable that the addition of rGO to the  $Ag_2S$  slowed the rate of decomposition between 520 °C and 850 °C; hence, rGO enhances the thermal stability of  $Ag_2S$  in the nanocomposite.

The derivative thermograms (DTG) of  $Ag_2S$  is shown in Fig. 5 (c). Fig. 5 (c) shows three significant peaks at 180 °C, 580 °C, and at 733 °C. These peaks correspond to the  $Ag_2S$  phase transitions [30]. Specifically, the monoclinic acanthite of  $Ag_2S$  shows stability up to 176 °C, after which the body-centered cubic argentite becomes stable between 176 °C and 622 °C, after which there is a face-centered cubic polymorph [30]. This explains the irregular pattern in weight loss of  $Ag_2S$  at 713 °C, in which there is an increase in weight, after which there is a fall from 766 °C as seen in Fig. 5 (a).

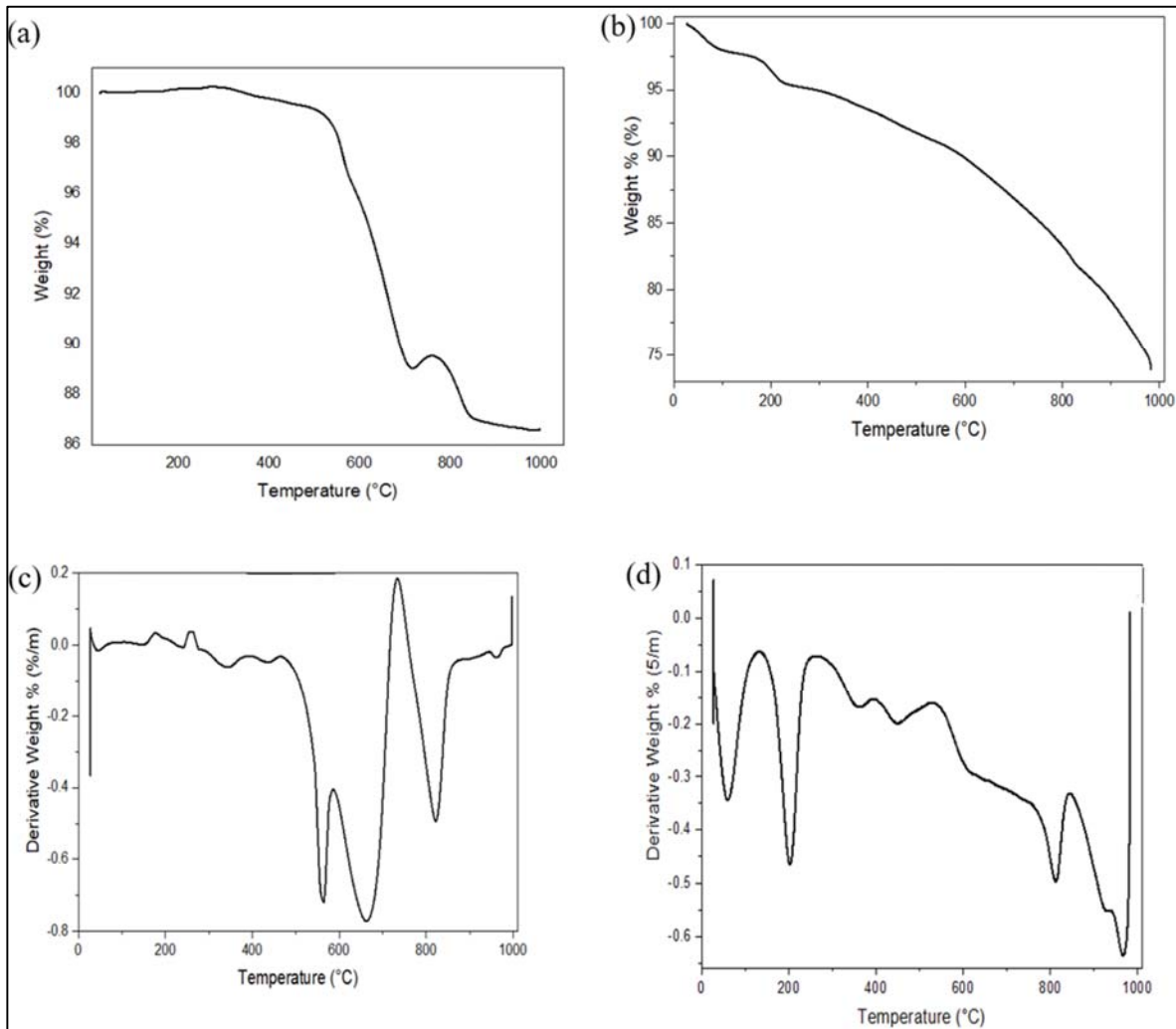


Fig. 5 TGA thermograms of (a)  $Ag_2S$  and (b)  $Ag_2S$ -rGO; and derivative thermogram of (c)  $Ag_2S$  and (d)  $Ag_2S$ -rGO

As shown in Fig. 5 (d), the DTA of  $Ag_2S$ -rGO shows peaks at 133 °C, 260 °C, 530 °C, and 845 °C. These peaks demonstrate the phase shifts of the nanocomposite. The peak at 845 °C indicate the combustion of graphite [32]. The peak at 260 °C can be associated with removing oxygen-containing groups from the nanocomposite [33]. The endothermal peak observed at 133 °C indicate the decomposition of water molecules from the surface of the nanocomposite [34]. Since there is no other DTA peak characteristic to another element other than the main components of  $Ag_2S$ -rGO, the nanocomposite can be considered to be pure.

### E. Optical Absorption

The  $Ag_2S$ -rGO nanocomposite was formed at different concentrations of rGO. Fig. 6 shows the UV-Vis spectra of  $Ag_2S$  and  $Ag_2S$ -rGO nanocomposite. Both the  $Ag_2S$  and  $Ag_2S$ -rGO nanocomposites show the highest absorption peak at 202

nm. The absorption peak at 202 nm for  $Ag_2S$  can be attributed to the surface plasmon resonance of the nanowire shape of the nanoparticle [4]. Silver nanowire effectively traps light through LSPR [5]. This can be confirmed by the observed nanowire shape of  $Ag_2S$  in the SEM image in Fig. 1. As observed in Fig. 6 (b), the peak at 202 nm is a superimposition of the absorbance of  $Ag_2S$  and rGO. For, rGO the peak is characteristic of  $\pi$ - $\pi^*$  transitions of the C-C aromatic bonds [35]-[37].

Research shows that the absorbance of  $Ag_2S$  increases with an increase in wavelength from the ultraviolet to the visible region [38]. This explains why the UV-Vis spectrum of  $Ag_2S$  shows a slight increase in absorbance from 202 nm. Fig. 6 (b) shows a slight increase in the absorbance of the nanocomposite from 250 nm. Such a change in the absorbance of  $Ag_2S$  with rGO indicates increased surface plasmon resonance excitation in the visible region [39].

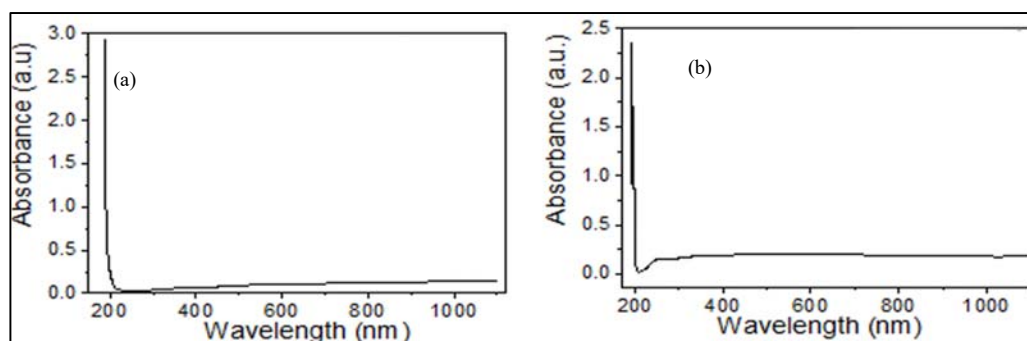


Fig. 6 Optical absorption of (a)  $Ag_2S$  and (b)  $Ag_2S$ -rGO

### F. Surface Chemistry

The FT-IR was conducted to determine the functional groups of the  $Ag_2S$ -rGO nanocomposite. Fig. 7 (a) shows the FT-IR spectrum of  $Ag_2S$  nanoparticles, while Fig. 7 (b) shows the spectrum for  $Ag_2S$ -rGO nanocomposite. For  $Ag_2S$  nanoparticles, the major bands were observed at 735  $cm^{-1}$ , 1048  $cm^{-1}$ , 1285  $cm^{-1}$ , 1438  $cm^{-1}$ , 1640  $cm^{-1}$ , 2090  $cm^{-1}$ , 2330  $cm^{-1}$ , 2660  $cm^{-1}$ , 2920  $cm^{-1}$ , and 3840  $cm^{-1}$ . The major peaks for  $Ag_2S$ -rGO spectrum were observed at 630  $cm^{-1}$ , 1055  $cm^{-1}$ , 1290  $cm^{-1}$ , 1320  $cm^{-1}$ , 1420  $cm^{-1}$ , 1640  $cm^{-1}$ , 1995  $cm^{-1}$ , 2100  $cm^{-1}$ , 2340  $cm^{-1}$ , 2680  $cm^{-1}$ , 2855  $cm^{-1}$ , 2920  $cm^{-1}$ , 3747  $cm^{-1}$ , and 3830  $cm^{-1}$ .

The band at 3747  $cm^{-1}$  for the nanocomposite indicates the presence of hydroxyl (OH) groups due to the addition of the rGO to the  $Ag_2S$  nanoparticles [40], [41]. The band at 630  $cm^{-1}$  in Fig. 7 (b) can be associated with the C-O-C and C-O bending vibrations, [40]. A common band appears at 1640  $cm^{-1}$  for both  $Ag_2S$  and  $Ag_2S$ -rGO. The peak is resonant to the stretching and bending of OH groups in water molecules [40], [42]. It is further observable that the band at 630  $cm^{-1}$  emerges in the  $Ag_2S$ -rGO nanocomposite nanoparticles. This occurrence can be explained by the chelation of blank orbits in  $Ag_2S$ , which are deficient in electrons to the atoms in O-H that are rich in O and the  $sp^2$ -conjugated bonds on the rGO surface [43]. The IR spectrum indicates a strong interaction between the  $Ag_2S$  and the matrix

of rGO confirming the immobility of  $Ag_2S$  on the GO matrix's surface.

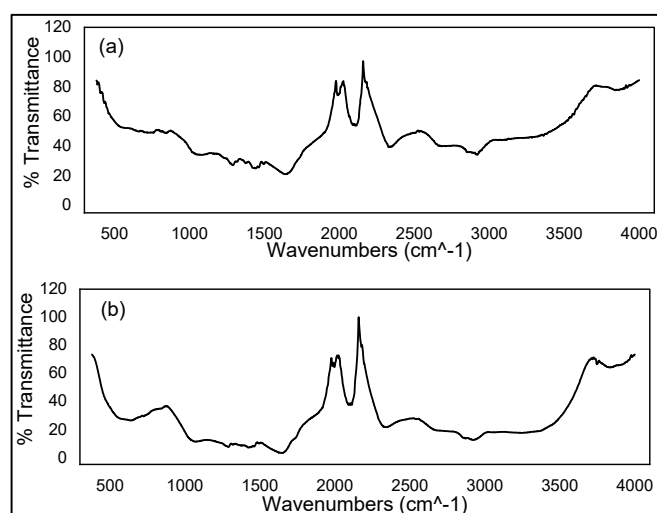


Fig. 7 IR Spectrum of (a)  $Ag_2S$  and (b)  $Ag_2S$ -rGO

## IV. CONCLUSION

$Ag_2S$ -rGO with high crystallinity was successfully synthesized using chemical reduction method. SEM image of the nanocomposite showed that rGO sheets were intercalated in

Ag<sub>2</sub>S nanoparticles during synthesis. The SEM analysis also showed that Ag<sub>2</sub>S had the shape of nanowires. The TGA analysis of the Ag<sub>2</sub>S-rGO nanocomposite indicated a significant weight loss at 157 °C to 211 °C, explaining the decomposition of the oxygenated functional groups. The TGA further showed that rGO enhances the thermal stability of Ag<sub>2</sub>S. The DTA analysis of the nanocomposite showed endothermic peaks at 133 °C, 260 °C, 530 °C, and 845 °C demonstrating the phase shifts of the nanocomposite with a DTA peak at 845 °C showing the combustion of graphite. The lack of DTA and EDX peaks for other elements other than those in the Ag<sub>2</sub>S-rGO showed the purity of the nanocomposite.

The d-spacing for the diffraction lines in XRD ranged between 1.0 Å and 5.2 Å, indicating that the composite formed was in nanoscale. The presence of rGO in the nanocomposite was confirmed by a strong XRD peak at  $2\theta = 26.3^\circ$ , corresponding to the miller index (002) of carbon. The nanocomposite exhibited strong optical absorbance in the UV region. FT-IR spectrum of the nanocomposite confirmed a strong interaction between Ag<sub>2</sub>S and the matrix of rGO. The nanocomposite is easily dispersed in polar and non-polar solvents. The nanocomposite is suitable for various applications including solar energy conversion given its thermal stability, optical absorption, LSPR effect and crystallinity.

#### CONFLICT OF INTEREST

The authors declare that no conflict of interest exists regarding this research article's submission and publication.

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