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# The structural and optical constants of $\text{Ag}_2\text{S}$ semiconductor nanostructure in the Far-Infrared

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## Abstract

**Background:** In this paper a template-free precipitation method was used as an easy and low cost way to synthesize  $\text{Ag}_2\text{S}$  semiconductor nanoparticles. The Kramers–Kronig method (K–K) and classical dispersion theory was applied to calculate the optical constants of the prepared samples, such as the reflective index  $n(\omega)$  and dielectric constant  $\epsilon(\omega)$  in Far-infrared regime.

**Results:** Nanocrystalline  $\text{Ag}_2\text{S}$  was synthesized by a wet chemical precipitation method.  $\text{Ag}_2\text{S}$  nanoparticle was characterized by X-ray diffraction, Scanning Electron Microscopy, UV-visible, and FT-IR spectroscopy. The refinement of the monoclinic  $\beta\text{-Ag}_2\text{S}$  phase yielded a structure solution similar to the structure reported by Sadanaga and Sueno. The band gap of  $\text{Ag}_2\text{S}$  nanoparticles is around 0.96 eV, which is in good agreement with previous reports for the band gap energy of  $\text{Ag}_2\text{S}$  nanoparticles (0.9–1.1 eV).

**Conclusion:** The crystallite size of the synthesized particles was obtained by Hall-Williamson plot for the synthesized  $\text{Ag}_2\text{S}$  nanoparticles and it was found to be 217 nm. The Far-infrared optical constants of the prepared  $\text{Ag}_2\text{S}$  semiconductor nanoparticles were evaluated by means of FTIR transmittance spectra data and K–K method.

**Keywords:** Nanostructures, Semiconductors, Raman spectroscopy, Infrared spectroscopy, Crystal structure, Optical properties

## Background

In recent years, nanometer-sized chalcogenide semiconductors have drawn attention as a component of nanotechnology, mainly due to their physical and chemical properties, heavily dependent on their shape and size. The  $\text{Ag}_2\text{S}$  is found amongst the most important chalcogenides and because of its unique optoelectronic properties. It has been extensively studied due to its many potential applications in optical and electronic devices such as infrared detectors, photoconductive cells, magnetic field sensors and photoconductors, amongst others [1–5].  $\text{Ag}_2\text{S}$  is an effective semiconductor material due to a large absorption coefficient and a direct band gap of 0.9 to 1.05 eV. It is a coinage mineral undergoes a structural phase transition. Above 183 °C,  $\text{Ag}_2\text{S}$  appear with a cubic

structure known as argentite ( $\alpha\text{-Ag}_2\text{S}$ ). At room temperature,  $\text{Ag}_2\text{S}$  have a monoclinic structure named acanthite, space group  $P2_1/c$  and  $Z = 4$  ( $\beta\text{-Ag}_2\text{S}$ ) [6, 7]. The  $\alpha\text{-Ag}_2\text{S}$  behaves like a metal ( $d\sigma/dT < 0$ ) while  $\beta\text{-Ag}_2\text{S}$  behaves like a semiconductor ( $d\sigma/dT > 0$ , with activation energy of 1.3 eV) [8–10]. Several methods have been developed for the synthesis of  $\text{Ag}_2\text{S}$  nanoparticles such as solvothermal method, hydrothermal route, and single-source precursor routes [11]. Yu *et al.* synthesized sub-micrometer  $\text{Ag}_2\text{S}$  particles thru a simple hydrothermal method but it is difficult to control the size and shape of the nanoparticles for the large-scale synthesis of high-quality nanoparticles [12]. Qin *et al.* successfully synthesized  $\text{Ag}_2\text{S}$  nanorods by a biomimetic route in the lysozyme solution at physiological temperature and atmospheric pressure [4]. In another work, Wang *et al.* synthesized spherical silver sulphide nanoparticles ( $\text{Ag}_2\text{S}$ ) at 205 °C under  $\text{N}_2$  atmosphere by a direct reacting silver acetate with n-dodecanethiol [13]. Therefore, there is a considerable challenge for the synthesis of  $\text{Ag}_2\text{S}$

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nanoparticles on a large scale through a simple and low-cost approach.

In this paper, a template-free precipitation method was used to prepare nanometric powders of  $\text{Ag}_2\text{S}$ . The structural and optical constants of the prepared  $\text{Ag}_2\text{S}$  nanometric powders in Far infrared were calculated and are presented for the first time.

## Experimental section

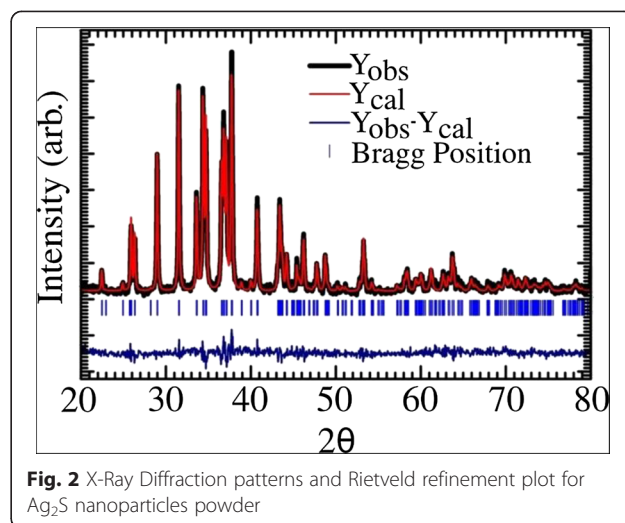
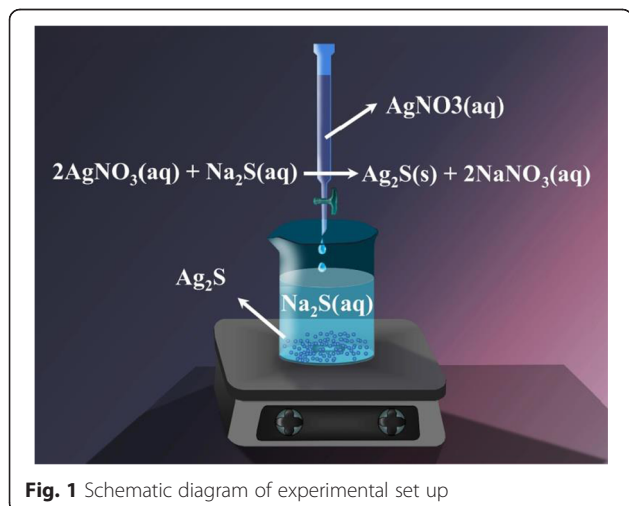
### Synthesis and characterization of $\text{Ag}_2\text{S}$ nanoparticles

Nanocrystalline  $\text{Ag}_2\text{S}$  was synthesized by a wet chemical precipitation method. Initially 0.1 mmol of  $\text{AgNO}_3$  (Aldrich, Germany) was dissolved in 50 ml of distilled water. The obtained solution was added drop wise into 50 mL 0.1 M  $\text{Na}_2\text{S}$  solution. Finally, the as prepared precipitated nanocrystalline powder was collected and dried after centrifugation at 80 °C during a 3 h period. The schematic diagram for the experimental set up and chemical reaction is shown in Fig. 1. The structure and morphology of the sample was studied by X-ray diffraction (Shimadzu XRD-6000, Tokyo, Japan) and Scanning Electron Microscopy (SEM, SU-70, Hitachi). The study of the optical properties of the samples was carried out by UV-visible (Perkin-Elmer, Lambda 35) and FT-IR spectrometry.

## Results and discussion

### Phase and compositional study (XRD)

Figure 2 shows the X-ray diffraction pattern for synthesized  $\text{Ag}_2\text{S}$  particles. A Rietveld refinement analysis was performed after x-ray diffraction pattern acquisition. The refinement of the monoclinic  $\beta\text{-Ag}_2\text{S}$  phase yielded a structure solution similar to the structure reported by Sadanaga and Sueno [8]. No impurity phase was observed in the X-ray diffraction pattern. However, the refined structure from this study showed a slight deviation in the xyz coordinates for Ag and S atom. The refined parameters are



listed in Table 1, and the Rietveld refinement diffraction pattern of  $\beta\text{-Ag}_2\text{S}$  structure is shown in Fig. 2.

To determine the strain and size effect associated to the synthesized  $\text{Ag}_2\text{S}$  particles, Hall-Williamson method was used as the estimation of the particle size. This is explained by the Scherrer equation not taking in consideration for

**Table 1** Structural details and refined parameters obtained by Rietveld refinement

Basic structural details							
Structure				Space group			
<i>Monoclinic</i>				<i>P 21/c</i>			
Lattice parameters (in Å) and angle (in °)							
<i>a</i>	<i>b</i>	<i>c</i>	$\alpha$	$\beta$	$\gamma$	Vol. (Å <sup>3</sup> )	
4.2278	6.9289	9.5323	90	125.58	90	227.11	
Atomic coordinates parameters							
Atom	<i>x/a</i>	<i>y/b</i>	<i>z/c</i>	SOF			
Ag1	0.07245	0.01478	0.30895	1			
Ag2	0.72498	0.32529	0.43819	1			
S1	0.49293	0.23577	0.13261	1			
Anisotropic displacement parameters, in Å <sup>2</sup>							
Atom	<i>U</i> <sub>11</sub>	<i>U</i> <sub>22</sub>	<i>U</i> <sub>33</sub>	<i>U</i> <sub>12</sub>	<i>U</i> <sub>13</sub>	<i>U</i> <sub>23</sub>	
Ag1	0.03732	0.04222	0.05706	0.01489	0.03081	0.01655	
Ag2	0.05167	0.05616	0.03745	-0.01454	0.04492	-0.00585	
S1	0.01985	0.01236	0.00734	0.01186	0.02756	-0.00555	
Other parameters							
<i>R</i> <sub>p</sub>	<i>R</i> <sub>wp</sub>	<i>R</i> <sub>exp</sub>	<i>R</i> <sub>b</sub>	<i>R</i> <sub>f</sub>	$\chi^2$	<i>c/a</i>	
18.8	22.7	20.14	7.94	6.72	1.27	2.2547	
Goodness of fit							
D-W statistics (d)			<i>Q</i> <sub>D</sub> = expected (d)	<i>S</i> (goodness of fit) = <i>R</i> <sub>wp</sub> / <i>R</i> <sub>exp</sub>			
1.6094			1.8251	1.13			

the broadening due to lattice strain presence. Generally, the observed peak broadening  $B_o$  can be attributed to

$$B_r = B_o - B_i \quad (1)$$

where  $B_o$  is the observed peak broadening in radians,  $B_i$  is the instrumental broadening in radians, and  $B_r$  is the broadening due to the small particle size and lattice strain. Using the Scherrer equation, the broadening caused by small crystallite size may be expressed as:

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

where:  $B$  is the broadening solely caused by small crystallite size,  $k$  is a constant whose value depends on particle shape and is usually taken as unity,  $d$  is the crystallite size,  $\theta$  is the Bragg angle and  $\lambda$  is the wavelength of the incident X-ray beam ( $1.5418 \text{ \AA}$ ). Similarly, according to Wilson, the broadening caused by lattice strain is expressed as:

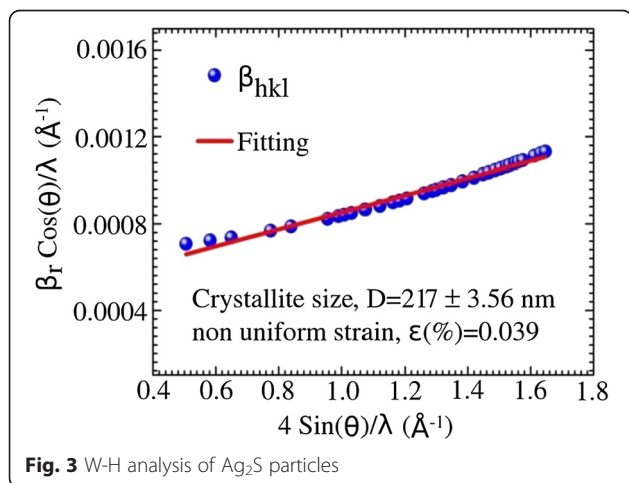
$$B_s = 4\varepsilon \tan\theta \quad (3)$$

where:  $B$  is the peak broadening caused by the lattice strain,  $\varepsilon$  the strain distribution within the material and  $\theta$  is the Bragg angle [14]. The instrumental broadening was estimated performing a XRD to a pure strain-free silicon standard under identical conditions. The total broadening excluding the instrumental broadening of the peak is expressed as the sum of eqn (2) and (3) [15]:

$$B_r = \frac{k\lambda}{t \cos\theta} + 4\varepsilon \tan\theta \quad (4)$$

$$\frac{B_r \cos\theta}{\lambda} = \frac{k}{t} + \varepsilon \frac{4\sin\theta}{\lambda} \quad (5)$$

The plot of  $B_r \cos(\theta)/\lambda$  versus  $4\sin(\theta)/\lambda$  is a straight line with slope equal to  $\varepsilon$  and hence the particle size can be estimated from the intercept. A typical Hall-Williamson plot for the synthesized  $\text{Ag}_2\text{S}$  nanoparticles is shown in Fig. 3.



**Fig. 3** W-H analysis of  $\text{Ag}_2\text{S}$  particles

The crystallite size of the synthesized particle was found to be 217 nm. A small non uniform lattice strain (0.039 %) was observed in the sample. The non-uniform strain and the crystallite size was calculated from the slope and the y-intercept of the fit, respectively.

#### Morphology study (SEM)

Figure 4 (left) depicts the SEM image of  $\text{Ag}_2\text{S}$  nanoparticles. Formation of agglomerated spherical  $\text{Ag}_2\text{S}$  nanoparticles can be seen from this Figure. Therefore it is difficult to estimate the real particles size. Energy dispersive X-ray spectroscopy (EDS) was also performed to determine the chemical composition of the prepared  $\text{Ag}_2\text{S}$  nanoparticles (shown in Fig. 4 (right)). The obtained EDS results confirmed the presence of Ag and S in the final products.

The absence of extra peaks, besides the expected ones for nanocrystals, suggests that the obtained powders are very pure.

#### UV-VIS reflectance

The UV-VIS reflectance spectrum of the sample is presented in Fig. 5a. The Kubelka-Munk function was used to convert the diffuse reflectance into the absorption coefficient and spectrum is presented in Fig. 5b.

$$\alpha = \frac{k}{s} = \frac{(1-R_\infty)^2}{2R_\infty} \equiv F(R_\infty) \quad (6)$$

where  $S$  and  $K$  are the scattering and absorption coefficients; the reflectance  $R_\infty$  is equal to:  $\frac{R_{\text{sample}}}{R_{\text{S standard}}}$  [16].

Bulk  $\text{Ag}_2\text{S}$  is a semiconductor with a direct band gap of 0.9 to 1.05 eV [17]. The following equation was used to determine the band gap of  $\text{Ag}_2\text{S}$  nanoparticles [18]:

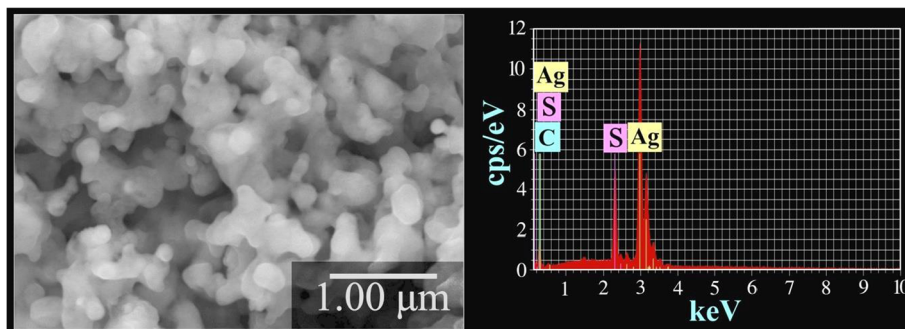
$$\alpha = A(h\nu - E_g)^n / h\nu \quad (7)$$

where  $A$  is constant,  $E_g$  is the absorption band gap,  $\alpha$  is the absorption coefficient, and  $n$  depends on the type of transition,  $n$  may assume the values 1/2, 2, 3/2 and 3 respectively corresponding to allowed direct, allowed indirect, forbidden direct and forbidden in direct transitions [19].

Since  $\text{Ag}_2\text{S}$  nanoparticles have direct allowed transitions so we choose  $n = 1/2$ . The band gap of  $\text{Ag}_2\text{S}$  nanoparticles was determined by extrapolating the function of  $(\alpha h\nu)^2$  in term of  $h\nu$  as shown in the Fig. 6 and it was found that the band gap of  $\text{Ag}_2\text{S}$  nanoparticles is around 0.96 eV, which is in good agreement with previous reports for the band gap energy of  $\text{Ag}_2\text{S}$  nanoparticles (0.9–1.1 eV) [19–21].

#### FT-IR analysis

Figure 7 shows FT-IR spectrum of  $\text{Ag}_2\text{S}$  nanometric powders. The characteristic vibration of Ag-S appears located at 500–600  $\text{cm}^{-1}$  while the broad and small peaks located



**Fig. 4** SEM image (left side) of and EDS analysis of the  $\text{Ag}_2\text{S}$  nanoparticle (right side)

at  $3400$  and  $1600\text{ cm}^{-1}$  can be attributed to the stretching and bending vibrations of the O–H bond of the adsorbed  $\text{H}_2\text{O}$  molecules on the surface of  $\text{Ag}_2\text{S}$  [22, 23].

#### Optical constants of $\text{Ag}_2\text{S}$ nanoparticles

The K–K method was used to determine the Far-infrared optical constants of the prepared  $\text{Ag}_2\text{S}$  semiconductor nanometric powders by using FT-IR transmittance spectral

data. The absorption ( $A$ ) can be obtained from transmittance according to Lambert's law [24]:

$$A(\omega) = \log \frac{I_0}{I} = \log_{10} \frac{1}{T(\omega)} = 2 - \log_{10}(T(\omega)\%) \quad (8)$$

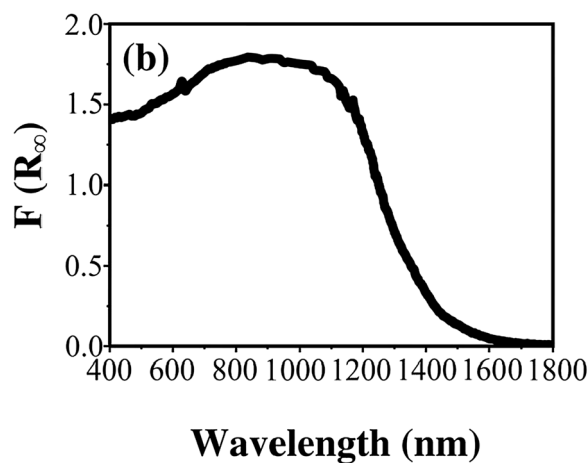
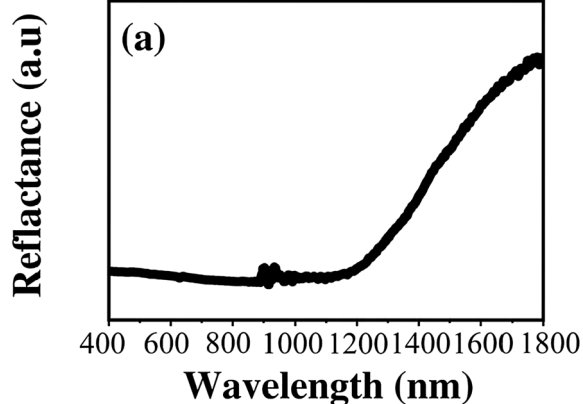
$$R(\omega) = 100 - [T(\omega) + A(\omega)] \quad (9)$$

where  $R(\omega)$  is the reflectance in the particular wave number. The reflective index  $n$  is an important physical quantity in optical design and generally is a complex quantity:

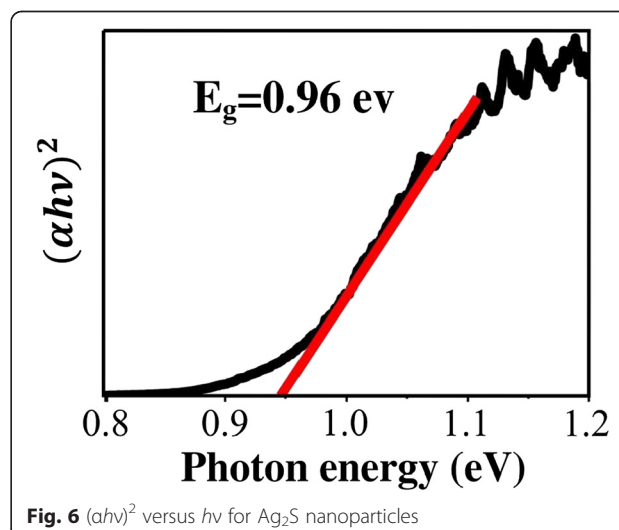
$$\tilde{n}(\omega) = n(\omega) + ik(\omega) \quad (10)$$

where  $n(\omega)$  and  $k(\omega)$  are the real and the imaginary parts of complex refractive index respectively, and can be obtained by the following equations:

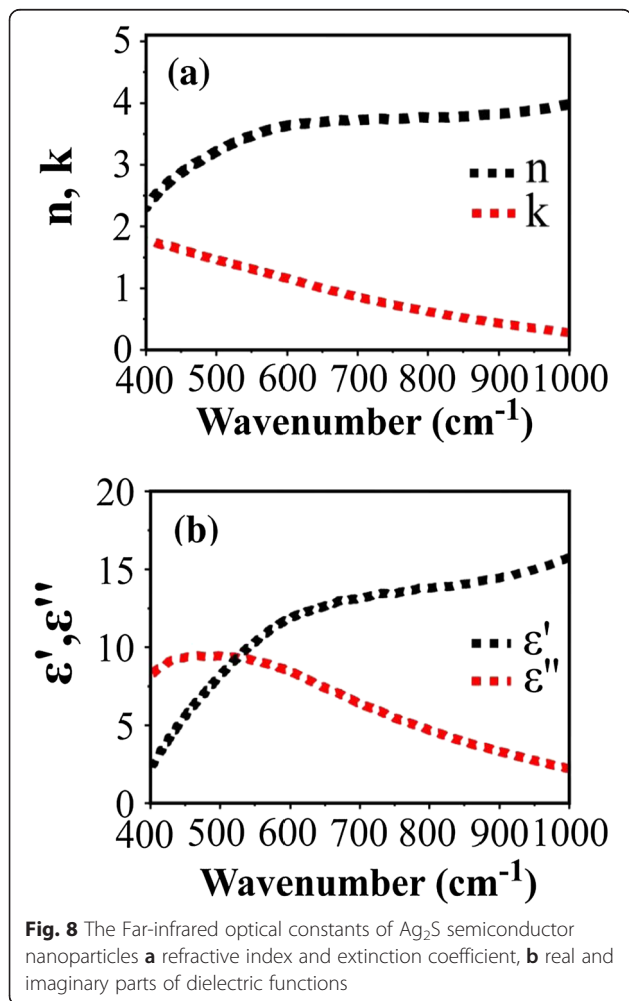
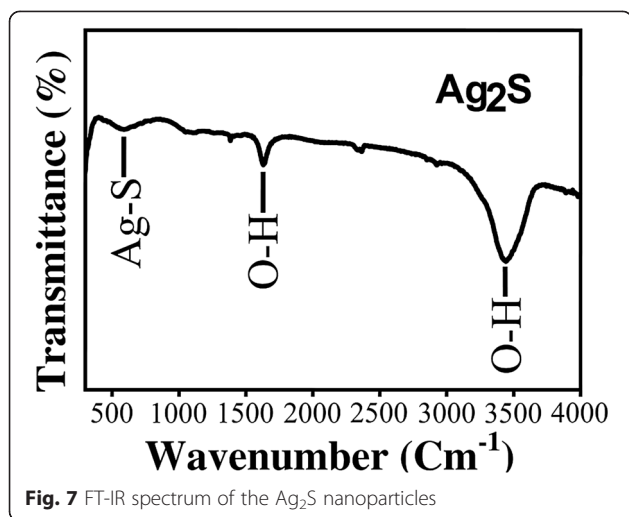
$$n(\omega) = \frac{1 - R(\omega)}{1 + R(\omega) - 2\sqrt{R(\omega)} \cos\phi(\omega)} \quad (11)$$



**Fig. 5** UV–VIS reflectance spectrum of  $\text{Ag}_2\text{S}$  nanoparticles **a** reflectance and **b** absorption coefficient



**Fig. 6**  $(\alpha h\nu)^2$  versus  $h\nu$  for  $\text{Ag}_2\text{S}$  nanoparticles



$$k(\omega) = \frac{2\sqrt{R(\omega)} \cos(\varphi)}{1 + R(\omega) - 2\sqrt{R(\omega)} \cos \varphi(\omega)} \quad (12)$$

Here,  $\varphi(\omega)$  is the phase change between the incident and the reflected signal at a particular wavenumber  $\omega$ . This phase change can be calculated from the K-K dispersion relation [25]:

$$\varphi(\omega) = \frac{-\omega}{\pi} \int_0^{\infty} \frac{\text{Ln}R(\omega') - \text{Ln}R(\omega)}{\omega'^2 - \omega^2} d\omega' \quad (13)$$

This integral can be precisely evaluated by Maclaurin's method [26]:

$$\varphi(\omega_j) = \frac{4\omega_j}{\pi} \times \Delta\omega \times \sum_i \frac{\text{Ln}(\sqrt{R(\omega)})}{\omega_i^2 - \omega_j^2} \quad (14)$$

here  $\Delta\omega = \omega_{j+1} - \omega_j$  and if  $j$  is an even number then  $i=1, 3, 5, 6, \dots, j-1, j+1, \dots$  while if  $j$  is an odd number then  $i=2, 4, 6, \dots, j-1, j+1, \dots$

In addition, the dielectric function can be obtained by the square of the refractive index. Therefore, the real and imaginary parts of the complex dielectric function are:

$$\bar{\epsilon} = [\tilde{n}(\omega)]^2 = [n(\omega) + ik(\omega)]^2 \quad (15)$$

$$\Rightarrow \epsilon' + i\epsilon'' = n^2(\omega) - k^2(\omega) + 2in(\omega)k(\omega) \quad (16)$$

$$\Rightarrow \begin{cases} \epsilon'(\omega) = n^2(\omega) - k^2(\omega) \\ \epsilon''(\omega) = 2n(\omega)k(\omega) \end{cases} \quad (17)$$

The Far-infrared optical constants of Ag<sub>2</sub>S semiconductor nanoparticles was calculated by the above equations and the spectrums are presented in Fig. 8a and 8b.

## Conclusion

We have successfully prepared Ag<sub>2</sub>S semiconductor nanometric powders by using a simple and low cost wet chemical precipitation technique. The micro-structural analysis of the sample was done through XRD pattern analysis and Rietveld refinement analysis. No impurity phase was observed in the X-ray diffraction pattern. The crystallite size of the synthesized particles was obtained by Hall-Williamson plot for the synthesized Ag<sub>2</sub>S nanoparticles and it was found to be 217 nm. The Far-infrared optical constants of the prepared Ag<sub>2</sub>S semiconductor nanoparticles were evaluated by means of FTIR transmittance spectra data and K-K method.

## Competing interests

The authors declare that they have no competing interests.

## Authors' contributions

RZ, HAA, and GZ prepared Ag<sub>2</sub>S nanoparticles, carried out the structural analyses of the samples and took part in the manuscript preparation. JMF, AZ, and BS organized the study, studied the data, and contributed to the manuscript writing. All authors read and approved the final manuscript.

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