



Silver Sulfide Nanostructures: Synthesis and Characterization

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ABSTRACT

Ag₂S semiconductor nanoparticles were prepared successfully using the chemical precipitation method. The XRD pattern shows that the Ag₂S particles are crystalline with a monoclinic phase. "Debye-Scherrer" formula was used to calculate the crystal size and it was found to be 64.22 nm. According to FE-SEM images, all particles have a spherical shape. The band gap for Ag₂S was estimated from the optical absorption curve and it was 3.89 eV.

1. INTRODUCTION

Characteristics of semiconductor nanostructured materials are well understood and they depend on their size, shape, and surface charge [1,2]. Chalcogenides of transition metals are used [3], with their remarkable photoelectron transformation capabilities and prospective applications, in chemistry, physics [4,5], biology, medicine, and materials science [6]. They are also used as important components in solar cells, photovoltaic devices, sensitive sensors, and slow-release medication [7,8]. Due to its excellent optoelectronic qualities, Ag₂S is one of the most significant chalcogenides [9]. At the nanoscale level, Ag₂S exhibits electronic and optoelectronic properties which lead to use them in many applications such as in electrical and optical devices [10]. Owing to the narrow band gap (0.9-1.05 eV) [11,12], silver sulfide nanoparticles reveal photoelectric and thermoelectric properties. Ag₂S is a significant chalcogenide that finds widespread use in a variety of scientific and technological fields [13,14], including IR detectors, photovoltaic cells, electrochemical storage cells, and photoconducting cells [15]. It is also widely known for its ability to function as mixed conductor of ions and electrons at temperatures higher than 200°C [16,17]. Some chemical and physical methods are used to synthesize Ag₂S nanoparticles, including chemical bath

deposition (CBD), spray pyrolysis deposition (SPD), sequential ionic layer adsorption reaction (SILAR), molecular beam epitaxy (MBE), gamma irradiation, thermal evaporation, and sol-gel, and ion implantation techniques [11, 18]. The purpose of this work was to use the chemical precipitation approach to explain the growth process of silver sulfide nanostructures. By adjusting variables including pH, precursors' concentration, reaction time, and reaction temperature, the structural and optical features of the synthesized Ag₂S nanoparticles can be controlled. Previously, chemical precipitation approach is used to prepare Ag₂S nanorods because it is an environmentally friendly and inexpensive method. The authors used silver nitrate and thiourea as starting materials with different molar concentration. [19] Emadi group used sonochemical method to fabricate Ag₂S nanoparticles. The authors used thioacetic acid as a source for sulphide and they found that the ultrasound radiation with 60 W for 20 minutes radiation was capable to produce nanoparticles with an average size of 11.8 nm. [20]

2. MATERIALS AND METHODS

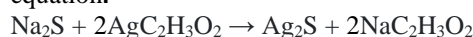
2.1 Chemical Substances

Silver acetate (99.9%, CH₃CO₂Ag) and sodium sulfide (99%, Na₂S) were purchased from THOMAS BAKER, deionized water was used as a solvent.

2.2 Preparation of Silver Sulfide

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To create silver sulfide nanoparticles, first. 0.16691 g of silver acetate was dissolved in 100 mL of deionized water and stirred. No particles were visible. This produced 0.01 M of silver acetate. Silver acetate solution was mixed with 100 mL of 0.01 M sodium sulfide solution. The solution was stirred for an hour at 50°C on the magnetic stirrer. Maintaining the temperature while stirring after the addition was finished. After centrifugation at 4000 rpm, the precipitate was finally recovered and dried at 50°C for three hours. preparation equation.



3. CHARACTERIZATION AND INSTRUMENTATIONS

To characterize the crystal properties of the prepared silver sulphide, X-ray diffraction, was used. Structural properties of Ag₂S nanoparticles were determined using field emission scanning electron microscopy (FE-SEM). Elemental analysis was performed using EDX. Fourier Transform Infrared Spectroscopy, also known as FTIR was used to study the functional groups of prepared sample. The band gap was investigated from the UV-Vis chart using tauc equation.

4. RESULT AND DISCUSSIONS

The XRD chart, which is shown in Figure 1, showed the appearance of many narrow diffraction peaks indicating the crystalline nature. According to JCPDS Card No .14-0072, peaks appeared at 26.32°, 28.97°, 31.51°, 33.62°, 34.39°, 36.82°, 37.73°, 40.75°, 43.40°, 47.77°, 48.76°, 53.27°, 58.36°, and 63.75° are corresponding to -101, 111, -112, 120, -121, 121, -103, 031, 200, 023, 113, 311, 212, 222, 024, and 034 diffraction planes.

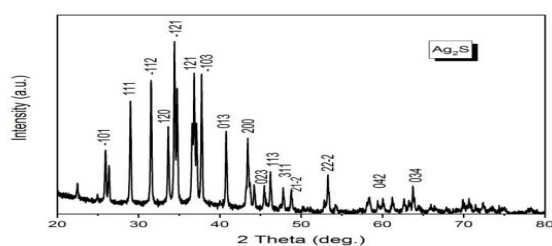


Figure 1. XRD pattern of Ag₂S nanoparticles.

The diffraction angle of 34.39° was used to obtain the crystallite size of Ag₂S particles Scherrer equation $2D \sin \theta = K\lambda / \beta \cos \theta$. Here, θ is the diffraction angle, K is a constant equal to 0.9 and β is the full width at half maximum., and the wavelength of the CuK α is 1.54056 Å [21].

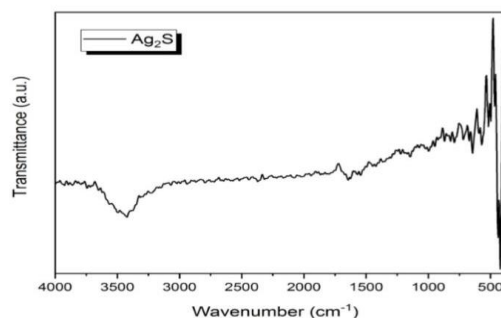


Figure 2. FTIR Spectrum of Ag₂S Nanoparticles.

Figure 2 shows the FTIR spectrum of Ag₂S nanoparticles. The peaks that appeared in the range of 400–600 cm⁻¹ are related to Ag–S bonds, supporting the formation of silver sulfide nanoparticles. The structural properties of the prepared particles were determined from the FE-SEM images. It is clear from the image in Figure 3 that the particles are spherical and their average size is calculated using imageJ program and it is found to be 64.3 nm.

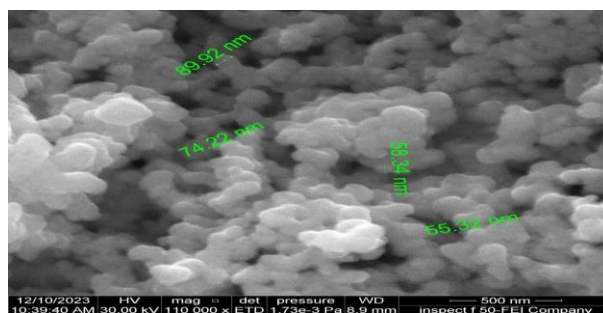


Figure 3. FE-SEM images of Ag₂S nanoparticles.

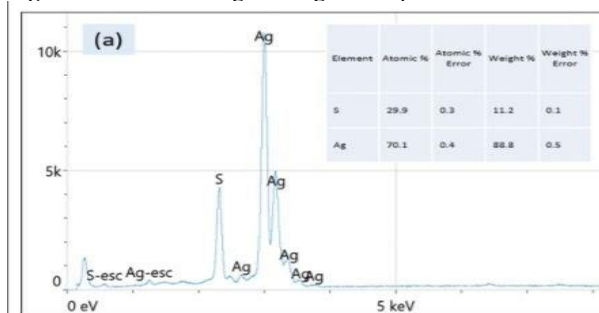


Figure 4. EDS chart of Ag₂S particles.

The presence of silver and sulphide elements within Ag₂S nanoparticles was confirmed using EDS chart. It is clear from Figure 4 that our sample has no impurities due to the absence of any peak except for silver and sulphide.[22] The atomic percentage for Ag is 70.1%, and for S is 29.9%.

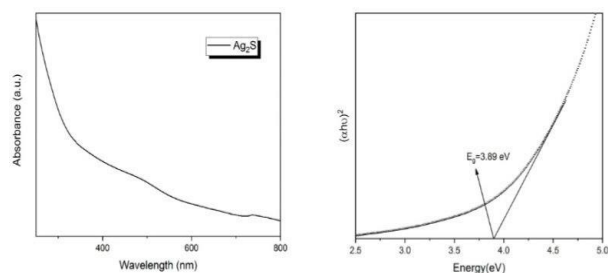


Figure 5. UV-Vis absorbance spectrum for silver sulfide nanoparticles and band gap energies of Ag₂S nanoparticles.

is absorbance spectrum of silver sulphide nanoparticles (left image) and the band gap energy of Ag₂S nanoparticles (right image).

The optical properties of Ag₂S were studied using UV-Vis spectroscopy. It is clear from Figure 5 that the absorption band edge [23] of the as-prepared nanoparticles is around 550 nm. The band gap energy, calculated using Tauc's equation, is 3.89 eV. This value is larger than that of bulk Ag₂S (0.9-1.05 eV) [24], suggesting the effect of quantum confinement as the size of the particles decreases.

5. CONCLUSION

In this study Ag₂S nanoparticles were fabricated using chemical precipitation technique. The crystal properties of particles were confirmed by XRD patterns. It seems that the particles are monoclinic with a crystallite size of 64.22356 nm. Most of Ag₂S particles are spherical in shape with an average diameter of 64.3 nm. Their optical properties show that the as-prepared particles are in nanoscale level due to the value of band gap (3.89 eV) which is greater than that of bulk Ag₂S that is possibly related to the quantum size effect.

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Arabic Abstract

في هذه الدراسة تم تصنيع الجسيمات النانوية Ag₂S باستخدام تقنية الترسيب الكيميائي. تم تأكيد الخصائص البلورية للجسيمات بواسطة مخطط XRD ويبدو أن الجسيمات أحادية الميل بحجم بلوري قدره 64.22 nm. معظم جسيمات Ag₂S كروية الشكل ويبلغ متوسط قطرها 64.3 nm. أظهرت خصائصها البصرية أن الجسيمات المحضرة موجودة على مستوى النانو بسبب قيمة فجوة النطاق (3.9 فولت) والتي هي أكبر من قيمة Ag₂S السائبة والتي ربما تكون مرتبطة بتأثير الحجم الكمي.
