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Organic-Medium Synthesis of $Cd_3Sb_2S_8$ and Its Thermogravimetric Analysis (TGA)

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Abstract. *In this study, the $Cd_3Sb_2S_8$ semiconductor compound was synthesized in a non-aqueous medium using antimony ore obtained from the Daridag deposit in the Julfa district of the Nakhchivan Autonomous Republic. The raw ore was initially enriched by flotation and subsequently converted into Sb_2S_3, Sb_2S_5, and Na_3SbS_4 precursor compounds through successive chemical transformations. The final $Cd_3Sb_2S_8$ phase was prepared by a solvothermal process in ethylene glycol at 150 °C, achieving an approximate product yield of 90%. A possible reaction pathway describing the formation of $Cd_3Sb_2S_8$ was proposed, and the sequential nucleation and crystal growth processes were discussed. The thermal behavior of the synthesized material was evaluated by thermogravimetric (TG) analysis, which demonstrated high thermal stability with a total mass loss of only 14.34% up to approximately 900 K. The obtained results indicate that the synthesized $Cd_3Sb_2S_8$ is a thermally stable semiconductor material with potential for further investigation in advanced functional applications.*

Keywords: *$Cd_3Sb_2S_8$, non-aqueous synthesis, solvothermal method, thermogravimetric analysis, semiconductor*

Introduction

The thioantimonate(III) compound $Cd_3Sb_2S_8$ was synthesized by an environmentally friendly ionothermal method (Gang et al., 2021). CdS thin films were prepared by the pyrolytic deposition method, and their structural, optical, and electrical properties were investigated (Abhijit & Elahipasha, 2011). The physicochemical properties of the samples were evaluated using spectrophotometry, X-ray diffraction (XRD), scanning electron microscopy (SEM), and other advanced analytical techniques (Chen et al., 2022). Sulfide-based semiconductor compounds of heavy metals exhibit high optical absorption coefficients, favorable electronic properties, and broad application potential in optoelectronics, solar energy conversion, and sensor technologies (Valiyeva et al., 2025; Fatemipayam et al., 2025).

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Materials and Methods

Antimony ore extracted from the Daridag deposit located in the Julfa district of the Nakhchivan Autonomous Republic was used as the starting material for the synthesis of $Cd_3Sb_2S_8$ (Ganiyev et al., 2026). The ore was first enriched by the flotation method (Aliyeva, 2021), after which high-purity Sb_2S_3 was obtained through successive chemical transformations (Aliyeva et al., 2026). In the next stage, Sb_2S_3 was oxidized to Sb_2S_5, and Na_3SbS_4 was subsequently synthesized from this product (Aliyeva, 2022; Aliyeva, 2024). The prepared Na_3SbS_4 served simultaneously as both the antimony and sulfur source during the synthesis process.

The $Cd_3Sb_2S_8$ sample was synthesized in a non-aqueous medium using ethylene glycol as the solvent via the solvothermal method (Hajiyeva et al., 2026). During the synthesis, Na_3SbS_4 and $Cd(NO_3)_2 \cdot 4H_2O$ were sequentially added to the reaction solution, while NH_4Cl was introduced to establish a homogeneous reaction system and to promote controlled crystal growth. The resulting mixture was sealed in a hermetically closed reactor and maintained at 150 °C for 8–10 h. During this period, $Cd_3Sb_2S_8$ crystals gradually formed through the interaction of Cd^{2+} ions with thioantimonate-derived complex species (Aliyeva et al., 2026).

After completion of the heat treatment, the reactor was allowed to cool naturally to room temperature. The resulting solid phase was separated from the solution by vacuum filtration. The precipitate was washed several times with distilled water followed by ethanol to remove soluble impurities. The purified product was dried at 110 °C, ground into a homogeneous powder, and prepared for subsequent physicochemical characterization. The calculated product yield was approximately 90%, indicating that the selected technological parameters and reaction conditions provided high synthesis efficiency.

The overall reaction can be expressed as follows:

$$2Na_3SbS_4 + 3Cd(NO_3) \xrightarrow{ethylene\ glycol} Cd_3Sb_2S_8\downarrow + 6NaNO_3$$

It should be noted that this equation represents the overall stoichiometric expression of the process (Yang et al., 2024; Zhou et al., 2013), while the reaction actually proceeds through several intermediate steps.

Formation Mechanism of $Cd_3Sb_2S_8$

Stage I. Dissociation of Sodium Thioantimonate

In the non-aqueous ethylene glycol medium, sodium thioantimonate dissociates to form the thioantimonate (Han et al., 2022) complex:

$$Na_3SbS_4 \rightarrow 3Na^+ + SbS_4^{3-}$$

Stage II. Ionization of Cadmium Nitrate

Cadmium nitrate dissolves in ethylene glycol, generating cadmium ions.

$$Cd(NO_3)_2 \rightarrow Cd^{2+} + 2NO_3^-$$

Stage III. Interaction of the Complexes

Upon heating, Cd^{2+} ions approach the SbS_4^{3-} complexes and initially form Cd–S coordination bonds.

$$3Cd^{2+} + 2SbS_4^{3-} \rightarrow Cd_3Sb_2S8\downarrow$$

This intermediate complex represents the initial stage of crystallization.

Stage IV. Nucleation

Heating the reaction medium to 150 °C decreases the stability of the intermediate complexes, and their condensation initiates the crystallization of $Cd_3Sb_2S_8$

$$[3Cd - SbS_4]^{3-} \rightarrow Cd3Sb_2S_8 \text{ (nuclei)}$$

At this stage, very fine dark-brown particles are observed in the solution.

Stage V. Crystal Growth

During the subsequent stages of the reaction, the SbS_4^{3-} and Cd^{2+} ions present in the solution attach to the surfaces of the previously formed crystallization nuclei, resulting in the gradual growth of the crystals.

$$Cd_3Sb_2S_8 \text{ (nuclei)} + Cd^{2+} + SbS_4^{3-} \rightarrow Cd_3Sb_2S_8 \text{ (crystal growth)}$$

Stage VI. Crystal Growth

During the solvothermal treatment, the previously formed $Cd_3Sb_2S_8$ nuclei gradually develop through the incorporation of Cd^{2+} and SbS_4^{3-} species from the solution onto their surfaces. The elevated temperature enhances ion mobility, promoting the orderly development of the crystal structure. As the reaction proceeds, the crystallinity increases, resulting in homogeneous $Cd_3Sb_2S_8$ particles.

Stage VII. Removal of By-products

After the separation of the main product, Na^+ and NO_3^- ions remain in the solution, while excess sulfur is present in elemental or polysulfide forms. During the washing process, these by-products are removed from the system, thereby increasing the purity of the $Cd_3Sb_2S_8$ sample.

Brief Description of the Reaction Mechanism

When Na_3SbS_4 dissolves in ethylene glycol, thioantimonate complexes are formed in the solution (Garayev & Aliyeva, 2022). Their interaction with Cd^{2+} ions leads to the formation of intermediate coordination compounds. Heating the reaction mixture to 150 °C accelerates the transformation of these complexes into $Cd_3Sb_2S_8$ crystal nuclei. Subsequent attachment of ions to the surfaces of these nuclei promotes crystal growth, resulting in the formation of a well-crystallized $Cd_3Sb_2S_8$ phase. Ethylene glycol maintains the stability of the reaction medium, whereas NH_4Cl regulates the crystallization process, facilitating the formation of homogeneous particles (Mamedova, 2021b).

Results and Discussion

Thermogravimetric (TG) analysis was performed to evaluate the thermal stability of the $Cd_3Sb_2S_8$ compound and to investigate the mass changes occurring during heating (Azimova et al., 2026). This technique enables the determination of the material's thermal stability (Mamedova, 2020a), decomposition stages, and the release of volatile components (Mamedova & Nasirli, 2021; Mamedova, 2021a). The TG results provide valuable information on the high-temperature behavior of the synthesized sample and allow the evaluation of the temperature range in which the material can be effectively applied.

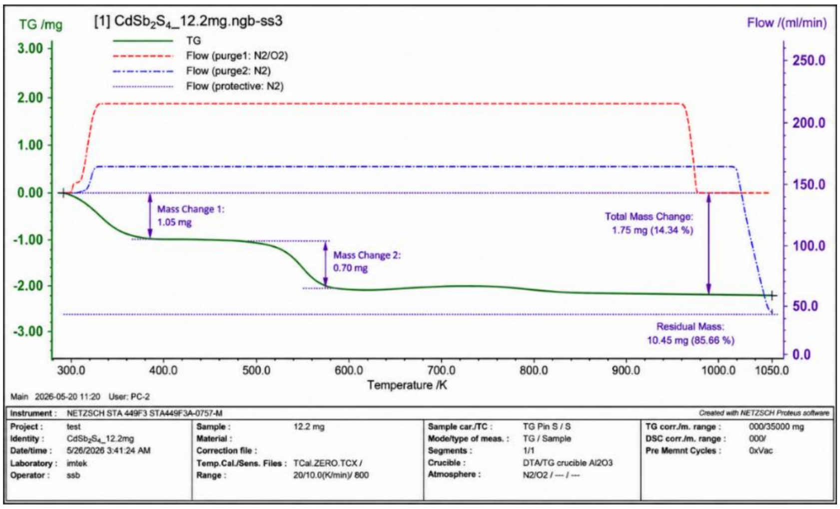

Figure 1

Thermogravimetric (TG) analysis of the $Cd_3Sb_2S_8$ compound

The TG analysis demonstrated that the mass change of the $Cd_3Sb_2S_8$ sample during heating occurred in a stepwise manner. A mass loss of 3.90 mg in the temperature range of 320–450 K was attributed to the removal of adsorbed water and volatile residual species. An additional mass loss of 1.65 mg was recorded within the 470–560 K range, indicating the onset of thermal transformations in the material. The TG curve remained nearly unchanged in the temperature interval of 700–920 K. In the temperature range of 920–1050 K, a further mass loss of 1.75 mg was observed, which was attributed to the partial decomposition of the crystal structure (Mamedova, 2020b) together with the release of sulfur-containing volatile species. These results confirm that the $Cd_3Sb_2S_8$ compound possesses high thermal stability over a wide temperature range (Mamedova, 2020b; Mamedova, 2016).

The results of this study demonstrated that the high-yield synthesis of $Cd_3Sb_2S_8$ in a non-aqueous medium is feasible using the Na_3SbS_4 precursor obtained from the antimony ore of the Daridag deposit. The sample synthesized by the solvothermal method exhibited high crystallinity and a homogeneous morphology, confirming the effectiveness of the selected synthesis conditions. Thermal analysis showed that the material retained its thermal stability over a broad temperature range, with gradual structural changes occurring only at elevated temperatures. The proposed formation mechanism consistently explains the nucleation and crystal growth stages in agreement with the experimental results.

Conclusion

In this study, the $Cd_3Sb_2S_8$ semiconductor compound was successfully synthesized from the antimony ore of the Daridag deposit using a non-aqueous solvothermal method, yielding a high product recovery. Structural and thermal analyses confirmed that the synthesized sample possesses high crystallinity and excellent thermal stability. The proposed reaction mechanism is consistent with the experimental findings and provides a reasonable explanation for the formation stages of the $Cd_3Sb_2S_8$ phase. The obtained results indicate that the synthesized $Cd_3Sb_2S_8$ material is a promising semiconductor for applications in photocatalysis, solar energy conversion, photodetectors, and other optoelectronic systems.

Conflict of Interest

The authors declare that they have no conflict of interest regarding this article.

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