

Synthetic Preparation of $\text{Cd}_3\text{Sb}_2\text{S}_8$ Phase from Darıdağ ore (Nakhchivan) and Investigation of its Physicochemical Properties

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Abstract. Chalcogenide compounds containing $\text{Cd}_3\text{Sb}_2\text{S}_8$ are of particular interest in modern materials science due to their semiconductor properties. In this work, the compound in question was obtained from a natural ore taken from the Darıdağ deposit located in the Nakhchivan Autonomous Republic. At the initial stage, the ore was processed by flotation and purified from arsenic impurities, and as a result, antimony (III) sulfide was isolated as the main component. The synthesis process was carried out at high temperature, in a protective argon environment, as a result of the joint interaction of cadmium, Sb_2S_3 and elemental sulfur. The reaction was carried out in stages, minimizing the possibility of oxidation, and no additional solvents or chemical reagents were used. The thermal behavior of the obtained sample was studied by differential thermal analysis, and its stability at high temperatures, as well as its melting properties, were determined. The surface structure and morphology were examined by scanning electron microscopy, and it was found that the material consists of small-sized crystals. The elemental composition was confirmed by energy dispersive analysis results and it was determined that the elements Cd, Sb and S were in accordance with the theoretical ratios. X-ray diffraction results showed that the synthesized material has a mainly crystalline structure, but also the presence of a slight amorphous phase. Overall, the obtained results demonstrate that the $\text{Cd}_3\text{Sb}_2\text{S}_8$ compound is promising as a functional material, especially for application in optoelectronic and thermoelectric fields.

Keywords: darıdağ mineral, X-ray phase analysis, SEM, EDS, melting temperature

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Received: 21 April 2026; Accepted: 25 August 2026; Published online: 22 October 2026

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Darıdağ filizindən (Naxçıvan) $\text{Cd}_3\text{Sb}_2\text{S}_8$ fazasının sintezi və onun fiziki-kimyəvi xassələrinin tədqiqi

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Xülasə. $\text{Cd}_3\text{Sb}_2\text{S}_8$ tərkibli xalkogenid birləşmələri yarımkeçirici xassələrinə görə müasir materialşünaslıqda xüsusi maraq doğurur. Bu işdə tədqiq olunan birləşmə Naxçıvan Muxtar Respublikasında yerləşən Darıdağ yatağından götürülmüş təbii filizdən əldə edilmişdir. İlk mərhələdə filiz flotasiya üsulu ilə emal edilmiş və arsen qarışıqlarından təmizlənmiş, nəticədə əsas komponent kimi sürmə(III) sulfid ayrılmışdır. Sintez prosesi yüksək temperaturda, qoruyucu argon mühitində kadmiyum, Sb_2S_3 və elementar kükürdün birgə qarşılıqlı təsiri nəticəsində həyata keçirilmişdir. Reaksiya oksidləşmə ehtimalını minimuma endirməklə mərhələli şəkildə aparılmış, əlavə həlledicilərdən və kimyəvi reagentlərdən istifadə edilməmişdir. Alınmış nümunənin termik davranışı diferensial termik analiz vasitəsilə öyrənilmiş, yüksək temperaturda davamlılığı və ərimə xassələri müəyyən edilmişdir. Səthin quruluşu və morfologiyası skanedic elektron mikroskopiyası ilə tədqiq edilmiş və materialın kiçikölçülü kristallardan ibarət olduğu müəyyən olunmuşdur. Element tərkibi enerji-dispersiv analiz nəticələri ilə təsdiqlənmiş, Cd, Sb və S elementlərinin nəzəri nisbətlərə uyğunluğu müəyyən edilmişdir. Rentgen difraksiyası nəticələri sintez olunmuş materialın əsasən kristallik quruluşa malik olduğunu, eyni zamanda az miqdarda amorf fazanın da mövcudluğunu göstərmişdir. Ümumilikdə, əldə edilən nəticələr $\text{Cd}_3\text{Sb}_2\text{S}_8$ birləşməsinin funksional material kimi, xüsusilə optoelektronika və termoelektrik sahələrində tətbiqi baxımından perspektivli olduğunu nümayiş etdirir.

Açar sözlər: Darıdağ mineralı, rentgen-faza analizi, skanedic elektron mikroskopiyası (SEM), enerji-dispersiv rentgen spektroskopiyası (EDS), ərimə temperaturu

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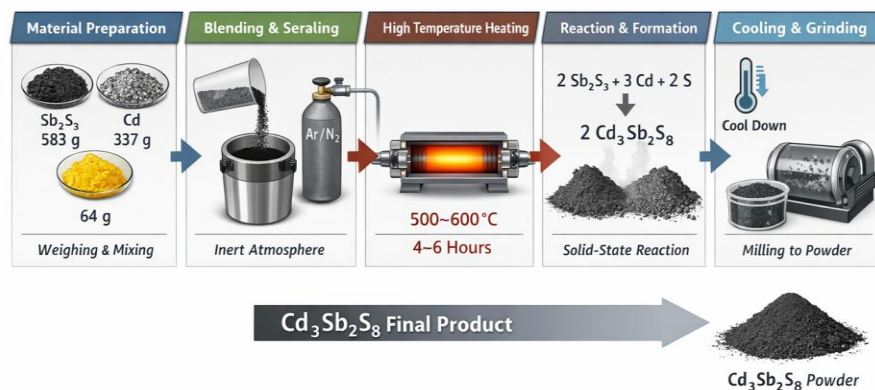
Daxil oldu: 21 Aprel 2026; Qəbul edildi: 25 Avqust 2026; Onlayn dərc edildi: 22 Oktyabr 2026

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Introduction

The deposited CdS thin films were found to be n-type polycrystalline and photoactive. Due to the high series resistance, the conversion efficiency for the PEC cell was 0.17%, the fill factor was 0.38, and a sharp peak was observed in the spectral response near 500 nm (Abhijit, 2011). The thermodynamic parameters of the CdSb compound were studied by the electromotive force method at different temperature ranges. The phase composition of the samples was determined by X-ray diffraction analysis and the results were compared with the available literature data (Babanly, 2024). The $\text{Sb}_2\text{S}_3/\text{CdS}/\text{CdIn}_2\text{S}_4$ cascaded S-scheme heterojunction was obtained by a combination of different synthesis methods. Hydrothermal, chemical bath deposition and microwave hydrothermal methods were used in the preparation of the structure (Chen, 2022).

Graphic abstract of the $\text{Cd}_3\text{Sb}_2\text{S}_8$ compound:



The Sb_2S_3 semiconductor is considered a promising light-absorbing material due to its suitable band gap, one-dimensional crystal structure and non-toxic composition. The structural properties of Sb_2S_3 thin films grown on a CdS layer and a glass substrate were compared by SEM and XRD analyses, and it was determined that the growth direction of the ribbon crystals depended on the substrate. The prepared CdS/ Sb_2S_3 heterojunction solar cells exhibited high photoelectric performance and maintained their stability even after long-term storage (Chen, 2017). A CdS, TiO_2 and MCM-41 based nanocomposite photocatalyst was prepared for the efficient degradation of Methylene Blue and characterized by various analytical methods. The highest photocatalytic activity was observed in the 15% loaded CdS/ TiO_2 /MCM-41 sample, and high MB degradation and significant mineralization were achieved in a short time in neutral media. Statistical modeling results showed that this material is a promising and applicable photocatalyst for wastewater treatment (Fatemipayam, 2025).

The structure of $\text{GeS}_2\text{--Sb}_2\text{S}_3\text{--CdS}$ chalcogenide systems was studied by Raman spectroscopy and it was found that the addition of Sb_2S_3 and CdS had a significant effect on the structure of the glass network. Although the addition of CdS increased the crystallization tendency, the presence of Sb_2S_3 improved the homogeneity of the glass by increasing the solubility of CdS (Hai-Tao, 2017). The crystallization and chemical resistance properties of $\text{GeS}_2\text{--Ga}_2\text{S}_3\text{--CdS}$ chalcogenide glasses have been investigated using various analytical methods. It was found that the glass containing GGC5 showed high thermal stability and good chemical resistance to water (Luh, 2008). It was found that new luminescence centers and deep energy levels were formed in single crystals of CdSe doped with Sb and Bi, and the photoconductivity spectrum shifted to the red region (Odin, 2000). In the Cd–In–Te–S system, CdS crystals were prepared by doping with In and Te in Cd and InTe vapors by a four-zone annealing method, resulting in orange–red luminescence bands instead of green emission and metastable sphalerite CdS structures showing high photosensitivity, and spectra shifted to longer wavelengths compared to stable samples (Odin, 2004).

Although photocatalytic technologies using solar energy play an effective role in reactions such as water splitting and CO_2 conversion under visible light thanks to semiconductors with a band gap of 2.4 eV, such as CdS, their application is limited by problems of rapid charge recombination and photocorrosion (Ren, 2024).

The innovative significance of Darıdağ ore in this context is related to its dual function, serving both as a raw mineral source and as an effective precursor for Sb_2S_3 formation. After flotation and subsequent purification, a high-purity Sb_2S_3 phase can be obtained directly from the processed ore. This strategy removes the necessity for independently synthesized Sb_2S_3 , thereby streamlining the overall technological workflow. As a result, the process becomes less complex and more economically viable. In addition, energy demands are reduced due to the elimination of separate synthesis steps. Production costs are consequently minimized, improving overall efficiency. Therefore, the utilization of Darıdağ ore represents a sustainable and resource-efficient pathway for the fabrication of chalcogenide materials derived from natural mineral sources (Gubbuk, 2026).

The present study is distinguished by the direct application of Sb_2S_3 obtained from Darıdağ antimony ore as a precursor for the synthesis of $\text{Cd}_3\text{Sb}_2\text{S}_8$ chalcogenide material. In contrast to earlier research that predominantly depends on chemically synthesized Sb_2S_3 or commercially purified precursors, this work introduces a more sustainable and resource-efficient route by employing a naturally sourced mineral after the removal of arsenic impurities.

Furthermore, this research systematically examines the phase evolution, thermal stability, and microstructural development of $\text{Cd}_3\text{Sb}_2\text{S}_8$ formed under controlled solid-state synthesis conditions. The combination of ore beneficiation processes with semiconductor material synthesis presents an innovative strategy in chalcogenide preparation, contributing to improved economic efficiency and reduced environmental impact.

2 Experimental.

2.1 Ore processing and Sb_2S_3 extraction

The Darıdağ antimony ore was processed by a flotation technique aimed at selectively eliminating arsenic-bearing mineral phases. The flotation was performed under controlled pH conditions (2.5–5.3) using xanthate-based collectors together with MIBC as a frothing agent for a duration of 20 minutes. This procedure enabled an estimated arsenic removal efficiency of approximately 90–95%, leading to the formation of a relatively enriched Sb_2S_3 concentrate.

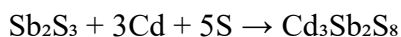
Subsequent X-ray diffraction (XRD) (Azimova, 2026) analysis indicated that the synthesized Sb_2S_3 phase possessed high crystallinity and purity (>90–95%), with no observable peaks corresponding to arsenic sulfide impurities.

The raw material used for the study was ore taken from the Darıdağ arsenic deposit of the Nakhchivan Autonomous Republic (Aliyeva, 2020; Aliyeva, 2025; Garayev, 2022, Hajiyeve, 2026). At the initial stage, the ore was processed by flotation to remove arsenic-containing components, and as a result, antimony (III) sulfide (Sb_2S_3) was obtained as the main phase. This purification stage plays an important role in preventing unwanted side reactions in the subsequent synthesis process and increasing the phase purity of the product to be obtained.

Darıdağ deposit constitutes an important natural source of antimony sulfide minerals suitable for semiconductor precursor production. In contrast to chemically synthesized Sb_2S_3 , the utilization of ore-derived material offers a more economical and resource-efficient alternative. Through beneficiation and subsequent purification procedures, arsenic-containing phases were substantially eliminated, resulting in the recovery of an Sb_2S_3 -rich concentrate with improved purity for further synthesis applications.

2.2. Synthesis of $\text{Cd}_3\text{Sb}_2\text{S}_8$

The synthesis of the $\text{Cd}_3\text{Sb}_2\text{S}_8$ compound was carried out as a result of the interaction of Cd metal, Sb_2S_3 and elemental sulfur under high temperature conditions. The molar ratio of the reagents was calculated in advance and stoichiometric compatibility was ensured, which creates conditions for the correct formation of the chemical composition of the final product. The overall reaction can be expressed as follows:



The synthesis process occurs according to a multi-stage solid-state mechanism, and this mechanism is determined by both kinetic and thermodynamic factors. In the initial stage, cadmium metal reacts with elemental sulfur to form the CdS intermediate phase. This stage is characterized by the formation of the first stable sulfide compound in the system and creates a chemically active environment for the onset of subsequent processes. Since the reactivity and diffusivity of sulfur increase at high temperatures, the interaction with Cd atoms accelerates and the CdS phase is easily formed. In the next stage, when the formed CdS particles come into contact with the Sb_2S_3 structure, solid-state diffusion processes are activated. Since the crystal structure of Sb_2S_3 is chain-like, with increasing temperature, its structure undergoes partial decomposition and reorganization. These structural changes allow Sb and S atoms to move more freely and intensify the interaction with CdS.

Elemental sulfur added to the system plays an important role, as it ensures the sulfur balance in the environment and contributes to the formation of a stable phase with a higher sulfur content. Under these conditions, the nucleation of a new crystalline phase - the $\text{Cd}_3\text{Sb}_2\text{S}_8$ compound - occurs as a result of mutual diffusion of Cd, Sb and S atoms. After the nucleation stage, the growth of crystal grains occurs, which is controlled by temperature and storage time.

The synthesis was carried out practically by a dry method. Cd and Sb_2S_3 were powdered, elemental sulfur was prepared separately, and all components were mixed homogeneously. Uniform distribution of the mixture is an important condition for the effectiveness of diffusion processes. The prepared mixture was placed in a tin capsule and sealed in an inert argon atmosphere to prevent oxidation.

The reaction was carried out in the temperature range of 550–650°C for 3–5 hours. This temperature range is high enough to ensure the diffusion of atoms and at the same time prevents excessive loss of sulfur. The staged heating regime allows the reaction to proceed more stably and homogeneously. Under these conditions, the Cd, Sb_2S_3 and S components interact to form the $\text{Cd}_3\text{Sb}_2\text{S}_8$ phase.

It is of particular importance to carry out the reaction in an inert environment, since at high temperatures, undesirable oxide phases such as CdO and Sb_2O_3 can form in the presence of oxygen. The argon environment prevents such side reactions, increases the selectivity of the synthesis process and maintains the phase purity of the product. After the synthesis was completed, the sample was gradually cooled inside the vessel, which helped to reduce thermal stresses and stabilize the crystal structure. The obtained $\text{Cd}_3\text{Sb}_2\text{S}_8$ granules were subsequently mechanically ground into a homogeneous powder and prepared for analytical studies.

Thus, the preparation of the $\text{Cd}_3\text{Sb}_2\text{S}_8$ compound is explained by the classical solid-phase synthesis mechanism, which includes the formation of the sulfide phase, activation of the structure, atomic diffusion and crystallization stages. The efficiency of this process is directly related to the purity of the reagents, homogeneous mixing, provision of an inert environment and selection of the optimal temperature regime.

2.3. DTA: thermal stability and phase transition behavior

The melting temperature of the sample was determined by the differential thermal analysis (DTA) method, and its diffractogram was also obtained (Aliyeva, 2026). The results of the DTA analysis are presented in Figure 1.

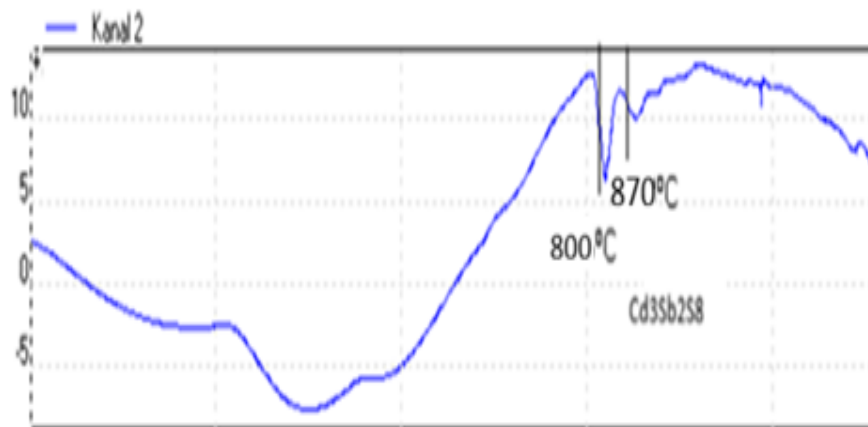


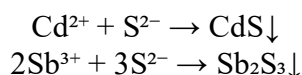
Figure 1
Melting temperature of cadmium–antimony sulfide

The presented DTA curve characterizes the thermal behavior of the $\text{Cd}_3\text{Sb}_2\text{S}_8$ -type sulfide system. The analysis reflects the energy changes occurring in the material during heating. Weak signals observed in the low-temperature region may be associated with the release of volatile components. At this stage, desorption of adsorbed species from the crystal surface takes place. With increasing temperature, the system gradually evolves toward a more stable crystalline state. In the intermediate temperature range, a reduction of internal lattice stresses can be observed. Ion diffusion becomes more active in this region. A noticeable thermal event appears around 800°C in the curve. This change may indicate the onset of structural rearrangement within the material. The endothermic response near 800°C is likely related to the weakening of crystal bonds.

At higher temperatures, the instability of the system increases. A sharp peak is detected at approximately 870°C , indicating a critical thermal event. This peak may correspond to a decomposition or phase transition temperature of the material. At 870°C , a significant part of the crystal structure undergoes reorganization. The probability of partial breaking of Sb–S and Cd–S bonds increases in this stage. At elevated temperatures, a transition toward a semi-amorphous state becomes possible. The change in the signal after the peak indicates that the system reaches a new equilibrium state. The DTA results define the limited thermal stability range of the material. This information is important for optimizing synthesis and application conditions. Overall, the curve shows that major thermal transformations in the $\text{Cd}_3\text{Sb}_2\text{S}_8$ system occur within the $800\text{--}870^\circ\text{C}$ temperature range.

2.4. Chemical analysis: gravimetric and EDTA titration methods

The chemical composition of the $\text{Cd}_3\text{Sb}_2\text{S}_8$ compound was analyzed by chemical methods, which are gravimetric (sulfide precipitation) and complexometric (EDTA) methods. The gravimetric method is based on the principle of precipitation of Cd^{2+} and Sb^{3+} ions in the $\text{Cd}_3\text{Sb}_2\text{S}_8$ compound in the form of sulfide. During the analysis, the sample is dissolved in an acidic medium, releasing metal ions into solution. H_2S or Na_2S is then added to the solution, resulting in the formation of sparingly soluble CdS and Sb_2S_3 precipitates:



The obtained precipitates are filtered, washed, dried and heated to constant mass and then weighed. The amount of Cd and Sb elements is calculated based on the mass of the precipitates. This method is distinguished by its high accuracy and simple implementation.

Complexometric (EDTA) analysis is based on the formation of stable complexes of metal ions with EDTA. At this stage, Cd^{2+} ions in the solution are directly titrated with EDTA solution and the equivalent point is determined using a suitable indicator (for example, eriochrome black T or murexide). Sb^{3+} ions are usually pre-oxidized or indirectly determined by placing them in a suitable complex medium. Since the complexes formed with EDTA are stable, the titration is completed with a color change at the end point.

Table 1

Chemical analysis of cadmium thioantimonate

Example, $\text{Cd}_3\text{Sb}_2\text{S}_8$, q		Amount of items, q				
		Cd		Sb		S
0.4200		Theor.	Exper.	Theor.	Exper.	Theor. Exper.
		0.1690	0.1681	0.1220	0.1211	0.1290 0.1281

2.5 XRD: crystal structure and phase identification

To determine the stoichiometric composition of the compound, we performed scanning electron microscopy (SEM) using an OXFORD device (Launch Trion XL ilution refrigerator) (Figure 2).

Элемент	Весовой %	Атомный%
S K	20.13	47.56
Cd L	53.35	35.95
Sb L	26.52	16.50
Итоги	100.00	

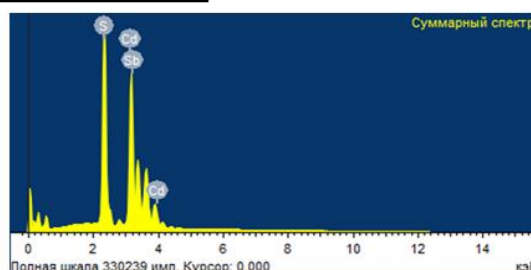
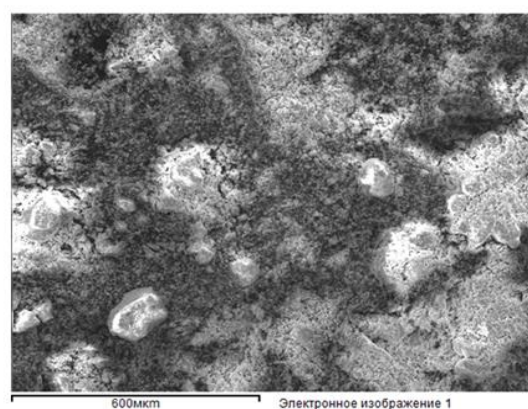


Figure 2
*Elemental composition, energy dispersive spectrum,
and microscope of cadmium–antimony sulfide*

The results obtained from the figure reflect both the chemical composition and morphological properties of the $\text{Cd}_3\text{Sb}_2\text{S}_8$ compound. Based on the scanning electron microscopy (SEM) (Imanov, 2020) analysis, it is determined that the surface of the material is mainly composed of small-sized

crystals and particles. The size of the particles varies in the range of approximately 5 μm , which indicates that the sample has a relatively homogeneous structural structure. The energy dispersive spectroscopy (EDS) results reveal that the elemental composition is consistent with the theoretical composition in terms of both mass and atomic percentages. Peaks belonging to the elements cadmium (Cd), antimony (Sb) and sulfur (S) are clearly observed in the spectra and they are mainly located in the energy range of 2.3–4 keV. The combined results of SEM and EDS analyses confirm both the morphological integrity and chemical correctness of the synthesized compound. These properties indicate that the $\text{Cd}_3\text{Sb}_2\text{S}_8$ material is a promising candidate for optoelectronic and thermoelectric applications.

Based on the 5 μm scale bar in the image, the particle size can be approximately estimated as follows:

The smallest distinguishable particles are in the range of 5–15 μm .

The majority of particles fall within 20–60 μm .

Larger agglomerates are observed in the range of 80–200 μm .

Occasional large structures reach approximately 200–300 μm .

Size (μm) Estimated fraction	
0–20	15–20%
20–50	40–50%
50–100	20–30%
100–200	10–15%
>200	<5%

Accordingly, based on this SEM image, the average particle size is estimated to be approximately 40–70 μm .

2.6 XRD: crystal structure and phase identification

X-ray diffraction was used to study the crystal structure and phase composition of the synthesized sample. The diffraction results showed that crystallinity was formed in the compound and the observed main peaks were consistent with standard parameters (Figure 3).

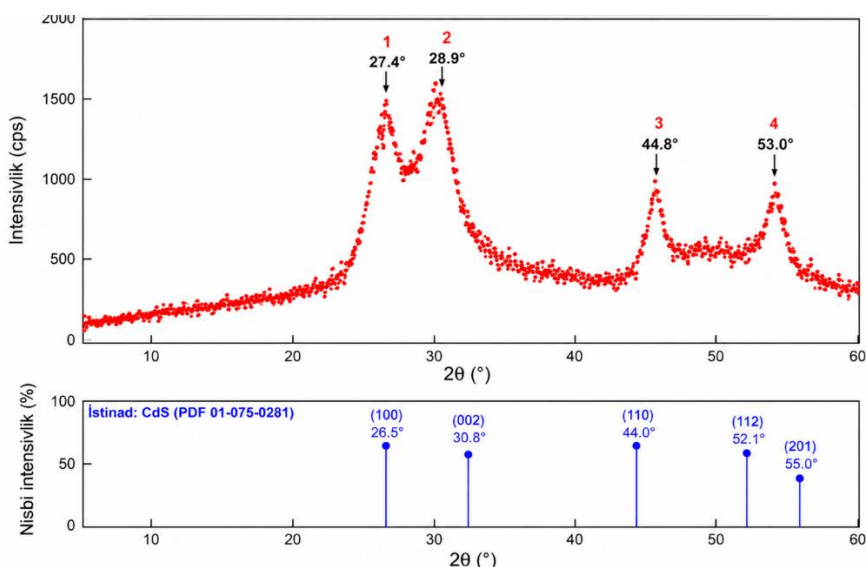


Figure 3
X-ray phase analysis of cadmium-antimony-sulfide

The presented diffractogram reflects the results of X-ray diffraction (XRD) (Mamedova, 2016; Mamedova, 2020; Mamedova, 2020) of the $\text{Cd}_3\text{Sb}_2\text{S}_8$ compound. The X-ray diffraction (XRD) pattern presented in Figure 3 was used to evaluate the crystal structure of the synthesized sample. Four prominent diffraction peaks were observed at approximately 27.4° , 28.9° , 44.8° , and 53.0° in the 2θ range. The most intense reflection appeared at 28.9° , indicating a preferred crystallographic orientation within the material. A strong peak was also detected at 27.4° , confirming the formation of a crystalline phase. The reflections at 44.8° and 53.0° correspond to characteristic lattice planes of the crystal structure. Comparison with the standard CdS diffraction data (PDF 01-075-0281) revealed good agreement between the experimental and reference peak positions. The standard pattern exhibits major reflections at 26.5° , 30.8° , 44.0° , 52.1° , and 55.0° . Slight shifts in the diffraction angles may be attributed to lattice strain, crystallite size effects, or structural defects. The relatively broad diffraction peaks suggest the formation of nanocrystalline domains. Furthermore, the elevated background intensity indicates the possible presence of a minor amorphous component. The well-defined diffraction peaks demonstrate a satisfactory degree of crystallinity. No significant impurity peaks were detected in the measured range, suggesting high phase purity of the synthesized material. Variations in peak intensity reflect differences in the diffraction contribution of individual crystal planes. The reflections near 44.8° and 53.0° closely match those reported for CdS-based materials. The observed peak positions and intensities confirm the successful formation of a CdS crystalline phase. Minor deviations from the standard pattern can be associated with synthesis conditions and crystal imperfections. Overall, the XRD analysis verifies that the sample possesses a predominantly crystalline CdS structure with good structural quality and phase stability (Gilshtein, 2025).

We calculated the crystallite size based on the Scherrer equation and presented it as follows:

The average crystallite size was evaluated using the Scherrer equation:

$$D = K\lambda / (\beta \cos\theta)$$

where K is the shape factor (0.9), λ represents the Cu $K\alpha$ radiation wavelength (0.15406 nm), β is the full width at half maximum (FWHM), and θ is the Bragg diffraction angle.

Using the most intense diffraction peak at approximately $2\theta \approx 28.9^\circ$ and an estimated FWHM of 2.0 – 2.5° , the crystallite size was determined to be roughly in the range of 10–25 nm. This value corresponds to the size of coherent diffracting domains rather than the overall particle dimensions observed in SEM images.

The difference between SEM results (showing micrometer-sized agglomerates) and XRD-derived crystallite sizes suggests that the material is composed of nanoscale crystallites that are aggregated into larger secondary particles.

The amorphous phase is estimated to be approximately $50 \pm 10\%$.

Overall, the results obtained show that the synthesized $\text{Cd}_3\text{Sb}_2\text{S}_8$ has a predominantly crystalline structure, but the presence of some non-crystalline (amorphous) regions in the structure cannot be ruled out. (Mamedova, 2021; Mamedova, 2021; Mamedova, 2021).

3 Result

3.1. Thermal analysis

Thermal analysis demonstrates that $\text{Cd}_3\text{Sb}_2\text{S}_8$ exhibits stability up to approximately 800°C. An intense endothermic effect observed in the range of 800–870°C is associated with melting behavior and subsequent structural rearrangement of the material. This response suggests a partial weakening of chemical bonding at elevated temperatures.

3.2. Morphology and composition

SEM observations reveal that the synthesized material is composed of aggregated microcrystalline particles with dimensions typically varying between 20 and 100 μm . Elemental mapping by EDS confirms a homogeneous distribution of Cd, Sb, and S throughout the sample, which is consistent with the expected stoichiometric composition of $\text{Cd}_3\text{Sb}_2\text{S}_8$.

3.3. Crystal structure

X-ray diffraction analysis verifies the successful formation of a well-crystallized $\text{Cd}_3\text{Sb}_2\text{S}_8$ phase. The main diffraction peaks located at 27.4° , 28.9° , 44.8° , and 53.0° are in good agreement with standard reference patterns (Yadav, 2011). The observed slight peak broadening indicates the presence of nanoscale crystallite domains together with a minor amorphous fraction, estimated at approximately $50 \pm 10\%$.

The average crystallite size calculated using the Scherrer approach falls within the range of 10–25 nm, confirming the coexistence of nanocrystalline domains within larger micrometer-scale aggregates (Ashok, 2020).

3.4. Role of ore-derived precursor

Utilization of Sb_2S_3 obtained from Darıdağ ore notably simplifies the synthesis route by eliminating the need for chemically synthesized precursors. This approach reduces both production cost and environmental burden. Importantly, the ore-derived Sb_2S_3 yields a phase purity comparable to that of conventional synthetic precursors, while contributing to a more sustainable and resource-efficient preparation strategy.

Discussion

The formation of $\text{Cd}_3\text{Sb}_2\text{S}_8$ in this work can be explained through a multi-stage solid-state reaction mechanism involving intermediate CdS formation and subsequent diffusion with Sb_2S_3 . At elevated temperatures, cadmium reacts readily with elemental sulfur, leading to the in situ generation of CdS as an active intermediate phase. This intermediate plays a key role in facilitating the incorporation of Sb and additional sulfur into the final ternary structure. The layered chain-like structure of Sb_2S_3 enhances atomic mobility, which supports solid-state diffusion processes during synthesis. The use of an argon atmosphere prevents oxidation reactions and helps maintain the chemical purity of the system. The X-ray diffraction analysis confirms that the synthesized material is mainly crystalline and corresponds to the $\text{Cd}_3\text{Sb}_2\text{S}_8$ phase (Ganiev 2026). The presence of broadened diffraction peaks indicates that the crystal domains are in the nanometer scale. Small variations in peak positions compared to standard patterns can be associated with internal stress, defects, and size-related effects developed during high-temperature synthesis. The simultaneous presence of ordered and slightly disordered regions suggests that the material has a partially crystalline structure. These structural characteristics are expected to affect the physical behavior of the compound. The scanning electron microscopy results show that the sample is composed of secondary particles formed by the aggregation of much smaller crystallites. This type of morphology is commonly observed in solid-state synthesized sulfide materials due to grain growth and particle coalescence at elevated temperatures. Energy-dispersive X-ray analysis confirms that cadmium, antimony, and sulfur are uniformly distributed throughout the sample. The absence of significant compositional fluctuations suggests that the reaction proceeded homogeneously across the material. When compared with similar Cd–S and Sb–S based chalcogenide systems reported in the literature, the present material demonstrates comparable structural behavior while benefiting from a naturally sourced Sb_2S_3 precursor. The use of Darıdağ ore reduces reliance on chemically synthesized reagents and contributes to a more sustainable synthesis strategy. This approach also simplifies the overall

preparation route and lowers material cost. The results confirm that ore-derived precursors can be effectively used in the fabrication of complex chalcogenide phases.

Conclusion

A new and sustainable synthesis pathway for $\text{Cd}_3\text{Sb}_2\text{S}_8$ has been established through the use of Sb_2S_3 obtained from Darıdağ ore. The resulting product shows a largely crystalline nature with nanometer-scale crystallite domains and demonstrates satisfactory thermal stability. These findings indicate that Sb_2S_3 derived from natural ore can serve as a viable substitute for conventionally prepared synthetic precursors in chalcogenide synthesis, enabling a more economical and environmentally benign production strategy.

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