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Comprehensive Investigation of the Physicochemical Properties of the $CdSb_2S_4$ Compound (Chemical and X-Ray Phase Analysis, Precipitation Kinetics)

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Abstract. *In this work, the physicochemical properties of the cadmium antimony sulfide compound $CdSb_2S_4$ in an aqueous environment were studied. To synthesize cadmium antimony sulfide, local raw materials were first used as the starting material. Antimony (III) sulfide was obtained from the Daridag antimony mine located in the Nakhchivan Autonomous Republic by filtration. Antimony (V) sulfide and cadmium sulfide were added to the obtained antimony (III) sulfide, and cadmium sulfide was synthesized. Cadmium antimony sulfide was X-ray diffraction (XRD) analysis of the obtained compound was performed. At the same time, the precipitation rate of the compound was calculated, the results of which are listed in the table. The effect of the amount of cadmium sulfide on the formation of cadmium antimony sulfide was studied and given in the table. The sample was chemically analyzed and its composition was determined. The application areas of cadmium antimony sulfide are defined, and brief information is provided (optoelectronics, thermoelectric materials, solar energy technologies, photocatalysis).*

Keywords: *cadmium sulfide, cadmium antimony sulfide, x-ray phase, precipitation, chemical analysis*

Introduction

A new thioantimonate(III) of the d10 transition metal $CdSb_2S_4$ was prepared by an environmentally friendly ionothermal method (Gang, 2021). The conditions for obtaining cadmium thiostibite from the interaction of cadmium acetate and antimony(III)chloride in an organic medium have been studied (Guliyev, 2017). A thin film of cadmium sulfide is deposited on a glass slide using a pyrolysis-deposition method at high temperature (400 ℃) using an aqueous solution of cadmium chloride and thiourea. The structural, optical and electrical properties of the deposited CdS thin film are investigated (Abhijit, 2011). To determine their physicochemical properties, measurements were made using spectrophotometer, X-ray diffraction (XRD), step height measurement device, digital four-point probe resistance measurement and scanning electron microscopy (SEM) (Chen, 2022).

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In the 21st century, the combination of heavy metals and sulfide-based semiconductor materials has attracted great interest due to its scientific and practical importance (Valiyeva, 2025). These antimony-based compounds are distinguished by their high optical absorption, wide band gap, and various physicochemical properties, and have wide application possibilities in optoelectronics, solar energy devices, and sensor technologies (Fatemipayam, 2025).

Methods

We used Daridag antimony ore as the starting raw material for the preparation of cadmium thiostibite compound (Garayev, 2017, 2022). Antimony (III) sulfide and then antimony (V) sulfide are synthesized from Daridag antimony ore (Garayev, 2016; Aliyeva, 2024). Cadmium thiostibite ($CdSb_2S_4$) is obtained from the interaction of antimony (V) sulfide and cadmium sulfide. The amount of cadmium sulfide added to antimony (V) sulfide is taken in a ratio of 1.2–1.4:1. The sample size varies between 0.17–0.182 mm, the ratio of antimony (V) sulfide to cadmium sulfide in the ore is 1:1.3 (Aliyeva, 2021; Aliyeva, 2022). The process proceeds at a temperature of 423–473 K with a solid-liquid phase ratio of 1:5–7. The completion time of the process is about 20–30 minutes, and the yield of antimony (V) sulfide is about 94.50%. Antimony (V) sulfide obtained from Daridag antimony ore is dissolved in HCl, and at this time the compound is purified from impurities. At about 430K, hydrogen sulfide (H_2S) is evaporated from antimony (V) sulfide. At this time, the solution changes color from dark brown to yellowish orange. Precipitation occurs and the precipitation of sulfide stops. The obtained antimony (V) sulfide is washed until chlorine and sulfide ions are purified, and finally washed again in 55ml of ethylene glycol. We dry it in vacuum at 467K until a constant mass is obtained. The preparation of cadmium thiostibite is as follows:

$$CdS + Sb_2S_5 \rightarrow CdSb_2S_4 + 2S$$

In this reaction, the substances taken are mixed, boiled, and water is added occasionally, and the boiling is continued until the dissolution process is complete. Finally, the solution is allowed to crystallize.

Results

The resulting grayish-yellow crystals are filtered through a Buchner funnel. If we take the amount of cadmium ions less than the equivalent amount, we will get a compound consisting of a mixture of cadmium sulfide and antimony (V) sulfide. This compound we get is not completely separated from the solution. In order for the pH to take a certain value, we must take the amount of antimony (V) sulfide according to the reaction. In this case, the reaction proceeds according to the exchange mechanism. It can be seen from the table below that the theoretical and experimental masses of the sample are close to each other (Sb_2S_3=20–35 ml). It can be seen from Table 1 that the process proceeds according to the exchange reaction.

Table 1
Effect of the amount of cadmium sulfide on the formation of cadmium thiostibit [Sb] $1{\cdot}10^{-1}$ M, [Cd] = $1{\cdot}10^{-1}$ M. Tem-r. 473K

No.	CdSb, ml	Sb_2S_5 ml	pH	$CdSb_2S_4$-teorik. mg	$CdSb_2S_4$-exsper. Mg
1	10.00	20	3-4	473.4	348.91
2	10.00	25	«__»	«__»	354.43
3	10.00	30	« »	« »	400.45
4	10.00	35	«__»	«__»	470.90

X-ray phase analysis of cadmium thiostibite obtained in aqueous medium (Mamedova, 2016; 2020; 2020; 2021; Mamedova & Nasirli, 2021; Mamedova, 2021) was performed (Fig. 1). The analysis results confirmed that the sample corresponds to a $CdSb_2S_4$ mixture.

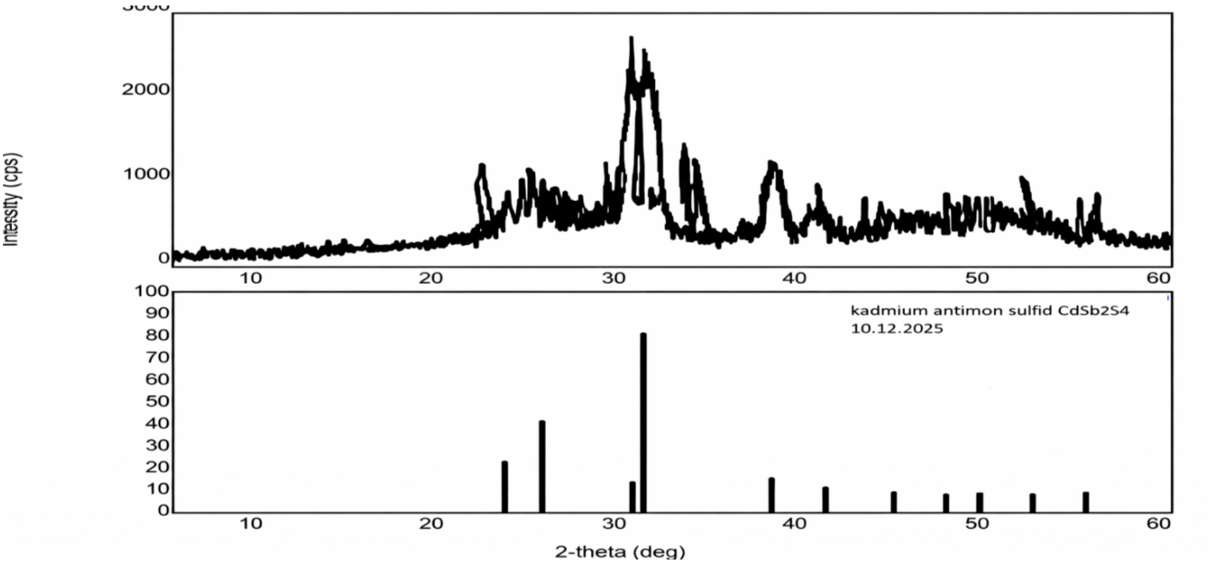

Figure 1. X-ray phase analysis of $CdSb_2S_4$

The strongest peak is observed around 2θ = 30°. This peak represents the main diffraction peak of the $CdSb_2S_4$ crystal. Other peaks are located around 2θ ≈ 27°, 38°, 44°, 48° and 57°. The position of the peaks corresponds to the crystal structure of $CdSb_2S_4$. The intensity of the peaks in the spectrum provides extensive information about the orientation and size of the crystal. The main phase is $CdSb_2S_4$ and there are no additional phases. This indicates that the sample has a tetragonal or orthorhombic structure. Based on the XRD analysis, we can say that the sample has a high crystallinity. Comparison with standard peaks confirms the accuracy of the experimental result. Such $CdSb_2S_4$ materials are promising for photovoltaic and semiconductor applications.

Application field: Cadmium thiostibite has a number of application fields. Table 2 lists the properties, advantages, and uses of cadmium thiostibite (Han, 2022; Zhou, 2013; Yang, 2024).

Table 2
Application area of $CdSb_2S_4$

Application	Area Properties of $CdSb_2S_4$	Advantages / Uses
Optoelectronics	High optical absorption, electron-hole pair separation efficiency	Photodiodes, infrared detectors, improve the performance of optoelectronic devices
Thermoelectric materials	High electronic conductivity, low thermal conductivity	Thermal energy conversion to electricity, energy saving, heat recovery applications
Solar energy technologies	Narrow band gap, photosensitizer property	Buffer or active layer in Sb_2S_3-based solar cells, improves photon absorption and electron-hole separation
Photocatalysis	Catalytic activity under UV and visible light, nanostructured forms	Accelerates water splitting, degradation of organic pollutants, catalysis processes

Discussion

In this study, the synthesis and physicochemical properties of the $CdSb_2S_4$ compound in an aqueous medium were comprehensively investigated. It was determined that $CdSb_2S_4$ can be synthesized with a high yield through the interaction of Sb_2S_5 and CdS obtained from the Daridag ore. The results of X-ray phase analysis confirmed that the sample possesses high crystallinity and that the main phase corresponds to $CdSb_2S_4$. It was observed that increasing the amount of cadmium sulfide leads to a closer agreement between theoretical and experimental results. Chemical analyses verified the stoichiometric composition of the compound and confirmed its formula as $CdSb_2S_4$. The obtained results indicate that $CdSb_2S_4$ is a promising semiconductor material with potential applications in optoelectronics and solar energy technologies.

Conclusion

The effect of the amount of cadmium sulfide on the formation of cadmium thiostibite When increasing the equivalent amount of cadmium Sb_2S_5 at a constant pH value, it was clearly seen that the theoretical and experimental masses overlapped. Chemical analysis confirmed the stoichiometric composition of cadmium thiostibite, and at the same time, its compliance with the formula $CdSb_2S_4$ was determined. The precipitation rate of cadmium thiostibite was calculated and the precipitation time of the reaction was determined. The specificity of the compound was confirmed by X-ray phase analysis.

Declaration of Competing Interests

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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