

Original Research Paper

STUDYING OF OBTAINING CONDITIONS OF BISMUTH SELENIDE IN THE WATER

Aliye Rzayeva^{1,2}

<https://orcid.org/0000-0001-7435-431X>

¹Nakhchivan State University, Nakhchivan, Azerbaijan

²Institute of Natural Resources, Nakhchivan, Azerbaijan

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*Corresponding Author: Aliye

Rzayeva, Nakhchivan State

University, Institute of Natural

Resources

Email: aliyerzayeva@ndu.edu.az

Abstract

This study investigates the conditions for synthesizing bismuth(III) selenide (Bi_2Se_3) from bismuth(III) oxide (Bi_2O_3) and elemental selenium in an aqueous medium using hydrazine monohydrate as a reducing agent. The effects of reagent quantity, reaction temperature, and reaction conditions on the formation and composition of the product were examined. The synthesis was performed by heating a mixture of bismuth(III) oxide, selenium powder, and diluted hydrazine monohydrate solution to the boiling point. The formation of a black precipitate indicated the production of bismuth selenide. The experimental results demonstrated that the amount of reducing agent significantly influenced the completeness of the reaction and the purity of the final product. Under suitable conditions, the obtained precipitate was identified as Bi_2Se_3 . The synthesized material was characterized using chemical analysis, thermogravimetric analysis, differential thermal analysis, and X-ray phase analysis. The X-ray diffraction results confirmed the crystalline nature of the product and its correspondence to the reference diffraction pattern of Bi_2Se_3 . Thermal analysis revealed changes in sample mass associated with oxidation and the subsequent volatilization of selenium oxide at elevated temperatures. The melting temperature was determined to be approximately 978 K, while the density measured by the pycnometric method was 6.78 g/cm³. The results demonstrate that bismuth(III) selenide can be obtained in an aqueous medium at relatively low temperatures. This approach may provide a useful alternative to high-temperature synthesis methods and contribute to the preparation of bismuth selenide for potential applications in semiconductor technology, thermoelectric devices, and thin-film production.

Keywords: *sedimentation, water environment, bismuth nitrate pentahydrate, x-ray analysis, chemical analysis*

INTRODUCTION

Currently, chalcogenide crystals—compounds of metals with S, Se, and Te, as well as compounds involving P, As, Sb, and Bi—are being intensively researched. This is due to their semiconducting and luminescent properties, which are of significant interest to fundamental science.

Furthermore, these materials are characterized as superconductors, magnets, topological insulators, catalysts, and other functional materials that hold broad potential for practical application.

A number of methods are used to obtain chalcogenide crystals, including growth from a molten solution, the chemical vapor transport method, direct synthesis in an ampoule, crystal growth from aqueous solutions, and hydrothermal synthesis. Since crystal growth from aqueous solutions and hydrothermal synthesis do not require high temperatures, complex equipment, or specialized conditions, we have chosen to employ these methods in our work.

In the synthesis of selenides in solution, a number of selenium compounds are used (sodium selenide, sodium selenosulfate, sodium selenite, potassium selenocyanate, hydrogen selenide, and elemental selenium).

Elemental selenium dissolves in a concentrated sodium sulfite solution to form sodium selenosulfate (Perov, 2002). Selenium dissolves in potassium cyanide to form potassium selenocyanate. The resulting white, needle-like, hygroscopic crystals are highly soluble in water, alcohol, and acetone (Waters, 2004). Dissolving elemental selenium in concentrated nitric acid yields selenious acid. We have established that elemental selenium dissolves in sodium borohydride and hydrazine monohydrate under ambient conditions (295 K) (Chemistry handbook, Jamafarli 2024). A method has been developed for the synthesis of metal selenides by reacting amorphous selenium with metal salts in a non-aqueous medium (using organic solvents, such as C_nH_{2n+2} hydrocarbons) at temperatures of 150–200°C for 4–8 hours, with a metal-to-selenium ratio of 1:(1–4) (Sharma, 2020).

The authors investigated the conditions for synthesizing bismuth selenide via the hydrothermal method. For the synthesis of Bi_2Se_3 , 1 mol of pure bismuth nitrate pentahydrate $[Bi(NO_3)_3 \cdot 5H_2O]$ and 1.5 mmol of selenium powder (in a 2:3 molar ratio) were placed into a Teflon liner. To control the pH of the medium, 4 mmol of sodium hydroxide and 2 mmol of ethylenediaminetetraacetic acid (EDTA) were added. The Teflon liner was filled with DMF to 80% of its total volume. The mixture was stirred in an ultrasonic homogenizer for 30 minutes to ensure uniformity, then sealed tightly and placed into an autoclave. The autoclave was maintained at temperatures of 140°C and 200°C for 24 hours. Following synthesis, the autoclave was allowed to cool to room temperature. The resulting black precipitate was filtered, washed several times with ethanol and distilled water, and dried under vacuum at 100°C for 4 hours to obtain the sample in powder form. The samples prepared at 140°C and 200°C for 24 hours were designated as Bi_2Se_3 -1 and Bi_2Se_3 -2, respectively. The thermoelectric properties of the materials were measured (Venkatasubramanian, 2001).



Semiconductors with high carrier mobility are leading candidates for thermoelectric materials. Bismuth(III) selenide is a semiconductor (Kadel, 2011) utilized in optical recording systems, photoelectrochemical devices, and thermoelectric devices (Rzayeva, 2026). In recent years, interest in studying various properties of bismuth chalcogenides at room temperature has increased significantly (Rowe, 1995). Since crystal growth from aqueous solutions and hydrothermal synthesis methods do not require high temperatures, complex equipment, or specialized conditions, we have chosen to employ these techniques in our work. In this context, the conditions for the conversion of bismuth oxide into bismuth selenide in an aqueous medium have been investigated for the first time.

MATERIALS AND METHODS

A specific amount of bismuth oxide is combined with a corresponding quantity of elemental selenium powder, maintaining a stoichiometric ratio. Subsequently, a specific volume of a hydrazine monohydrate solution of a known concentration is added to the mixture, which is then heated. As the solution reaches its boiling point, the yellowish Bi_2O_3 gradually transforms into bismuth selenide. A decrease in the amount of selenium in the solution is observed (as selenium dissolves in the hydrazine monohydrate). Ultimately, the formation of black-colored Bi_2Se_3 is confirmed.

The experiments were conducted quantitatively as follows: 46.6 mg of Bi_2O_3 is mixed with 23.7 mg of elemental selenium, and 5 ml of a hydrazine monohydrate solution (diluted in a 1:4 ratio) is added. The mixture is heated until it boils. At the end of the process, 64.1 mg of Bi_2Se_3 is obtained (fig.1). This corresponds exactly to the theoretical amount (Rzayeva, 2024).

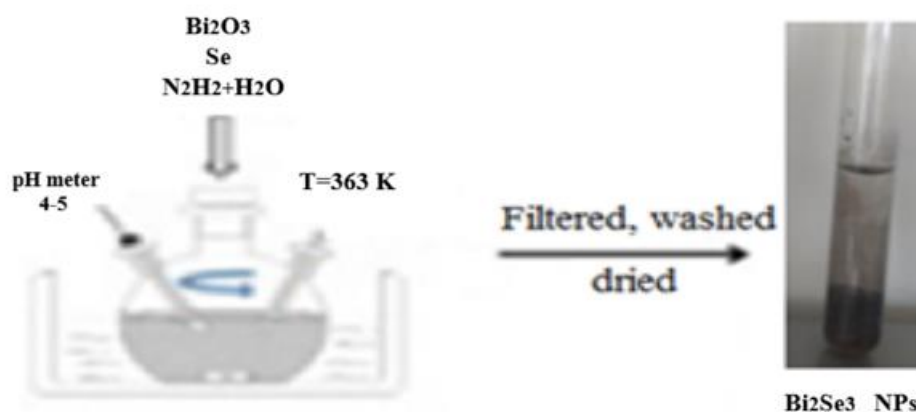
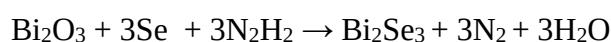


Figure.1.Obtaining of Bi_2Se_3

The reaction equation for the transformation process can be represented as follows.



RESULTS AND DISCUSSION

In this process, a black, crystalline precipitate is formed. The analysis of the precipitate for bismuth and selenium confirmed that both elements are incorporated into its composition. Since hydrazine monohydrate possesses strongly basic properties, even its diluted solution creates a basic environment; consequently, the reaction proceeds in a basic medium. Given that hydrazine monohydrate acts as the primary reducing agent in the reaction, the influence of the amount of this reducing agent ($\text{N}_2\text{H}_4 \cdot \text{H}_2\text{O}$) on the formation of the compound was investigated, and the results are presented in table 1.

Table 1. Dependence of bismuth selenide formation on the amount of hydrazine monohydrate in the medium $\text{C}_{\text{N}_2\text{H}_4 \cdot \text{H}_2\text{O}} = 10\%$, temp. 363-373 K

№	Bi_2O_3 mg	Se, mg	$\text{N}_2\text{H}_4 \cdot \text{H}_2\text{O}$, ml	Theoretical mass of the precipitate (mg)	Experimental mass of the precipitate (mg)	The formula for the precipitate
1	93.2	47.4	2+5ml water	131.0	135.41	$\text{Bi}_2\text{Se}_3 + \text{Bi}_2\text{O}_3$
2	-	-	3+5ml water	-	132.22	Bi_2Se_3
3	-	-	5+5ml water	-	132.06	Bi_2Se_3
4	-	-	7+5ml water	-	131.50	Bi_2Se_3
5	-	-	10+5ml water	-	131.25	Bi_2Se_3

As indicated by the figures in the table, in the first experiment, selenium failed to completely convert the bismuth oxide; in other words, the amount of hydrazine solution was insufficient. In subsequent experiments, the quantity of hydrazine solution proved adequate, and the experimental values were very close to the theoretical values. In the final two experiments, although the masses of the precipitates were identical, the precipitate proved difficult to separate from the solution. The precipitate obtained in the second and third experiments consisted of Bi_2Se_3 . The precipitates were filtered, thoroughly washed with distilled water, and dried at a temperature of 383 K. The influence of temperature on the process was investigated; it was determined that the reaction rate is very low at room temperature but increases with rising temperature, with the process reaching completion within a few minutes at the boiling point of the solution.



The bismuth(III) selenide obtained in an aqueous medium was subjected to thermal analysis in the presence of air using a derivatograph (NETZSCH STA 449F3). The results are presented in Figure 1.

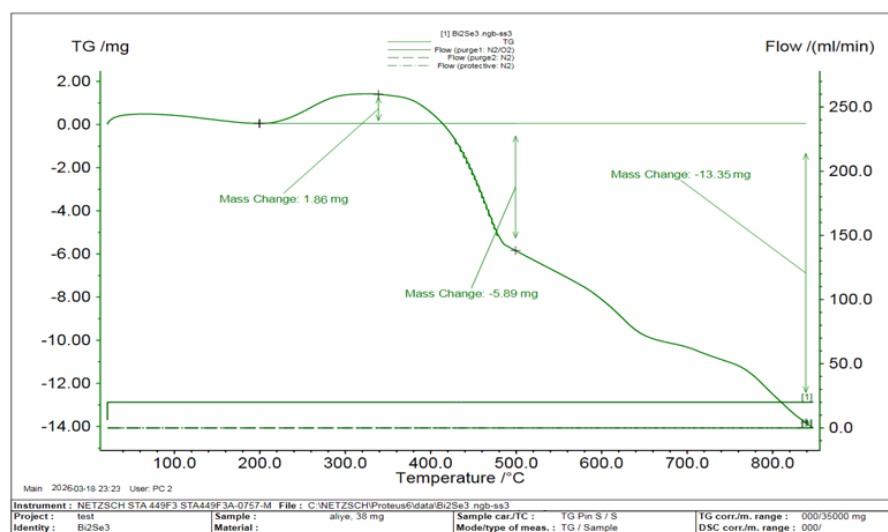


Figure. 2. The thermal analysis of Bi_2Se_3

As shown in Figure 1, no compositional changes occur in a 38 mg sample of Bi_2Se_3 (Bi: 24.25 mg, Se: 13.75 mg) at temperatures up to 200°C. In the 200–300°C temperature range, a mass increase of 1.35 mg is observed, which is attributed to the formation of selenium oxide. A mass loss of 13.86 mg occurs between 300°C and 850°C; this is due to the sublimation of the selenium oxide formed following the oxidation of bismuth(III) selenide. The remaining residue consists of bismuth (24.14 mg). Calculations indicate that the mass ratio of bismuth to selenium (Bi:Se) is 90.65:49.98. These findings confirm that the formula for bismuth selenide corresponds to Bi_2Se_3 .

The melting point of the compound was determined using an NTR-73 pyrometer, and the results are presented in Figure 2.

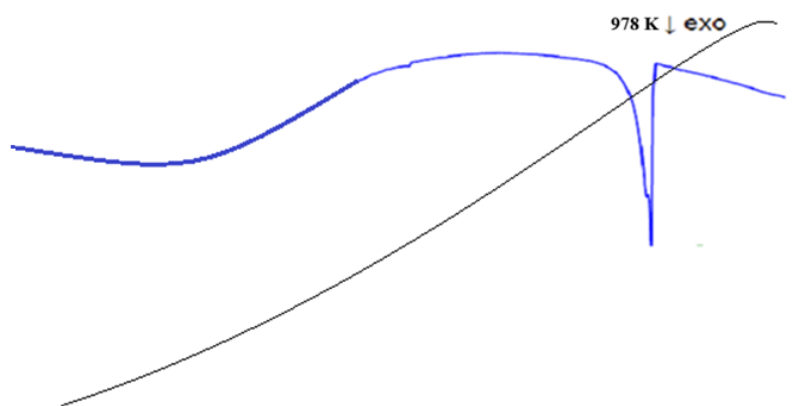


Figure. 4. The DTA of Bi_2Se_3

The analysis results indicate that the melting temperature of the sample is 978 K. Furthermore, X-ray phase analysis of the compound confirmed that it is Bi_2Se_3 (Fig. 5).

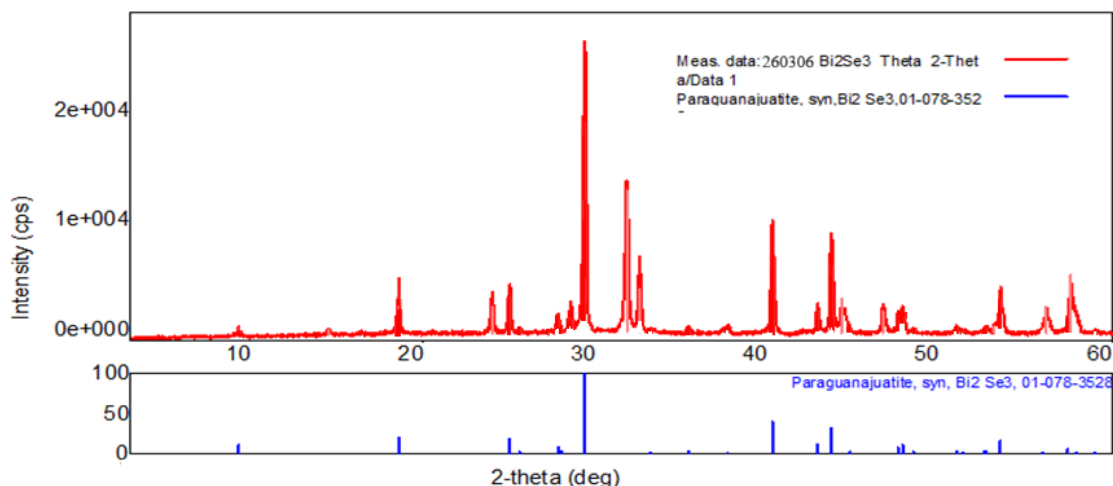


Figure. 5. X-ray of Bi_2Se_3

As can be seen from the figure, the positions and intensities of the peaks correspond to the standard, indicating the unique identity of the substance (JCPDS 01-078-3528) and confirming that it possesses a crystalline structure.

The chemical analysis of the compound was conducted as follows. The sample is decomposed using nitric acid, and the mixture is evaporated until a residue of salts remains. It is then transferred to a volumetric flask, with the addition of distilled water and a few drops of hydrochloric acid. Bismuth is precipitated using ammonia in the form of bismuth hydroxide. The resulting precipitate is filtered, washed with distilled water, and ignited in a muffle furnace at 350°C . The resulting Bi_2O_3 is weighed to determine its mass. Selenite ions present in the filtrate are determined using the hydroxylamine method. The obtained results are presented in Table 2.

Table 2. Chemical analysis of bismuth(III) selenide sample

Bi_2Se_3 Sample, g	Components, g			
	Bismuth		Selenide	
	Theoretic.	Pract.	Theoretic.	Pract.
0.6552	0.4180	0.4011	0.2371	0.2209

Note: the average value of the results of four experiments.

The results indicate that the values obtained experimentally align with theoretical calculations. This demonstrates that the composition of the synthesized bismuth(III) selenide corresponds to the

formula Bi_2Se_3 . Furthermore, the density of the bismuth(III) selenide was determined using the pycnometric method (6.78 g/cm^3).

Thus, the transformation of bismuth(III) oxide into bismuth(III) selenide was carried out for the first time in a solution (aqueous medium) at a temperature of $80\text{--}90^\circ\text{C}$, and its composition was confirmed by physicochemical analysis to correspond to the formula Bi_2Se_3 . These findings suggest that bismuth(III) selenide could be utilized as a chemical reagent, a semiconductor material, or a raw material for the production of thin films.

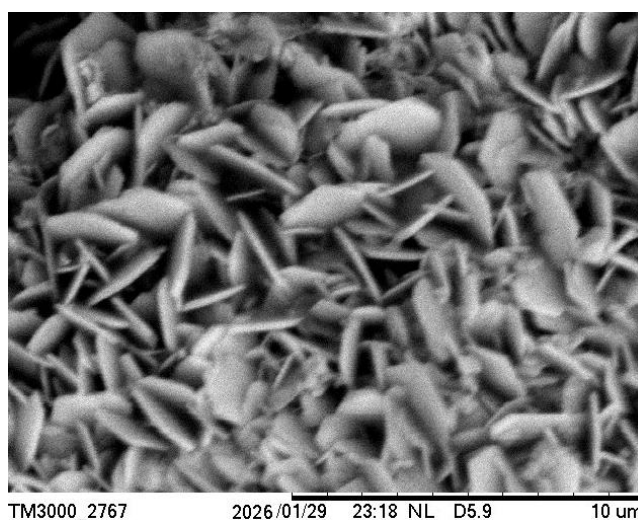


Figure 6. In water medium at the during 1 hours 60°C

The crystallinity of the alloys was controlled by differential thermal analysis with an HTP-70 pyrometer with a Thermoskan-2 device.

CONCLUSION

Chemical analysis, thermogravimetric analysis, differential thermal analysis, and X-ray phase analysis were used to investigate the composition and physicochemical properties of the synthesized material analysis. The X-ray diffraction pattern was consistent with the reference data for crystalline Bi_2Se_3 . Chemical also supported the formation of bismuth selenide, although some differences between theoretical and experimental elemental contents were observed. The measured density was 6.78 g/cm^3 , and the thermal analysis indicated a characteristic melting temperature of approximately 978 K .

Overall, the findings demonstrate the feasibility of preparing Bi_2Se_3 through a comparatively low-temperature solution-based process. The method may offer a practical alternative to conventional synthesis techniques that require more demanding thermal conditions. Further studies should optimize the reagent ratios, investigate the reaction mechanism, and evaluate the morphology, particle



size, phase purity, and thermoelectric properties of the synthesized product. These investigations would help establish the suitability of the material for semiconductor applications and the fabrication of functional thin films.

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XÜLASƏ

SU MÜHİTİNDƏ BİSMUT SELENİDİN ALINMA ŞƏRATİNİN ÖYRƏNİLMƏSİ

Aliyə Rzayeva^{1,2}

¹Naxçıvan Dövlət Universiteti, Naxçıvan, Azərbaycan

²Təbii Sərvətlər İnstitutu, Naxçıvan, Azərbaycan

Bu tədqiqatda hidrazin monohidratın reduksiyaedici agent kimi istifadə edildiyi sulu mühitdə, bismut(III) oksid (Bi_2O_3) və elementar selenə bismut(III) selenidin (Bi_2Se_3) sintezi şərtləri araşdırılmışdır. Reagentlərin miqdarının, reaksiya temperaturunun və reaksiya şərtlərinin məhsulun əmələ gəlməsinə və tərkibinə təsiri öyrənilmişdir. Sintez prosesi bismut(III) oksid, selen tozu və durulaşdırılmış hidrazin monohidrat məhlulundan ibarət qarışığın qaynama temperaturuna qədər qızdırılması yolu ilə həyata keçirilmişdir. Qara çöküntünün əmələ gəlməsi bismut selenidin alındığını



göstərmişdir. Eksperimental nəticələr göstərmişdir ki, reduksiyaedici agentin miqdarı reaksiyanın tamlığına və son məhsulun təmizliyinə əhəmiyyətli dərəcədə təsir edir. Müvafiq şərtlər daxilində alınan çöküntünün Bi_2Se_3 olduğu müəyyən edilmişdir. Sintez olunmuş material kimyəvi analiz, termoqrammetrik analiz, differensial termik analiz və rentgen-faza analizi üsulları ilə xarakterizə edilmişdir. Rentgen difraksiyası nəticələri məhsulun kristal quruluşa malik olduğunu və Bi_2Se_3 üçün mövcud olan standart difraksiya mənzərəsinə uyğun gəldiyini təsdiqləmişdir. Termik analiz, yüksək temperaturalarda oksidləşmə və ardınca selen oksidinin uçması ilə əlaqədar nümunə kütləsində baş verən dəyişiklikləri aşkar etmişdir. Ərimə temperaturu təxminən 978 K, piknometrik üsulla ölçülmüş sıxlıq isə $6,78 \text{ q/sm}^3$ olaraq müəyyən edilmişdir. Əldə olunan nəticələr göstərir ki, bismut(III) selenidi sulu mühitdə, nisbətən aşağı temperaturlarda almaq mümkündür. Bu yanaşma yüksək temperaturlu sintez üsullarına faydalı bir alternativ ola bilər və yarımkeçirici texnologiyalar, termoelektrik qurğular və nazik təbəqələrin istehsalı kimi sahələrdə tətbiq üçün bismut selenidin hazırlanmasına töhfə verə bilər.

Açar sözlər: *çökdürmə, su mühiti, bismut nitrat pentahidrat, rentgen analizi, kimyəvi analiz*

