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Investigation of nitric acid dissolution of stibnite in the presence of tartaric acid

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Abstract: This study explores the nitric acid leaching of stibnite in the presence of tartaric acid, which acts as a complexing agent. The proposed approach is of considerable interest, as antimony is widely used across industries, from electronics to alloying applications. Thermodynamic analysis showed that nitric acid dissolution of stibnite inevitably leads to the formation of antimony oxides, which markedly reduces the extraction of the target metal into solution. To counteract these losses and enhance process efficiency, tartaric acid was introduced as an additive. The results demonstrated that tartaric acid promotes the formation of stable complexes with antimony ions, thereby retaining the metal in solution and minimizing the risk of oxide precipitation. Experimental design analysis revealed that the mass ratio of tartaric acid to antimony and the nitric acid concentration exert a stronger influence on leaching efficiency than temperature and leaching duration. Optimal conditions were established, achieving an antimony extraction of 87 %: temperature 35 °C, nitric acid concentration 5 mol/dm^3, leaching time 45 min, and a tartaric acid-to-antimony mass ratio of 4.5 : 1.0.

Keywords: stibnite, antimony, leaching, nitric acid, tartaric acid, optimal parameters.

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Исследование азотно-кислотного растворения стибнита с добавлением винной кислоты

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Аннотация: Работа посвящена изучению процесса азотно-кислотного выщелачивания стибнита с добавлением винной кислоты, выполняющей функцию комплексообразующего агента. Предлагаемый способ переработки стибнита обладает высокой актуальностью, поскольку сурьма широко используется в промышленности – от электроники до применения в качестве легирующих добавок. В ходе термодинамического анализа выявлено, что в процессе азотно-кислотного растворения стибнита неизбежно образуются оксиды сурьмы, что приводит к существенному снижению извлечения целевого металла в раствор. Для того чтобы снизить эти потери и повысить эффективность процесса, в качестве добавки предложено использовать винную кислоту. Исследования показали, что она способствует образованию стабильных комплексов с ионами сурьмы, что позволяет сохранить металл в растворе и минимизировать риск осаждения оксидов. С использованием математического планирования эксперимента установлено, что массовое соотношение винной кислоты к сурьме, а также концентрация азотной кислоты оказывают большее влияние на эффективность процесса выщелачивания, чем температура и продолжительность процесса. Определены оптимальные условия для достижения максимального значения извлечения сурьмы в раствор – 87 %: температура 35 °C, концентрация азотной кислоты 5 моль/дм^3, время выщелачивания 45 мин и массовое соотношение винной кислоты к сурьме 4,5 : 1,0.

Ключевые слова: стибнит, сурьма, выщелачивание, азотная кислота, винная кислота, оптимальные параметры.

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Introduction

Antimony, a critical element in various industrial applications, remains in high demand due to its unique physicochemical properties [1; 2]. It is used in the production of flame-retardant materials, lead—acid batteries, semiconductor devices, and as an alloying element in metallurgy. The primary source of antimony is the mineral stibnite (Sb2S3). However, efficient recovery of antimony from stibnite is complicated by a number of technological and environmental challenges [3—8], which has driven the search for innovative processing methods [9—14].

One promising approach to antimony extraction is nitric acid leaching [15—20]. Despite its advantages, this method has drawbacks such as slow reaction kinetics and the formation of sparingly soluble compounds. A potential strategy to improve acid leaching is the addition of organic complexing agents that stabilize metal ions in solution, thereby preventing their premature precipitation. In this regard, tartaric acid ($C_4H_6O_6$) is of particular interest [21; 22] Its molecules readily form stable complexes with metal cations, including antimony, which may enhance extraction efficiency and process selectivity.

Previous research has shown the successful application of organic acids in hydrometallurgical processes. For instance, citric and oxalic acids have been used to improve the leaching of oxide and sulfide ores of nickel, copper, and zinc [23—26]. However, in the case of antimony — particularly in combination with nitric acid — such studies remain limited. Most existing work has focused on single-agent systems or combinations of inorganic acids, whereas the synergistic effect of introducing tartaric acid into a HNO_3 medium has received little attention. This defines the relevance of the present study, which examines the effects of temperature, nitric acid concentration, leaching duration, and tartaric acid dosage on the efficiency of nitric acid dissolution of stibnite. The optimal conditions for maximum antimony recovery were also established.

The findings of this study may contribute to the development of more efficient and sustainable methods for processing antimony-bearing raw materials, thereby promoting more rational use of natural resources and reducing environmental impact.

Experimental procedure

Materials, equipment, and methods

The chemical composition of stibnite was determined by complete dissolution of a 0.2—0.3 g portion of the material in a PreeKem M3 microwave digestion system (PreeKem, China). The resulting solutions were analyzed using an EXPEC 6500 inductively coupled plasma optical emission spectrometer (ICP—OES) (Focused Photonics Inc., China), which was also used to determine the composition of the leach solutions.

The phase composition of the stibnite sample (Fig. 1) and the leach residue was analyzed with an XRD 7000 Maxima diffractometer (Shimadzu Corp., Tokyo, Japan).

A natural stibnite sample (Sb2S3) served as the starting material. The mineral was ground in a Pulverisette 6 planetary mill (FRITSCH GmbH, Germany) and sieved through laboratory screens to obtain a –56 μm working fraction. All other reagents were of analytical grade.

Nitric acid leaching experiments were carried out under laboratory conditions using a 500 mL Lenz Minni-60 reactor (Lenz Laborglas GmbH, Germany). The leaching solution was preheated to the de-

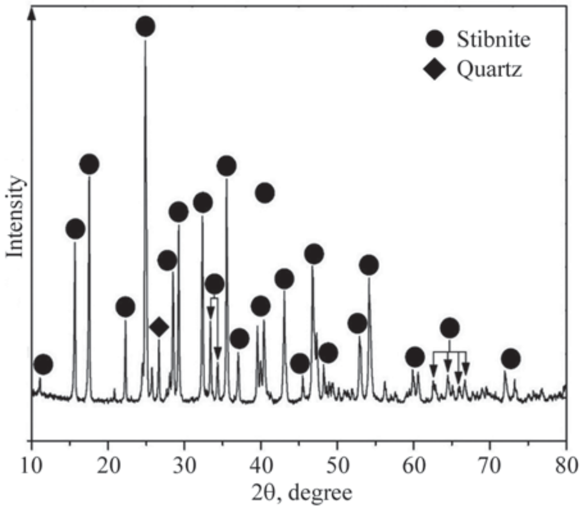

Fig. 1. Phase composition of stibnite

Рис. 1. Фазовый состав стибнита

sired temperature with a Huber Kiss K6 precision thermostat (Peter Huber Kältemaschinenbau, Germany). After thermostating, the weighed sample of stibnite was introduced into the reactor. Slurry homogenization was achieved using an IKA Eurostar 20 digital overhead stirrer (IKA-Werke GmbH, Germany).

After the reaction, the slurry was vacuum-filtered through a Büchner funnel. The filtrate was analyzed for antimony content by ICP—OES. The leach residue was sequentially washed with distilled water and then dried in a convection oven at 80 °C to constant weight.

Thermodynamic calculations were performed using HSC Chemistry Software v. 9.5 (Metso Outotec Finland Oy, Tampere, Finland).

Results and discussion

Thermodynamics of nitric acid leaching of stibnite

To establish the possibility of stibnite interacting with nitric acid solution, Gibbs free energy changes (ΔG) were calculated in the temperature range of 25—85 °C for the following reactions:

$$Sb_2S_3 + 6HNO_3 = Sb_2O_3 + 6NO_2 + 3S + 3H_2O, \quad (1)$$

$$Sb_2S_3 + 28HNO_3 = Sb_2O_5 + 3H_2SO_4 + \\ + 28NO_2 + 11H_2O, \quad (2)$$

$$Sb_2S_3 + 24HNO_3 = 2HSbO_2 + 3H_2SO_4 + \\ + 24NO_2 + 8H_2O, \quad (3)$$

$$2HSbO_2 + 4HNO_3 = Sb_2O_5 + 3H_2O + 4NO_2, \quad (4)$$

$$Sb_2O_3 + 4HNO_3 = Sb_2O_5 + 4NO_2 + 2H_2O. \quad (5)$$

As shown in Table 1, stibnite dissolution in nitric acid can proceed along several pathways, leading either to sparingly soluble antimony oxides (Sb_2O_3, Sb_2O_5) or to antimonous acid ($HSbO_2$) via reactions (1)—(3). Subsequently, both antimonous acid and antimony (III) oxide can be oxidized by nitric acid to antimony (V) oxide.

Pourbaix (Eh—pH) diagrams are a useful tool for predicting the thermodynamic stability of chemical species in aqueous media. To examine the behavior of antimony species at different pH and Eh values, an Eh—pH diagram was constructed for the S—Sb—H_2O system at 25 °C (Fig. 2, *a*). In addition, to more accurately predict the products of nitric acid leaching of stibnite, a distribution diagram was constructed (Fig. 2, *b*), showing the equilibrium concentrations of various antimony species as a function of nitric acid consumption.

The Pourbaix diagram (Fig. 2, *a*) indicates that in acidic media, dissolution of stibnite begins at a potential of about –0.4 V with the formation of antimonous acid ($HSbO_2$). A further increase in oxidation potential to approximately –0.61 V results in the conversion of $HSbO_2$ to Sb_2O_5, indicating more complete oxidation. These transformations are consistent with Gibbs free energy calculations for reactions (3) and (4).

The distribution diagram (Fig. 2, *b*), unlike the Pourbaix diagram, highlights the effect of nitric acid (HNO_3) consumption on the composition of stibnite dissolution products. At the initial stages, both $HSbO_2$ and Sb_2O_3 are formed. With nitric acid consumption exceeding 7 M,

Table 1. **Gibbs free energy change (ΔG) for reactions (1)–(5)**

Таблица 1. Результаты расчета изменения энергии Гиббса для реакций (1)–(5)

Reaction	ΔG, kJ/mol				
	25 °C	40 °C	55 °C	70 °C	85 °C
(1)	–344	–355	–367	–378	–389
(2)	–1630	–1683	–1735	–1787	–1839
(3)	–1411	–1455	–1498	–1541	–1583
(4)	–219	–228	–237	–246	–255
(5)	–168	–175	–182	–189	–196

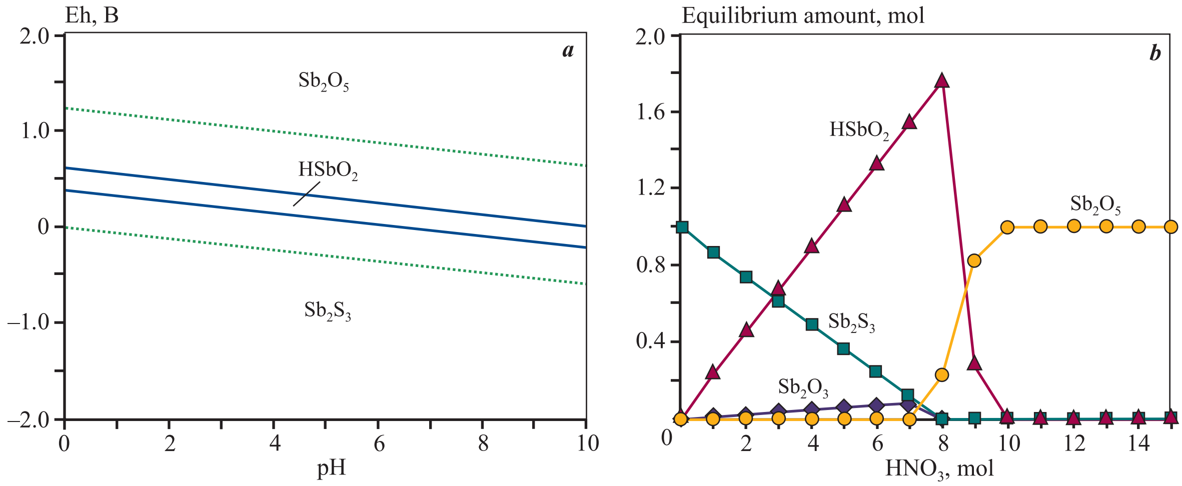

Fig. 2. Eh–pH diagram for the S–Sb–H_2O system (***a***), equilibrium distribution diagram of antimony species during nitric acid leaching of stibnite (***b***)

Рис. 2. Диаграмма Eh–pH для системы S–Sb–H_2O (***a***), диаграмма равновесного распределения соединений сурьмы при азотно-кислотном выщелачивании стибнита (***b***)

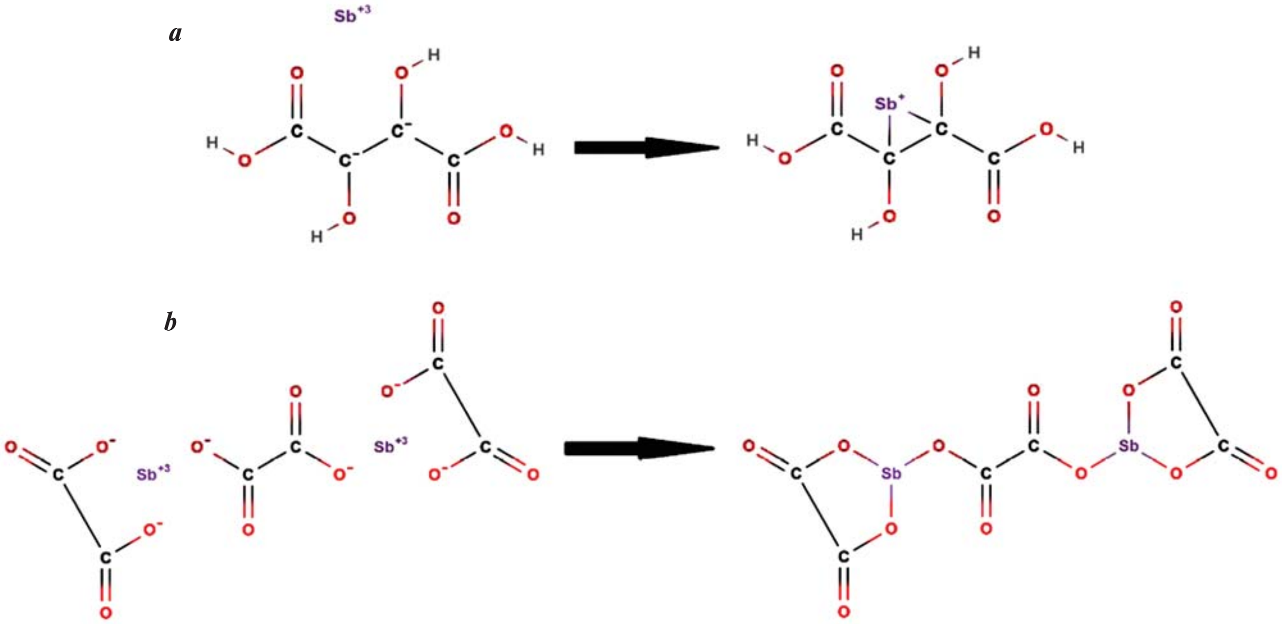

Fig. 3. Interaction schemes of antimony with tartaric acid (***a***) and oxalic acid (***b***)

Рис. 3. Схема взаимодействия сурьмы с винной (***a***) и щавелевой (***b***) кислотами

antimony is predominantly present as Sb_2O_5. This is attributed to the sharp increase in oxidation potential at high HNO_3 concentrations, which drives further oxidation of Sb(III) to Sb(V). At concentrations above 8 M, nearly complete oxidation of Sb_2O_3 and $HSbO_2$ to Sb_2O_5 is observed.

The thermodynamic analysis shows that nitric acid leaching of stibnite results primarily in the formation of antimony oxides, followed by their precipitation. To prevent antimony losses and improve leaching efficiency, it is therefore advisable to introduce additives that intensify the process and increase the degree of metal extraction. According to the literature [22], oxalic and tartaric acids are among the most effective complexing agents. They promote the formation of stable antimony complexes (Fig. 3), which helps retain the metal in solution and minimize oxide precipitation.

Investigation of nitric acid leaching and determination of optimal parameters

To evaluate the effect of tartaric acid on stibnite dissolution in nitric acid solution, experiments were conducted under the following conditions: nitric acid concentration — 6 mol/dm^3, liquid-to-solid ratio (L : S) = = 6 : 1, leaching duration — 60 min, temperature — 50 °C, and tartaric and oxalic acid concentrations — 50 g/dm^3 each.

As shown in Fig. 4, in the experiment without additives, antimony extraction into solution reached 44 % within the first 2 min, but subsequently decreased to 13 %. This effect is most likely associated with the transformation of antimony from antimonous acid ($HSbO_2$) into the insoluble oxide form (Sb_2O_5) according to reaction (4). The addition of tartaric and oxalic acids improved stibnite dissolution in nitric acid. Acting as complexing agents, they formed stable antimony complexes and prevented its conversion into insoluble oxide. However, tartaric acid achieved higher extraction (52 %) than oxalic acid (45 %), so further experiments were carried out with tartaric acid.

To determine the optimal parameters for nitric acid leaching of stibnite with tartaric acid, a statistical experimental design method was applied using the StatGraphics software package. A second-order orthogonal matrix was employed, comprising four variables: process temperature (35—85 °C), nitric acid concentration (1—9 mol/dm^3), leaching duration (10—70 min), and the tartaric acid-to-antimony mass ratio in stibnite (0.5÷4.5 : 1.0). The liquid-to-solid ratio (L : S) was maintained at 6 : 1 in all experiments.

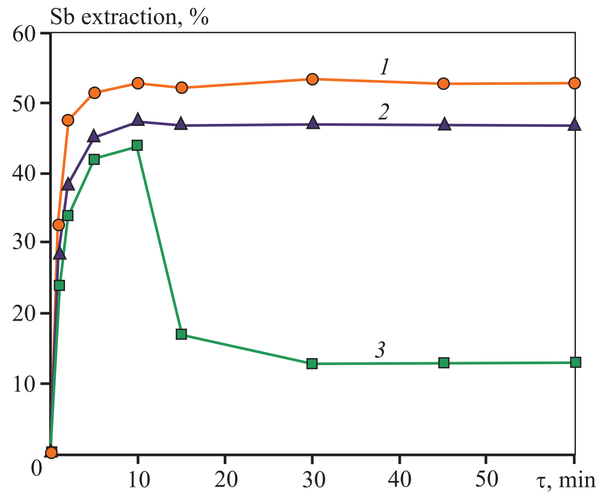

Fig. 4. Dependence of Sb extraction on time during nitric acid leaching with tartaric acid (*1*), oxalic acid (*2*), and without additives (*3*)

Рис. 4. Зависимость извлечения Sb от времени при азотно-кислотном выщелачивании с добавлением винной (*1*) и щавелевой (*2*) кислот, а также без добавок (*3*)

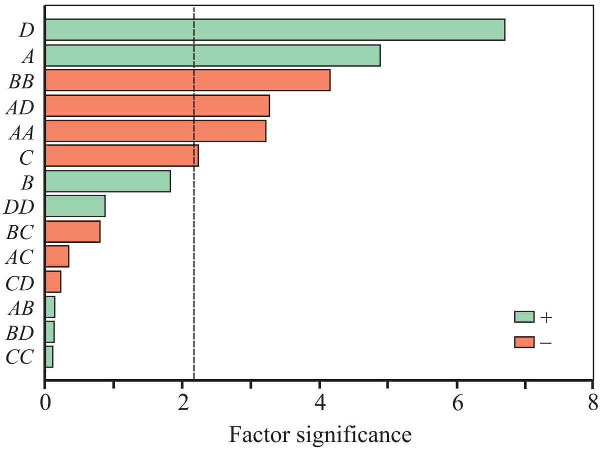

Fig. 5. Pareto chart for the parameters of nitric acid leaching of stibnite

D – tartaric acid-to-antimony ratio; *A* –nitric acid concentration; *C* – temperature; B – leaching duration

Рис. 5. Диаграмма Парето для изменяемых параметров азотно-кислотного выщелачивания стибнита

D – соотношение винной кислоты к сурьме; *A* – концентрация азотной кислоты; *C* – температура; *B* – продолжительность

Fig. 5 presents the Pareto chart for antimony leaching under the experimental conditions. The results indicate that the tartaric acid-to-antimony ratio and nitric acid concentration are the most statistically significant factors affecting antimony leaching, while leaching duration and temperature are less significant.

The dependence of antimony extraction on temperature, nitric acid concentration, leaching duration, and tartaric acid-to-antimony ratio is shown in Fig. 6.

As shown in Fig. 6, maximum antimony extraction from stibnite (>85 %) can be achieved under the following conditions: liquid-to-solid ratio — 6 : 1, temperature — 35 °C, nitric acid concentration — 5 mol/dm^3, leaching duration — 45 min, and tartaric acid-to-antimony mass ratio — 4.5 : 1.0. The regression equation for antimony leaching is

$$\begin{gathered}\mathrm{Sb} = -36.4551 + 13.2264A + 1.4411B - 0.0825433C + \\ + 0.609196D - 0.631207A^2 + 0.00311979AB - \\ - 0.0101319AC - 0.0722656AD - 0.0144331B^2 - \\ - 0.00313426BC + 0.000390972BD + \\ + 0.000639506C^2 - 0.00089537CD + 0.00306981D^2, \quad (6)\end{gathered}$$

where *A* — nitric acid concentration, mol/dm^3; *B* — leaching duration, min; *C* — temperature, °C; *D* — tar-

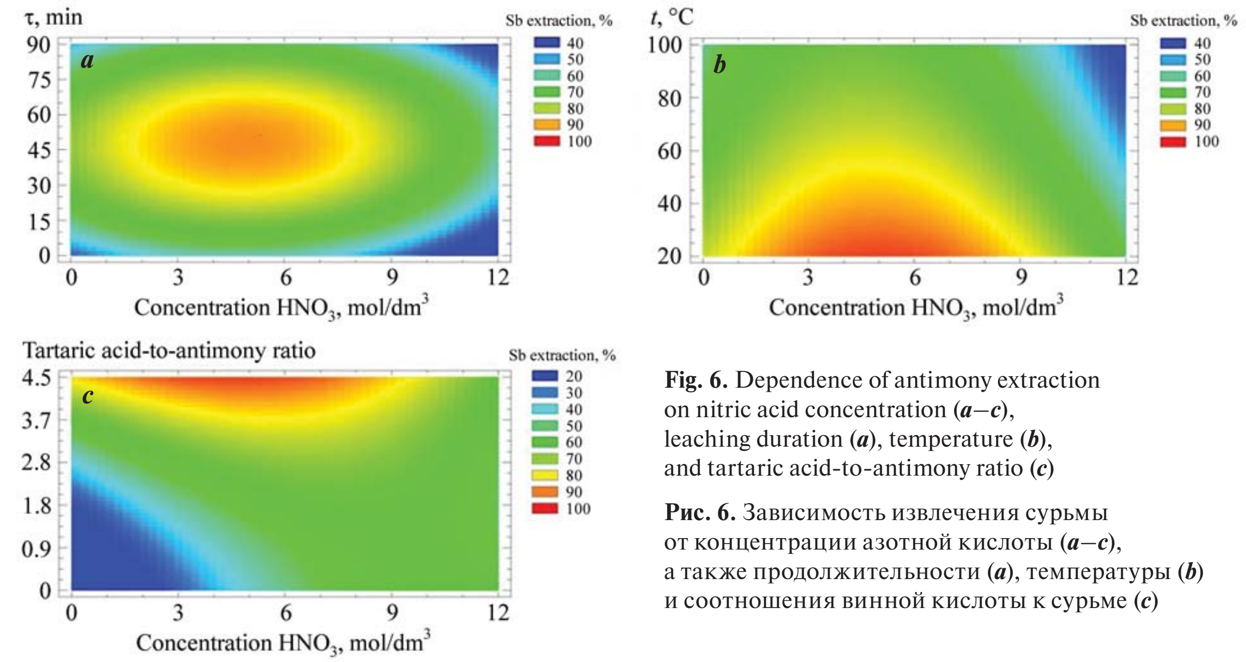

Fig. 6. Dependence of antimony extraction on nitric acid concentration (***a***–***c***), leaching duration (***a***), temperature (***b***), and tartaric acid-to-antimony ratio (***c***)

Рис. 6. Зависимость извлечения сурьмы от концентрации азотной кислоты (***a***–***c***), а также продолжительности (***a***), температуры (***b***) и соотношения винной кислоты к сурьме (***c***)

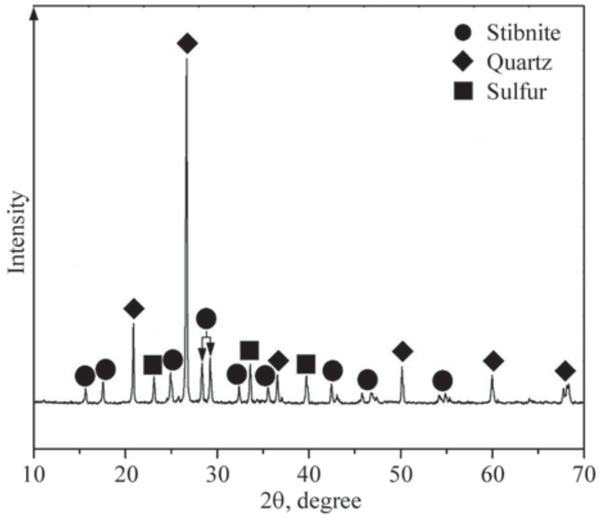

Fig. 7. XRD pattern of the leach residue after nitric acid leaching of stibnite with tartaric acid under optimal conditions

Рис. 7. Рентгенограмма нерастворенного остатка после азотно-кислотного выщелачивания стибнита с добавлением винной кислоты при оптимальных условиях

Table 2. **EDS analysis results of nitric acid leach residues of stibnite**

Таблица 2. Результаты исследования спектров кеков азотно-кислотного выщелачивания стибнита

Spectrum	Content, %			Identified phases
	O	Sb	S	
Without acid (see Fig. 8, *a*)				
1	15.1	56.6	28.3	$Sb_2O_5 + Sb_2S_3 + S^0$
2	17.5	55.3	27.2	$Sb_2O_5 + Sb_2S_3 + S^0$
3	16.4	57.2	26.4	$Sb_2O_5 + Sb_2S_3 + S^0$
With tartaric acid (see Fig. 8, *b*)				
1	–	48.6	51.4	$Sb_2S_3 + S^0$
2	–	44.8	55.2	$Sb_2S_3 + S^0$
3	–	54.3	45.7	$Sb_2S_3 + S^0$
4	–	67.1	32.9	$Sb_2S_3 + S^0$

taric acid-to-antimony mass ratio. The adequacy of the chosen model and regression equation is confirmed by a correlation coefficient of 0.92.

To validate the optimal parameters of nitric acid leaching of stibnite, an experiment was carried out under these conditions. The results showed antimony extraction of 87 % and sulfur extraction of 77 %. Of the total sulfur remaining in the leach residue, 61 % was present as sulfide and 39 % as elemental sulfur. The XRD pattern of the leach residue is shown in Fig. 7.

Fig. 8 presents SEM images and EDS analysis results at selected points (Table 2) of the leach residues after nitric acid leaching (L : S = 6 : 1, $T = 35$ °C,

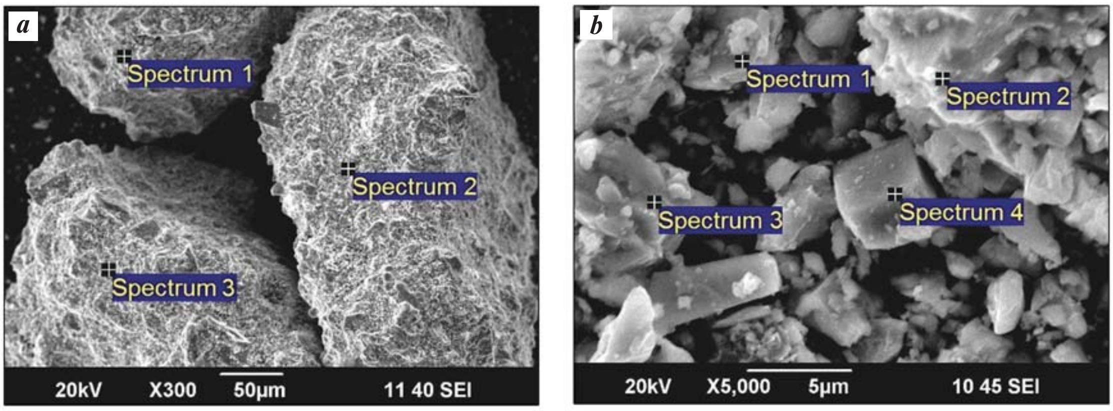

Fig. 8. SEM images of the leach residue from nitric acid leaching of stibnite without tartaric acid (***a***) and with tartaric acid (***b***)

Рис. 8. SEM-изображения кека азотно-кислотного выщелачивания стибнита без винной кислоты (***a***) и при ее наличии (***b***)

$[HNO_3] = 5$ mol/dm^3, $\tau = 15$ min) without and with tartaric acid (tartaric acid-to-antimony mass ratio 4.5 : 1.0).

The SEM images in Fig. 8 highlight differences in the composition of the leach residue depending on tartaric acid addition. In the sample without tartaric acid (Fig. 8, *a*), the residue contains antimony pentoxide (Sb_2O_5), stibnite (Sb_2S_3), and elemental sulfur (S^0). In contrast, in the presence of tartaric acid (Fig. 8, *b*), the residue consists mainly of unreacted stibnite and elemental sulfur, while antimony oxides are absent. This confirms that tartaric acid binds Sb ions in solution, preventing their hydrolysis and precipitation as oxides.

Conclusion

The present study on nitric acid leaching of stibnite with tartaric acid confirmed the effectiveness of this complexing agent in improving antimony extraction.

Thermodynamic analysis demonstrated that direct nitric acid leaching inevitably leads to the formation of antimony oxides, resulting in metal losses. The addition of tartaric acid promotes the formation of stable water-soluble complexes with antimony, which significantly reduces oxide precipitation.

Statistical experimental design showed that the tartaric acid-to-antimony mass ratio and nitric acid concentration are the most significant factors affecting leaching efficiency. Maximum antimony extraction of 87 % was achieved under the following conditions: temperature — 35 °C, $[HNO_3] = 5$ mol/dm^3, leaching duration — 45 min, and tartaric acid-to-antimony mass ratio — 4.5 : 1.0.

SEM and XRD analyses of the leach residue further confirmed that tartaric acid addition minimizes antimony losses. In the absence of tartaric acid, the residue contains antimony pentoxide (Sb_2O_5), indicating oxidation and hydrolysis of Sb ions with their conversion to insoluble forms, leading to metal losses. In contrast, in the presence of tartaric acid, oxide phases were not detected; the residue consisted mainly of unreacted stibnite (Sb_2S_3) and elemental sulfur (S^0).

Thus, tartaric acid ensures that antimony remains in a recoverable form by preventing its conversion into stable oxides, thereby improving leaching efficiency. This approach is relevant for developing more effective processing technologies for antimony-bearing raw materials.

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