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RESEARCH OF SULFIDE AND CARBON MATERIAL WITH THE PURPOSE OF STUDYING PHYSICO-CHEMICAL TRANSFORMATIONS DURING ITS ROASTING

Abstract. Over the past decades, due to the decrease in the quality of mineral raw materials from developed deposits, the share of gold extracted from refractory ores has increased, the effective processing of which requires the development of more complex and combined schemes that ensure the most complete extraction of gold. For the same purpose, in 2019, Navoi Mining and Metallurgical Combine (NMMC) successfully launched a workshop for roasting sorption cyanidation tailings during the processing of gold-bearing ores from the Kokpatas and Daugyztau deposits. At this stage, the NMMC specialists face the task of intensifying and optimizing the roasting process. This paper briefly presents the results of a detailed study of the effect of temperature on chemical transformations occurring during roasting of sorption cyanidation tails, carried out in order to identify the reasons that prevent the disclosure of gold particles during roasting and its encapsulation.

Key words: gold, sorption cyanidation tails, roasting, refractory gold, calcine, cyanide, encapsulation, pyrite, arsenopyrite, fluidized bed furnace with circulating roasting.

Сүлфид және көміртек материалдарын қуыру кезіндегі физикалық-химиялық түрлістерді зерттеу мақсаты

Аңдатпа. Соңғы онжылдықтарда игерілетін кен орындарындағы минералды шикізат сапасының төмендеуіне байланысты отқа төзімді кендерден алынатын алтынның үлесі артты, оларды тиімді өңдеу барынша толықты қамтамасыз ететін күрделі және аралас схемаларды әзірлеуді талап етеді алтын өндіру. Дәл осы мақсатта 2019 жылы Науаи кен металлургия комбинаты (НКМК) Көкпатас және Дауғызтау кен орындарының құрамында алтын бар кендерді өңдеу кезінде сорбциялық цианидтеу қалдықтарын күйдіру цехын сәтті іске қосты. Осы кезеңде НКМК мамандарының алдында қуыру процесін күшейту және оңтайландыру міндеті тұр. Бұл жұмыста күйдіру және оны инкапсуляциялау кезінде алтын бөлшектерінің ашылуын болдырмайтын себептерді анықтау мақсатында жүргізілген сорбциялық цианидтеу құйрықтарын қуыру кезінде болатын химиялық өзгерістерге температураның әсерін егжей-тегжейлі зерттеу нәтижелері қысқаша берілген.

Түйінді сөздер: алтын, сорбциялық цианидтеу құйрықтары, күйдіру, қыңыр алтын, кальцин, цианид, инкапсуляция, пирит, арсенопирит, циркуляциялық күйдірумен сұйық қабаты бар пеш.

Исследование сульфидно-углеродистого материала с целью изучения физико-химических превращений при его обжиге

Аннотация. В течение последних десятилетий в виду снижения качества минерального сырья разрабатываемых месторождений увеличивается доля золота, извлекаемого из упорных руд, эффективная обработка которых требует разработку более сложных и комбинированных схем, обеспечивающих максимально полное извлечения золота. С этой же целью в Навоийском горно-металлургическом комбинате (НГМК) в 2019 году был успешно запущен цех по обжигу хвостов сорбционного цианирования при переработке золотосодержащих руд месторождений Кокпатас и Даугызтау. На данном этапе перед специалистами НГМК стоит задача: интенсификация и оптимизация процесса обжига. В данной работе кратко представлены результаты детального исследования влияния температуры на химические превращения, происходящие при обжиге хвостов сорбционного цианирования, проведенного с целью выявления причин, препятствующих раскрытию частиц золота при обжиге и его инкапсуляции.

Ключевые слова: золото, хвосты сорбционного цианирования, обжиг, упорное золото, огарок, цианид, инкапсуляция, пирит, арсенопирит, печь кипящего слоя с циркулирующим обжигом.

Introduction

Today, special attention is paid to the processing of refractory gold ores and concentrates. To reduce the cost of the product, increase the through extraction of the base metal and reduce the cost of processing, new methods and technological solutions are being developed¹ [1, 2].

One of the innovative technological solutions in the processing of gold-bearing ores from the Kokpatas and Daugyztau deposits was the construction of a plant for roasting sorption cyanidation tailings. The prerequisites for the introduction of roasting technology were studies on the search for combined options for the oxidation of sulfide concentrates.

According to the results of laboratory tests, the highest throughput gold recovery rates are achieved when using oxidative roasting of sorption cyanidation tailings of the biooxidation product. Thus, at the end of 2019, the circulating fluidized bed kiln was successfully put into operation² [3].

Experiment Methods

The availability of reliable information about the chemical reactions occurring during roasting is the basis for improving the improvement of the quality indicators of roasting. In this regard, in order to study the ongoing physicochemical transformations during the roasting process and identify the reasons that prevent the extraction of gold

in the subsequent leaching with cyanide solutions, a study was made of the behavior of the constituent components of the sample taken from the sorption cyanidation tailings subject to oxidative roasting. The studies were carried out comprehensively in the temperature range of 200-800°C step by step, every 50°C, using modern physical and chemical methods, including DTA (differential thermal analysis-derivatography), IR spectroscopy, RA (X-ray phase analysis), SEM (scanning electron microscopy).

A mineralogical study is a critical and integral aspect of any pre-feasibility study, as well as one of the quality control measures during the process

¹Baudouin A. Ya., Fokina S. B., Petrov G. V., Serebryakov M. A. Modern hydrometallurgical technologies for processing resistant gold-containing raw materials. / Modern problems of science and education. – 2014. Electronic resource: <https://www.science-education.ru/ru/article/view?id=15619> (in Russian)

²Sanakulov K. S., Ergashev U. A., Dobersek A., Wotzka N., Mishina O. Industrial plant for oxidative roasting of gold sorption leaching tailings. – 2021. Electronic resource: https://zolteh.ru/technology_equipment/promyshlennaya_ustanovka_okislitel'nogo_obzhiga_khvostov_sorbtsionnogo_vyshchelachivaniya_zolota/ (in Russian)

Table 1
Weight percentages of minerals in a KEMIX sample calculated by the Rietveld method

Кесте 1
Rietveld әдісімен есептелген KEMIX үлгісіндегі минералдардың салмақтық пайызы

Таблица 1
Массовые проценты минералов в образце KEMIX, рассчитанные по методу Ритвельда

Mineral	Content, %	RSO, %
Quartz	30.9	0.4
Gypsum	1.6	0.1
CaSO ₄	1.5	0.1
Pyrite	3.8	0.1
Arsenopyrite	0.6	0.1
Chlorite	3.4	0.2
Muscovite/Ilit	30.5	0.3
Albite	7.3	0.2
TiO ₂	1.9	0.1
Calcite	1.9	0.1
Jarosite	3.3	0.1
Graphite (3R)	2.2	0.5
CaSO ₄ × 0.625(0.5)H ₂ O (bassanite)	5.2	0.1
Shabazit	1.7	0.1
Kaolinite	4.4	0.2

optimization phase. The mineralogical composition must be considered together with the results of the metallurgical testing program to ensure that the statements and justifications of the research results are valid.

To determine the mineral composition of the studied sample, the method of powder X-ray diffraction analysis was used. This method is based on the phenomenon of X-ray diffraction on the three-dimensional crystal lattice of minerals [4, 5].

The study of the mineralogical composition of the sample was carried out on a Panalytical Empyrean diffractometer using a «Scintillation» detector. Quantitative X-ray phase analysis will be performed by the Rietveld method on the software «Profex – Open source XRD and Rietveld Refinement» [6].

Infrared spectroscopy is based on the interaction of infrared radiation with substances. When infrared radiation is passed through a substance, vibrational movements of molecules or their individual fragments are excited. In this case, a decrease in the

intensity of light transmitted through the sample is observed. However, absorption does not occur over the entire spectrum of incident radiation, but only at those wavelengths whose energy corresponds to the excitation energies of vibrations in the molecules under study. Infrared spectroscopy is a valuable analytical method and is used to study the structure of organic molecules, inorganic and coordination, as well as high-molecular ones. The infrared spectra of the samples were obtained using a Nicolet Continuum FTIR microscope (Thermo Fisher Scientific, USA) in the range from 450 cm⁻¹ to 2200 cm⁻¹.

Scanning electron microscopy is one of the most widely used for diagnosing nanomaterials and nanostructures. SEM allows one to simultaneously study the size and shape of grains, the size distribution of grains and phases, determine the composition of the phase and the distribution of chemical elements over its area and over the area of the sample under study, and also obtain an image of an object in a wide range of magnifications in secondary

and reflected electrons. SEM analysis of samples was carried out in the low vacuum mode of a scanning electron microscope, which makes it possible to analyze carbon particles in samples. For imaging, a backscattered electron detector was used.

The study of the behavior of individual substances and sample compositions under conditions of programmed heating was carried out by thermogravimetric methods of analysis (TG and DTG) and by scanning calorimetry (DSC). The thermogravimetry (TG) method makes it possible to record the mass loss of a substance during heating. The essence of this method lies in the periodic or continuous weighing of a sample of the test substance in the process of heating using special balances. The method of differential thermogravimetry (DTG) makes it possible to determine the rate of change in the mass of a sample upon heating.

Results and Discussion

Research on a detailed study of physical and chemical transformations in the temperature range from 200°C to 800°C makes it possible to purposefully influence the course of the roasting process, making it possible to prevent factors affecting the disclosure of gold particles and its encapsulation during the roasting process, thereby improving the quality of the resulting calcine.

The powder X-ray diffraction pattern of the studied sample is shown in Figure 1, and the semi-quantitative mass percentages of minerals obtained as a result of the refinement by the Rietveld method of the powder X-ray diffraction pattern are shown in table 1.

As can be seen from Table 1, the sample contains pyrite, arsenopyrite, and possibly carbonaceous material as gold minerals. The total amount of pyrite and arsenopyrite in the sample is calculated as 4.4%. The presence of these minerals allows us to conclude that pyrite is not completely oxidized.

We can also observe the presence of calcium sulphate minerals (gypsum, anhydride and bassanite) and the mineral jarosite (total 3.3%), which are identified as minerals determining the persistence of the biooxidation

Table 2

Semi-quantitative elemental analysis from the sample surface focused on sulfide minerals at up to 1200x magnification

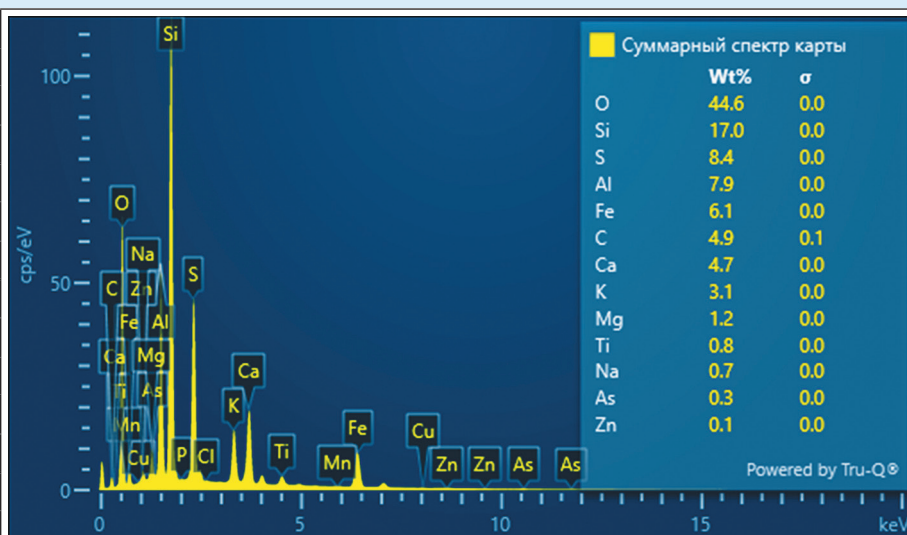
Кесте 2

1200 есе үлкейту кезінде сульфидті минералдарға бағытталған үлгі бетінен жартылай сандық элементтік талдау

Таблица 2

Полуколичественный элементный анализ с поверхности образца с акцентом на сульфидные минералы при увеличении до 1200x

Element	Weight, %	Sigma Wt, %
O	44.58	0.04
Si	17.04	0.02
S	8.43	0.01
Al	7.86	0.01
Fe	6.14	0.01
C	4.92	0.07
Ca	4.73	0.01
K	3.06	0.01
Mg	1.19	0.01
Ti	0.78	0.01
Na	0.66	0.01
As	0.31	0.01
Zn	0.10	0.01
Cu	0.09	0.01
Cl	0.05	0.00
P	0.04	0.00
Mn	0.04	0.01
Total	100.00	



product. Jarosite induces the greatest resistance by forming a thin film layer around pyrite and arsenopyrite, thus blocking bacterial access to the surface of these minerals, and by encapsulating fine gold particles within its matrix. Quartz, muscovite, albite, chlorite, chabazite, kaolinite, rutile, pyrite, arsenopyrite and graphite are ore minerals. Calcite and all sulphate minerals are the result of oxidation of sulphide minerals and subsequent neutralization with calcite and an increase in the pH of the medium using lime.

To obtain more detailed information about the number of elements on the surface of the sample, elemental analysis was carried out on a scanning electron microscope (table 2).

Elemental maps of SEM analysis showed that sulfur is associated either with the Ca-forming mineral $CaSO_4$ (gypsum, gypsum, hemihydrate or anhydrite), or with the Fe-forming – mainly arsenopyrite, the particle size

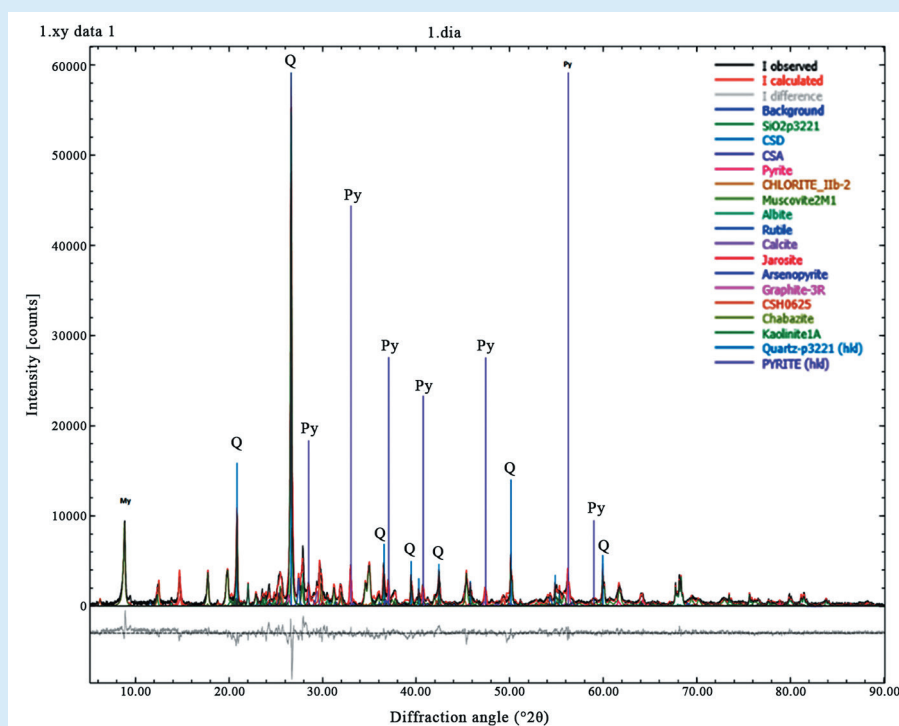


Figure 1. Powder diffraction pattern of a KEMIX sample.
Сурет. 1. KEMIX үлгісінің ұнтақты дифракциялық үлгісі.
Рис. 1. Порошковая дифрактограмма образца KEMIX.

of which is about 25 microns. Longer X-ray spectrum collection along with higher electrical current (2 nanoamps) results in improved signal from arsenic as well as signal from gold associated with arsenic in the sulfide mineral. We can also observe rather small sulfide minerals, less than 5 microns. At this magnification, many small carbon particles, about 5 microns in size or less, can be observed. Other minerals are associations of silicon, aluminium, potassium, magnesium, sodium, magnesium and titanium, which we can refer to as quartz, muscovite, albite, chlorite and rutile.

An analysis of the main transformations during sample roasting at temperatures from 200°C to 800°C with an interval of 50°C shows that the oxidation of pyrite and arsenopyrite is the main thermal process that can affect the recovery of gold in the sample. Dehydration of calcium sulfate minerals does not lead to the formation of larger aggregates as a result of water loss. The kaolinite and chlorite minerals also weaken interlayer water molecules within the crystal structure and are unlikely to trap gold particles within their matrix.

The phase transition of quartz from alpha to beta form includes centering and increasing the symmetry of atoms within the same crystallographic structure and does not include the destruction and formation of new minerals. Roasting the carbon particles should only increase the recovery of gold from the samples.

For the above reasons, the main interest in the study was to identify the minerals formed during pyrite/arsenopyrite roasting and which minerals are associated with sulfur and arsenic, and to investigate if any of these minerals interfere with gold extraction during the roasting process.

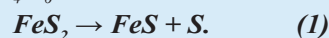
Raise temperatures of the beginning of pyrite/arsenopyrite oxidation with increasing heating rate can be explained by the lack of oxygen corresponding to the heating rate. For these reasons, it is best to use a rate of 5°C/min as a reference for determining the roasting temperatures of arsenopyrite and pyrite.

In samples, the onset of oxidation is observed at temperatures between

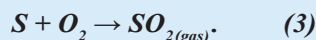
300°C and 350°C, pyrite/arsenopyrite is completely oxidized at temperatures below 500°C, while only 0.3% pyrite is observed in the X-ray powder diffraction experiment (semi-quantitative). which is probably the result of introducing additional air into the kiln and stirring the samples during the experiment.

In an X-ray diffraction experiment with heating in situ, the oxidation of arsenopyrite/pyrite starts at 400°C to 450°C, and is completely oxidized below 500°C. Heating the sample from the bottom of the 0.1 mm thick sample and collecting data from the top of the sample could explain the reason for the higher oxidation initiation temperatures for pyrite/arsenopyrite in this setup.

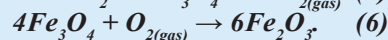
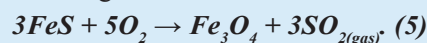
Arsenopyrite and arsenian pyrites decompose to volatilize arsenic, which absorbs heat and leaves behind. The arsenic vapor then diffuses to the outer perimeter, where it reacts with oxygen to form $As_4O_6(g)$ and releases heat [7].



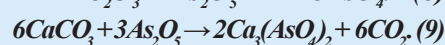
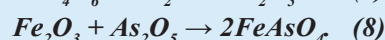
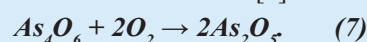
Arsenic and sulfur are released as a result of exothermic reactions from decomposition oxides along the outer perimeter of the grains. Reaction areas below:



Ideally, pyrrhotite is oxidized to magnetite, and magnetite is further oxidized to hematite, following the reactions:



Reaction between arsenic effluent gas phase and iron oxide interfaces to form non-porous iron arsenate or iron arsenite. Based on roasting studies of typical roast leach tailings at Barrick's Goldstrike, it was concluded that in a highly oxidizing environment in low climates, arsenic could potentially be trapped as non-porous iron arsenate ($FeAsO_4$) or iron pyroarsenite (Fe_2AsO_5). The resulting As_2O_5 can further react with Fe_2O_3 or $CaCO_3$ or CaO in the sample to form $FeAsO_4$ and $Ca_3(AsO_4)_2$ as shown in the reactions below [8].



Iron and calcium compounds with a high content of arsenic were observed in samples prepared at temperatures of 650°C and above.

Raise temperatures of the beginning of pyrite/arsenopyrite oxidation with increasing heating rate can be explained by the lack of oxygen corresponding to the heating rate. For these reasons, it is best to use a rate of 5°C/min as a reference for determining the roasting temperatures of arsenopyrite and pyrite.

Analysis of samples fired from 200°C to 800°C shows the formation of maghemite and hematite as the main products of the oxidation process. Arsenic is evenly distributed in the sample, with the ratio of iron to arsenic in most samples being approximately 12:1 when analyzed from the entire surface of the sample, and the ratio of iron to arsenic can reach 56:1 when measured from the surface of iron oxide particles in samples with temperatures above 500°C. Iron arsenate and calcium arsenate have been observed in samples prepared at 650°C and above.

Fourier transform infrared spectroscopy has been instrumental in identifying the formation of the mineral maghemite by roasting. Optical microscopy used during measurements in micro-infrared showed that colorless minerals such as calcium sulfate, quartz, and others were coated with a very thin layer of hematite, giving them a red-brown color under the microscope. This also explains the change in color of the sample.

According to [9] maghemite, an intermediate oxidation phase between magnetite and hematite, is considered the main carrier of gold, present both as liberated particles and as small (10 µm) inclusions in gangue minerals, demonstrated that maghemite is massive and immune to cyanidation. Gold in maghemite tends to remain solid in solution, which correlates with elevated arsenic concentrations. Incomplete oxidation, as evidenced by the residual ore structure of the pyrite inclusions in the gangue, also contributes to the formation of the remaining solid gold in the pyrite, as does pyrite. During a study

of the Barrick Goldstrike roasting products, they showed that 50% of the unrecovered gold was left with maghemite particles.

Conclusions

Thus, the studies carried out in the temperature range made it possible to study the behavior of the

constituent components of a sample of sorption cyanidation tailings subject to oxidative roasting and to identify possible causes of gold encapsulation.

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