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Satbayev University

ХАБАРЛАРЫ

ИЗВЕСТИЯ

НАЦИОНАЛЬНОЙ АКАДЕМИИ
НАУК РЕСПУБЛИКИ
КАЗАХСТАН
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INVESTIGATION OF THE USE OF ION FLOTATION FOR THE EXTRACTION OF COPPER FROM SULFURIC ACID SOLUTIONS

Abstract. The purpose of this study is to use heap leaching followed by recovery of copper from solution by ionic flotation for efficient processing of off-balance copper ores. The object of research was the off-balance copper ore of the JSC Almalyk Mining and Metallurgical Combine. Since ion flotation is one of the promising methods for concentrating non-ferrous metals from various technological solutions, studies were carried out on the use of ion flotation to extract copper from sulfuric acid solutions. The air flow was controlled by changing the rotation speed of the impeller. The extraction of metals into foam (foam is a product of ionic flotation, consisting of a chemical compound of the collector with metal ions and from a certain amount that did not react with metal ions) was judged by its residual concentration in the solution after flotation. Sulfuric acid solution obtained by leaching had pH=4.5 and contained (g/l): copper - 10 ÷10,5; iron – 5÷5,5; zinc - 0,5-1,5. Copper, iron and zinc are in solution in sulfate form ($CuSO_4$, $FeSO_4$, $ZnSO_4$). In order to select a collector, we studied the dependence of the extraction of copper ions into the "foam" on the pH of the solution, the consumption of the collector, and the duration of flotation for the following collectors: amyl xanthate, ethyl xanthate, C_7–C_9 xanthates, isopropyl xanthate, and sodium diethyldithiocarbamate. As follows from the data presented, a relatively low extraction of ions was obtained with amyl xanthate. A mixture of high molecular weight xanthates (C_7 - C_9) forms a liquid "foam" during flotation. The remaining collectors form a dry "foam" with a high extraction of copper ions. During ion flotation, 98-100% of the metal is extracted from solutions. By adjusting the pH of the medium, it was possible to selectively extract 98.-99.5% of iron, 88-95% of zinc (at pH=3.5-4), 99-99.8% of copper (at pH=5-5.5). The collector consumption was 50 mg/1 g of the recovered metal.

Key words: copper, off-balance ore, heap leaching, solution, ion flotation, collector, recovery, concentrate.

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ИССЛЕДОВАНИЕ ПРИМЕНЕНИЯ ИОННОЙ ФЛОТАЦИИ ДЛЯ ИЗВЛЕЧЕНИЯ МЕДИ ИЗ СЕРНОКИСЛЫХ РАСТВОРОВ

Аннотация. Цель данного исследования заключается в использовании кучного выщелачивания с последующим извлечением меди из раствора ионной флотацией для эффективной переработки забалансовых медных руд. Объектом исследований послужила забалансовая медная руда ОАО «Алмалыкский ГМК». Так как ионная флотация является одним из перспективных методов концентрирования цветных металлов из различных технологических растворов проводились исследования по применению ионной флотации для извлечения меди из сернокислых растворов. Расход воздуха регулировался изменением скорости вращения импеллера. Об извлечении металлов в пенку (пенка – продукт ионной флотации, состоящий из химического соединения собирателя с ионами металлов и из некоторого количества, не прореагировавшего с ионами металла) судили по ее остаточной концентрации в растворе после флотации. Сернокислый раствор, полученный при выщелачивании имел pH=4,5 и содержал (г/л): меди - 10÷10,5; железа – 5÷5,5; цинк – 0,5-1,5. Медь, железо и цинк находятся в растворе сернокислой форме ($CuSO_4$, $FeSO_4$, $ZnSO_4$). С целью выбора собирателя исследовалась зависимость извлечения ионов меди в «пенку» от pH раствора, расхода собирателя и продолжительности флотации для следующих собирателей: амиловый ксантогенат, этиловый ксантогенат, ксантогенаты C_7–C_9, изопропиловый ксантогенат и диэтилдитиокарбамат натрия (ДЭДТКН). Как следует из приведенных данных, относительно низкое извлечение ионов получено с амиловым ксантогенатом. Смесь высокомолекулярных ксантогенатов (C_7 – C_9) при флотации образует жидкую «пенку». Остальные собиратели образуют сухую «пенку» при высоком извлечении ионов меди. При ионной флотации из растворов извлечено 98-100% металла. Регулированием pH среды достигнуто избирательно извлечь 98,-99,5% железа, 88-95% цинка (при pH=3,5-4), 99-99,8% меди (при pH=5-5,5). Расход собирателя составил 50 мг/1 г извлекаемого металла.

Ключевые слова: медь, забалансовая руда, кучное выщелачивание, раствор, ионная флотация, собиратель, извлечение, концентрат.

Introduction. One of the main conditions for the further expansion of the domestic production of non-ferrous metals, including copper, is the improvement of existing and the introduction of new technological processes. At present, the republic has accumulated a huge amount of off-balance copper ores, which are additional raw materials for the production of copper at the Almalyk Mining and Metallurgical Plant OJSC.

To date, the republic has accumulated a huge amount of off-balance copper ores,

which are additional raw materials for the production of copper at the Almalyk Mining and Metallurgical Plant OJSC.

To obtain copper from off-balance copper ores of the required quality, an appropriate technology is needed. Despite the severity and relevance of the problem, there is still no modern, efficient, cost-effective technology for extracting copper from off-balance ores.

In addition, gold and silver can be extracted from ores. This determines the relevance of the tasks set, which correspond to the main directions of the economic and social development of Uzbekistan to create the foundations for the processing of off-balance copper ores.

The well-known hydrometallurgical schemes for the processing of concentrates into non-ferrous metals are characterized by common disadvantages, which are determined by poor multicomponent raw materials, which require complex technological schemes for processing with a large expenditure of reagents and energy. On an industrial scale, ion flotation is used to purify industrial and domestic wastewater from cations of non-ferrous, rare radioactive, and other metals. Ionic flotation allows the extraction of valuable components and associated metal cations (Dzhevaga, 2012: 161).

Materials and methods. When studying the existing literature, it was found that: the flotation scheme is the simplest in design, reliable in operation and economically favorable. When enriching refractory oxidized ores, the flotation scheme does not provide satisfactory performance, in particular, the extraction of copper is only from 27 to 40%, depending on the quality of the ore; the combined scheme (Mostovich's scheme) does not allow achieving high performance in the enrichment of refractory ores; sorption-flotation technology has not been implemented on an industrial scale.

The purpose of this study is to use heap leaching followed by recovery of copper from solution by ionic flotation for efficient processing of off-balance copper ores.

The object of research was the off-balance copper ore of the JSC Almalyk Mining and Metallurgical Combine (Dzhevaga, 2012: 161; Gonçalves and others, 2003: 1213; Ivanova, 2013:35; Jeldres and others, 2019: 57; Kadirov and others, 2021: 304; Khairullaev and others, 2021: 36; Kholikulov and others, 2007: 187; Kirjavainen and others, 2007: 629).

The studies were carried out under laboratory conditions in a cast-iron bath, the size of which is as follows: width - 400 mm, length - 400 mm, height - 400 mm.

Preliminarily, large ore was stored in the lower part of the bath, then medium and small. The pile was gradually irrigated with a solution of sulfuric acid. During the experiment, a chemical analysis for copper was carried out every day. After a one-day pause, irrigation was continued. During the experiments, the bottom cock (drain) was kept open all the time. The solution was applied dropwise from above, which prevents the solution from accumulating on top of the ore.

Since ion flotation is one of the promising methods for concentrating non-ferrous metals from various technological solutions (Dzhevaga, 2012: 161; Dzhevaga, 2012: 161), studies were carried out on the use of ion flotation to extract copper from sulfuric acid solutions.

The flotation of copper, iron, and zinc ions from solutions was carried out in a flotation

machine with a capacity of 200 ml. The flotation chamber is made of organic glass, the impeller and the air suction pipe are made of ordinary glass. Air was supplied through a tube inside which the impeller shaft rotated.

Changing the pH of the solution was carried out by adding sulfuric acid. To control the concentration of hydrogen ions, an LPU-01 universal laboratory pH meter with a silver chloride electrode was used (Kozlowski and others, 2002: 677; Santos and others, 2010: 15).

Results. The ores of the Kalmakyr deposit are divided into three technological types: oxidized, mixed (also including secondary sulfide) and primary sulfide. It has been established that off-balance oxidized ores have a complex structure (Sergeev and others, 2018: 214). The content of copper in oxidized ores is from 0.1-0.33%, in the zone of secondary sulfide enrichment - from 0.3 to 4%. The share of secondary balance ores in relation to all reserves is 2.2%, mixed - 4.4% and secondary sulfide - 1.9%.

Off-balance oxidized ores accumulate in dumps. Dumps of overburden and off-balance ores at the Almalyk Mining and Metallurgical Plant, excluding dumps in the mined-out space of the quarry, contain about 5 million tons. mountain mass.

Highly mineralized copper-bearing effluents can also be attributed to copper-bearing resources. Natural leaching waters contain up to 0.5 g/dm^3 of copper. Effluent is neutral to very acidic. Before being dumped, they are cleaned, usually hydrolytically, and the metals are lost in the sludge. An alternative physical and chemical technology can be applied to extract copper from all the named man-caused objects.

The study of the chemical and mineral composition of the ore showed that copper is present in oxidized ores - in the form of malachite, cuprite, tenorite. Copper-bearing minerals are in fine intergrowth with silicates of host rocks and other minerals of non-ferrous metals and iron. The copper content usually does not exceed 0.5%, but mostly tenths of a percent.

To establish the regularities of physical and chemical processes, the influence of the following main factors on the results of leaching of minerals and ores was analyzed: size of the ore mass, structural features of the ore massif, solvent concentration, concentration of intensifiers. Leaching of malachite and oxidized copper ore occurs in a wide range of temperatures, even at their negative values, which determines the possibility of using heap leaching. The leaching of sulfide ores proceeds effectively only at high positive temperatures.

The previously established experimentally good solubility of copper oxides in sulfuric acid solutions served as the basis for studying the process of ore leaching depending on the duration, temperature, pulp density and solvent concentration. In addition, taking into account the complex nature of the feedstock, it was important to study the behavior of the accompanying valuable copper components in sulfuric acid solutions in order to ensure the complete separation of copper into a separate product and the selectivity of copper recovery (Kolesnikov, 2015: 131; Kozlowski and others, 2002: 677; Makhmudov and others, 2022: 2432; Makhmudov and others, 2022: 2432; Malghan, 1986: 158; Nasirov and others, 2020: 2251; Norov and others, 2018: 42; Pavlov and others, 2012: 11).

Thus, the pulp always contains a certain amount of ferric sulphate, an oxidizer

of sulfides, but the dissolution of sulfides proceeds more slowly than the reaction of dissolution of oxides. This allows additional oxidation of copper minerals.

It has been established that a change in the concentration of ferric iron in the solution and a change in the size of the mineral have practically no effect on the extraction of copper into the solution (Khayitov and others, 2022: 46). A significant increase in the extraction of copper into the solution is observed with increasing temperature. So, in one day of leaching at 35^0 C, 22% of copper is extracted, and at 50^0 C, already 32% of copper is extracted.

With an increase in the concentration of sulfuric acid in the solution (up to 75 g/l), the solubility of the ore components increases linearly and reaches a maximum. A further increase in concentration (from 75 g/l) does not increase the rate of copper dissolution and, on the contrary, after some time causes some slowdown in the process. Increasing the concentration of sulfuric acid from 75 g/l only slightly affects the dissolution of copper. For ore leaching with sulfuric acid, we recommend that the concentration of sulfuric acid not exceed 75 g/l, this can control the degree of dissolution of associated minerals.

Irrigation density has a significant impact on the leaching process. With its increase, the concentration of copper in the productive solution decreases (Khairullaev and others, 2021: 36). Based on the test, it was found that in order to achieve the optimal ratio between the copper content in the productive solution and the existing capacities for its processing, it is necessary to differentiate the irrigation density over time.

Ion flotation was carried out to extract copper, iron, and zinc from the sulfuric acid solution. Ionic flotation is achieved by introducing into water, together with air, compounds having a charge opposite to that of the extracted ions.

The air flow was controlled by changing the rotation speed of the impeller (Zhurinov and others, 2020: 94). The extraction of metals into foam (foam is a product of ionic flotation, consisting of a chemical compound of the collector with metal ions and from a certain amount that did not react with metal ions) was judged by its residual concentration in the solution after flotation.

Sulfuric acid solution obtained by leaching had pH=4.5 and contained (g/l): copper - 10÷ 10,5; iron - 5÷ 5,5; zinc - 0.5-1.5. Copper, iron and zinc are in solution in sulfate form ($CuSO_4$, $FeSO_4$, $ZnSO_4$). In order to select a collector, the dependence of the extraction of copper ions into the «foam» on the pH of the solution (Fig. 1), the consumption of the collector (Fig. 2) and the duration of flotation (Fig. 3.) was studied for the following collectors: amyl xanthate, ethyl xanthate, C_7 xanthates C_9, isopropyl xanthate and sodium diethyldithiocarbamate. As follows from the data presented, a relatively low extraction of ions was obtained with amyl xanthate. A mixture of high molecular weight xanthogens (C_7 - C_9) forms a liquid “foam” during flotation.

The remaining collectors form a dry “foam” with a high extraction of copper ions.

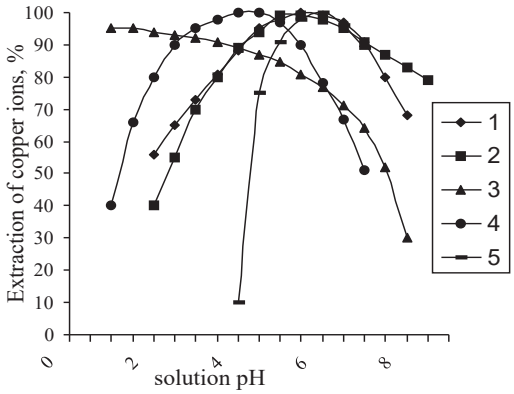
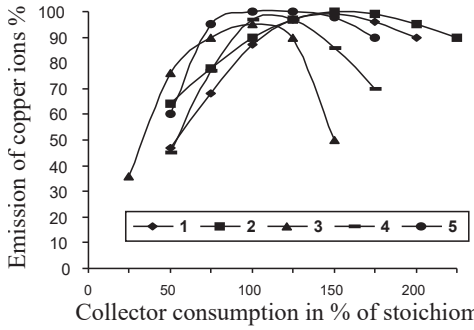


Fig.1. The dependence of the extraction of copper ions from the sulfuric acid solution on the pH of the medium, xanthates.

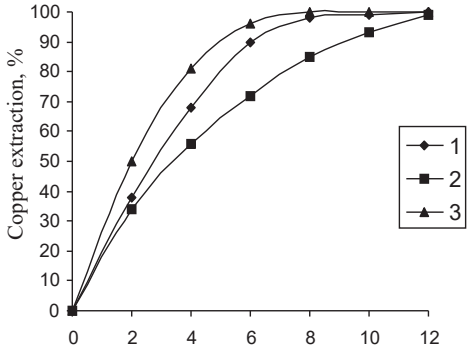
As the results of the experiments showed, of all the tested collectors, sodium diethyldithiocarbamate turned out to be the most acceptable. With its optimal consumption (110% of the stoichiometric), the extraction of copper is 99.6-100%.

For further research, we chose the collector sodium diethyldithiocarbamate.



1- ethyl; 2-isopropyl; 3- amyl; 4-C_7–C_9; 5- Sodium diethyldithiocarbamate

Fig.2. The dependence of the extraction of copper ions from the sulfuric acid solution on the consumption of the collector, xanthates.



1- ethyl xanthate; 2- isopropyl xanthate; 3- Sodium diethyldithiocarbamate

Fig.3. Dependence of extraction of copper ions from sulfuric acid solution on the duration of flotation.

Sodium diethyldithiocarbamate is a well-known surfactant (surfactant) collector, it is a salt of weak diethyldithiocarboxylic acid and has long been used in conventional flotation processes and has the following advantages: Sodium diethyldithiocarbamate forms very strong, acid-stable, insoluble compounds with all heavy metals and enough stable in solutions with low pH; reagent available; relatively non-toxic (the maximum allowable concentration is 0.5 mg/dm^3); there is a possibility of partial regeneration; the reagent is relatively inexpensive.

Studies have shown the possibility of using the sodium diethyldithiocarbamate collector in the ionic flotation of copper from acidic solutions obtained during the hydrometallurgical processing of copper ores (Fig. 4.). It was found that the extraction of copper from the solution occurs in a certain range of values, at pH ≅5,5-6 (Fig. 4).

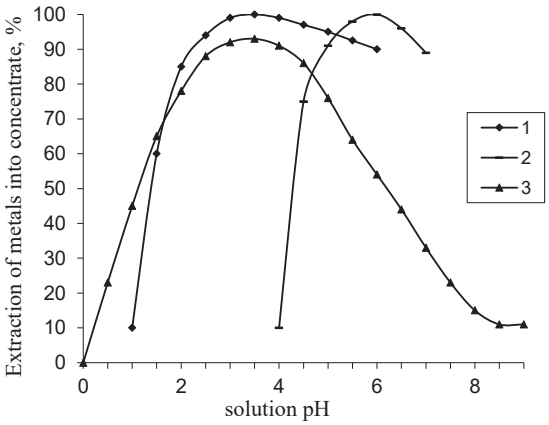


Fig. 4. Influence of the solution pH on the extraction of metals into the product (1-iron; 2-copper; 3-zinc)

In ionic flotation, each extracted ion interacts with the collector. As a result, the consumption of the collector per unit mass of colligen is much higher than in the case of mineral flotation. In ion flotation, complete extraction of colligen requires at least a stoichiometric flow rate of the collector; the minimum possible weight ratio of collector to colligend depends on their equivalent weights (Santos and others, 2010: 15; Sergeev and others, 2018: 214; Soltanbekova, 2021: 113; Zhurinov and others, 2020: 94).

In most cases, for the flotation of ions and molecules, gas is needed only as an inert phase for the formation of a liquid-gas interface. Therefore, in laboratory studies, nitrogen or air is most often used. In our case, air was used.

Discussion. As a result of the studies, it was found that with an increase in gas consumption, the concentration in the lower product decreases, and the extraction into the upper product increases. In froth flotation, an increase in gas flow is usually accompanied by an increase in bed height in the separating vessel. However, sometimes the foam is so unstable that the foam is formed directly on the surface of the solution. In such cases, at high gas flow rates, when the liquid surface is restless, there is a serious danger of foam dispersion and precipitation of its particles into the solution volume.

In addition, the air flow rate affects the stability of the foam. The gas flow rate should

only slightly exceed the foam breakdown rate in order to maintain a foam layer of the desired thickness.

Losses of individual metals in tailings range from 0 to 1.6%, and the sum of losses of all metals does not exceed 1%.

Table 1

Optimal regime of copper ions flotation from sulfuric acid solutions by various collectors.

Gatherers	pH	Collector consumption, in % of stoichiometric	Flotation duration, in min	Extraction, in %
Potassium amyl xanthate	2-3	130	10	96,0
Ethyl xanthate	5-6	125	10	98,0 - 99,0
Xanthate $C_7 – C_9$	3-4	130	10	98,0
Isopropyl xanthate	5,0	140	12	99,0 - 99,2
Sodium diethyldithiocarbamate	6	110	10	99,2 - 99,8

Conclusion. Thus, in the course of the study, the fundamental possibility of using sodium diethyldithiocarbamate as flotation collectors of Cu (II), Fe (II), and Zn (II) ions was established. The use of sodium diethyldithiocarbamate as a collector in copper ion flotation makes it possible to extract 98-100% of the metal from solutions. Due to the double charge of its ion, copper is an element that gives an easily processed foam. By adjusting the pH of the medium, it was achieved to selectively extract 98.-99.5% iron, 88-95% zinc (at pH = 3.5-4), 99-99.8% copper (at pH = 5-5.5) (table 1). The collector consumption was 50 mg/1 g of the recovered metal.

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