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Metallurgy

Flotation processing of copper-containing technogenic raw materials using a composite flotation reagent

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ABSTRACT

The article presents the results of laboratory research on the possibility of flotation processing of copper-containing technogenic raw materials. Studies were performed using a sample of copper-containing tailings obtained after the processing of copper ore from the Kazakhstan deposit and a composite flotation reagent. The application of a combination of various collectors allows for raising technological indicators of flotation. The purpose of the research is the increase extraction of copper at flotation of copper-containing technogenic raw materials with the application of a composite reagent. The studied sample of tailings contains 0.23% of copper. A mixture of sodium butyl xanthate and thionocarbamate in the ratio of 1:1 was used as a composite flotation reagent. Parameters of flotation of copper-containing tailings were worked out: degree of regrinding, charges of sodium butyl xanthate, blowing agent T-92, and a composite reagent. Composite flotation reagent was fed into the flotation process of copper-containing tailings in the form of emulsion produced in a T 18 digital ULTRA-TURRAX dispersant. The optimal emulsification time of the composite flotation reagent was 1 min. Without emulsification in the composite reagent the percentage of particles smaller than 1.192 μm is 55.047%. After emulsification of the composite reagent for 1 minute, the percentage of particles smaller than 1.192 μm is 91.134%. In optimum basic mode the rough copper concentrate with a copper content of 4.2% was obtained with the extraction of 61.56%. With the use of a composite reagent, a blister copper concentrate with a copper content of 4.5% and a recovery of 66.54% was obtained. Extraction of copper increases by 4,98%.

Keywords: copper-containing tails, flotation, flotation agent, emulsion, concentrate, recovery.

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Introduction

Technogenic wastes from mining and processing plants, metallurgical plants, oil refineries, and enterprises to process the natural minerals contain millions of tons of valuable metals in sufficiently high concentrations suitable for their

highly profitable processing and use as secondary raw materials.

Due to the depletion of the mineral resource base, it is necessary to develop new and optimize existing methods of extraction of valuable components from ore materials [[1], [2], [3], [4]]. In recent years the task of localization and

neutralization of technogenic and natural-technogenic objects especially saturated with harmful substances (mercury, radionuclides, oil products, flotation reagents, etc.) has become extremely acute. Such objects include tailing dumps, sludge storages and waste dumps. Anthropogenic formations may serve and have served as sources of raw materials for the industry. Being an important reserve for replenishing the volume of mineral raw materials, technogenic deposits or formations have a very aggressive impact on the natural environment. Therefore, the interest in their processing is caused not only by commercial calculations but also by increased environmental requirements [[5], [6], [7], [8]]. The concentration of technogenic formations in areas with a developed infrastructure and communication network in the absence of the need for stripping works serves as an additional factor in reducing the energy and material charges of mining. It is necessary to develop highly efficient, fast-payback technological schemes for complex processing, ensuring the creation of low-waste and non-waste production, the implementation of which will significantly expand the mineral resource base without disturbing the subsoil and land [[9], [10], [11], [12]].

Enrichment wastes are generated in the processes of the same name, which are usually intermediate between mining technologies and their deep physical and chemical processing. A significant part of them, about 80 percent, is used for backfilling of mines and quarries as part of the execution of industrial mining technologies. The rest part of them, also considerable, is accumulated in the tailing dumps of beneficiation plants.

The main difficulties are related to the processing of the slurry part of the tailings, which is to a great extent enriched with non-ferrous, rare and precious metals [[13], [14], [15], [16], [17]].

In this regard, the use of modern approaches, new flotation reagents and modernized equipment for the beneficiation of technogenic raw materials is important in creating promising innovative technologies [[18], [19], [20], [21]].

The purpose of the research presented in this article is to increase copper recovery during flotation of copper-containing technogenic raw materials with the use of a composite reagent/

Experimental part

Researches on studying the possibility of flotation processing of copper-containing

technogenic raw materials with application of a composite reagent were performed.

Researches were performed with use of the sample of copper-containing tailings received after processing the copper ore of the Kazakhstan deposit and a composite flotation reagent representing a mix of sodium butyl xanthate and thionocarbamate (non-ionogenic collector with thioamide group).

In the process of conducting research, various methods were used using modern technological and analytical equipment such as mineralogical and dispersion analysis, X-ray phase, X-ray fluorescence, electron-probe, particle size analysis, chemical analysis, flotation.

For analysis, a Venus 200 PANalytical B.V. spectrometer, a D8 ADVANCE X-ray diffractometer, and a JEOL JXA-8230 scanning electron microscope were used. Chemical analysis was performed using an Optima 2000 DV optical emission spectrometer.

The initial tailings were regrind in a ball mill 40ML-000PS (40МЛ-000ПС) to perform the studies. Flotation was carried out on laboratory flotation machines in open and closed cycles. The sample weight for flotation was 0.5 kg. The scheme of flotation included regrinding, rougher floatation, control and three retreatment operations of the rough copper concentrate.

Sodium sulfide was fed into the regrinding process to sulfidize oxidized minerals. The rougher floatation was performed for 15 min, the control flotation for 10 min, and the refining operations for 6-8 min. In the basic mode the following reagents were used as a collector - sodium butyl xanthate; foaming agent - T-92.

Total consumption of basic reagents: sodium sulfide (sulfideizer) - 400 g/t; sodium butyl xanthate (collector) - 240 g/t; T-92 (blowing agent) - 120 g/t. Sodium sulfide was fed into the grinding process in a ball mill. Sodium butyl xanthate and T-92 were fed into the flotation operation. The reclamation operations were performed without addition of reagents.

In flotation of tailings using the composite flotation reagent it was fed to the rougher and control flotations instead of the basic collector sodium butyl xanthate. Composite flotation reagent was a mixture of sodium butyl xanthate and thionocarbamate in the ratio 1:1. Before flotation the composite flotation reagent for reception of a microemulsion was passed through the disperser T 18 digital ULTRA-TURRAX. The optimum particle size of microemulsion for flotation of tailings was

selected. The particle size was determined on a laser particle analyzer Winner2000E.

After regrinding in a ball mill in the presence of sodium sulfide, the mineral slurry was placed in the chamber of the flotation machine. Then sodium butyl xanthate (or composite flotation reagent) and foaming agent T-92 were added to the flotation machine. The mineral slurry was stirred for 0.5 minutes without air supply at a rotor speed of 1,300 rpm. After atmospheric air supply (3.5 dm³/min) pulp flotation was performed according to the scheme of tailings processing. Foam product of the rougher flotation was subjected to three refinements with receiving the draft copper-containing concentrate.

Discussion of results

The use of a mixture of various flotation reagents makes it possible to increase the technological parameters of flotation. Combination of reagents, their dispersion and optimization of composition (increase or reduction of length of a hydrocarbon radical and its branching, introduction of an additional component) leads to improvement of foaming, collecting and other properties of used flotation reagents [[9], [10], [11], [12], [13]].

In the present work efficiency of application of sodium butyl xanthate and thionocarbamate composition for flotation processing of copper-containing technogenic raw materials is shown. The combination of these reagents will not only improve the quality of the resulting concentrates due to the action of thionocarbamate, but also increase the recovery of copper by improving the flotation of slurry particles of sulfide minerals.

Mineralogical analysis of the initial tailings showed that copper minerals are represented mainly by chalcopyrite; secondary sulfides and copper oxides account for about 15%. Tailings contain about 20% of zinc oxide compounds. The main amount of sulfides is open, about 15-20% of copper and zinc minerals are present in the form of intergrowths with pyrite and rock-forming minerals. The surface of the sulfides is partially oxidized and covered with iron oxide films.

The X-ray phase analysis has shown that the main part of the initial sample of copper-containing tailings of the beneficiation plant is represented by rock-forming minerals, such as quartz (72.2%), illite (11.2%), clinoferrosilite (5.9%), albite (4.3%), muscovite (3.2%), microcline (1.9%), clinochlor (1.3%) (Table 1).

Table 1 - Results of X-ray phase analysis of the initial sample of copper-containing tailings

Compound Name, Formula	S-Q
Quartz, syn, SiO ₂	72.2
Illite, KAl ₂ Si ₃ AlO ₁₀ (OH) ₂	11.2
Clinoferrosilite, syn, Fe(SiO ₃)	5.9
Albite, low, Na(AlSi ₃ O ₈)	4.3
Muscovite-2M1, KAl ₂ (Si,Al) ₄ O ₁₀ (OH) ₂	3.2
Microcline, intermediate, KAlSi ₃ O ₈	1.9
Clinochlore (Ilb-4), Mg _{4.882} Fe _{0.22} Al _{1.881} Si _{2.96} O ₁₀ (OH) ₈	1.3

According to the results of chemical analysis, the analyzed sample of tailings contains 0.23% copper. The results of X-ray fluorescence analysis are presented in the table 2.

Table 2 - Results of X-ray fluorescence analysis of the initial sample of copper-containing tailings

Item name	Contents,%
O	51.468
Na	0.412
Mg	0.650
Al	6.621
Si	27.020
P	0.046
S	0.137
Cl	0.012
K	1.146
Ca	2.810
Ti	0.393
V	0.006
Cr	0.020
Mn	0.050
Fe	4.473
Cu	0.230
Zn	0.215
As	0.016
Rb	0.006
Sr	0.012
Zr	0.010
Mo	0.007
Pb	0.071

According to the results of X-ray fluorescence analysis in the initial sample of copper-containing tailings of the beneficiation plant, the content of copper is 0.23%, molybdenum 0.007%. The main mass is composed of silicon - 27.02%, oxygen - 51.468%, aluminum - 6.621%, iron - 4.473%, calcium - 2.81% etc.

A sample of the original tailings was analyzed on a JXA-8230 electron probe microanalyzer (Figure 1).

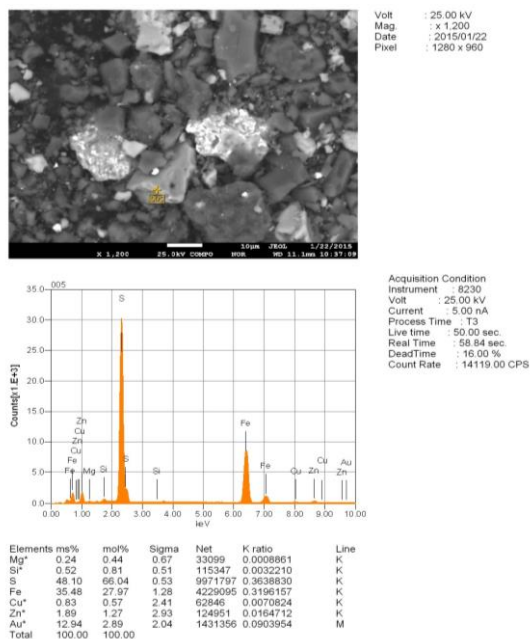


Figure 1 - Analysis of initial copper-containing tailings on electron-probe microanalyzer JXA-8230 by JEOL (chalcopyrite)

As a result of studies on a microanalyzer, it was shown that copper is presented mainly in the form of chalcopyrite and its content is less than one percent. They are most often found as inclusions in non-metallic minerals.

The grain-size composition of copper-containing tailings with distribution of copper by grain-size classes using sieve method was determined. The results are given in table 3. The results of the grain-size analysis showed that 68.94% of copper is in the class larger than 0.071 mm. This shows that it is necessary to include a regrinding operation into the scheme of tailings processing.

Table 3 - Grain size composition and grain-size classes distribution of copper in source copper-containing tailings

Grain-size class, μm	Yield %	Cu Content, %	Cu extraction, %
+0.4	6.96	0.23	7.38
-0.4+ 0.2	29.93	0.21	29.62
-0.2 + 0.1	30.33	0.19	26.90
-0.1 + 0.071	8.3	0.13	5.04
-0.071+ 0.05	6.88	0.20	6.44
-0.05+ 0	17.58	0.30	24.62
Initial Tails	100.0	0.21	100.0

Dispersion analysis of the initial sample of tailings was carried out on the FSH-6K photometric sedimentometer, the results of which are shown in Figure 2.

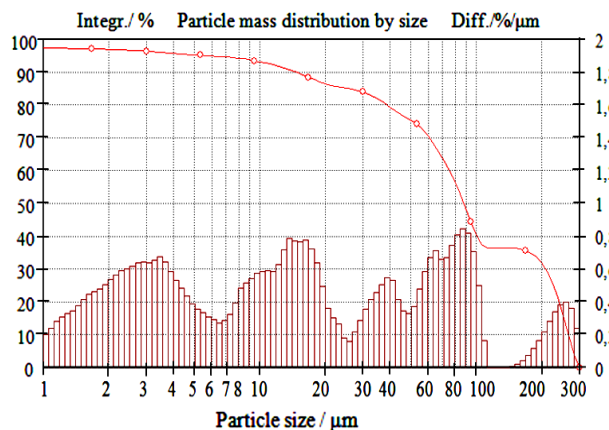


Figure 2- Dispersion analysis of initial copper-containing ore tailings on FSKh-6K

The dispersion analysis results show that in the initial sample of tailings the grain-size classes in the area of 70-80 μm make up 40-45%, the fine grain classes of 3-5 μm make up 30-35%. Up to 20-25% are the grain-size classes of 100-300 μm .

Thus, the results of mineralogical, granulometric, chemical and phase analyses of copper-containing tailings of the beneficiation plant indicate that part of the valuable components is concentrated in coarse grain classes and is in the form of aggregates. The regrinding process in the technological scheme of tailings processing shall be included to open such aggregates.

The initial coarseness of the flotation tailings of the beneficiation plant by the class -0.071 mm was 31%. Studies to determine the optimal degree of regrinding of the initial tailings lot were performed. Regrinding of initial tailings in a ball mill 40МЛ-000ПС was performed from 10 to 30 minutes, which corresponded to the degree of grinding 66.04-96.82% of class -0.071 mm.

It is shown that the best results were achieved when tails were regrind up to 95% of class -0.074 mm (table 4).

A rough copper-containing concentrate containing 2.60% of copper with the extraction of 30.05% was obtained without regrinding. At the optimum degree of regrinding of 95% of class - 0.071 mm a copper-containing concentrate containing 3.23% of copper was obtained with the extraction of 48.75%.

The research results show that regrinding of copper-containing flotation tailings of the

beneficiation plant allows to increase in the extraction degree of copper in the rough copper concentrate by 18.7%.

Table 4 – Results of flotation of copper-containing tailings using regrind

Product name	Yield %	Cu content, %	Cu extraction, %
Without regrind			
Rough Cu conc.	2.46	2.60	30.05
Intermediate product 1	3.14	0.37	5.44
Intermediate product 2	1.2	0.71	4.00
Intermediate product 3	0.66	0.88	2.72
Cont. fl. conc.	2.38	0.59	6.60
Final tails	90.16	0.12	51.19
Initial Tails	100.0	0.21	100.0
With regrind			
Rough Cu conc.	3.10	3.23	48.75
Intermediate product 1	4.34	0.20	4.26
Intermediate product 2	2.24	0.33	3.57
Intermediate product 3	1.14	0.48	2.65
Cont. fl. conc.	2.52	0.37	4.54
Final tails	86.66	0.08	36.23
Initial Tails	100.0	0.21	100.0

For the flotation process, the optimal costs of sodium butyl xanthate, foaming agent T-92, and composite reagent were selected.

Optimal flotation conditions in the basic mode with the use of sodium butyl xanthate as collector are: regrinding of tailings to 95% of class -0.071 mm, Na_2S - 400 g/t; charge of butyl xanthate in the rougher floatation is 160 g/t, T-92 is 80 g/t; charge of butyl xanthate in the control floatation is 80 g/t; T-92 is 40 g/t. In optimum basic mode in an open cycle the rough copper concentrate with copper content of 4,03% at extraction 51,9%.

The reagent mode of flotation of copper-containing tailings using a composite reagent which is a mixture of sodium butyl xanthate and thionocarbamate in the ratio of 1:1 was processed.

The composite flotation reagent was fed into the process of flotation of copper-containing tailings in the form of an emulsion generated in a T 18 digital ULTRA-TURRAX dispersant. The consumption rate of the composite reagent in the rougher copper floatation was 120 g/t, in the control floatation - 60 g/t. Optimal time of emulsification of composite flotation reagent was 1 minute.

Composite reagent without emulsification and after emulsification studied on a laser particle size analyzer Winner2000E (Figure 3-4).

The results showed that without emulsification in the composite reagent the percentage of particles smaller than 1.192 μm is 55.047% (Figure 3). After emulsification of the composite reagent for 1 minute, the percentage of particles smaller than 1.192 μm is 91.134% (Figure 4).

The study of the particle distribution of the proposed flotation reagent on a Winner2000E laser particle size analyzer shows the need to obtain a reagent in the form of an emulsion for its more effective action during floatation. The floatation agent emulsion enhances the hydrophobization of fine copper mineral particles, contributing to an increase in floatation performance.

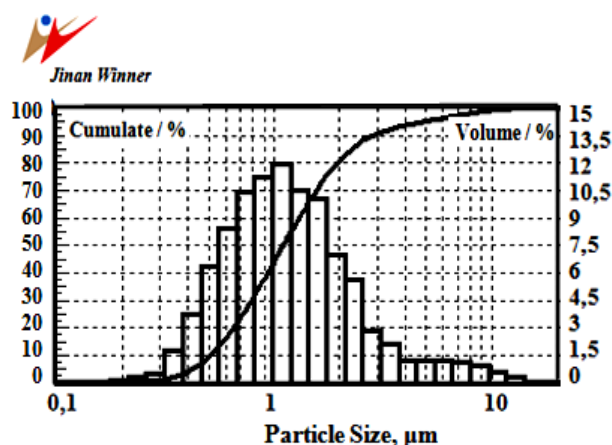


Figure 3 - Composite reagent particle distribution obtained on the Winner 2000E without emulsification

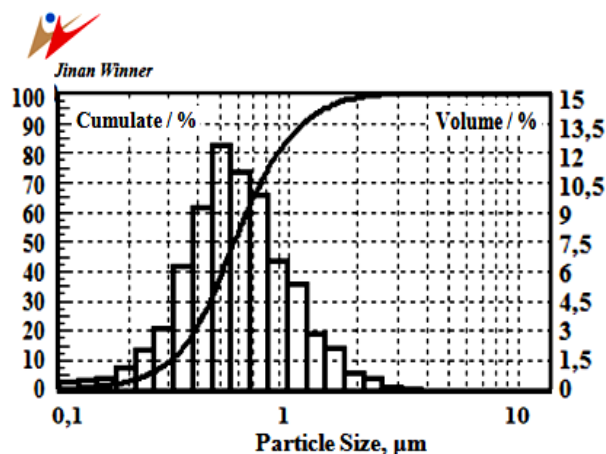


Figure 4 - Distribution of composite reagent particles obtained on Winner 2000E after emulsification

Studies on the processing of copper-containing technogenic raw materials using the proposed composite flotation reagent were carried out in comparison with basic flotation reagents.

According to the basic mode with application of sodium butyl xanthate, the rough copper concentrate with copper content 4.2% with copper extraction of 61.56% was obtained. Rough copper concentrate with copper content of 4.5% and extraction of 66.54% was obtained using a composite reagent. Extraction of copper increases by 4,98%.

The dispersion analysis of flotation tails of the basic mode and the optimal mode of flotation with the dispersed microemulsion of composite flotation reagent was conducted in order to establish the dependence of the degree of dispersion of the emulsion of composite flotation reagent on the extraction degree of slime particles of copper-bearing minerals. Results of dispersion analysis are given in the table 5.

Table 5 – Results of dispersion analysis of flotation tailings of basic mode and with dispersed emulsion of composite reagent

Product name	Yield %	Cu contents, %	Cu distribution, %
Flotation tailings in basic mode			
-0.071+0.050	24.55	0.084	27.07
-0.050+0.030	17.9	0.060	14.10
-0.030+0.020	12.85	0.052	8.77
-0.020+0.010	26.9	0.067	23.66
-0.010+0	17.8	0.113	26.40
Initial Tails	100.0	0.076	100.0
Flotation tailings with a composite reagent			
-0.071+0.050	28.95	0.083	33.29
-0.050+0.030	19.45	0.058	15.63
-0.030+0.020	10.25	0.031	4.40
-0.020+0.010	26.45	0.062	22.72
-0.010+0	14.9	0.116	23.95
Initial Tails	100.0	0.072	100.0

The dispersion analysis of flotation tails showed that the use of dispersed emulsion of composite reagent allows to increase the extraction degree of slam particles of copper-containing minerals in the rough copper concentrate. The flotation tailings received in the basic mode contain 58.83% of copper in the grain-size classes less than 30 μm. The flotation tailings received with the use of the composite reagent contain 51,07% of copper in the grain-size classes less than 30 μm. The content of copper in sludge classes (less than 30 μm) in the final flotation tailings is reduced by 7.76% when using a composite flotation reagent.

Thus, results of researches show perspective of application of a composite reagent to process the copper-containing technogenic raw materials.

Conclusions

The influence of the composite flotation reagent on flotation of copper-containing technogenic raw materials was studied. As object of researches the sample of mature tailings of flotation of one of Kazakhstan beneficiation plants with the contents of copper 0.23% was taken.

Composite reagent is a mixture of sodium butyl xanthate and thionocarbamate in a ratio of 1:1. Composite reagent in the process of flotation was fed in the form of dispersed microemulsion. Optimal dispersion time of the composite flotation reagent was 1 min.

Microemulsion of a composite flotation reagent allows to strengthen hydrophobization of slurry particles of copper minerals, thereby to improve their flotation and to increase technological parameters of flotation.

The percentage of particles with a size less than 1.192 μm in the emulsion is 91.134% at optimum dispersion time as defined on the laser particle size analyzer Winner2000E.

In optimum basic mode in a closed cycle rough copper concentrate with copper content of 4.2% was received at extraction of 61.56%. Rough copper concentrate with copper content of 4.5% and extraction of 66.54% was obtained with application of composite reagent. Extraction of copper increases by 4.98%.

Dispersion analysis of flotation tailings showed that the use of dispersed emulsion of composite reagent allows to increase the extraction degree of slime particles of copper-containing minerals in the rough copper concentrate.

The results of the research can be used at beneficiation plants in the development of flotation technology to process the thin-rocked technogenic waste using microemulsion of flotation reagents.

Conflict of interest

The corresponding author, on behalf of the authors of this study, declares that there is no conflict of interest.

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Комбинирленген флотореагентті қолдана отырып, мысқұрамды техногендік шикізатты флотациялық өңдеу

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Сараптамадан өтті: 04 мамыр 2022
Қабылданды: 14 шілде 2022

ТҮЙІНДЕМЕ

Мақалада мысқұрамды техногендік шикізатты флотациялық өңдеу мүмкіндігі бойынша зертханалық зерттеулердің нәтижелері ұсынылған. Зерттеулер Қазақстан кенорнындағы мыс кенін өңдеуде алынған мысқұрамды қалдықтардың сынамасын және композиттік флотациялық реагентті қолдану арқылы жүргізілді. Әр түрлі жинағыштардың қоспасын қолдану флотацияның технологиялық көрсеткіштерін жақсартуға мүмкіндік береді. Зерттеудің мақсаты комбинирленген реагентті қолдана отырып, мысқұрамды техногендік шикізатты флотациялауда мыстың бөліп алу дәрежесін арттыру болып табылады. Зерттелетін қалдықтардың сынамасында мыстың үлесі 0,23% құрайды. Комбинирленген флотореагент ретінде 1:1 қатынасында натрий бутил ксантогенаты мен тионокарбамат қоспасы қолданылды. Мысқұрамды қалдықтарды флотациялау параметрлері анықталды: қайта ұсақтау дәрежесі, натрий бутил ксантогенатының, Т-92 көбіктендіргіштің, комбинирленген реагенттің шығындары. Комбинирленген флотациялық реагент мыс қалдықтарын флотациялау процесіне эмульсия түрінде жіберілді, эмульсия Т 18 сандық ULTRA-TURRAX диспергаторында алынған. Комбинирленген флотореагентті эмульсиялаудың оңтайлы уақыты 1 минутты құрады. Эмульсияға айналмағанда, комбинирленген реагентте 1,192 мкм-ден кем бөлшектердің пайыздық мөлшері 55,047 % құрайды. Комбинирленген реагентті 1 минут эмульгирлеуден кейін 1,192 мкм-ден кем бөлшектердің пайыздық мөлшері 91,134% құрайды. Оңтайлы базалық режимде өрескел мыс концентраты алынды, ондағы мыстың үлесі 4,2%, бөліп алу дәрежесі 61,56 % құрайды. Комбинирленген реагентті қолдану барысында мыстың үлесі 4,5 %, бөліп алу дәрежесі 66,54 % құрайтын өрескел мыс концентраты алынды. Мыстың бөліп алу дәрежесі 4,98 % - ға артады.

Түйін сөздер: мысқұрамды қалдықтар, флотация, флотациялық реагент, эмульсия, концентрат, бөліп алу.

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Флотационная переработка медьсодержащего техногенного сырья с применением композиционного флотореагента

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АННОТАЦИЯ

В статье представлены результаты лабораторных исследований по возможности флотационной переработки медьсодержащего техногенного сырья. Исследования проведены с использованием пробы медьсодержащих хвостов, полученных после переработки медной руды казахстанского месторождения, и композиционного флотационного реагента. Применение сочетания различных собирателей позволяет повысить технологические показатели флотации. Целью исследований является повышение извлечения меди при флотации медьсодержащего техногенного сырья с применением композиционного реагента. В исследуемой пробе хвостов содержится 0,23 % меди. В качестве композиционного флотореагента применена смесь бутилового ксантогената натрия и тионоккарбамата в соотношении 1:1. Отработаны параметры флотации медьсодержащих хвостов: степень доизмельчения, расходы бутилового ксантогената натрия, пенообразователя Т-92, композиционного реагента. Композиционный флотореагент подавался в процесс флотации медьсодержащих хвостов в виде эмульсии, полученной в диспергаторе Т 18 digital ULTRA-TURRAX. Оптимальное время эмульгирования композиционного флотореагента составило 1 мин. Без эмульгации в композиционном реагенте процентное содержание частиц крупностью менее 1,192 мкм составляет 55,047 %. После эмульгации композиционного реагента в течение 1 минуты процентное содержание частиц крупностью менее 1,192 мкм составляет 91,134 %. В оптимальном базовом режиме получен черновой медный концентрат с содержанием меди 4,2 % при извлечении 61,56 %. С применением композиционного реагента получен черновой медный концентрат с содержанием меди 4,5 % при извлечении 66,54 %. Извлечение меди повышается на 4,98 %.

Ключевые слова: медьсодержащие хвосты, флотация, флотореагент, эмульсия, концентрат, извлечение.

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