

ФІЗИКА ТВЕРДОГО ТІЛА, ЗБАГАЧЕННЯ КОРИСНИХ КОПАЛИН

UDC 553.4:351.823

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EVALUATION OF FLOTATION ABILITY OF A SULFHYDRYL COLLECTOR MIXTURE IN THE PROCESSING OF COPPER-BEARING ORES

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ОЦІНКА ФЛОТАЦІЙНОЇ ЗДАТНОСТІ СУМІШІ СУЛЬФІДРИЛЬНИХ ЗБИРАЧІВ ПРИ ПЕРЕРОБЦІ МІДЬВМІСНИХ РУД

Purpose. Establishing the criteria for the selectivity of flotation enrichment of copper- copper-bearing ores with a mixture of sulfhydryl collectors.

Methodology. Atomic absorption, X-ray fluorescence analysis.

Findings. Investigations of flotation ability of collector mixtures of potassium butyl xanthate and sodium isobuthyldithiophosphate in relation to the copper-bearing ore samples are carried out. Mathematical models describing the enrichment process of copper ore are obtained. Optimal conditions of froth flotation in particular air flow rate of 40 l/h⁻¹, frequency of impeller rotation of 35 Hz, consumption of lime of 3000 g/t⁻¹, consumption of a collector mixture of 100 g/t⁻¹ are determined.

Originality. For the first time, the regression equations simulating the effect of oxygen amount in the pulp, pH of medium, contact time of solid phase with a solution of a collector mixture, of collector mixture concentration, are obtained. The flotation analysis of a “copper ore – solution of collector mixture” system is carried out. It is established that the effect of air flow rate on the fractional composition of the pulp by floatability, which is to change the redox potential, leads to a change in the strength of securing of collectors on the ore surface.

Practical value. Obtained mathematical relationships will enable to carry out the forecast of the change in chemical-technological parameters of enrichment under the influence of air flow rate, consumptions of medium regulator (lime) and mixture of collectors, frequency of impeller rotation. Results of the flotation analysis can be used in developing and improving flowsheets.

Keywords: *copper ore, X-ray fluorescence analysis, potassium butyl xanthate, sodium diisobuthyldithiophosphate, froth flotation, mathematical models, flotation analysis*

Introduction. In the mining industry one of the key tasks is increasing the yields and quality of the concentrate at the expense of creating reagents with pronounced selective properties in relation to a certain type of ore, developing technological regimes with adjusting the flow of collectors, environmental regulators and other flotation agents, as well as using various combinations of both traditional and alternative reagents.

On the other hand, the flotation capacity of reagents in relation to different types of ores depends not only on the chemical composition of the ore, but also

on the form of minerals in the ore, which is determined by the genesis of the deposit. For example, the Zhezkazgan deposits ores are localized in gray-colored rocks exclusively, regardless of their lithologic composition. The content of copper, lead and zinc in the gray-colored rocks is on average 4–5 times higher than that in the red-colored rocks. The industrial mineralization in the field is extended to a depth of about 600 m, hypsometrically below; only the poor sulphide mineralization is noted [1].

The basic bulk of copper is concentrated in three widely distributed minerals – chalcopyrite, bornite and chalcocite. There is a clear vertical zoning in the distri-

bution of these minerals at the deposit. In the upper horizons of the deposit the main role is played by chalcopyrite, in the deeper horizons, bornite prevails, and at last, in the lowest horizons there is chalcocite.

Moreover, increased lead and zinc amounts are characteristic of the lower horizons. A similar sequence of copper minerals changing is observed in the individual ore bodies; in the central parts of them chalcocite prevails. Chalcocite is replaced by bornite, and the latter, in turn, by chalcopyrite as it moves to the periphery. The edge parts of ore bodies are characterized by an increase in the content of lead and zinc. These are deposits such as copper sandstones (chalcocite, bornite, and chalcopyrite).

The Zhezkazgan deposit is epigenetic, with high-temperature, that was formed in two stages: 1) densely clayed and massive ores arose in sandstones from fluids (melt solutions) of 5 % H₂O, 20 % H₂S, 19 % CO₂, 5 % (CO, NH₃, H₂, N₂, Ar), 51 % of the salts (Na₂SO₄) at 500–400 °C; 2) mineralization in sandstones was formed at 650–400 °C in sulphide veins.

The process was completed by the carbonate-sulfide veins at progressive decrease of the temperature in the range of 500–700 °C and with the participation of residual water-salt solutions [2].

Therefore, these ores are characterized by a high dispersion degree of mineral particles, the intergrowth of minerals among themselves, and, consequently, they are difficult for enrichment. In connection with this, the work purpose is establishment of the selectivity criteria of flotation enrichment of copper-containing ores with the mixture of sulphhydryl collectors.

Unsolved aspects of the problem. Currently, establishment of an optimal reagent regime and selectivity criteria for the copper ore enrichment, which ensures the greatest possible degree of mineral separation and the production of conditioning concentrates remains an unsolved problem.

Experimental part. Froth flotation was carried out on a laboratory flotation machine FML-1 with a chamber volume of 0.5 l by the following procedure: a sample of the ore (75 % of a fraction of 0.074 mm) with a mass of 10 g was loaded into the flotation chamber and it was mixed with water. Lime was added to maintain the desired pH. Then, a solution of collector mixture with the given concentration and a foaming agent were added into the chamber, and stirring was continued for 9 minutes. The collector mixture consisting of potassium butyl xanthate (active substance content of 57.6 %) and AFI-4 (basic substance sodium diisobutyl dithiophosphate of 65 %) was used as the flotation agent in a ratio of 9:1. Froth generator T-92 had consumption of 15 g/t⁻¹. Flotation experiments were carried out on the basis of a 4-factor 3-level matrix (Table 1).

Decomposition of the initial ore samples and the resulting concentrates (0.1 g) was carried out with a mixture of concentrated hydrochloric and nitric acids (3:1) [3]. Determination of metal ions Cu²⁺, Zn²⁺, Pb²⁺ concentration was carried out on the Varian AA140 atomic absorption spectrometer. Elemental analysis was performed on the Olympus Delta XRF X-ray fluorescence analyzer (Table 2).

Table 1

Conditions for froth flotation

Experiment number	Air flow rate	Impeller rotation frequency	Lime consumption	Collector mixture consumption (AFI-4 and potassium butyl xanthate)
№	$U_{air}, l/h^{-1}$	v_{rot}, Hz	$m_{lime}, g/t^{-1}$	$C_{flot.reagents}, g/t^{-1}$
1	20	30	1000	50
2	20	35	2000	100
3	20	40	3000	150
4	40	30	2000	150
5	40	35	3000	50
6	40	40	1000	100
7	60	30	3000	100
8	60	35	1000	150
9	60	40	2000	50

Table 2

Results of elemental analysis of copper ore

Compound	Mass fraction, %	Element	Mass fraction, %
CaO	6.62	Ca	8.89
SiO ₂	62.46	Si	54.78
SO ₃	0.76	S	0.57
Fe ₂ O ₃	5.40	Fe	7.05
Al ₂ O ₃	14.28	Al	14.12
MgO	1.96	Mg	2.19
K ₂ O	2.98	K	4.62
TiO ₂	0.63	Ti	0.71
Na ₂ O	2.63	Na	3.63
MnO	0.20	Mn	0.29
SrO	0.04	Sr	0.06
CuO	1.23	Cu	1.83
ZnO	0.28	Zn	0.42
BaO	0.09	Ba	0.15
PbO	0.29	Pb	0.50
P ₂ O ₅	0.15	P	0.12
Rb ₂ O	0.02	Rb	0.02
ZrO ₂	0.03	Zr	0.04

The technological parameters of enrichment were calculated by the formulas

$$Y_k = 100(\alpha - \theta)/(\beta - \theta); \quad (1)$$

$$E_k = Y_k/\beta/\alpha; \quad (2)$$

$$K = \beta/\alpha. \quad (3)$$

Where Y_k is concentrate yield, %, E_k is metal recovery into the concentrate, %, β is metal content in the concentrate, %, K is degree of concentration [4].

Explanation of scientific results. On the basis of the probabilistic-deterministic approach, the optimization of the copper ore enrichment process was carried out; some partial dependencies of the chemical-technological parameters on the variable factors of the air flow rate, the impeller rotation frequency, collector mixture consumption (AFI-4 and potassium butyl xanthate), and the lime consumption were obtained (Table 3).

In Table 3, y is the response function (copper recovering in concentrate ($\epsilon_{conc.}$), concentration degree (K)), x is the designation of the parameter presented, the impeller rotation frequency is denoted as v_{rot} , the air flow rate is denoted as v_{air} , the lime consumption is m_{lime} , flo-toreagent mixture consumption is $C_{flot.reagent}$, the correlation coefficient for the partial dependence between response function and the values of the set parameter (factor) is R .

Table 3

Partial dependencies between the copper recovering degree and the concentration ratio on each factor for the copper ore enrichment process

Factor	Copper recovering in concentrate (response function), %	Correlation coefficient	Concentration ratio (response function)	Correlation coefficient
x	$\epsilon_{conc.} (y)$	R	$K (y)$	R
$v_{air}, l/h^{-1}$	$y = -0.01x^2 + 1.44x - 1.83$	0.99	$y = 0.08x + 2.86$	0.99
v_{rot}, Hz	$y = -0.50x^2 + 35.67x - 593.53$	0.99	$y = -0.01x^2 + 0.76x - 8.69$	0.99
$m_{lime}, g/t^{-1}$	$y = 2 \cdot 10^{-6}x^2 + 16.06$	0.99	$y = 0.01x + 0.63$	0.99
$C_{flot.reagent}, g/t^{-1}$	$y = -0.01x^2 + 2.15x - 52.45$	0.99	$y = -2 \cdot 10^{-5}x^2 + 0.01x + 3$	0.98

Optimum parameters of copper ore flotation enrichment, the air flow rate of $40 l/h^{-1}$, the impeller rotation frequency of $35 Hz$, the lime consumption of $3000 g/t^{-1}$, and the collector mixture consumption of $100 g/t^{-1}$ were determined. On the other hand, the air flow rate determines the value of the oxidation-reduction potential of the pulp, through the amount of oxygen moles held by the pulp, the lime consumption determines the pH of the medium (Table 4).

In Table 4 the number of moles of oxygen held by the pulp is (O_2) and the contact time of pulp phases is τ , hydrogen index volume of the pulp is pH, the collector mixture concentration is C .

It is seen from Table 5 that partial dependencies of the chemical-technological enrichment parameters on the variable factors can be used for modeling the copper ore enrichment process.

In Table 5, y is the response function (concentration yield ($\gamma_{conc.}$), copper content in the concentrate (Cu)), x is the designation of the parameter presented, the number of moles of oxygen held by the pulp is (O_2) , the contact time of pulp phases is (τ) , the hydrogen index volume of the pulp (pH), the collector mixture concentra-

tion is $C_{flot.reagent}$, the correlation coefficient for the partial dependence between response function and the parameter presented values (factor) is R .

Table 4

Results of conversions of variable factors into the medium physicochemical parameters

The number of moles of oxygen held by the pulp	Contact time of pulp phases	Hydrogen index volume of the pulp	Collector mixture concentration
$n(O_2) \cdot 10^3, mole$	τ, min	pH	$C, mg/l^{-1}$
5.74	10	10.96	1
5.74	8.57	11.11	2
5.74	7.5	11.20	3
11.49	10	10.96	3
11.49	8.57	11.11	1
11.49	7.5	11.20	2
17.23	10	10.96	2
17.23	8.57	11.11	3
17.23	7.5	11.20	1

Table 5

Mathematical models of the copper ore enrichment process

Factor	Concentration yield, % (response function)	Correlation coefficient	Copper content in the concentrate, % (response function)	Correlation coefficient
x	$\gamma_{conc.} (y)$	R	$Cu (y)$	R
$n(O_2) \cdot 10^3, mole$	$y = 661.88x^2 - 9.14x + 0.25$	0.99	$y = -904.11x^2 + 15.09x + 0.14$	0.99
τ, min	$y = 2.02 \times x^{-0.986}$	0.80	$y = -0.01x^2 + 0.21x - 0.77$	0.99
pH	$y = 0.38x^2 - 8.47x + 47.31$	0.99	$y = -1.66x^2 + 37.06x - 206.36$	0.99
$C_{flot.reagent}, mg/l^{-1}$	$y = 0.04x^2 - 0.18x + 0.41$	0.99	$y = 0.01x + 0.16$	0.98

The optimum parameters of the enrichment process were also determined: for the concentrate yield the number of moles of oxygen held by the pulp is 17.23 mole (40 l/h^{-1}), the contact time of pulp phases is 7.5 min (40 Hz), the pH of the medium is 10.96 (1000 g/t^{-1}), the collector mixture concentration is 1 mg/l^{-1} (50 g/t^{-1}).

For copper content in the concentrate – the number of moles of oxygen held by the pulp is 5.74 mole (20 l/h^{-1}), the contact time of pulp phases is 8.57 (40 Hz), the pH of the medium is 11.11 (2000 g/t^{-1}), the collector mixture concentration is 3 mg/l^{-1} (150 g/t^{-1}).

During the formation of enrichment process model, simultaneous consideration of the number of moles of oxygen held by the pulp, the pH of the medium, the collector mixture concentration are taken into account, this allows the oxidation-reduction potential of the pulp to be calculated.

While constructing the surfaces in the coordinates the pulp oxidation-reduction potential – pH makes it possible to predict the bubble-particle complex stability in a wide range of pH of the medium, and adding of the time parameter into the generalized equation makes it possible to carry out a kinetic monitoring of the system, which is directly related to the ore enrichment [5].

Consequently, the ratios given in Table 5 are more informative than those given in Table 3 for the following analysis of copper ore flotation ability.

Further the copper ore flotation was carried out at the optimal conditions. The analysis of the obtained copper concentrate was carried out by X-ray fluorescent method (Fig. 1).

Analysis of the spectrum showed that a high content of copper and iron is observed in the concentrate, this is due to the presence of chalcopyrite (K_{α} peaks of the copper line and K_{β} of the iron line which have intensity of 55 and $40 \text{ kpm}\cdot\text{s}^{-1}$, respectively), the presence of the K_{α} iron line at $1850 \text{ m}\text{\AA}$ suggests the presence of oxidized iron compounds on the surface of chalcopyrite.

The results of a multifactor experiment demonstrated that the efficiency of flotation enrichment depends on the pH of the medium and the number of moles of oxygen held by the pulp (the air flow rate is 10, 20, 40, 60, 70 l/h^{-1}) at the optimum concentration of the collector (mixture of AFI-4 – potassium butyl xanthate (1:1)) (100 g/t^{-1}).

Therefore, realization of flotation analysis of the copper ore enrichment process under the conditions indicated above is necessary [6]. The results are shown in Figs. 2–6.

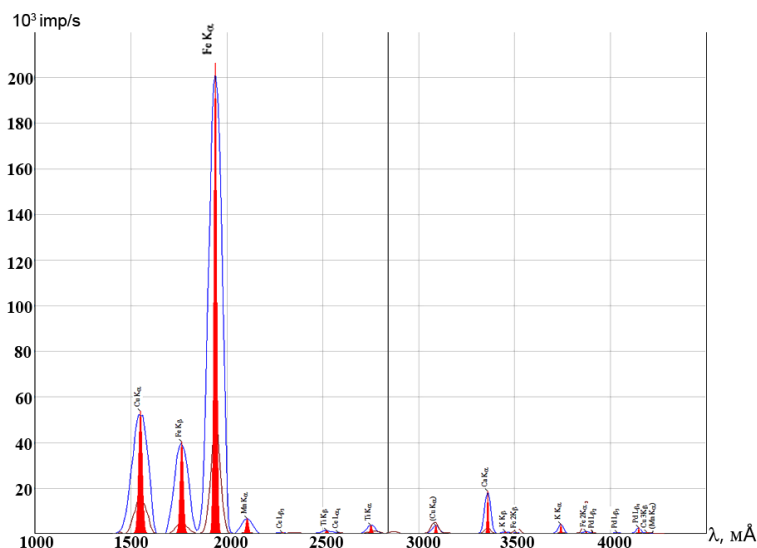


Fig. 1. XRF spectrum of copper concentrate

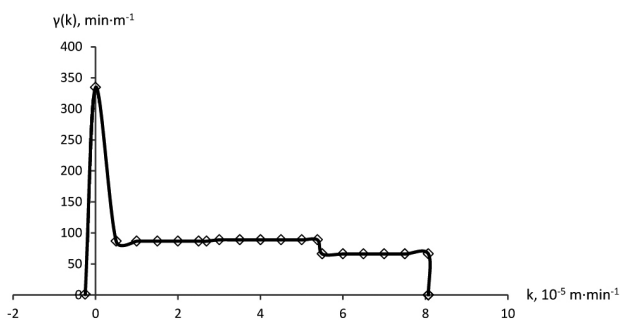


Fig. 2. Fractional composition of the copper ore sample by floatability with flotation agents mixture of AFI-4 – potassium butyl xanthate at a lime consumption of 1000 g/t^{-1} , the air flow rate of 10 l/h^{-1}

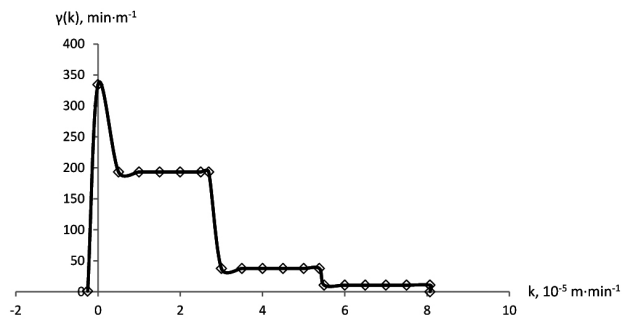


Fig. 3. Fractional composition of the copper ore sample by floatability with flotation agents mixture of AFI-4 – potassium butyl xanthate at a lime consumption of 1000 g/t^{-1} , the air flow rate of 20 l/h^{-1}

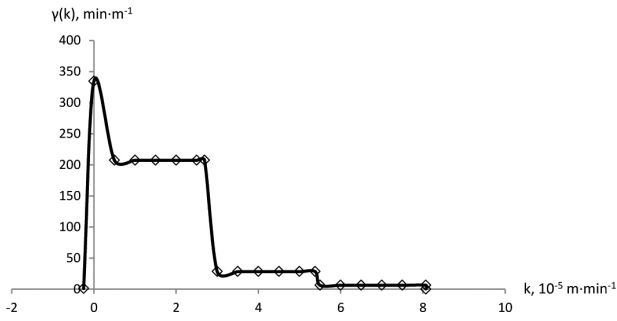


Fig. 4. Fractional composition of the copper ore sample by floatability with flotation agents mixture of AFI-4 – potassium butyl xanthate at a lime consumption of 1000 g/t⁻¹, the air flow rate of 40 l/h⁻¹

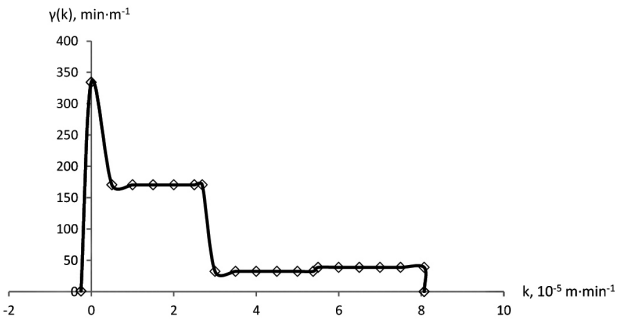


Fig. 5. Fractional composition of the copper ore sample by floatability with flotation agents' mixture of AFI-4 – potassium butyl xanthate at a lime consumption of 1000 g/t⁻¹, the air flow rate of 60 l/h⁻¹

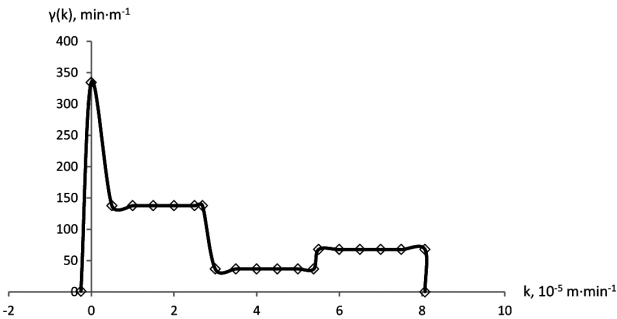


Fig. 6. Fractional composition of the copper ore sample by floatability with flotation agents mixture of AFI-4 – potassium butyl xanthate at a lime consumption of 1000 g/t⁻¹, the air flow rate of 70 l/h⁻¹

Analysis of the data presented in Fig. 2 showed that these conditions characterized by availability of only two fractions with medium and light flotation, the harnessing of AFI-4 – potassium butyl xanthate mixture permitting the difficult-to-float and moderately floatable fractions to be extracted. The fractions given above can contain chalcocite-copper oxides splices, whereas lightly floatable fraction is chalcopyrite.

However, a difference of 88 min/m⁻¹ for the distribution function between medium and lightly floatable fractions indicates a low selectivity of the reagent under the given conditions. It is caused by the insufficient oxidation degree of minerals and the surface proportion occupied by the collector hydrophobic molecules, correspondingly.

From the data in Figs. 3 and 4 it is seen that increasing the air feed rate from 20 to 40 l/h⁻¹ does not lead to significant changes in the fractional composition of the pulp.

Consequently, at the indicated conditions (Figs. 3, 4) the gradient of the distribution function between difficult-to-float and moderately floatable fractions increases, that results from the chalcopyrite oxidation to copper and iron oxides, followed by lime depressions and the formation of strong metal hydroxides. Whereas for chalcocite, as less stable mineral, is characterized by the oxidation to sulfate and thiosulphate ions, which are replaced by anions of collectors.

According to the results shown in Fig. 5, the difficult-to-float fraction decreases in share, the share of the moderately floatable fraction remains practically constant, while the lightly floatable fraction increases in share.

It has been revealed that at the indicated conditions, the maximum yield of difficult-to-float fraction is caused by redox processes at the metal sulphide surface and formation of sufficiently strong hydrophobic layer on the particles of mixed composition (rich intergrowths cuprite-chalcocite, chalcopyrite-cuprite).

At the same time, a share of low fraction of the lightly floatable fraction was associated with the formation of sparingly soluble iron hydroxides on the chalcopyrite surface [8] which sharply reduce the collector sorption amount.

This also was associated with the shift of equilibrium to sideways formation of more stable compound which has high value of the average atomic Gibbs energy [7] and energy density [9].

With reference to Fig. 6, it can be seen that increasing the air feed rate leads to the increase in the share of the lightly floatable fraction, and does not have impact on the distribution of the moderately floatable fraction, and reduces the share of the difficult-to-float fraction.

In the presence of strong and weak collectors the number of poor splices containing both copper oxides and sulphides has been passed into concentrate increases, due to sulphides oxidation and the formation of hydroxides on the copper oxides surface, whereas the number of ordinary splices in the concentrate remains practically constant.

Conclusions and recommendations for further research. Thus, the mathematical models adequately describing the flotation process for all the chemical-technological parameters (concentrate yield, metal content in the concentrate, recover degree) were obtained as a result of performing research studies on the collector mixture (AFI-4 and potassium butyl xanthate) flotation ability in relation to copper ore samples. Optimal conditions for conducting flotation and obtaining a collective concentrate were determined. Flotometric analysis of the enrichment process was carried out. It made it possible to establish the presence of three fractions, namely, difficult-to-float, moderately floatable, and lightly floatable in the ore sample.

It was shown that the difference in enrichment is determined by the composition of the fraction, the oxida-

tion-reduction processes in alkaline and strongly alkaline pH regions.

Therefore, the criteria for the selectivity of the collectors will be the pulp flotation ability; the controlling factors are the air flow rate, the pH of the medium, the impeller rotation frequency, and the collector consumption.

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Мета. Встановлення критеріїв селективності флотаційного збагачення мідьвмісних руд сумішшю сульфідрильних збирачів.

Методика. Атомно-абсорбційний, рентгенофлуоресцентний аналіз.

Результати. Проведено дослідження флотаційної здатності сумішей збирачів бутилового ксантогенату калію та ізобутілдітіофосфату натрію по відношенню до зразків мідьвмісної руди. Отримані математичні моделі, що описують процес збагачення мідної руди. Визначені оптимальні умови здійснення пінної флотації: швидкість подачі повітря 40 л/год, частота обертання імPELLера 35 Гц, витрата вапна 3000 г/т., витрата суміші збирачів 100 г/т.

Наукова новизна. Уперше отримані рівняння регресії, що моделюють вплив кількості утримуваного пульпою кисню, рН середовища, часу контакту твердої фази з розчином сумішей збирачів, концентрації суміші збирачів. Проведено флотаційний ана-

ліз системи „мідна руда – розчин суміші збирачів“. Встановлено вплив швидкості подачі повітря на фракційний склад пульпи за флотуємістю, полягає у зміні окисно-відновного потенціалу, що приводить до зміни міцності закріплення збирачів на поверхні руди.

Практична значимість. Отримані математичні співвідношення дозволять проводити прогноз зміни хіміко-технологічних параметрів збагачення під впливом швидкості подачі повітря, витрати регулятора середовища й суміші збирачів, частоти обертання імPELLера. Результати флотаційного аналізу можуть бути використані в розробці та вдосконаленні технологічних схем.

Ключові слова: мідна руда, рентгенофлуоресцентний аналіз, бутилований ксантогенат калію, діізобутілдітіофосфат натрію, пінна флотація, математичні моделі, флотаційний аналіз

Цель. Установление критериев селективности флотамионного обогащения медьсодержащих руд смесью сульфидрильных собирателей.

Методика. Атомно-абсорбционный, рентгенофлуоресцентный анализ.

Результаты. Проведены исследования флотационной способности смесей собирателей бутилового ксантогената калия и изобутилдитиофосфата натрия по отношению к образцам медьсодержащей руды. Получены математические модели описывающие процесс обогащения медной руды. Определены оптимальные условия осуществления пенной флотации: скорость подачи воздуха 40 л/ч, частота вращения имPELLера 35 Гц, расход извести 3000 г/т., расход смеси собирателей 100 г/т.

Научная новизна. Впервые получены уравнения регрессии, моделирующие влияние количества удерживаемого пульпой кислорода, рН среды, времени контакта твердой фазы с раствором смесей собирателей, концентрация смеси собирателей. Проведен флотационный анализ системы „медная руда – раствор смеси собирателей“. Установлено влияние скорости подачи воздуха на фракционный состав пульпы по флотуємістю, которое заключается в изменении окислительно-восстановительного потенциала, приводящее к изменению прочности закрепления собирателей на поверхности руды.

Практическая значимость. Полученные математические соотношения позволят проводить прогноз изменения химико-технологических параметров обогащения под влиянием скорости подачи воздуха, расхода регулятора среды и смеси собирателей, частоты вращения имPELLера. Результаты флотационного анализа могут быть использованы в разработке и усовершенствовании технологических схем.

Ключевые слова: медная руда, рентгенофлуоресцентный анализ, бутилованный ксантогенат калия, диізобутилдітіофосфат натрію, пенная флотація, математические модели, флотационный анализ

Рекомендовано до публікації докт. хім. наук С. Н. Нікольським. Дата надходження рукопису 19.10.16.