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# Development of enrichment reagent regime of Py-Cu-Zn ore using collectors' mixtures

Abstract. The article presents results of flotation enrichment of pyrite-copper-zinc ore in a selective mode. It allows predicting the choice of technology for enrichment of non-ferrous metal ores based on mathematical models describing the influence of hydrodynamic and reagent modes on the flotation parameters of the ore under study. The use of a mixture of flotation reagents excludes the use of special foaming agents in the process of selective flotation enrichment because the function of foaming agents and collectors is performed by flotation reagents. At the same time, an increase in the yield of the concentrate and the degree of metal extraction into the concentrate is achieved, and an increase in the selectivity of metal extraction and the degree of flotation enrichment of polymetallic ore. As a result of the processing and analysis of data on the flotation collective-selective enrichment of pyrite-copper-zinc ore, there have been developed recommendations for the technological evaluation of the choice of a selective reagent mode of flotation enrichment of ores.

**Key words:** pyrite-copper-zinc ore, flotation reagents, sodium diisopropyldithiophosphate, sodium diisobutyldithiophosphate, flotation scheme, reagent mode.

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#### Introduction

The development of the world economy is constantly accompanied by an increase in the consumption of fuel energy and other types of mineral resources. The consumption of non-ferrous and alloying metals has increased 3-5 times over the past 100 years. In the XXI century there continues the intensive growth of consumption of almost all types of mineral raw materials. It is predicted that in the next 50 years the volume of mining operations will increase by 5 times.

At the same time, the quality of processed ores and the content of metals in them continuously decrease. Over the past 20 years, the content of non-ferrous metals in ores has decreased by 1.3-1.5 times, the share of difficult-to-refiner ores has returned up to 40% of the total mass of raw materials entering enrichment. Ore processing is involved in processing, characterized by a low content of valuable components, fine dissemination, and close technological properties of minerals. On the other hand, a cycle of natural raw materials can be extended both to individual technological processes and the production of products because a wide range of wastes is generated in various industries. Therefore, it is necessary to focus on the sources of waste, according to the data provided by the Statistical Office of the European Union and North American industry classification system (NAICS)) [1-2].

Also Waste containing heavy metal ions, according to the FCCW, has the code 9 41 406 13 31 3 / 94140613313;, the transcript is given below: Block 9. Waste from performing other activities that are not included in blocks 1-3, 6-8 / Waste from technical tests, measurements, research / Laboratory waste and chemical residues / Waste of inorganic salts and their mixtures during technical tests and measurements / waste of aqueous solutions of heavy metal salts, including chromium salts (VI), during technical tests and measurements (the total content of heavy metals is less than 10%), is waste of hazard class III, (31) Aggregate state, physical form is liquid in liquid / Emulsion. The issues of resource-saving can be solved not only by recycling waste, but also by the complex use of ore raw materials and water in separating mineral raw materials.

### **Experimental procedures**

Pyrite-copper-zinc ore with a grain size of 0.074 mm was used in the work. The collectively selective flotation of Py-Cu-Zn ore, a mixture of sodium diisopropyl dithiophosphate and sodium diisobutyldithiophosphate was carried out in ratio 1:1 as collector, variable parameters change of the hydrodynamic (impeller rotation frequancy, air flow rate) and reagent regimes (medium regulator's consumption, collector consumption) at the flotation: airflow rate of 20-60 l/h, impeller rotation frequancy of 30-40 Hz, lime consumption of 1000-3000 g/t, collector consumption of 50-150 g/t. Ore enrichment was carried out on a laboratory chamber-type flotation machine with mechanical mixing of the FML brand according to the method [3]. Elemental analysis was performed on the ore grade instrument (Spektrolab). FT-IR spectroscopic analysis of the samples was conducted on the FSM 1201 device.

#### Results and discussion

It was shown that using a mixture of sodium diisopropyldithiophosphate and sodium diisobutyldithiophosphate in the selective flotation and separation of copper-zinc concentrate under the following conditions: air flow rate 20 l/h, impeller rotation frequancy 25 Hz, collector consumption 50 g/t, soda consumption 700 g/t allows to extract copper in an amount of 88%. It should be noted that the predominant influence on the concentrate quality is provided by the flotoreagent consumption. It has been shown that a high copper content in the concentrate at low concentrations of flotoreagent in 2 mg/l (50 g/t) pulp is associated with the formation of a monomolecular layer of a chemically adsorbed collector on the copper minerals' surface [4].

Further increase in the collector consumption leads to copper complexes formation desorbed into the solution volume and, as a result, to an increase in hydrophilicity. Thus, the probability of securing the particle on the bubble decreases. On the other hand, the increase in hydrophilicity drastically reduces the yield of copper-containing particles in the concentrate at low air flow rates.

To confirm the above results, IR-spectra were obtained. The collective concentrate samples of the Py-Cu-Zn ore flotation after its interaction with AFI-4 (sodium diisobutyldithiophosphate) in a mixture with TAF-7 (sodium diisopropyldithiophosphate) recorded absorption bands at wave numbers of 547 cm-1 (based on the FT-IR-spectroscopy), which are characteristic of the -S-S- bonds in alkyl disulfides. The absence of absorption bands of the P=S group (700-650 cm-1) suggests the formation of Cu complexes with diisobutyldithiophosphate and diisopropyldithiophosphate ion. Consequently, dialkyldithiophosphate complexes are formed on the minerals' surface, bound by strong coordination bonds with non-ferrous sulfides and physically sorbed disulphides, which causes high surface hydrophobicity [5].

These processes cause a sufficiently high recovery of copper in the collective concentrate at a soda consumption of 700 g/t, the impeller rotation frequency of 25 Hz. Whereas in order to achieve a high zinc extraction rate of 85%, and air flow of 50 l/h, an impeller rotation frequency of 25 Hz, collector consumption of 75 g/t, soda consumption of 1750 g/t are necessary. It was found that the separation process of collective concentrate in the sphalerite depression consists in the fine particles' formation, which is fixed on the active centers of the ore surface free from collector molecules. It is shown that potassium ferrocyanide can be used as effective depressor together with zinc sulfate. In this regard, the reagent mode of feeding a mixture of sodium diisopropyldithiophosphate and sodium diisobutyldithiophosphate into selective copper flotation was changed, soda should be used as a medium regulator, and potassium ferrocyanide should be used for the depression stage of zinc-containing minerals. Based on the above, recommendations for a technological evaluation of the choice of selective reagent enrichment (flotation) regimes of Fe-Cu-Zn ore were developed using separate collectors and/or their mixtures.

The optimal conditions for Py-Cu-Zn ore flotation using a mixture diisopropyldithiophosphate - potassium butylxanthate, providing the maximum pulp separation rate are: air flow rate of 35 l/h, impeller rotation frequency of 28.75 Hz, lime consumption of 1.5 kg/t, flotoreagent consumption of 0.26 kg/t. It is shown that the separation rate of solid into a foam product depends on the strength of the particle fixation on the bubble, which is completely determined by the hydrophobicity degree of the surface layer consisting of adsorbed collector complexes on the surface [6].

From the position of hydrodynamics, the rate of ascent of the bubble-particle complex influences the indicated parameter under the turbulent motion regime of the fluid, which in turn depends on the density of the pulp [7].

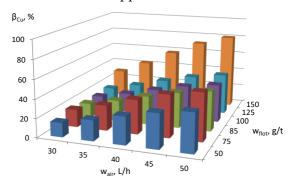
Further, the values of the flotation process rate of of Py-Cu-Zn ore were calculated with a variation of air flow rate and impeller rotation frequency from 25 to 40 Hz, with an optimum lime consumption of 1.5 kg/t and flotoreagent consumption of 0.26 kg/t. It is shown that a decrease in the residence time of bubbles in the chamber and, correspondingly, a decrease in the mineralization time and the collisions' number of the particle with bubble with the parallel motion of the phases leads to a decrease in the flotation rate of individual classes. The longer the residence time of the bubbles in the chamber is the higher the flotation rate is. Therefore, the values of rate constants of the flotation process were calculated.

It is shown that the rate constant of flotation process increases with increasing flotation time, and also depends on collectors' mixture consumption, lime consumption. It has been revealed that the Py-Cu-Zn ore flotation occurs at a high rate if one of the following conditions is satisfied: Q or Er is large, the ratio E/E0 is small, then b=1/E. The more b is, the better the flotation process will be. Since the coefficients E, E0, and Q depend on flotrivability of particles, size of air bubbles, collector type, and hydrodynamics of flotation cell, it is the energy E that determines the probability of the formation of the flotation complex, which is the main parameter in predicting the choice of the technology for the enrichment of non-ferrous and precious metals [8].

It is shown that with increasing the soda consumption, the barrier energy of flotation adhesion increases from 3.381 to 6.334 kJ, b=2.95•10-4 and 1.5•10-4. This indicates a lower flotation capacity of dialkyldithiophosphates in a strongly alkaline medium (medium regulator consumption of 2100 g/t) at low impeller rotation frequancy, due to the formation of sparingly soluble iron and copper hydroxides that enhance the hydrophilicity of the particles.

An increase in the impeller rotation frequancy from 25 to 36.25 Hz also increases the energy barrier, which is associated with an increase in the bubble upflow speed, a decrease in the contact time of the particles with the air bubble, but at a rotation frequency of 40 Hz, the activation energy decreases, which is associated with the stabilization processes of the turbulent flow.

Figure 1 shows that the use of sodium diisopropyldithiophosphate - potassium butyl xanthate mixture in the selective flotation and separation of copper-zinc concentrates under the following conditions: air flow rate of 50 l/h, the impeller rotation frequency of 23 Hz, collector consumption of 75 g/t, lime consumption of 1100 g/t allows to extract copper in an amount of 83%.



1-0.075; 2-0.1; 3-0.25; 4-0.5; 5-0.75 моль/л

Figure 1. Effect of the consumption of mixture of sodium diisopropyl dithiophosphate-butyl xanthate and air consumption to the collective concentrate quality

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Based on the results of the collective flotation of Py-Cu-Zn ore samples using a mixture of sulfhydryl collectors with different sorption and complexing capacity (butyl potassium xanthate and TAF-7) with optimization of experiment number based on the probabilistic-deterministic approach, some generalized mathematical models describing ore enrichment process under the influence of hydrodynamic conditions, reagent mode have been obtained. The prevailing influence of impeller rotation frequency and air flow rate on the concentrate quality and metal recovery degree in concentrate are shown. Varying the medium regulator and collector consumptions under constant hydrodynamic conditions made it possible to establish that copper recovery into the collective concentrate is increasing with the increase of butyl xanthate consumption from 50 to 150 g/t.

Copper extraction is 85% at butyl xanthate consumption of 25 g/t, and 96% - at 75 g/t. As a result, the optimal consumption of butyl xanthate is 50 g/t. Technological schemes and separation modes of sulfide minerals with similar flotation properties have been developed using separate collectors and/or their combinations based on pilot laboratory tests. Table 1 shows the results of Py-Cu-Zn enrichment according to the collective-selective scheme.

Table 1
Results of collectively selective enrichment of Py-Cu-Zn ore using butyl potassium xanthate
and sodium diisopropyldithiophosphate as main reagent

Product	γ,%	β, %		ε, %	
		Cu	Zn	Cu	Zn
Cu	6.12	9.64	0.56	87.99	10.38
concentrate	0.12	7.04	0.50	07.55	10.50
Zn	4.92	0.99	5.71	7.27	85.12
concentrate	4.92	0.99	5.71	1.21	65.12
Tailings	88.97	0.04	0.02	4.74	4.50
Initial ore	100	0.67	0.33	100	100

The indicated results of calculations of flotation indices allow proposing a flotation scheme, according to which tailings of selective flotation are subjected to control flotation with additional extraction of copper minerals, as well as a technological map for the enrichment of Py-Cu-Zn ore (Table 2).

Table 2
The technological map of collectively selective enrichment of Py-Cu-Zn ore (separation modes of minerals with similar flotation properties)

Stage	Reagent	Consumption, g/t	Reagent	Consumption, g/t	Stage of reagent entrance
Mode 1 Mode 2  Impregnated ore, non-cyanide technology					entrance
	Soda	700	Lime	1100	II grinding degree (60%, - 0.074 mm)
Collective flotation	(i-C <sub>4</sub> H <sub>9</sub> O) <sub>2</sub> PS <sub>2</sub> Na -(i-C <sub>3</sub> H <sub>7</sub> O) <sub>2</sub> PS <sub>2</sub> Na T-92	50 15	(i-C <sub>3</sub> H <sub>7</sub> O) <sub>2</sub> PS <sub>2</sub> Na -C <sub>17</sub> H <sub>33</sub> COONa T-92	50 15	Main and control flotation

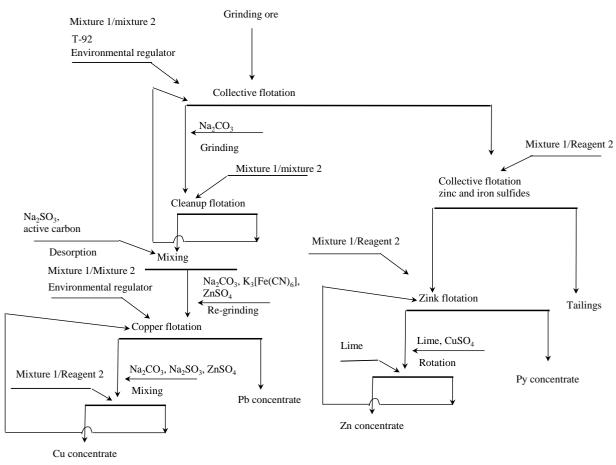
	Soda	700	Lime	1000	Re-grinding the collective concentrate (80% - 0.074 mm)
	(i-C4H9O)2PS2 Na -(i-C3H7O)2PS2 Na	5	(i-C3H7O)2PS2 Na -C17H33COONa	5	Cleanup flotation of the collective concentrate
	Na <sub>2</sub> SO <sub>3</sub>	1200	Na <sub>2</sub> SO <sub>3</sub>	1200	Mixing the collective
	Active carbon	120	Active carbon	120	concentrate
Preparation of collective concentrate to separation	Soda	1400	Lime	1000	Re-grinding the collective concentrate
	Potassium ferrocyanide	200	Na <sub>2</sub> SO <sub>3</sub>	200	Re-grinding the collective concentrate
	Zinc sulfate	500	Zinc sulfate	500	Re-grinding the collective concentrate
Copper flotation	(i-C <sub>4</sub> H <sub>9</sub> O) <sub>2</sub> PS <sub>2</sub> Na - (i-C <sub>3</sub> H <sub>7</sub> O) <sub>2</sub> PS <sub>2</sub> Na Na <sub>2</sub> SO <sub>3</sub>	8 200	(i-C <sub>3</sub> H <sub>7</sub> O) <sub>2</sub> PS <sub>2</sub> Na Na <sub>2</sub> SO <sub>3</sub>	8 200	Main and control flotation
	Soda Na <sub>2</sub> SO <sub>3</sub> Zinc sulfate	1750 150 250	Lime Na <sub>2</sub> SO <sub>3</sub> Zinc sulfate	1000 150 250	Mixing before cleanup flotation of concentrate
Zinc flotation	Lime Copper sulfate	2000 100	Lime Copper sulfate	2000 100	Mixing
	(i-C4H9O)2PS2 Na - (i-C3H7O)2PS2 Na	26	(i-C <sub>3</sub> H <sub>7</sub> O) <sub>2</sub> PS <sub>2</sub> Na	13	Main and control flotation
	Lime	2100	Lime	2100	Cleanup flotation of zinc concentrate

Figure 2 shows the technological scheme for flotation enrichment of ores, developed based on pilot laboratory tests, on the basis of which the following recommendations were proposed:

Py-Cu-Zn enrichment: it is recommended to use a mixture of 1  $(i-C_4H_9O)_2PS_2Na-(i-C_3H_7O)_2PS_2Na$  as a collector at a rate of 50 g/t (1:1) according to technological map (Table 2). The collector  $(i-C_4H_9O)_2PS_2Na-(i-C_3H_7O)_2PS_2Na$  is selective to copper, zinc and reduces losses of copper and zinc and improves the rough concentrate quality with copper and zinc recovery of 88% and 85%.

Optimal parameters are aeration rate of 35 l/h; impeller rotation frequancy of 22 Hz.

Py-Cu-Zn enrichment: it is recommended to use mixture 2 (i-C3H7O)2PS2Na-C17H33COONa as a collector at a rate of 50 g/t (1:1) according to technological map (Table 2). The collector (i-C3H7O)2PS2Na is selective to copper, zinc and reduces losses of copper and zinc and improves the rough concentrate quality with copper and lead recovery of 60% and 56%. Optimum parameters are aeration rate 50 l/h; impeller rotation frequancy of 22 Hz.



Mixture 1: (i-C<sub>4</sub>H<sub>9</sub>O)<sub>2</sub>PS<sub>2</sub>Na (25 g/t) - (i-C<sub>3</sub>H<sub>7</sub>O)<sub>2</sub>PS<sub>2</sub>Na (25 g/t);

Mixture 2: (i-C<sub>3</sub>H<sub>7</sub>O)<sub>2</sub>PS<sub>2</sub>Na (25 g/t) - C<sub>17</sub>H<sub>33</sub>COONa (25 g/t);

Reagent 2: (i-C<sub>3</sub>H<sub>7</sub>O)<sub>2</sub>PS<sub>2</sub>Na (8 g/t).

Figure 2. Modified scheme of collectively selective flotation of Py-Cu-Zn ore

The results obtained indicate an increase in the concentrate yield and metal extraction degree.

#### Conclusion

A correlation between metals extraction degree to concentrate and impeller rotation frequency, confirming the enhancement of the interaction of the collector with metal ions in the pulp at a high rotation frequency corresponding to the stationary turbulent regime of fluid motion was found. It has been established that the use of reagent AFI-4 (sodium diisobutyl dithiophosphate) as an additional flotation reagent in the enrichment of copper-lead ore makes it possible to achieve the maximum recovery of Pb (>70%) under the following conditions: impeller rotation frequency of 30 Hz, air flow rate of 20 l/h, lime consumption of 3000 g/t, reagent consumption of 3000 g/t, reagent consumption of 150 g/t.

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## Жинағыш қоспаларын қолдану арқылы Ру-Сu-Zn кенін байытудың реагенттік режимін әзірлеу

Аңдатпа. Колчедан-мыс-мырыш кендерін селективті режимде флотациялық байыту нәтижелері келтірілген, бұл зерттелетін кенді флотациялау көрсеткіштеріне гидродинамикалық және реагенттік режимдердің әсерін сипаттайтын математикалық модельдер негізінде түсті металл кендерін байыту технологиясын таңдауды болжауға мүмкіндік береді. Флотореагенттер қоспасын қолдану нәтижесінде селективті флотациялық байыту процесінде арнайы көбіктендіргіштерді пайдаланбайды, өйткені көбіктендіргіштер мен жинағыштардың функциясын флотореагенттер орындайды. Бұл концентраттың шығымдылығын және металды концентратқа алу дәрежесін арттыруға, сондай-ақ металды алудың селективтілігін және полиметалл кенін флотациялық байыту дәрежесін арттыруға мүмкіндік береді. Колчедан-мыс-мырыш кендерін флотациялық ұжымдық-селективті байыту жөніндегі мәліметтерді өңдеу және талдау нәтижесінде кендерді флотациялық байытудың селективті реагенттік режимін таңдауды технологиялық бағалау бойынша ұсынымдар әзірленді.

**Түйін сөздер:** Колчедан-мыс-мырыш кені, флотореагенттер, натрий диизопропилдитиофосфаты, натрий диизобутилдитиофосфаты, флотация сұлбасы, реагенттік режим.

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## Разработка реагентного режима обогащения Ру-Сu-Zn руды с использованием смесей собирателей

Аннотация. Приведены результаты флотационного обогащения колчеданно-медноцинковой руды в селективном режиме, который позволяет проводить прогнозирование выбора технологии обогащения руд цветных металлов на основе математических моделей, описывающих влияние гидродинамического и реагентного режимов на показатели флотации исследуемой руды. Применение смеси флотореагентов исключает использование в процессе селективного флотационного обогащения специальные пенообразователи, так как функцию пенообразователей и собирателей выполняют флотореагенты; при этом достигается увеличение выхода концентрата и степени извлечения металла в концентрат, а также повышение селективности извлечения металла и степени флотационного обогащения полиметаллической руды. В результате обработки и анализа данных по флотационному коллективно-селективному обогащению колчеданно-медно –цинковой руды разработаны рекомендации по технологической оценке выбора селективного реагентного режима флотационного обогащения руд.

**Ключевые слова:** колчеданно-медно-цинковая руда, флотореагенты, диизопропилдитиофосфат натрия, диизобутилдитиофосфат натрия, схема флотации, реагентный режим

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