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RESEARCH PAPER

Hydrometallurgical Processing of Substandard Mixed Copper Ore with a High Sulfide Copper Content

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ABSTRACT

The depletion of rich copper raw material reserves makes the processing of low-grade oxidized and mixed ores from Kazakhstan using SX - EW hydrometallurgical technology particularly relevant. The object of the study was low-grade mixed copper ore from one of the deposits in the Karaganda region, characterized by a high sulfide copper content. The article presents the results of physicochemical studies of the initial ore, its leaching with sulfuric acid at different concentrations, and investigations of copper extraction and stripping processes. The application of modern LIX-type oxime extractants was analyzed. Model solutions were used to study the characteristics of the extraction process that ensure high copper recovery and the subsequent production of strip liquors suitable for electrolytic metal recovery. The study established the effect of pH and extractant concentration on the degree of copper extraction, demonstrated the possible formation of copper complexes with the extractant, and determined their composition. An extractant was selected to study the recovery of copper from pregnant solutions. The data obtained confirmed the efficiency of the SX-EW hydrometallurgical technology for processing substandard mixed copper-bearing ore with high sulfide copper content.

Keywords: copper, copper-bearing solution, extraction, extractant, organic phase, raffinate, stripping

INTRODUCTION

The depletion of the raw material base and the limited reserves of high-quality copper ores require the involvement of substandard raw materials in processing. This task has important economic significance for the Republic of Kazakhstan. Its solution requires the introduction of modern geotechnological and hydrometallurgical methods, enabling the processing of reserves previously considered unprofitable.

In recent years, copper production has shown a clear trend toward the development of hydrometallurgical methods, which are more economical and safer for the environment than conventional pyrometallurgical processes. Solvent Extraction - Electrowinning (SX-EW), which combines liquid-liquid extraction and electrowinning, has become the most widely used technology.

The range of raw materials involved in production using this technology is expanding every year. Earlier, this technology was used only to process oxidized copper ores. Now, it is increasingly applied to process mixed ores with different ratios of oxidized and sulfide forms.

SX-EW technology comprises four main stages: copper leaching, extraction of copper into the organic phase, stripping, and electrolytic deposition. The leaching process is the key stage because its efficiency largely determines the performance of subsequent stages and the technology's overall economic efficiency. The extraction-stripping stage is also important because a poorly selected extractant can significantly reduce overall recovery. In this regard, the present study aims to optimize the technological parameters and select rational conditions for the leaching and extraction-stripping processes.

The development of extraction technologies was made possible by the creation of highly selective extractants for copper. Foreign companies, including General Mills, Ashland Chemical, Shell, Henkel, and Cognis, as well as research institutes of the former USSR, including VNIIKhT, Gintsvetmet, and Gidrotsvetmet, made a significant contribution to their development. Oxime extractants of various types, such as Lix 984, Lix 984N, Lix 84-I, and others, are widely used [1-4]. At the early stages of research, various classes of organic reagents were considered, including carboxylic, naphthenic, and alkylphosphoric acids. However, their application was limited by low selectivity and high solubility in the aqueous phase. This limitation led to a shift toward celebrating extractants that provide high selectivity for copper [5-8].

A wide range of effective chelating extractants for copper is available, and Lix-type reagents and Cytec reagents have found broad application in the copper industry [9, 10]. A significant number of studies have focused on extracting copper from sulfate solutions using Lix-type extractants [10-12].

Modern oxime extractants form stable complexes with copper ions through coordination via oxime and hydroxyl groups. Their use enables efficient extraction of copper from acidic leach solutions at low pH, yielding high recovery and good phase separation [13-18]. Study [18] examined the applicability of SX-EW technology to overburden and waste rock from the copper deposits of Cerovo, Eastern Serbia. A decrease in acidity from 2.0 to 1.0 increased copper extraction from 68% to 79% over a duration of 120 hours. The addition of iron (III) as an oxidizing agent to the leaching solution had a strong effect on chalcopyrite solubility.

Henkel developed a new generation of extractants for copper recovery. Earlier systems were based on aldoximes combined with alcohols and phenols; for example, Lix-622 is a mixture of 5-dodecylsalicylaldoxime and tridecanol. In contrast, modern formulations consist of combinations of ketoxime and aldoxime components. This approach helps avoid the formation of interfacial sludge, which is typical of conventional mixtures.

Lix-864 is one such extractant. It is a composition of a ketoxime, 5-nonylphenol-2-hydroxybenzophenone oxime, and the aldoxime 5-DDSA. At low levels of organic phase loading, ketoximes act as modifiers and simultaneously reduce the system's tendency to form interfacial sludge. The Lix-864 extractant has found industrial application, and it has been introduced at seven enterprises over the past two years. At the same time, several production facilities continue to use aldoxime and ketoxime extractants. Further development in this area led to the creation of the improved extractant Lix-984, a mixture of 5-nonyl-2-hydroxyacetophenone oxime and 5-DDSA. This reagent offers greater selectivity and a higher rate of copper extraction, making it promising for industrial applications [13-18].

Thus, the brief review presented above shows the direction of research aimed at improving the extraction stage of hydrometallurgical technology. The leaching process also remains highly relevant because processing low-grade ores dominated by oxide copper is well known, whereas processing mixed oxide-sulfide ores in production, especially those with high sulfide copper content, has been studied only to a very limited extent.

In this regard, the present work aims to investigate the applicability of SX-EW technology for recovering copper from substandard mixed ore with high sulfide copper content by justifying the selection of an extractant and subsequently confirming its efficiency using process solutions.

MATERIAL AND METHODS

Substandard, mixed, copper-bearing ore from one of the deposits in the Karaganda region was used as the study subject. Physicochemical studies of the initial ore were conducted using the following instruments.

X-ray fluorescence analysis (XRF) was used to determine the elemental composition and was performed on a Venus 200 spectrometer (PANalytical B.V., Netherlands) based on the wavelength-dispersive principle.

Inductively coupled plasma optical emission spectrometry (ICP-OES), performed on an Optima 8300 DV spectrometer (PerkinElmer, USA), was used to quantitatively analyse the initial ore samples. The measurement error did not exceed 2.0%.

X-ray phase analysis was used to identify crystalline phases and was performed on a D8 Advance diffractometer (Bruker, USA) with Cu Kα radiation at 40 kV and 40 mA. EVA software and the PDF-2 powder diffraction database were used to process the diffraction patterns, calculate interplanar spacings, and identify the phases.

Microphotographs of the ore samples were obtained using a JSM-6610LV scanning electron microscope (JEOL) with a LaB6 thermionic cathode in low-vacuum mode. The microscope was equipped with detectors for energy-dispersive spectroscopy (EDS) and wavelength-dispersive spectroscopy (WDS) microanalysis, an electron backscatter diffraction (EBSD) analysis system, and backscattered and secondary electron detectors, including a detector for low-vacuum mode.

Fourier-transform infrared spectroscopy (FTIR) was performed on an Avatar 370 CsI spectrometer with a Transmission E.S.P. Accessory.

Mineralogical analysis was performed using an AxioScope.A1 microscope. A polished section was prepared from the sample material to conduct mineralogical analysis.

The quantitative copper content was determined using an AA240 atomic emission spectrometer manufactured by Varian Optical Spectroscopy Instruments, Australia.

The initial ore was leached using both agitation and percolation methods. Agitation leaching was performed for 90 minutes at a solid-to-liquid ratio of 1:4 after the ore had been ground to a particle size of -0.1 mm. The sulfuric acid concentration was 5, 10, and 15 g/dm3. Percolation leaching of the ore is shown in Figure 11.

The extraction process was studied using model solutions. Copper and iron salts were used to prepare model aqueous solutions because iron is the primary impurity in the ore. The reagents used to prepare the model solutions were of analytical grade or chemically pure grade. Sulfuric acid or sodium hydroxide solutions were used to adjust the pH values. The composition of the aqueous solutions was modeled considering the composition of the initial ore and the objectives of the study.

LIX 984 and LIX 864 reagents were selected as extractants. KO-30 kerosene was used as the diluent. The organic phase consisted of extractant solutions in kerosene at a concentration of 2.0 × 10-1 M.

The metal-ion extraction capacity of the extractant was determined using the saturation method. The extraction phase ratio was O = 1:0.5, and the phase contact time for one cycle was 2 minutes. Stripping was performed at an O ratio of 1:1 and a phase contact time of 10 minutes.

The experiments on copper extraction from pregnant solutions were performed in 12 cycles. The extracts obtained were analyzed for copper content and acidity. The copper concentration in the organic phase was determined from the metal balance, accounting for the difference between the initial solution and the extract.

The component analyses were performed using generally accepted methods. The metal concentration in the aqueous phase was determined directly, whereas the metal concentration in the organic phase was determined after stripping into an aqueous solution. Stripping was performed using 5-20% sulfuric acid solutions, and the organic phase was washed with water to remove acid impurities. The pH values of the solutions were determined using an IP67-class pH meter.

The composition of the extracted complex was determined using the equilibrium shift method, the isomolar series method (Job-Ostromyslensky method), and the molar ratio method.

RESULTS AND DISCUSSION

At the first stage, an extractant was selected for the subsequent studies. For this purpose, the comparative characteristics of LIX 984 and LIX 864 extractants were determined using model solutions. The comparison of our experimental results with published data for the two extractants shows that the pH value required for copper extraction with LIX 984 is significantly lower than that required for LIX 864. This feature makes it possible to work efficiently with more acidic solutions without additional neutralization **(Fig. 1)**.

The comparison of copper extraction isotherms showed that LIX 984 can efficiently extract copper even at low pH values while maintaining high organic-phase loading **(Fig. 2)**. The degree of copper extraction with LIX 984 can reach 80%. At the same time, LIX 864 achieves similar indicators only at higher pH values, which limits its application in acidic solutions. The copper loading capacity of LIX 984 reaches 6.0-6.5 g/dm3, which is higher than that of LIX 864. The distribution coefficient (D) confirms the higher loading capacity of LIX 984 (**Fig. 3**).

Fig. 1 shows the effect of the pH of the aqueous solution and the concentration of LIX 984 in the organic phase on the degree of copper extraction. The curves are presented for pH values of 3.3, 2.8, 2.4, 2.1, and 1.8. The figure shows that increases in pH and extractant concentration increase the degree of copper extraction. More acidic conditions noticeably reduce the efficiency of the process. This factor directly reduces the number of extraction stages required in an industrial flowsheet.

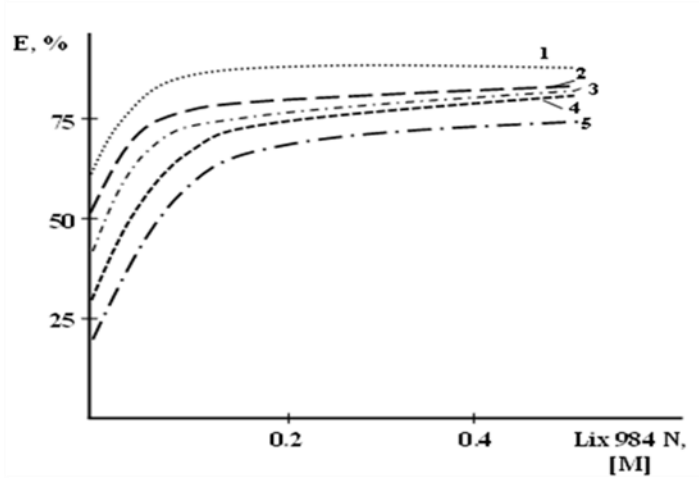

Fig. 1. Effect of the pH of the aqueous solution and the concentration of LIX 984 in the organic phase on the degree of copper extraction. pH: 1, 3.3; 2, 2.8; 3, 2.4; 4, 2.1; 5, 1.8

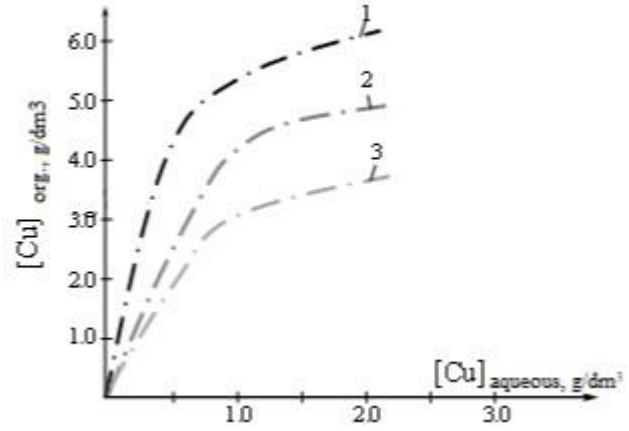

Fig. 2. Copper loading isotherms of 10 vol. % LIX 984N in kerosene depending on the pH of the aqueous solution: 1) pH = 2.1; 2) pH = 1.5; 3) pH = 1.3.

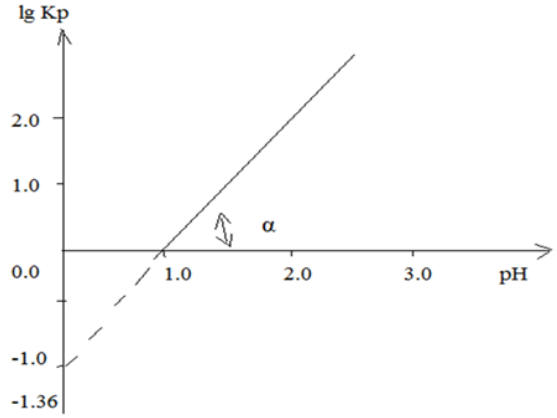

Fig. 3 Copper distribution coefficient as a function of the pH of the equilibrium aqueous phase during extraction with substituted 2-hydroxybenzophenone oximes

The studies were performed to determine the optimal concentration of LIX 984 in the organic phase (**Fig. 2**) and the dependence of D_Cu on the pH of the aqueous phase (**Fig. 3**). **Fig. 2** presents the copper-loading isotherms for LIX 984 in kerosene at different aqueous-phase acidity levels. The curves correspond to pH values of 2.1, 1.5, and 1.3. The figure shows that an increase in pH increases the copper loading capacity of the organic phase, which may be associated with more favorable conditions for complex formation. Lower pH values reduce the degree of loading because hydrogen ions compete with copper ions for the extractant.

Fig. 3 shows the dependence of the copper distribution coefficient (D) on the pH of the equilibrium aqueous phase during extraction. The results show that increasing pH increases the distribution coefficient, indicating enhanced transfer of copper into the organic phase. This dependence reflects the key role of medium acidity in the extraction process.

Thus, the studies showed that LIX 984 has a high copper extraction capacity and achieves high extraction in acidic media. It is also characterized by a higher loading capacity of the organic phase. The efficiency of LIX 984 at low pH values and its potential to reduce the number of extraction stages justified selecting it as the primary extractant for further studies.

The next stage involved comparing the infrared spectra of the initial LIX 984 reagent **(Fig. 4)**, the LIX 984 solution in kerosene **(Fig. 5)**, and the copper-loaded extract **(Fig. 6).**

The spectrum of the initial extractant **(Fig. 4)** contains a set of characteristic bands corresponding to the functional groups of the organic reagent. In particular, the band at 1267 cm-1 corresponds to stretching vibrations of the C-N bond in ArNHR-type compounds and partly to vibrations of the C-O-H group.

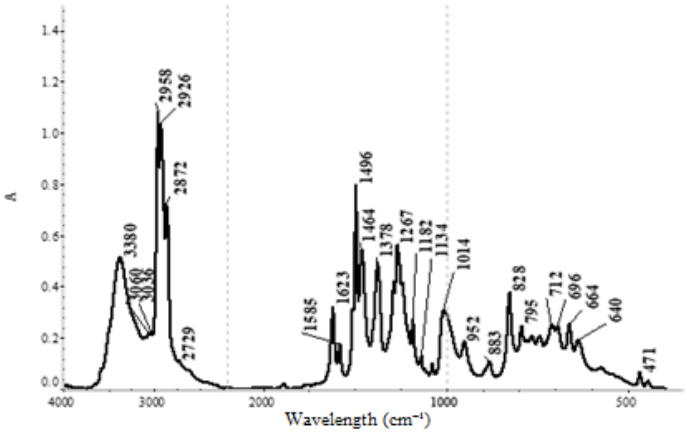

Fig. 4 Infrared spectrum of pure LIX

The transition to the extractant solution in kerosene **(Fig. 5)** causes almost no change in the positions of the main bands. The band corresponding to the indicated vibrations is recorded at 1,268 cm-1. This result indicates that dilution in the organic solvent does not significantly alter the structure of the reagent or its functional groups.

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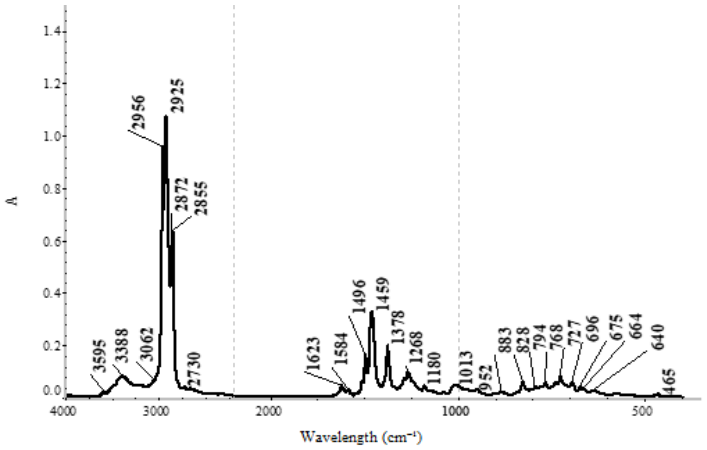

Fig. 5 Infrared spectrum of LIX 984 in kerosene

The most significant changes are observed in the spectrum of the copper-loaded extract (**Fig. 6**). A noticeable shift of the absorption band from 1,267 - 1,268 to 1,298 cm-1, approximately 30 cm-1 toward the high-frequency region, occurs in the range of 1,400 - 1,000 cm-1. This shift indicates a change in the electronic state of the donor atoms and confirms their participation in coordination with the copper ion.

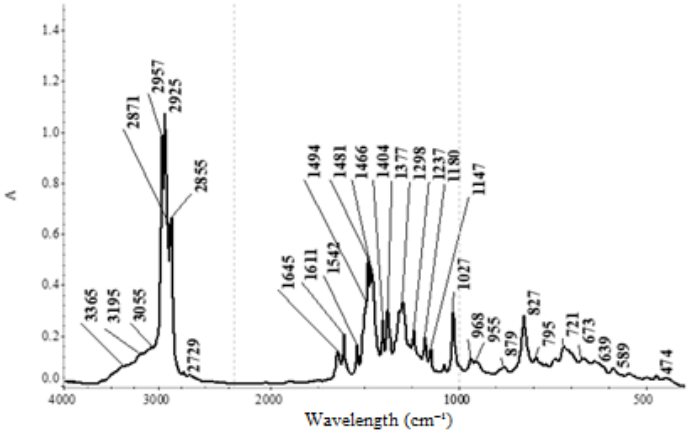

Fig. 6 Infrared spectrum of copper-loaded LIX 984

In addition, the preservation of the general spectral structure, with simultaneous displacement of individual bands, indicates that complex formation proceeds without destruction of the molecule's organic framework and is accompanied by a redistribution of electron density in the donor groups.

The absence of bands characteristic of coordinated water in all spectra, as well as the absence of new intense bands associated with hydrate forms, confirms that water molecules are not included in the coordination sphere of the complex. This finding supports the assumption that a planar CuR2 complex forms with a copper coordination number of 4.

The comparative analysis of the three spectra shows that the key sign of complex formation is a shift of the band in the 1,267 - 1,298 cm-1 region, reflecting the participation of LIX 984 functional groups in coordination with the copper ion.
At the next stage, studies were performed to determine the composition of the complex formed during copper extraction **(Fig. 7-9).**
Fig. 7 shows that an increase in copper concentration is initially accompanied by an increase in the copper content in the complex, after which this content reaches a limit indicating saturation of the organic phase.
Fig. 8 presents the determination of the composition of the extracted complex by the isomolar series method, or Ostromyslensky-Job method. The total concentration of the components was kept constant, and changes in lg E were recorded as their ratio was varied. The point of intersection of the straight lines drawn through the ascending and descending sections corresponds to the mole fraction of the components at which the most stable complex is formed.
The position of the maximum, $\chi(Cu) \approx 0.31 - 0.33$, was used to determine the stoichiometry of the complex.

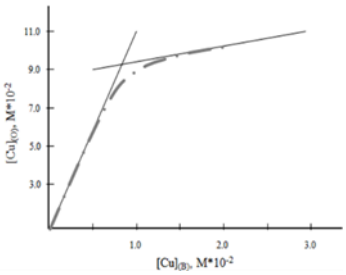

Fig. 7 Determination of the composition of the extracted complex by the molar ratio method

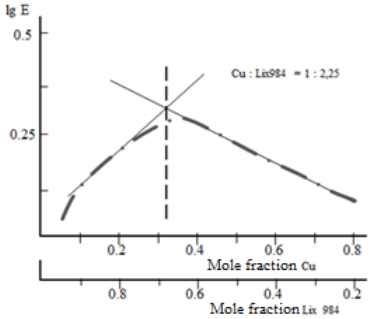

Fig. 8 Determination of the composition of the extracted complex by the isomolar series method, or Ostromyslensky-Job method

The Cu:LIX 984 ratio of 1:2.25 shown in the figure indicates that the extracted complex contains approximately two extractant molecules per copper ion, corresponding to a composition close to 1:2.

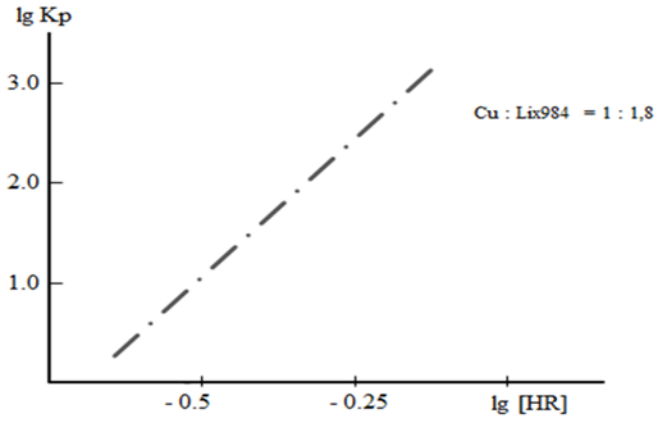

Fig. 9 Determination of the composition of the extracted complex by the equilibrium shift method

Fig. 9 shows the dependence of the logarithms of the distribution coefficient (lg K_p) and reagent concentration (lg [HR]). The linear nature of the plot indicates that the stoichiometry of the extracted compound remains constant in the concentration range investigated. The slope of the straight line is close to 1.8, which indicates that the component ratio in the complex is Cu:LIX984 ≈ 1:1.8. The data obtained confirms the formation of an extractable complex of variable composition close to the 1:2 stoichiometry.

Thus, the studies conducted supported the selection of LIX 984 as the preferred extractant, as it forms a copper complex of variable composition near 1:2 stoichiometry during extraction.
Further studies focused on the physicochemical characterization of the initial ore. According to the phase analysis (**Table 1**), quartz is the main component of the ore. Therefore, silicate rocks predominate in the ore, while albite, microcline, clinochlore, and muscovite occur in smaller amounts in descending order.

Table 1 Phase composition of the initial ore

Compound Name	**Formula**	**S-Q**
Quartz, syn	SiO_2	39.4%
Albite	$Na(AlSi_3O_8)$	21.5%
Microcline, intermediate	$KAlSi_3O_8$	16.8%
Clinochlore-1MIIb, ferroan	$(Mg,Fe)_6(Si,Al)_4O_{10}(OH)_8$	11.4%
Muscovite-2M1	$KAl_2(AlSi_3O_{10})(OH)_2$	10.8%

The elemental composition of the ore is presented in **Table 2**, which shows that silicon, potassium, calcium, sodium, magnesium, and iron are the main elements.

Table 2. Results of X-ray fluorescence analysis of the initial ore

O	Na	Mg	Al	Si	P	S	Cl	K	Ca
56.142	1.413	1.385	7.077	25.813	0.078	1.142	0.027	1.837	0.753
Mn	Fe	Ti	Zn	As	Rb	Sr	Zr	Ni	Pb
0.018	1.660	0.614	0.04	0.014	0.011	0.011	0.012	0.175	0.007

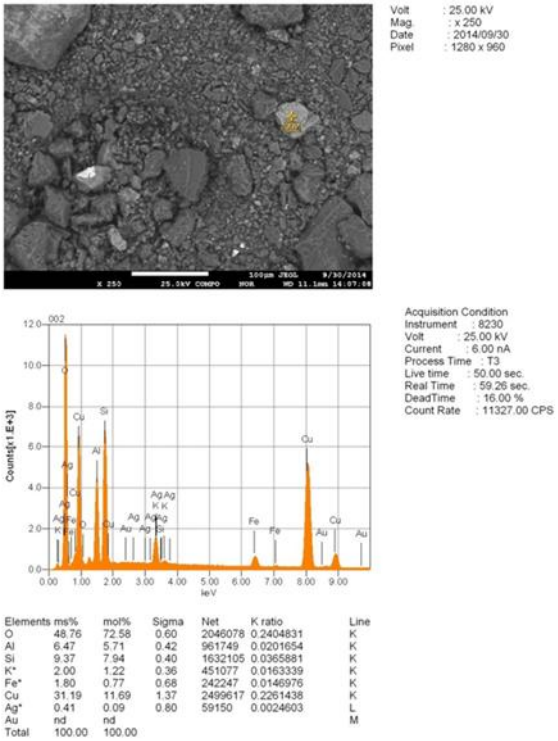

Fig. 10 Electron microscopic analysis of the initial ore. Fragments with the most pronounced atomic copper spectra

Chemical analysis showed that the copper content in the ore sample was 0.399%, while the oxide phase accounted for 0.09-0.1%. Therefore, most copper ores occur as sulfides.
The presence of the main components was confirmed by the results of ore studies performed using a scanning electron microscope **(Fig. 10).**

According to the mineralogical analysis, the sulfide phase consists of chalcopyrite, chalcocite, and bornite, with chalcocite being the predominant mineral. The oxide phase is represented mainly by azurite.
Preliminary agitation leaching of the initial ore was performed for 90 minutes at a solid-to-liquid ratio of 1:4 after the ore had been ground to a particle size of -0.1 mm. The process was used to study the efficiency of copper extraction at sulfuric acid concentrations of 5, 10, and 15 g/dm3. The results obtained are presented in **Table 3**.

Table 3 Results of agitation leaching of the initial ore

H_2SO_4 concentration, g/dm^3	Initial Cu, %	Sample weight, g	Cu concentration, g/l	Solution volume, L	Cu extraction, %
5.0	0.377	100	0.35	0.37	33.89
10.0	0.377	100	0.4	0.37	39.26
15.0	0.377	100	0.48	0.36	45.84

The table shows that the best results were obtained when the ore was leached with sulfuric acid at a concentration of 15 g/dm3.
The percolation leaching method was selected to reduce the acid concentration and achieve more complete copper extraction **(Fig. 11)**.

Fig. 11 Pilot setup for copper extraction by the percolation method 1, percolator; 2, pregnant solution collection tank; 3, peristaltic pump; 4, solution storage tanks; 5, dispensers; 6, extraction chambers; 7, stripping chambers; 8, raffinate discharge tank

The studies tested two variants of ore sample leaching: ore crushed to a particle size of -25 mm and ore in the form of dump material with a particle size of 150-200 mm (**Fig. 12**).

Crushed ore, 25 mm

Uncrushed dump material, 150 - 200 mm

Fig. 12 Representative ore samples used in the experiments

The results obtained are presented in **Tables 4 and 5.**

Table 4 Leaching of crushed ore

Days	Cu, g/L	H_2SO_4 in pregnant solution, g/L	H_2SO_4 consumption, g per 1 g Cu	Extracted Cu mass, g	Cu extraction, %
10	5.2	10.3	10.39	244.4	30.29
20	6.8	11.0	11.19	319.6	39.61
30	7.8	13.2	13.65	358.8	44.47
40	8.3	15.0	13.51	398.4	49.38
50	9.2	15.0	14.21	414.0	51.32
60	9.0	15.0	14.20	432.0	53.55

Table 5 Leaching of ore in the form of dump material

Days	Cu, g/L	H_2SO_4 in pregnant solution, g/l	H_2SO_4 consumption, g per 1 g Cu	Extracted Cu mass, g	Cu extraction, %
10	4.7	12.3	9.21	216.2	27.84
20	6.5	11.0	9.11	299.0	38.50
30	7.8	14.2	10.88	358.8	46.20
40	9.4	15.0	9.36	441.8	56.89
50	11.1	15.0	9.09	499.5	64.32
60	10.7	15.0	9.44	513.6	66.13

Tables 4 and 5 show that copper was leached more actively from crushed ore during the first 20 days, whereas copper leaching from dump material became more intensive over time. This factor is associated with the gradual dissolution of copper from large mineral fragments during acid irrigation. The study also established that additional ore crushing does not improve copper extraction in percolation leaching because finer crushing increases sulfuric acid consumption by gangue minerals.
The pregnant solutions obtained during leaching were tested to determine their suitability for subsequent technological stages, including extraction and stripping. LIX 984 was used as the extractant, as described above. The raffinate obtained after extraction, as well as the water used to wash the organic phase, can be recycled to the ore leaching stage.

The average composition of the strip liquor obtained during the studies is given in **Table 6.**

Table 6 Chemical composition of the strip liquor

Cu, g/L	Au, mg/L	Ag, mg/L	Fe, g/L	Zn, mg/L	Pb, mg/L	As, g/L	H_2SO_4, g/L
45.2	0.04	0.02	0.001	0.015	0.018	0.003	182.4

Strip liquors of this composition are fully suitable for the electrolytic production of commercial cathode copper. The use of the LIX 984 extractant enabled the preparation of an electrolyte solution containing 45 g/dm3 copper and 180 g/dm^3 sulfuric acid, with minimal impurity content. This composition enables the production of high-quality cathode copper with a purity of at least 99.99%.
Based on the conducted studies, we proposed the following conceptual process flowsheet for the hydrometallurgical recovery of copper from this ore (**Fig. 13**).
Thus, the studies conducted and the results obtained confirmed the feasibility of applying hydrometallurgical technology to efficiently process substandard mixed copper-bearing ore with the stated composition and high sulfide copper content.

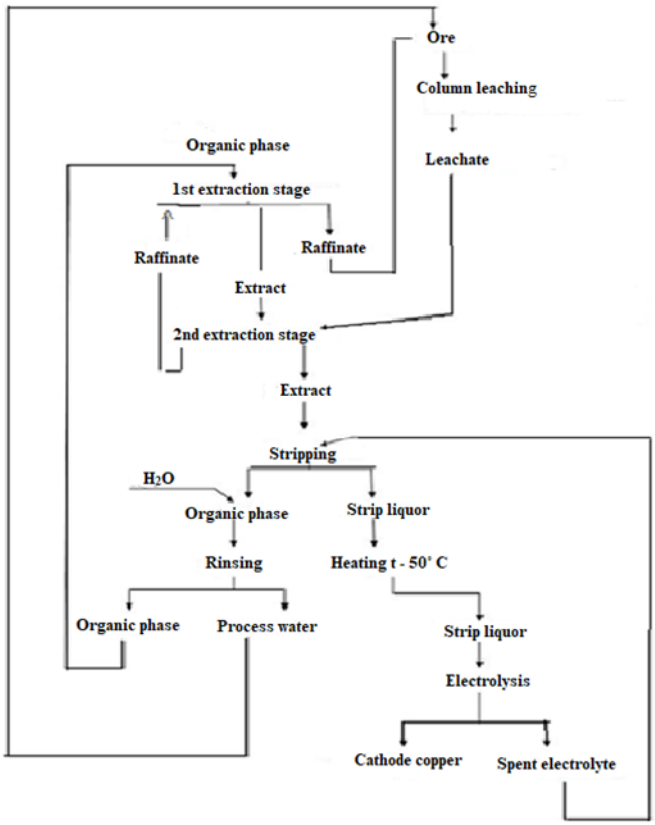

Fig. 13 Process flowsheet for extraction and stripping of ore from the Baiskoye deposit

CONCLUSIONS

Physicochemical studies of substandard mixed copper-bearing ore were performed. The chemical and phase composition of the ore was established. The study showed that sulfide copper predominates in the ore.
The application of modern LIX-type oxime extractants was analyzed.
Model solutions were used to study the characteristics of the extraction process that ensure high copper recovery and the subsequent production of strip liquors suitable for electrolytic metal recovery. The study established the effect of pH and extractant concentration on the degree of copper extraction, demonstrated the possible formation of copper complexes with the extractant, and determined their composition. An extractant was selected to recover copper from pregnant solutions.
The leaching of the initial ore was studied using agitation and percolation methods, and the optimal conditions for subsequent copper recovery by extraction and stripping were established.
The study proposed a conceptual process flowsheet for the hydrometallurgical recovery of copper from substandard mixed ore with a high sulfide copper content and confirmed its efficiency.

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