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RESEARCH PAPER

STUDIES ON EXTRACTION OF COPPER FROM SULFURIC ACID SOLUTION AFTER THERMOCHEMICAL ENRICHMENT OF ROUGH CONCENTRATE

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ABSTRACT

One of the processes of complete and complex extraction of valuable components is the desiliconization of final tailings to increase the content of concentrate and obtain marketable products in the form of white soot and copper cathode. This study aimed to investigate the possibility of further extraction of copper from the sulfuric acid solution obtained after the thermochemical enrichment of rough copper concentrate on a temperature range of 300-500°C. Acorga M5774 extractant and Elixore 205 diluent served to extract copper from the pregnant solution containing 9.59 g/l copper and 24.6 g/l iron. The study of the extractant performance and parameters involved plotting the isotherms of extraction and re-extraction curves. The result shows thermochemical enrichment of the rough concentrate using alkali and subsequent leaching with further copper extraction. The concentrate water leaching yielded 88.0% silicon recovery and 96.15% copper recovery. The copper recovery was 97.9%, with an organic-to-water phase ratio of 1.2. From the dependence of iron transfer on the copper saturation of the organic phase, it follows that if the latter is low, the rest of the extractant will absorb iron, so some excess copper content is required.

Keywords: pregnant solution; copper extraction; Acorga M5774; diagram; Elixore 205, electrolyte

INTRODUCTION

An extended period of field development reduces the volume of balance ore reserves, while the volume of accumulated waste (tailings) of mining and processing production is increasing [1]. The presence of associated non-ferrous, noble, and rare metals in the accumulated tailings, along with copper, as well as the replenishment of retired capacities of operating mines, has necessitated the development of innovative technologies for mining and processing mature copper ore tailings. In addition, the waste of enrichment production stored on the surface is a source of regional environmental pollution. In this regard, the involvement in the effective industrial exploitation of the mature volumes of tailings of ore-processing plants based on the development of innovative integrated technology is an urgent problem whose solution will ensure the expansion of the raw material base and reduce the environmental load.

Copper concentrator tailings currently occupy a significant area due to the lack of efficient solutions in processing technology [2-5]. Studies [6-8] investigated methods of processing tailings with the combination of grinding processes and flotation enrichment with the leaching process of the mature tailings of the Zhezkazgan enrichment plant (Republic of Kazakhstan). The complete cycle of integrated subsoil development should include mining and ore enrichment and deep processing of technogenic raw materials in a completed, environmentally balanced cycle, including the goaf stowing.

One process for the complete and integrated recovery of valuable components is desalinizing final copper-mine tailings to improve concentrate quality in terms of copper and other elements and obtain additional marketable products. Sulfuric acid leaching of concentrate provides complex extraction of valuable components with further marketable products (copper cathode, white soot).

Studies [9-11] investigated the known processes for desiliconization of copper and zinc ores using autoclave leaching methods. However, [12-17] showed that alkaline sintering eliminates the need for autoclave leaching and high-temperature treatment of alumina minerals and aluminosilicates. Studies [13, 18] investigated the alkaline sintering method for treating copper-bearing ores and showed the possibility of significant enrichment of the raw materials. Studies [19, 20] showed the alkaline sintering method with further leaching of copper concentrate with the extraction of 62% Si, 70% Fe, 95% Ag, 97% Al, and 98% Cu into

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the solution. Alkaline leaching of silicon-containing raw materials makes it possible to extract silicon into a solution for further precipitation of white soot. Studies [21] use ammonium nitrate or carbon dioxide to precipitate silicon dioxide [22].

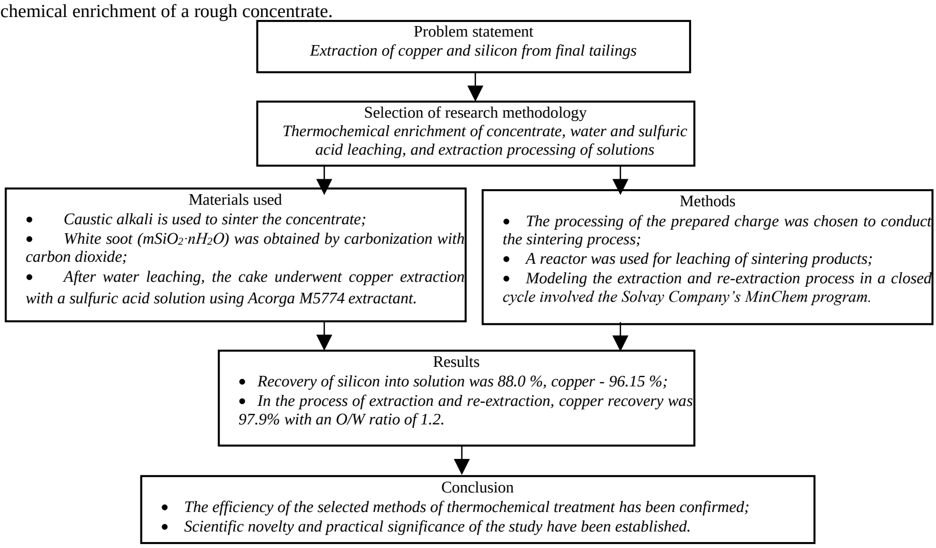
After leaching and transferring silicon into a solution, the concentrate goes to further extraction of valuable components. Studies [19, 20] have shown that sulfuric acid leaching makes transferring copper into a solution possible. In addition, this process dissolves many impurities (Al, Fe), which are part of ores and concentrates. Liquid extraction and electrolysis most often apply to process such solutions [23-25]. Liquid extraction of copper is the most widely used in the metallurgical industry [26-29]. The hydrometallurgical method of copper cathode production uses extraction to remove impurities from the pregnant leaching solution. The efficiency of the extraction process mainly depends on the composition of the organic extraction reagent [30-34].

This study aims to investigate the possibility of further copper extraction from a sulfuric acid solution obtained after the thermochemical enrichment of a rough concentrate.

MATERIAL AND METHODS

The peculiarity of this research is the possibility of integrated processing due to the optimal combination of flotation and chemical enrichment of the concentrate, which provides the best interdependent extraction of metals. The method includes concentrate sintering with sodium hydroxide and subsequent leaching of the solid residue of sintering first with water and then with a sulfuric acid solution, with subsequent obtaining of marketable products: white soot and copper cathode.

The research objective (compared to the known) is to increase the degree of desalinization of concentrates while simultaneously decreasing the temperature of thermal treatment and extraction of valuable components. **Fig. 1** presents a flowchart of the research process.


Problem statement

Extraction of copper and silicon from final tailings

Selection of research methodology

Thermochemical enrichment of concentrate, water and sulfuric acid leaching, and extraction processing of solutions

Materials used

- *Caustic alkali is used to sinter the concentrate;*
- *White soot (mSiO_2·nH_2O) was obtained by carbonization with carbon dioxide;*
- *After water leaching, the cake underwent copper extraction with a sulfuric acid solution using Acorga M5774 extractant.*

Methods

- *The processing of the prepared charge was chosen to conduct the sintering process;*
- *A reactor was used for leaching of sintering products;*
- *Modeling the extraction and re-extraction process in a closed cycle involved the Solvay Company's MinChem program.*

Results

- *Recovery of silicon into solution was 88.0 %, copper - 96.15 %;*
- *In the process of extraction and re-extraction, copper recovery was 97.9% with an O/W ratio of 1.2.*

Conclusion

- *The efficiency of the selected methods of thermochemical treatment has been confirmed;*
- *Scientific novelty and practical significance of the study have been established.*

Fig. 1 Flowchart of the research process (developed by the authors)

The research used rough copper concentrate from flotation from the enrichment's final tailings. **Table 1** presents the composition of the sample.

Table 1 Content of main components in rough copper concentrate

Content of components, %		Content of components, %	
Cu	4.857	Al /Al_2O_3	5.399/10.204
Fe	5.388	Ag	123.7g/t
Zn	0.293	Si /SiO_2	15.07/32.25
Pb	0.314	S	3.484

The main valuable components were 4.857% copper, 15.07% silicon, and 123.7 g/t silver.

Copper in the concentrate is represented by sulfide minerals – 88.347%, oxidized – 11.653%.

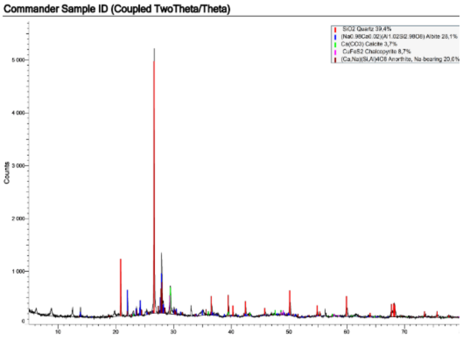


Fig. 2 Diffractogram of rough copper concentrate (developed by the authors)

The phase composition of the concentrate was determined using D2 Phaser diffractometer: quartz SiO_2 – 39.4%, albite

$K[AlSi_3O_8]$ – 28.1%, chalcopyrite $CuFeS_2$ – 8.7%, anorthite $Ca[Al_2Si_2O_8]$ – 20.0%, calcite $CaCO_3$ – 3.7% (**Fig. 2**).

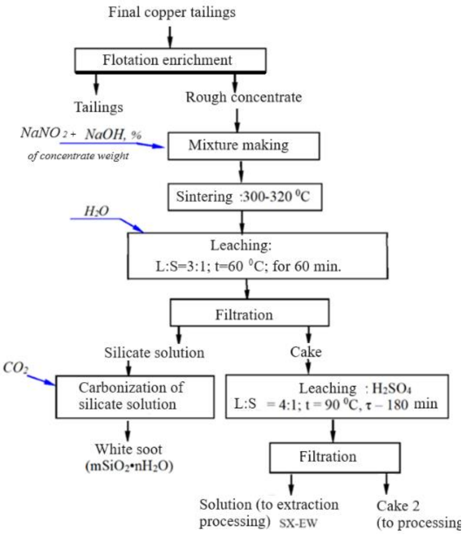


Fig. 3 Process flow diagram of rough copper concentrate processing (developed by the authors)

Fig. 3 shows the scheme of integrated extraction of valuable components from rough copper concentrate. The novelty of this method lies in the sequence of technological operations, conditions of rough concentrate sintering, and extraction of valuable components. The method of processing rough copper concentrates of final tailings with the transfer of silicon and copper into marketable products is possible because the necessary completeness of extraction of each of these elements during sintering is achievable only by selective transfer of them into products, namely silicon during water leaching and copper during sulfuric acid leaching with further extraction processing.

The temperature of the sintering concentrate with alkali was 300-500°C; the ratio of concentrate to alkali was 1:2 with the addition of 0.1% $NaNO_2$ of concentrate by weight to improve oxidation processes. The temperature of water leaching of sinter was 60°C, L:S was 3:1, and the duration was 60 minutes.

White soot ($mSiO_2 \cdot nH_2O$) manufacturing involved the two-step carbonization of silicate solution (liquid glass) by carbon dioxide in a recirculating system: 1) bringing the pH to 9-10 for 30 min and 2) carbonization for 60 min to a residual alkali content in the solution of 90 g/l.

After water leaching, the cake underwent extraction of the target components by sulfuric acid solution at L: S = 4:1, 85-90°C, and a sulfuric acid concentration of 80-150 g/l. The next step after filtration of the solution containing copper was extraction.

The extractant used for copper extraction by liquid extraction was Acorga M5774 Solvay extractant (Sytec, Belgium/USA), a modified aldoxime (5-nonylsalicylaldoxime). **Table 2** presents the technical specifications.

The pregnant leach solution (PLS) collection followed sulfuric acid leaching of the copper concentrate. The extractant (organic phase) included 30% Acorga M5774 and 70% diluent. Elixore 205 diluent is similar in purpose, close in physical, chemical, and operational properties to Shellsol D70, and consists of hydrocarbons C_{11}-C_{14} (paraffins/n-alkanes, cyclanes/naphthenes) [33] (**Table 3**).

Table 2 Technical specifications of Acorga M5774 extractant

Copper capacity, g/l per vol. %	0.56−0.59
Extraction kinetics at 25°C	
for 15 s:	≥ 90%
for 30 s:	≥ 95%
Re-extraction kinetics at 25°C	
for 15 s:	≥ 95%
Extraction at 25°C, g/l Cu	
Organic phase:	At least 4.2
Water phase:	No more than 1.6
Re-extraction at 25°C, g/l Cu	
Organic phase:	No more than 2.1
Water phase:	At least 32.0
Selectivity, copper/iron	At least 2000
Phase separation, s	
Extraction:	No more than 60%
Re-extraction	No more than 60%
Crystallization/solubility at 0°C	
After 24 h	Precipitate was absent

Table 3 Main characteristics of Elixore 205 and Shellsol D70 diluents

Characteristics	Unit	Method	Elixore 205	Shellsol D70
Density at 15°C	kg/m^3	ASTM D 4052	0.820	0.796
Viscosity at 20°C	mm^2/s	ASTM D 445	2.4	−
Viscosity at25°C			−	2,0
Viscosity at 40°C			1.7	−
Flash point	°C	ASTM D 93	76	78
Content of aromatic compounds	mg/kg	SMS 2728	−	<200

Extractant performance and operability studies involved plotting the extraction and re-extraction isotherm curves. Based on the obtained data, modelling the extraction–re-extraction circuit in a closed cycle was conducted, determining copper recovery at the liquid extraction process.

Eight points are usually required to obtain a good extraction isotherm curve. The data for determining the points are taken from analyses of carefully mixed different volumes of initial solution and organic phase in separating funnels (thorough mixing time is 3-5 minutes). Determining the selectivity of copper by iron involved measuring the iron contents in the organic phase and superimposing these values on the dependency graph on the percentage of copper loading of the reagent, taking the maximum loading of the extractant at point 1:10 as a basis.

Four points determined from the analyses of carefully mixed different volumes of poor electrolyte and maximally loaded organic phase in the separating funnels (thorough mixing time was 3-5 min) provided the curve of the re-extraction isotherm.

RESULTS AND DISCUSSION

Thermochemical enrichment of rough concentrate with alkali adding sodium nitrite ($NaNO_2$) followed by water and sulfuric acid leaching converted the target components into acid-soluble form and made it possible to obtain marketable products. Oxidation of sulfide minerals accompanied the sintering of charge at 300-350°C.

The main sintering reactions are as follows:

$$SiO_2 + 2NaOH_{liquid} = Na_2SiO_3 + H_2O\uparrow \quad (1.)$$

$$Al_2O_3 + 2NaOH = 2NaAlO_2 + H_2O\uparrow \quad (2.)$$

$$Cu_2S + 2NaOH + 2{,}5O_2 = 2CuO + Na_2SO_4 + H_2O\uparrow \quad (3.)$$

$$FeS_2 + 2NaOH + 3O_2 = FeO + Na_2SO_4 + H_2O\uparrow + SO_2\uparrow \quad (4.)$$

The conversion of sulfides into oxides due to air oxygen makes it possible to significantly simplify the leaching of copper from concentrate without the need for additional oxidizing processes. The concentrate was mixed with sodium hydroxide in a given ratio (according to the reaction stoichiometry), followed by placing the investigated material in the furnace. **Table 4** presents the results of experiments on concentrate sintering with alkali in the temperature range of 300-500℃.

Table 4 Conditions and results of water leaching

t, °C	Cake yield,%	Chemical composition, %					Extraction of *Si*,%
		Cu	*Fe*	*Si*	*Ag, g/t*	*Zn*	
300	22.64	6.53	7.12	7.90	118.9	0.23	88.13
350	22.45	6.50	7.36	8.03	119.2	0.23	88.04
400	22.50	6.34	7.77	8.10	120.0	0.26	87.91
500	20.61	6.38	8.69	8.25	125.5	0.27	88.72

As **Table 4** shows, the copper content in the cake after water leaching increased on average by 1.34 times. Silicon extraction into solution was around 88% depending on the sintering temperature.

According to the results of X-ray phase analysis performed on D2 Phaser, sodium silicate and aluminosilicate, iron, and copper oxides are formed in the concentrate after thermochemical enrichment (**Fig. 4**).

The silicate solution containing 126.5 g/l Na_2O, 107.7 g/l SiO_2, and 3.1 g/l Al_2O_3 was directed to obtain white soot ($mSiO_2 \cdot nH_2O$). Experiments showed that white soot precipitate formed during 90 min with final pH=9.5 at room temperature. The white soot precipitated from the silicate solution contains many impurities, including aluminum oxide, so after carbonization, it underwent washing with a sulfuric acid solution. **Table 5** shows the chemical composition of white soot after acid leaching.

White soot after acid leaching corresponds to WS-100 grade of GOST 18307-78 [35]. The cake enriched by waste rock removal after water leaching (**Table 4**) underwent sulfuric acid leaching. **Table 6** shows the conditions and results of studies on concentrate leaching in sulfuric acid solution.

Table 5 Chemical composition of white soot

Conditions	**Na_2O**	**MgO**	**Al_2O_3**	**SiO_2**	**CaO**	**Fe_2O_3**	**Total**
Precipitate	1.96	0.04	3.16	94.57	0.06	0.21	99.70
After leaching	0.61	0.01	0.05	98.84	0.2	0.06	99.8

Table 6 Results of sulfuric acid leaching of concentrate

$C_{H_2SO_4}$, g/l	Cake yield, %	β_{Cu}, % in cake	ε_{Cu}, %
80	62.5	1.41	86.50
120	58.5	0.43	96.15
130	58.5	0.43	96.15
150	58.3	0.42	96.25

Note: β_{Cu}– copper content in cake; ε_{Cu} – copper extraction into solution

Sulfuric acid leaching yielded a pregnant solution for copper recovery by extraction and re-extraction. **Table 7** summarises its chemical composition.

Table 7 Chemical composition of pregnant solution, g/l

H_2SO_4	**Cu^{2+}**	**Fe**	**Ag**	**Zn**	**Pb**	**Cl**
30.8	9.59	24.6	1.7	0.019	0.006	8.16
Al	**Cr**	**Ni**	**Ti**	**Ca**	**K**	**Na**
6.31	0.009	0.006	0.29	0.55	0.26	25.8

Table 8 presents the chemical composition of extraction and re-extraction solutions for copper and iron.

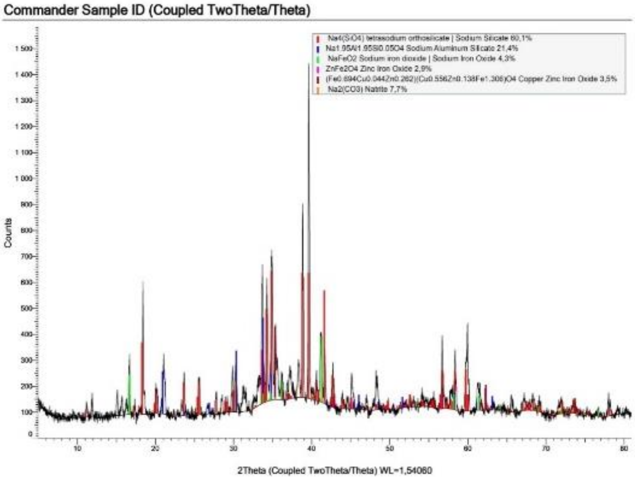


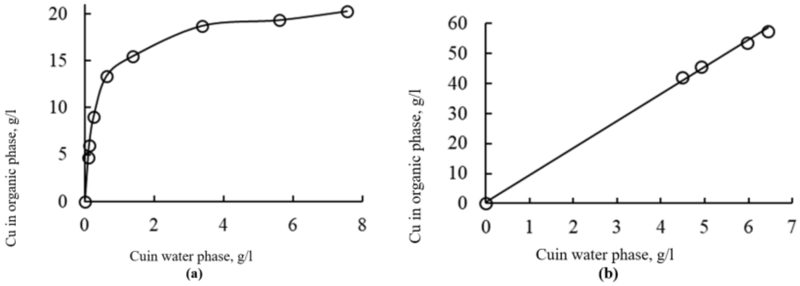
Fig. 4 Diffractogram of rough copper concentrate after thermochemical enrichment (developed by the authors)

Table 8 Chemical composition of extraction and re-extraction solutions

Volume, ml	Content, g/l

Organicphase (O)	Waterphase (W)	O:W ratio	Organicphase		Waterphase	
			Cu	Fe (mg/l)	Cu	Fe
		Extraction isotherms				
50	25	2:1	4.710	347.610	0.089	29.067
45	30	1.5:1	5.980	189.820	0.117	30.823
35	35	1:1	8.990	172.030	0.245	30.894
30	45	1:1.5	13.340	139.990	0.615	30.419
25	50	1:2	15.500	88.980	1.387	30.941
20	60	1:3	18.720	71.180	3.385	31.297
12	60	1:5	19.320	53.390	5.610	30.811
12	120	1:10	20.260	17.800	7.560	30.989
		Re-extraction isotherms				
45	15	3:1	6.440	-	57.460	-
40	20	2:1	5.960	-	53.720	-
25	25	1:1	4.920	-	45.650	-
20	40	1:2	4.480	-	42.150	-

Fig. 5(a) shows the extraction curves based on the data on copper content in organic (g/l) and water phases (g/l – x-axis). **Fig. 5(b)** shows the extraction curve based on the data on copper content in the water (g/l – y-axis) and organic phases (g/l – x-axis).



(a) **(b)**

Fig. 5 Copper extraction (a) and re-extraction (b) curves (developed by the authors)

Modeling the extraction–re-extraction process in a closed cycle involved the Solvay Company’s MinChem program.

Based on the obtained isotherms, a model (**Fig. 6**) and a McCabe-Thiele diagram (**Fig. 7**) were built using the laboratory method, with the ratio of organic and water phases (O/W) = 1.1:1 in extraction and 4.5:1 in re-extraction. Table 9 summarises the data.

According to the data in Table 9, at the O/W ratio of 1.2, the extraction using Acorga M5774 extractant was 97.9%, which is very high. Higher recovery is possible by increasing the O/W ratio, but it is necessary to consider the decreasing load on the extractant, which will lead to increased iron transfer chemically [36]. Cu:Fe selectivity is the main parameter in the choice of extractant. Since iron transfers from PLS solutions to electrolytes, it is essential to reduce this in extraction. For this purpose, it is necessary to saturate the organic phase with copper as much as possible and to use a selective extractant. If iron in electrolytes exceeds the permissible concentration (2.5-3 g/l), it is necessary to renew a part of the electrolyte, thereby bearing losses in cobalt sulfate and other additives dosed into the electrolyte. According to the conducted tests, the dependence of iron concentration in the organic phase on its saturation with copper is plotted (**Fig. 8**).

Table 9 Copper recovery results with 30% organic phase

Scheme configuration	**O/W ratio**		**Copper content, g/l**				**Recovery, %**
	E	R	Pregnant solution	Spent electrolyte	Electrolyte	Raffinate E2	
2(E) + 1(R)	1.2	2	9.7	35	50.83	0.204	97.9

Note: E – extraction; R – re-extraction

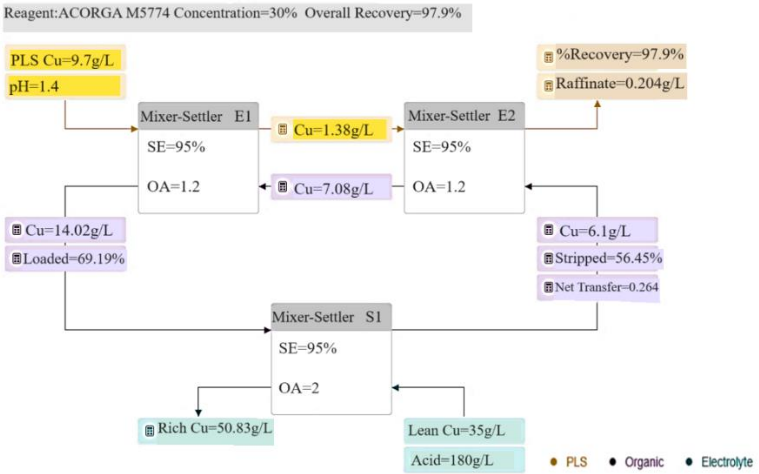
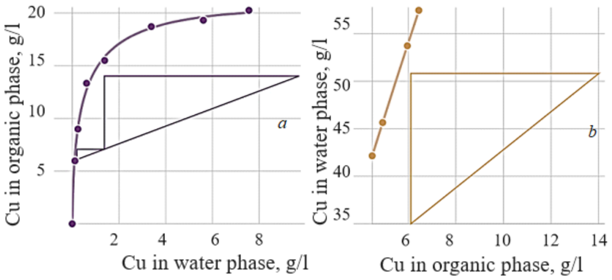


Fig. 6 Copper extraction model (developed by the authors using Solvay Company's MinChem program)



(a) **(b)**

Fig. 7 McCabe-Thiele diagram: *(a)* – extraction; *(b)* – re-extraction (developed by the authors)

Fig. 8 shows that iron transfer depends on the copper saturation of the organic phase. If the copper saturation in the organic phase is low, then the rest of the extractant will absorb iron. After re-extraction, the electrolyte went to the copper cathode production stage, and the extractant returned to the pregnant solution extraction stage. The technology of copper cathode production by electrolysis of sulfuric acid copper electrolyte is widely known. As a result of copper extraction and re-extraction studies, an electrolyte was obtained (**Table 10**), further useable for copper cathode production.

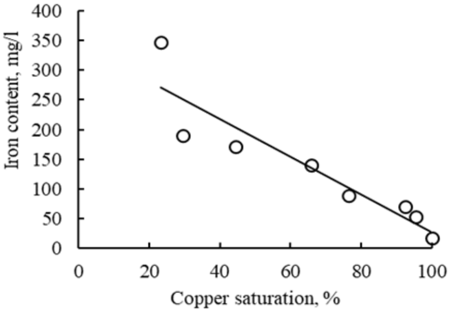


Fig. 8 Dependence of iron transfer on copper saturation of the organic phase (developed by the authors)

Table 10 Chemical composition of copper electrolyte, g/l

Cu	Ni	H_2SO_4	SO_4	Cl	Pb	As
45.28	<0.01	142.5	217.5	0.05	<0.05	<0.05
Mg	**Al**	**Bi**	**Ca**	**Zn**	**Sb**	**Fe**
<0.01	<0.05	<0.05	0.01	<0.01	<0.05	0.03

In industrial conditions, it is possible to organize a continuous process of copper extraction and re-extraction by the spent electrolyte and its return in a copper-saturated state to the electrolysis section, i.e., the organization of a closed system according to the SX-EW principle.

The continued depletion of deposits reduces the volume of balance reserves, accompanied by a constant decline in the quality of ores under development. At the same time, the volume of accumulation of mining and processing wastes placed on the surface is constantly growing. The accumulated technogenic georesources, in terms of their volume and content of valuable components, represent a promising mineral resource base expansion source. However, to minimize capital expenditures, more efficient and cost-effective technologies are necessary to obtain marketable products.

The known methods for desiliconization of iron-containing concentrates [37] by their melting with soda ash at temperatures of 700-850°C with forming Na_2SiO_3 and with further treatment with water or acid with the recovery of 88-94%, have the disadvantage of using high temperatures of charge melting (more than 700°C). Autoclave desiliconization of off-balance sulfide copper ore [38] uses alkali with a concentration of 160 g/l, L:S ratio

= 5:1, and temperature 230°C. The disadvantage of the method is the low degree of desiliconization of concentrates (57.1% SiO_2) and the lack of integrated extraction of other non-ferrous metals such as silver and rhenium. In contrast to similar methods, this study proposes integrated processing of raw materials, including a sintering stage at 300-350°C and double leaching with 88% and 96.15% recovery of silicon and copper into solution, respectively.

Selecting the most effective product and process scheme for combined technologies is essential. Therefore, it is critical to consider the peculiarities of the processing type, timely study data, and possible causes of malfunctions in the process for the future development of hydrometallurgical processing.

It is necessary to mention that the choice of a specific method depends on the type of raw material, the production size, and technological requirements for the purity of the marketable product, which determines the price of processing. It is also necessary to increase the financing and qualification of employees, introduce new technologies to improve the design and modeling of hydrometallurgical processing of final copper tailings and reduce errors during the complex technological processes in these productions.

Studies on the thermochemical enrichment of concentrate include the following technological operations:

Thermal treatment of charge (rough concentrate) at 300-350°C, using sodium hydroxide as a reagent forming a chemical compound with silicon dioxide, with further water leaching and silicon extraction;

Sulfuric acid leaching of enriched cake (residue after water leaching) and copper extraction;

Extraction of copper from solution.

CONCLUSION

Low-temperature concentrate sintering from final tailings makes it possible to extract valuable components without using high-temperature pyrometallurgical processes and obtain marketable products.

By sintering the rough concentrate with sodium hydroxide at 300-350°C followed by water leaching, 88% of the silicon was recovered into solution.

The obtained cake (after water leaching), containing 6.53% Cu, underwent leaching with a sulfuric acid solution with a recovery of 96.15% Cu. The filtrate containing copper and other components went to extraction. Copper extraction involved the organic phase in a ratio of 30% Acorga M5774 extractant and 70% Elixore 205 diluent consisting of C_{11}-C_{14} hydrocarbons (paraffins/n-alkanes, cyclanes / naphthenes).

On the basis of the conducted studies using Acorga M5774 extractant and Elixore 205 diluent, copper extraction and re-extraction isotherms were plotted, obtaining a model of the liquid extraction process based on these isotherms. The copper extraction was 97.9% (at O/W ratio = 1.2), with an extractant loading of 69.19%.

The dependence of iron transfer with the organic phase (extractant) on the degree of its saturation with copper was plotted using the obtained extraction isotherms. According to these data, at saturation of the extractant with copper up to 69.19%, the iron transfer is only a few mg/l. A specific stage applies to wash iron from the organic phase if the iron content in the pregnant solutions is more than 5-6 g/l. Acorga M5774 minimizes iron transfer to the electrolyte without a washing step.

Strengths and limitations of the study

Processing of rough concentrates of final copper tailings with extraction of silicon and copper is possible because the necessary completeness of transfer of these elements at sintering is achievable only by their selective transfer into products, namely silicon at the operation of water leaching and copper – at sulfuric acid leaching. At that, the degree of desiliconization of concentrates increases with a simultaneous decrease in the thermal treatment temperature.

Thermochemical enrichment of rough copper concentrate occurred in the range of 300-500°C because below 300°C the crystallization of alkali occurs, and above 500°C – the decomposition and impact on refractory materials.

The water leaching of silicon after the concentrate sintering occurred at 60°C because of high solution coagulation. After water leaching, the cake underwent copper extraction using the sulfuric acid solution. In the studied concentrations of 80-150 g/l, the optimal concentration of sulfuric acid was 130 g/l, providing copper recovery of 96.15%.

Copper extraction from sulfuric acid solutions was performed by liquid extraction using Solvay Company's MinChem program, which provides a cycle passage based on extractant data for copper-containing solutions only.

Recommendations and further studies

Further studies relate to the integrated processing of final copper tailings according to the technological scheme of the final product, with further theoretical justification of the processes of thermochemical enrichment, leaching, and electroextraction. They also involve multifactor analysis of technological operations with the identification of the optimal process modes.

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