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A study of copper leaching from the tailings of the Karagaily (Republic of Kazakhstan) concentrating factory using an electric hydropulse discharge

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Abstract: The method for using concentration plants tailings is proposed for the first time in this study. These tailings contain a number of valuable metals, such as Cu, Fe, Zn, *etc*., and are a potential raw material for obtaining concentrates of some elements. An electrohydropulse discharge was used to intensify the process of copper leaching and other metals. Ammonium bifluoride, the most effective of the ammonium salts used in copper leaching by the ammonization method, was chosen as the reagent. The influences of significant leaching parameters were studied and optimized using probabilistic deterministic planning of experiment. Based on the study findings, the following process conditions were found to be optimal: mass ratio of solid to liquid (S:L) of 1:1; Cu:F = 1:6; sulphuric acid concentration 40 g L^{-1}; experiment duration 30 min; discharge voltage 10 kV and a leaching efficiency of 80-85 % could be achieved. Comparative features of tailings samples from the Karagaily (Republic of Kazakhstan) concentration plant were studied using X-ray diffraction (XRD), scanning electron microscope (SEM) and atomic emission spectral analysis. The study results showed that copper was maximally transferred to the aqueous phase.

Keywords: chalcopyrite; ammonium bifluoride; water leaching; copper tetra-amine sulfate.

INTRODUCTION

The tailings of concentration plants of non-ferrous metallurgy enterprises negatively affect the environment, especially water bodies, air basin, as well as agriculture, being sources of pollution with heavy metals, silicates, sulphates, *etc.* Today, special attention is paid to the problem of the utilization of mining waste,

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since the use of such accumulations of technogenic minerals is not only sources of raw materials for the mining industry, but also an important part of the state policy of resource conservation and environmental protection. The flotation tailings consist mainly of vein minerals in the form of quartz, pyrite and silicates with low copper content and other valuable metals that have not been recovered from the ore by froth flotation.

Heap and underground leaching, as well as vat dissolution with stirring or percolation, are widely used in the extraction of copper from lean and off-balance ores or tailings. The main solvent in heap leaching is iron sulphate solutions which are obtained by irrigating heaps with water as a result of pyrite oxidation. Irrigation is carried out alter-natively with water and solution, followed by cementation of copper with iron scrap.[1–4] Ammonization methods (ammonia–ammonium extraction) using leaching solutions of ammonia and ammonium salts in the presence of free oxygen or air are known. First of all, an effective and complete uncovering of raw materials and deep separation of the target and by-products occur due to this method.[5–8]

However, all known leaching methods require large amounts of reagents, high temperatures, high pressures, and a long duration of the process itself. For the treatment of mining waste in particular the tailings of concentrating plants that are technogenic mineral raw materials in terms of resource value, new technologies are required that allow deep selective separation of metals with a high degree of their extraction from the feedstock. Ensuring environmental friendliness and wastelessness are the main conditions for the method of processing manmade material that are been developed. Therefore, for the first time, an electrohydropulse discharge was to extract copper from the current tailings of the Karagaily concentrating plant (Republic of Kazakhstan). Based on the analysis of the nature of the reagent used in the known methods for leaching copper from technogenic raw materials, ammonium hydrobifluoride was chosen as a reagent. The goal of leaching experiments using ammonium hydrobifluoride in combination with sulphuric acid was to estimate the total amount of copper available for leaching from oxide and sulphide mineral phases using an electro-pulse discharge.

EXPERIMENTAL

Materials and methods

The object of the research was the current tailings of the Karagaily Concentrating Plant (KCP). X-Ray diffraction studies of the samples were performed on a D8 Advance Eco powder diffractometer (Bruker, Germany). The shooting mode is Bragg–Brentano geometry, the angular range was from 15 to 75°, with a step of 0.03°, the spectrum acquisition time was 2 s. X-Ray radiation was generated using a copper tube with a CuK wavelength λ = 1.5406 Å. DiffracEVA decryption software, v.4.2, PDF-2 matching phase search database (2016). The weight ratio of the phases was estimated using the standard Eq. (1), which is based on the determination of the values of integral intensities and an assessment of their contributions:

$$V_{\text{admixture}} = \frac{RI_{\text{phase}}}{I_{\text{admixture}} + RI_{\text{phase}}} \tag{1}$$

I_{phase} – average integrated intensity of the main phase of the diffraction line, $I_{\text{admixture}}$ – average integral intensity of the additional phase, R – structural factor equal to 1.45.

The content of copper and other valuable metals in the original tailings, solid residues after leaching, and in the working solution were measured on atomic emission spectrometer with microwave plasma Agilent 4210 MP-AES ("Agilent Technologies Bayan Lepas Free", Malaysia). The mineralogical composition of the tailings samples was determined using a Micromed POLAR 2 polarizing microscope (Micromed, China).

The electrohydraulic effect (EH, the Yutkin effect) for the leaching of copper from the tailings of the copper concentration plant was used for the first time in this work.[9] The advantages of EH technologies are the following: the ability to directly convert electrical energy into mechanical energy (with high efficiency – up to 80 %); energy can simply be realized at a given point (both in the bladder – for crushing stones, and in water well, at a depth of tens of meters); the process is easily regulated by changing the discharge parameters (operating voltage and capacitance of the capacitor bank). The process of copper leaching from tailings was studied using probabilistic-deterministic planning of the experiment,[10] and a five-factor matrix at five levels. The leaching experiments were performed in a 1 L cell. In each experiment, 250 g of tailings were leached. At the end of the experiment, the mixture was filtered, the solid residue was washed with water and dried in an oven.

Experiments of the tailings leaching were realized using a laboratory setup, the scheme of which is shown in Fig. 1. A hydro-impulse discharge in cell (6) was created by the laboratory setup developed and assembled in house with a high-voltage generator. It allows experiments to be performed over a wide range of changes in the characteristics of an electric discharge. A general view of the laboratory cell for electrohydro-impulse treatment of the copper concentration plant tailings is shown in Fig. 1. It has a hermetically sealed lid made of high-strength insulating material. On the lid with a plug an electrode of positive polarity (platinum) is screwed in, connected by means of a high-voltage cable with a controlled spark gap of an electrohydraulic installation. The negative electrode is a graphite rod, fixed to the inner rod, the body of which is electrically connected to the negative pole of the generator by means of grounding buses. The oval container made of Teflon contains the working pulp. The distance between the electrodes that is the working discharge gap can be changed by screwing in the plug. The height of the rod is chosen so that the working gap is located in the geometric centre of the object being processed.

In order to confirm the formation of tetraammine copper(II) sulphate in the working solution, it was dried under vacuum. The spectral characteristics of the sediment were obtained on an FSM1201 infrared Fourier spectrometer (LLC "Infraspek", Russia). The sediments obtained after drying were mixed with potassium bromide (Fluka, Germany) in the ratio of 1:100, and a disk was formed using a hydraulic press. The measurement was performed in the range of 4000–400 cm^{-1}, with a resolution of 2 cm^{-1} and the number of scans was 25. The device was controlled and data processed using the Fspec 4.0 program.

The results of X-ray diffraction of the studied samples before and after leaching are shown in Fig. 2. According to the X-ray phase analysis of the obtained diffraction patterns, the most intense reflections at 2θ 20.9, 27.4, 37.2, 39.2, 50.5, 60.3, 68.2 and 68.7° correspond to the structure of silicon dioxide, which is characteristic of most silicon-containing or silicate rocks. Reflections characteristic of FeS_2 (pyrite), $CaSiO_3$ (wollastonite), Cu_2S (chalcocite) phases were also observed. A new reflection for the KCP (Karagaily Concentrating Plant) samples after leaching was not observed, which indicates the absence of phase transformation processes as a result of the actions performed on the sample.

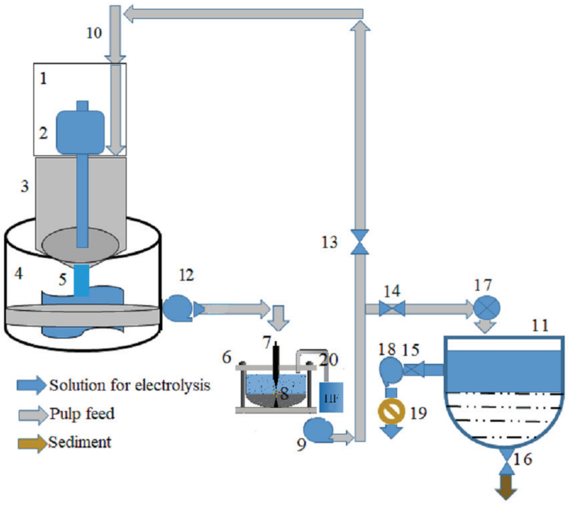

Fig. 1. The flowchart of the laboratory setup for extracting copper from the tailings of the concentrating plant (1 – casing; 2 – electric drive; 3 – pulp and reagent supply tank; 4 – mixing vessel with a lid; 5 – stirrer; 6 – cell for HID; 7 – electrodes; 8 – discharge; 9 – slurry pump; 10, 13, 14, 15, 16 – taps; 11 – tank for settling; 12 – pump for supplying slurry to the reactor; 17 – coarse filter; 18 – pump for removing copper-containing solution for further processing; 19 – fine filter; 20 – a vessel for capturing ammonia).

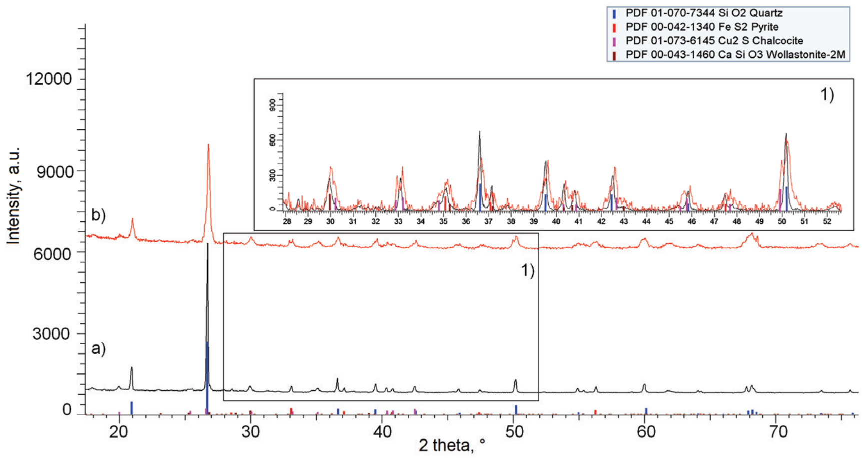

Fig. 2. X-Ray diffraction patterns of KCP tailings before (a) and after leaching (b); roasting temperature was 500 °C.

The main changes of the KCP sample after leaching are associated with the change in the intensity of the reflections, which indicates the processes of changes in the concentration of phases (see Fig. 2), as well as the appearance of impurity phases in the form of changes in their concentration (see the results presented in the diagram in Fig. 3). The phase composition, as well as the concentration of phases were determined using Eq. (1). The presented data showed a decrease in the content of phases FeS_2 from 5.9 to 4.4 % and Cu_2S from 10.9 to 4.1 % after leaching. The diagram shows the content of metals decreases in the sediment after leaching which characterizes their transition into the solution.

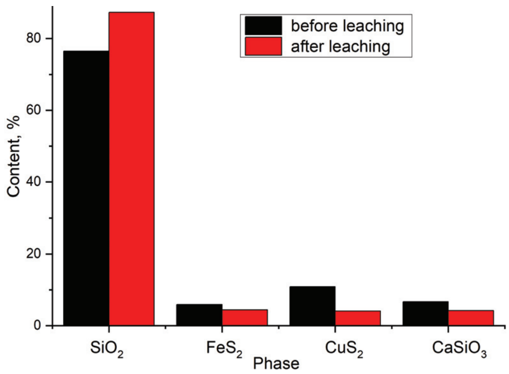

Fig. 3. Results of changes in phase composition.

The mineralogy of copper compounds in the studied samples KCP was determined on a polarizing microscope Micromed POLAR 2 (Micromed, China). The results are given in Table I, which indicate that copper is contained in the samples of the current KCP tailings before leaching mainly in the form of chalcopyrite. After leaching, mineralogical analysis of the solid phase indicates complete destruction of chalcopyrite.

TABLE I. Mineralogy of copper compounds in samples of current KCP tailings

Copper form	Content, %	
	Before leaching	After leaching
Total	0.210	<0.02
CuΣ-oxidized phase	<0.02	<0.02
Free oxidized	<0.02	<0.02
Associated oxidized	<0.02	<0.02
CuΣ malachite + azurite + atacamite ($Cu_2CO_3(OH)_2$+2Cu[CO_3]×Cu[OH]$_2$+$Cu_2Cl(OH)_3$)	<0.02	<0.02
Chrysocolla $Cu_{2-x}Al_x(H_{2-x}Si_2O_5)(OH)_4\times nH_2O)$, $x < 1$	<0.02	<0.02
Chalcocite (Cu_2S)	<0.02	<0.02
Bornite (Cu_5FeS_4)	<0.02	<0.02
Copper iron sulfide (chalcopyrite) ($CuFeS_2$)	<0.143	<0.014

Quantitative indicators of the main valuable components in the original tailings, solid residues after leaching and in the working solution are presented in the Table II. The amount of copper in the working solution was determined using a microwave plasma atomic emission spectrometer Agilent 4210 MP-AES (Agilent Technologies Bayan Lepas Free, Malaysia). The process of leaching copper from the tailings was performed in a cell with a volume of 1 L. 250

g of raw materials (tailings, waste) was poured then water 1:1 was added to prepare the working pulp (mixture). 4 g of ammonium bifluoride, NH_4HF_2, was added and the pulp mixed thoroughly for 5 min, then it was pumped into the reactor (cell), where through the built-in electrodes periodically in 1–2 s. A high-voltage discharge of 10 kV was produced, for 15 min and then the slurry was pumped into a mixing vessel. Then the mixing was stopped and 40 ml of sulphuric acid was added to the pulp, mixing continued for another 5 min, and then the pulp was pumped into the reactor. After processing the HID for another 15 min, the slurry was fed through a coarse filter into a settling tank.

TABLE II. Chemical composition of KCP tailings from Karagaily Concentrating Plant

Source	Content, %							
	Cu	Fe	Cr	Mn	Zn	SiO_2	Al_2O_3	Ag
In tailings before leaching	0.210	4.44	0.03	0.05	0.13	62.81	11.02	–
In the solid phase after leaching	0.035	3.68	0.0024	0.0025	0.012	62.81	11.02	–
	Concentration, g/L							
In the working solution after leaching	1.02	2.79	–	–	1.67	–	–	–

Leaching of concentrator tailings

The extraction of copper from the tailings was realised under the following conditions: a mass ratio of S:L (x_1) – 1:2.5, 1:2, 1:1.5, 1:1 and 1:0.5; the mass ratio of Cu:F (x_2) – 1:8, 1:7, 1:6, 1:5 and 1:4; sulphuric acid concentration (x_3) – 20, 40, 60, 80 and 100 g L^{-1}; the duration of the experiments (x_4) – 10, 20, 30, 40 and 50 min, and discharge voltage, (x_5) – 5, 7, 10, 12 and 15 kV. The experimental conditions and the obtained results are given in Table III.

TABLE III. Plan – matrix and results of a five-factor experiment at five levels (α – copper leaching indicator)

No.	x_1	x_2	x_3	x_4	x_5	α_{Cu} / %	
						Experimental	Theoretical
1	1:2.5	1:8	20	10	5	60.5	66.95
2	1:2.5	1:6	60	30	10	84.0	80.71
3	1:2.5	1:7	40	20	7	70.2	74.32
4	1:2.5	1:4	100	50	15	74.5	83.25
5	1:2.5	1:5	80	40	12	65.5	84.40
6	1:1.5	1:8	60	20	15	79.3	75.69
7	1:1.5	1:6	40	50	12	82.9	81.95
8	1:1.5	1:7	100	40	5	78.2	75.88
9	1:1.5	1:4	80	10	10	77.0	78.05
10	1:1.5	1:5	20	30	7	70.6	75.93
11	1:2	1:8	40	40	10	70.6	74.60
12	1:2	1:6	100	10	7	71.2	75.10
13	1:2	1:7	80	30	15	69.2	79.35
14	1:2	1:4	20	20	12	75.6	74.57
15	1:2	1:5	60	50	5	77.5	82.73
16	1:0.5	1:8	100	30	12	76.2	75.11
17	1:0.5	1:6	80	20	5	74.3	78.41
18	1:0.5	1:7	20	50	10	73.0	76.03

TABLE III. Continued

No.	x_1	x_2	x_3	x_4	x_5	α_{Cu} / %	
						Experimental	Theoretical
19	1:0.5	1:4	60	40	7	79.2	81.67
20	1:0.5	1:5	40	10	15	80.3	80.39
21	1:1	1:8	80	50	7	81.2	77.09
22	1:1	1:6	20	40	15	83.0	78.53
23	1:1	1:7	60	10	12	77.5	76.55
24	1:1	1:4	40	30	5	83.5	77.59
25	1:1	1:5	100	20	10	82.3	79.59

Based on the experiments results, a sample of the obtained data was made and partial dependencies on the relevant factors were built (Figs. 4–8).

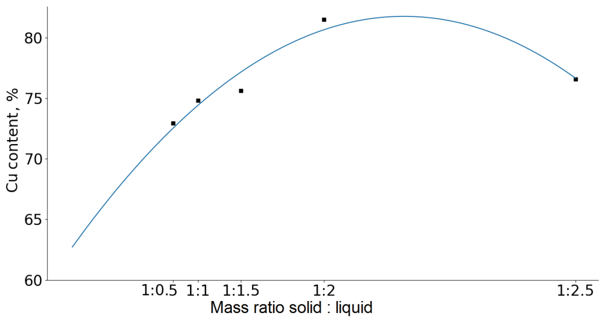

Fig. 4. Dependencies of the degree of copper leaching on mass ratio S:L.

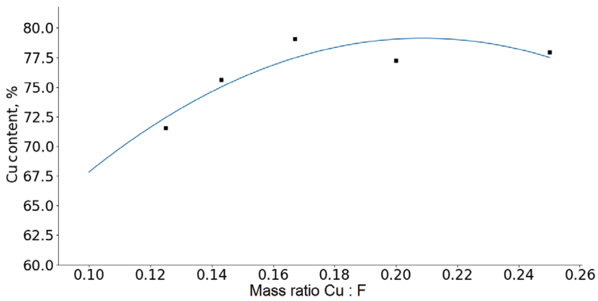

Fig. 5. Dependencies of the degree of copper leaching on mass ratio Cu:F.

The calculated correlation coefficients and their significance are presented in Table IV.

The statistical description of the partial dependencies of copper leaching is presented by the generalized Protodyakonov Equation (2):

$$Up = (-0.6057x_1^2 + 2.2343x_1 + 76.256)(-955.5x_2^2 + 398.86x_2 + 37.506)\times$$
$$\times(-0.0021x_3^2 + 0.2941x_3 + 67.996)(0.12x_4 + 72.692)(0.3222x_5 + 73.135)/75.9^{-4} \quad (2)$$

where the correlation coefficient $R = 0.67$, significance $t_R = 2.04$, the error of the equation $\sigma =$ = 8.62 %.

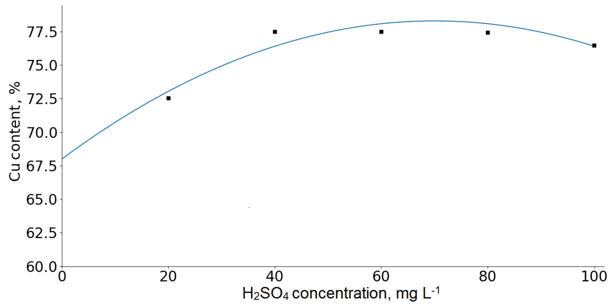

Fig. 6. Dependencies of the degree of copper leaching on sulphuric acid concentration.

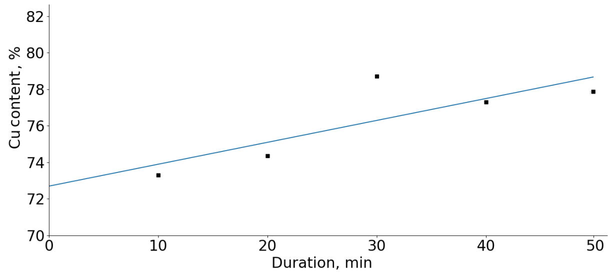

Fig. 7. Dependencies of the degree of copper leaching on process duration.

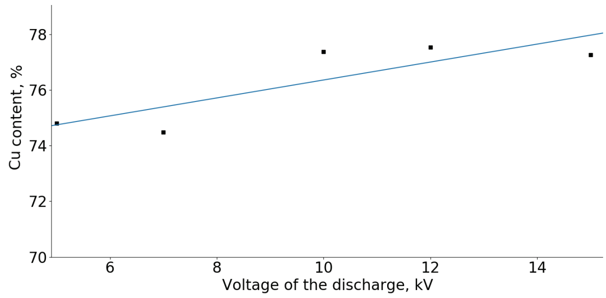

Fig. 8. Dependencies of the degree of copper leaching on the discharge voltage.

TABLE IV. Correlation coefficients (R) and their significance (t_R) for the partial dependencies of the degree of copper leaching

Function	R	t_R
$\alpha (x_1) = -0.6057x_1^2 + 2.2343x_1 + 76.256$	0.78	>0.66
$\alpha (x_2) = -955.5x_2^2 + 398.86x_2 + 37.506$	0.89	>0.66
$\alpha (x_3) = -0.0021x_3^2 + 0.2941x_3 + 67.996$	0.94	>0.66
$\alpha (x_4) = 0.12x_4 + 72.692$	0.81	>0.66
$\alpha (x_5) = 0.3222x_5 + 73.135$	0.84	>0.66

An analysis of the partial dependences in accordance with Figs. 4–6 shows that the functions of the degree of copper leaching on the S:L ratio, the Cu:F ratio and the sulphuric acid concentration pass through a maximum. The influences of the process duration and the discharge voltage (Figs. 7 and 8) are characterized by increases in the rates of copper extraction into solution. However, increases in their values over 40 g L^{-1} and 10 kV, respectively, does not give any significant additional effect. Based on the study findings, the following parameters of copper leaching from tailings of the Karagaily concentration plant were found to be optimal: S:L mass ratio of 1:1; Cu:F mass ratio of 1:6; sulphuric acid concentration, 40 g L^{-1}; the duration of the experiments, 30 min; discharge voltage, 10 kV. Under these conditions, the copper leaching efficiency was 80–85 %.

RESULTS AND DISCUSSION

The physicochemical basis of the fluorination process of ammonium hydrodifluoride is that transition oxygen-containing compounds and many non-transition elements interact with NH_4HF_2 to form ammonium fluoro- or oxofluorometallates. They are very convenient for processing[11] and due to their physicochemical properties, ensure the solubility of products and the possibility of separating mixtures by sublimation. A great advantage of these complex salts is the selective tendency to sublimate or thermally dissociate to non-volatile fluorides, which guarantees a wide separation of the components. Thus, taking into account the differences in the physicochemical properties of ammonium fluorometallates, it is possible to select the conditions for separating the mineral product fluorinated with ammonium hydrodifluoride into individual components or to concentrate some of the valuable components in the residue.

The uncovering of "encapsulated" metals of mineral raw materials with ammonium bifluoride has undeniable advantages: fluorination occurs at a temperature not higher than 200 °C, and some reactions do not require heating at all, fluorination by-products (water and ammonia vapours) do not contain fluorine, which ensures environmental safety of the production and allows their use in the condensed state in the processes of ammonia hydrolysis.[5–8] The release of NH_4F during the hydrolysis of fluoroammonium salts or NH_3 and HF during the thermal decomposition of these substances create favourable conditions for the regeneration of NH_4HF_2 and the creation of environmentally friendly technological industries. The transition of copper into solution is a complex and staged process, which begins with the opening of the silicates surface under the action of ammonium bifluoride:[12,13]

$$SiO_2 + 3NH_4HF_2 \rightarrow (NH_4)_2SiF_6 + NH_3 + 2H_2O \quad (3)$$

The use of a hydro-impulse discharge (HID) in the process of tailings leaching in an aqueous solution in the presence of ammonium hydrodifluoride and sulphuric acid creates the necessary redox medium. Under conditions of direct polarity (minimum for positive and maximum for negative electrodes), the processes in the volume between the electrodes will be characterized by a predominance of oxidative reactions. Thus, conditions are created under which metallogenic associates are actively decomposed, access to them is provided by the action of fluorides on silicates, in which metal-containing minerals are "encapsulated", in the present case, chalcopyrite.

Oxygen formation

$$2H_2O \rightarrow 2H_2 + O_2 \quad (4)$$

further is the oxidation of chalcopyrite:

$$CuFeS_2 + 3O_2 \rightarrow CuO + FeO + 2SO_2 \quad (5)$$

formation of copper fluoride:[14]

$$CuO + NH_4HF_2 \rightarrow CuF_2 + NH_3 + H_2O \quad (6)$$

formation of a complex – tetraamine copper sulphate (II):[15,16]

$$CuF_2 + H_2SO_4 + 4NH_4OH \rightarrow [Cu(NH_3)_4]SO_4 + 2HF + 4H_2O \quad (7)$$

The IR spectra (Fig. 9) of the studied sediment revealed absorption bands in the region of 1400 and 1643 cm^{-1}, characteristic of NH_4^+ , and in the region of 578–1114 cm^{-1}, characteristic of SO_4^{2-}. These results are consistent with literature data.[17,18] Comparative analysis of the IR spectra of three objects – the precipitate obtained by evaporation of the working solution, copper sulphate and tetraammine copper (II) sulphate (Fig. 6) also confirms the participation of NH_4^+ and SO_4^{2-} in the formation of the composition of the working solution in the process of leaching of KCP tailings using HID. Samples of tetraamminemed sulphate were obtained in accordance with the procedure.[15] The reagent $CuSO_4 \cdot 5H_2O$ (reagent grade) was used as samples of copper(II) sulphate.

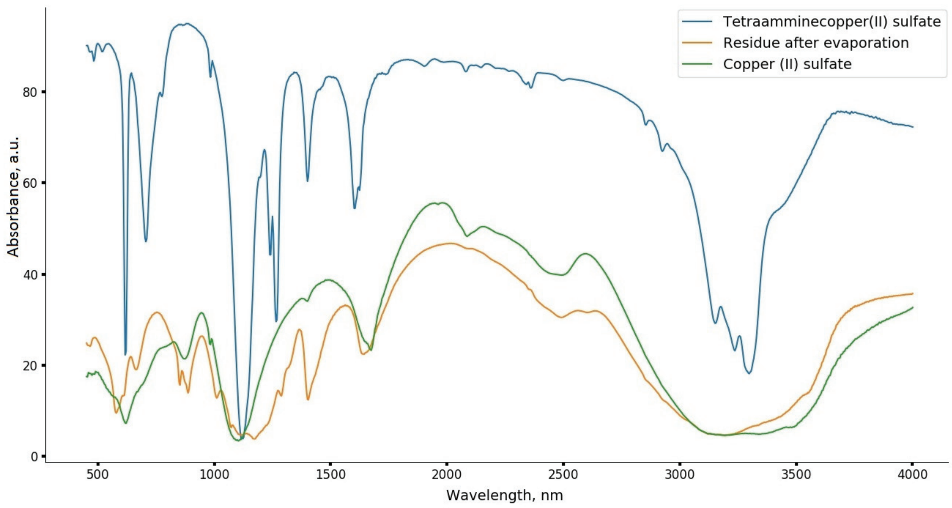

Fig. 9. IR spectra of samples of tetraamminecopper (II) sulphate (blue line), copper (II) sulphate (green line), precipitate obtained by evaporation of the working solution (orange line).

Quantitative analysis of metals in the working solution (Table I) after separation of the solid phase showed the presence of copper, iron and zinc. Al, Cr, Mn and partly Fe, forming the following fluorides that remain in the solid phase,[11,14] reactions (8)–(11):

$$Al_2O_3+3NH_4HF_2 \rightarrow 2AlF_3+ 3NH_3+3H_2O \quad (8)$$

$$Fe_2O_3+3NH_4HF_2 \rightarrow 2FeF_3+ 3NH_3+3H_2O \quad (9)$$

$$MnO_2 + 2NH_4HF_2 \rightarrow MnF_4 + 2NH_3 + 2H_2O \quad (10)$$

$$Cr_2O_3+3NH_4HF_2 \rightarrow 2CrF_3+ 3NH_3+3H_2O \quad (11)$$

Selective extraction of copper from a working solution containing iron and zinc is not difficult and could be realised by known methods.[19,20]

The sediment was studied before and after leaching using a scanning electron microscope (Tescan Mira, Czech Republic). SEM analysis of the samples is shown in Fig. 10.

a b

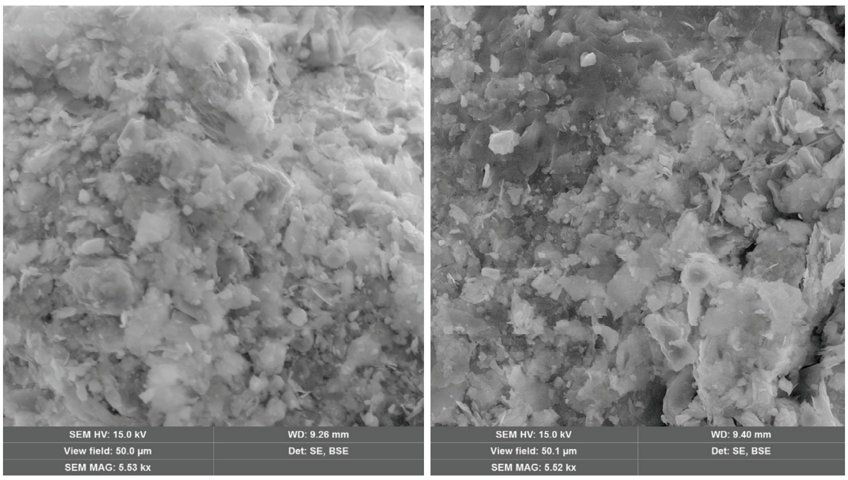

Fig. 10. SEM images of KCP tailings before (a) and after leaching (b).

There were noticeable differences between tailings samples before and after HID treatment: all clay fine particles crumbled and dissolved, all fibrous formations disappeared, there was no adhesion of particles to each other, and quartz crystals were clearly visible.

CONCLUSIONS

As a result of the experiments, it was found that of the five studied parameters the following ones determine the process of copper leaching from the tailings of the Karagaily concentrating plant (KCP) using a hydro-pulse discharge (HID): S:L ratio, Cu:F ratio and sulfuric acid concentration. The optimal leaching conditions were established under which ensures 80–85 % of copper extraction into the aqueous phase. The main advantage of using an electrohydroimpulse discharge is a significant reduction in the experiment time with a high yield of copper into the solution and low energy consumption.

X-ray, scanning electron microscope (SEM) and spectral studies of the KCP tailings before and after leaching with the use of HID indicate deep processes in the reaction zone occur. They aimed at the formation of developed microcracks and micropores in the tailings, which leads to the uncovering of copper minerals "encapsulated" in silicates, their oxidation, fluorination and the formation of copper ammonia complexes in the aqueous phase.

ИЗВОД

ПРОУЧАВАЊЕ ИСПИРАЊА БАКРА ИЗ ЈАЛОВИНЕ КОНЦЕНТРАЦИОНОГ ПОСТРОЈЕЊА KARAGAILY (РЕПУБЛИКА КАЗАХСТАН) ПОМОЋУ ЕЛЕКТРОХИДРОПУЛСНОГ ПРАЖЊЕЊА

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У овом раду први пут је предложен начин коришћења постројења за концентрацију јаловине. Ова јаловина садржи низ вредних метала, као што су Cu, Fe, Zn, и др., и потенцијална је сировина за добијање концентрата неких елемената. Електрохидропулсно пражњење је искоришћено за интензивирање процеса лужења бакра и других метала. За реагенс је изабран амонијум бифлуорид, који је ефикаснији од амонијум соли, које се користе у лужењу бакра методом амонизације. Утицај значајних параметара испирања је проучаван и оптимизован коришћењем вероватноће детерминистичког планирања експеримента. На основу добијених резултата, утврђено је да су следећи процесни услови оптимални: однос масе чврсто:течно (S:L) = 1:1; Cu:F = 1:6; концентрација сумпорне киселине – 40 g/l; трајање експеримента – 30 min; напон пражњења – 10 kV и ефикасност испирања може достићи 80-85 %. Упоредне карактеристике узорака јаловине из Karagaily постројења за концентрацију (Република Казахстан) проучаване су уз помоћ рендгенске дифракције (XRD), скенирајућег електронског микроскопа (SEM) и атомске емисионе спектралне анализе. Утврђено је да су минерали бакра садржани у јаловини потпуно разбијени и бакар је максимално прешао водену фазу.

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