Electrochemical Polarization of Complex Anthropogenic Dump Oxidized Copper Ores Using a Pyrite Sulfidizer

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Abstract. There was for the first time determined the possibility of using the natural mineral pyrite as a sulfidizer. The mechanism of sulfur transfer from the pyrite mineral to oxidized minerals with the formation of easily floatable copper sulfides was studied. To supplement the sulfur content in refractory oxidized copper ore, pyrite (FeS₂) was used. It was introduced into the pulp based on the reaction of formation of easily floatable copper sulfides. In order to achieve the maximum values of the copper recovery into the concentrate and to determine the optimal parameters of the current density, the concentration of sulfuric acid, the sulfidizing agent, pyrite, the electrolyte temperature, the duration of the electrolysis, there was studied electrochemical sulfiding of the Zhezkazgan oxidized copper ore according to the method of mathematical planning of the experiment.

Keywords: oxidized copper ore, grinding, electrolysis, cathode, anode, sulfidizer, natural mineral pyrite, current density, electrolyte, sulfuric acid, flotation, recovery, copper sulphide concentrate.

Introduction

At present, due to the gradual depletion and limited reserves of high-quality mineral raw materials and the growing needs of the national economy in non-ferrous metals, it is necessary to involve in the processing of more complex and difficult-to-dress ores, including oxidized ones. There is a large stock of flotation stale dump tailings where copper is in the form of oxidized minerals and it is time to re-process them. From the practice of copper concentrating plants it is known that with direct flotation of oxidized ores, the copper content is 0.5%. The degree of the recovery does not exceed 75% and the quality of concentrates deteriorates. In this regard, various combined schemes of processing oxidized ores are used by scientists to maximize the recovery of valuable components [1-4]. The main disadvantages of the known methods are relatively low copper recovery into the concentrate, the technological scheme complexity, a relatively high temperature and high energy costs. Sulfidation compared to other methods of processing oxidized minerals has the following main advantages: the degree of copper recovery into the flotation concentrate increases to 66 90% or more; sulfidated oxidized refractory ores are

further processed as sulfide ores according to the existing generally accepted technology. The authors of works [4, 5] carried out studying electrochemical processes during the directed transformation of oxidized minerals into sulfide ones, i.e. when changing the technical and technological properties of copper minerals, which allows increasing its recovery into the flotation concentrate by more than 10-15%, which in turn will lead to expansion of the raw material base of the mining industry, due to the involvement in the processing of complex substandard raw materials, will increase the use of minerals through the disposal of associated valuable components.

The purpose of our research was to determine the possibility of using pyrite, a widespread natural mineral, which is a part of refractory oxidized copper ores, as a sulfidizing agent; studying the mechanism of sulfur transition from the pyrite mineral to oxidized minerals with the formation of easily floatable copper sulfides.

Experimental techniques

Anthropogenic complex dump oxidized copper ores of the Zhezkazgan deposit are mainly represented by such copper minerals as chrysocolla malachite,

azurite, turquoise, chalcosiderite, cuprite, tenorite, etc. that are practically not amenable to traditional methods of flotation dressing. In addition, significant intergrowth of minerals is observed in these ores, which significantly complicates the processes of their dressing by the existing methods. In the world practice difficult-to-dress oxidized copper ores are processed in various ways [1-4]. The main disadvantages of the known methods are relatively low extraction of copper into the concentrate, complexity of the technological scheme, a relatively high temperature and high energy costs.

The promising methods of preparing complex copper ores for flotation are electrochemical methods. The authors of works [2, 3] carried out electrochemical studies aimed at changing the surface properties of minerals that are part of difficult-to-dress copper ores

The basis for the development of new electrochemical methods of preparing oxidized copper ores for flotation was the work carried out for studying the voltammetric behavior of copper and its oxidized and sulfide compounds: chrysocolla, cuprous and cupric oxides, copper sulfides: Cu₂S, CuFeS₂ in aqueous solutions [4-8].

Scientific results

The process of electrochemical sulfiding was carried out in a thermostated glass electrolyzer with the volume of 0.5 l, a titanium cathode and a lead anode were used as electrodes. The anode and the cathode spaces are separated by an acid-resistant filter cloth. Crushed ore (class content – 0.074 mm in ore 65%) was mixed with the solution of sulfuric acid, at the W:S ratio equal to 3:1. The chemical composition of the oxidized copper ore of the Zhezkazgan deposit is

shown in Table 1.

The resulting pulp is loaded into the cathode space of the electrolyzer and subjected to electrical treatment under conditions of intense mixing. After electrical treatment, the pulp is fed into the flotation machine. During flotation, the consumption of butyl xanthate is 120 g/l, the blowing agent T-66 is 70-80 g/t. Flotation is carried out on the laboratory F-237 FL-A flotation machine. The experimental data obtained are shown in Table 2.

The results of chemical analysis show that the ore under study contains mainly a finely disseminated difficult-to-open mineral: chrysocolla that is practically not amenable to flotation. In addition, the content of total copper and sulfur in it is relatively insignificant. As a result, the amount of sulfur present in the ore is insufficient for copper sulfiding. Therefore, for the first time, to supplement the sulfur concentration in the ore, a natural mineral, pyrite (FeS₂) is used as a sulfidizer. It is introduced into the ore pulp based on the reaction of formation of easily floatable copper sulfides.

The sulfidizing mechanism consists in the following:

- during the cathodic polarization of oxidized copper minerals, the latter is upon impact restored to the elemental state according to the reactions:

chrysocolla
$$CuSiO_3 + 2e + 2H^+ \rightarrow Cu^0 + H_2SiO_3$$
, (1)

malachite
$$CuCO_3$$
 (2)
 $Cu(OH)_2 + 4e + 4H^+ \rightarrow 2Cu^0 + H_2CO_3 + 2H_2O$,

azurite
$$2CuCO_3$$

 $Cu(OH)_2 + 6e + 6H^+ \rightarrow 3Cu^0 + 2H_2CO_3 + 2H_2O.$ (3

Table 1 – Chemical composition (in %) of oxidized copper ore											
Cu _{tot}	Cu _{oxid}	S _{tot}	Fe	Na₂O	TiO ₂	SiO ₂	CaO	Al ₂ O ₃	MgO		
1.2	0.96	0.16	1.46	0.10	0.21	76.1	1.86	8.4	0.21		

Table 2 – Copper recovery into concentrate. Experimental data $(\epsilon_{Cu,9})$ and calculated data $(\epsilon_{Cu,p})$												
No.	ε _{Cu, э}	ε _{Cu, p}	No.	ε _{Cu, э}	ε _{Cu, p}		ε _{Cu, э}	ε _{Cu, p}				
1	89.63	89.0828	10	92.34	90.8395	19	82.37	85.6829				
2	88.15	91.7254	11	97.03	95.7244	20	91.34	87.7998				
3	85.64	90.0301	12	94.66	89.2916	21	96.77	93.4242				
4	95.81	93.7934	13	96.38	96.4862	22	90.82	95.5358				
5	97.11	93.6056	14	95.67	93.3069	23	87.67	93.2513				
6	95.98	97.5403	15	84.00	89.6737	24	89.94	88.4815				
7	98.87	95.7483	16	93.78	92.2085	25	95.63	90.9082				
8	93.69	91.3126	17	87.99	84.2875							
9	86.41	90.1359	18	89.08	88.8665							

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Then, freshly formed very active copper, upon contact with the natural iron sulfide mineral, pyrite (FeS₂) introduced as a sulfidizer, passes into easily floatable copper sulfide:

$$Cu^0 + FeS_2 \rightarrow CuS + FeS,$$
 (4)

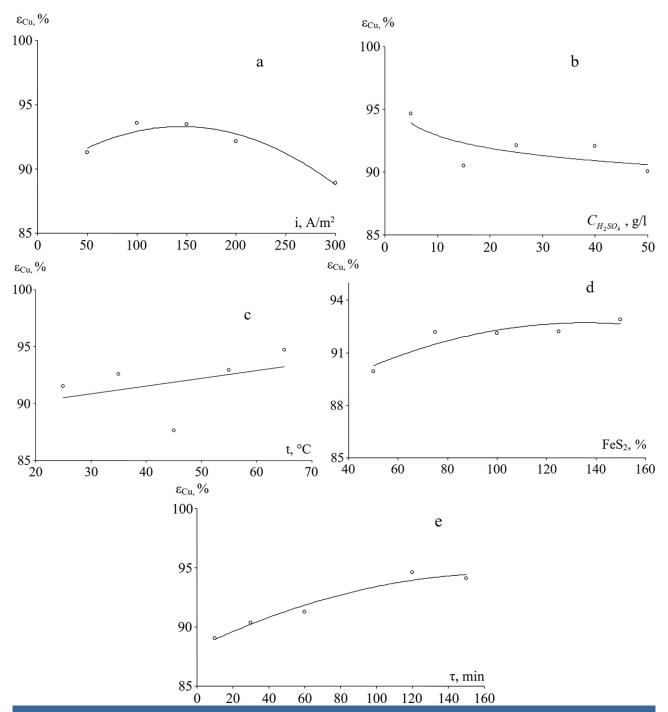
$$FeS + Cu^{0} - 2e \rightarrow CuS + Fe^{2+}$$
. (5)

In order to achieve the maximum values for copper recovery into the concentrate and to determine the optimal parameters, electrochemical sulfidation of

the Zhezkazgan oxidized copper ore was carried out according to the method of mathematical planning of the experiment [9], which independent variable values are given in Table 3.

After sampling the experimental data (from Table 2) to the levels of factors and averaging the samples, scatter plots of partial dependences were constructed (shown in Figure 1). The approximating functions were selected taking into account the physical meaning of the studied dependences.

Figure 1 shows that the dependence of copper



a – on the current density; b – on the concentration of sulfuric acid; c – on the temperature; d – on the amount of addition of FeS2; e – on the duration; points – experimental data, lines – according to the equation

recovery on the current density has a clearly defined For the degree of copper recovery into the maximum in the region of 150 A/m². Apparently, at concentrate, the equations obtained from Table current densities above this value at the cathode, the 4, taking into account significant functions, are rate of the competing reaction of hydrogen evolution generalized by the Protodyakonov equation: increases, which leads to a significant decrease in the copper recovery rates. With increasing the concentration of sulfuric acid (Figure 1, b), the reverse dissolution of oxidized reduced minerals and freshly formed sulfides occurs, i.e. copper is contained in the pulp mainly in the form of ions, therefore, the copper recovery indicators deteriorate significantly. The effect of the temperature (Figure 1, c) leads to acceleration of the process of interaction of all minerals with the electrolyte solution, while the process of solution neutralization is accelerated, which in turn reduces the opening of the surfaces of minerals and the electrical conductivity of the pulp. The sulfidizing agent, pyrite FeS₂ has a significant effect on the copper recovery and tends to saturation, the most complete sulfiding of copper minerals occurs at 70%

formation of copper sulfides proceeds completely. The resulting dependences were determined by the correlation coefficient R and its significance t_R (Table 4).

sulfur content in pyrite. Probably, at the indicated concentration, the amount of sulfur is sufficient for

the process of sulfiding of all the oxidized copper

minerals. Further increasing the concentration of the

FeS₂ sulfidizing agent has practically no effect on the

copper recovery into the flotation concentrate. With

increasing the duration of the process (Figure 1, e),

the recovery of copper naturally increases. It follows

that in the range of 60-120 min, the reduction reaction

of oxidized copper minerals and the process of

$\varepsilon_{Cu} = \frac{\left[(-0,0002i^2 + 0,0544i + 89,375) \times \right.}{\left[91,8704^3 (96,341C_{H_2SO_4}^{-0,0157}) \times \right.} \\ \left. \times (-0,0003FeS_2^2 + 0,0907FeS_2 + 86,564) \right]$ $\times (0.0002\tau^2 + 0.0724\tau + 88.238)^{-1}$

Comparison with the data calculated directly by direct substitution of experimentally determined values gives R = 0.9152 and $t_R = 3.75 > 2$, indicating the adequacy of the description of the experimental data by this equation. The confidence interval calculated through t_R [10] is 4.165%. Using the resulting equation (6), a nomogram was constructed for the copper recovery into concentrate (Table 5). According to the nomogram, the optimal areas of the factors taken into account are visible at other values.

Conclusion

Thus, a new method of electrochemical sulfiding of complex oxidized copper ores was developed. As a result of the studies, for the first time, natural iron sulfide, pyrite, was used as a sulfidizing agent for oxidized copper ores. A mathematical model of the copper recovery into the flotation concentrate was obtained and a nomogram for the extraction of copper into the concentrate was calculated. It follows that the given sufficiently high degree of copper extraction ≥ 95.0% can be achieved at the current density of 25-150 A/m², with the addition of sulfidizer, pyrite (FeS₂) 75-125% and with the duration of 60-120 minutes.

Table 3 – Factors and their levels											
No.	Factors	Factor levels									
1	Current density, A/m ²	50	100	150	200	300					
2	H ₂ SO ₄ concentration, g/l	5	15	25	40	50					
3	Temperature, °C	25	35	45	55	65					
4	Sulfur content in pyrite added as a sulfidizing agent, %	50	75	100	125	150					
5	Duration, min	10	30	60	120	150					

Table 4 – Correlation coefficient R and its significance $t_{\scriptscriptstyle R}$ for particular functions of the degree of copper recovery into concentrate											
Function	R	Condition t _R > 2	Significance								
$\epsilon_{Cu} = -0.0002i^2 + 0.0544i + 89.375$	0.888	7.250 > 2	significant								
$\epsilon_{\text{Cu}} = 96,341 C_{\text{H}_2 \text{SO}_4}^{-0.0157}$	0.737	2.06 > 2	significant								
ε _{cu} = 0,0678t + 88,819	0.000	0 < 2	insignificant								
$\varepsilon_{Cu} = -0,0003 \text{FeS}_2^2 + 0,0907 \text{FeS}_2 + 86,564$	0.879	3.319 > 2	significant								
$\epsilon_{Cu} = -0,0002\tau^2 + 0,0724\tau + 88,238$	0.971	28.930 > 2	significant								

Table	e 5 – Nomogram o	of cop	per re	covery	y into	conce	ntrate	e (extra	acted	≤ 95,0)						
FeS ₂ ,	i, A/m²	i, A/m² 25 75							1!	50		200					
%	C _{H₂SO₄} τ, min	5	30	45	60	5	30	45	60	5	30	45	60	5	30	45	60
		87.8	85.4	84.8	84.5	89.5	87	86.5	86.1	90.2	87.7	87.1	86.7	89.4	86.9	86.4	86
	5	87.8	85.4	84.8	84.5	89.5	87	86.5	86.1	90.2	87.7	87.1	86.7	89.4	86.9	86.4	86
	_	87.8	85.4	84.8	84.5	89.5	87	86.5	86.1	90.2	87.7	87.1	86.7	89.4	86.9	86.4	86
		87.8	85.4	84.8	84.5	89.5	87 88.6	86.5	86.1	90.2	87.7	87.1	86.7	89.4	86.9	86.4	86
		89.4 89.4	87 87	86.4 86.4	86 86	91.1	88.6	88	87.7 87.7	91.8	89.3 89.3	88.7 88.7	88.3 88.3	91.1	88.5 88.5	88	87.6 87.6
	30	89.4	87	86.4	86	91.1	88.6	88	87.7	91.8	89.3	88.7	88.3	91.1	88.5	88	87.6
		89.4	87	86.4	86	91.1	88.6	88	87.7	91.8	89.3	88.7	88.3	91.1	88.5	88	87.6
50		91.1	88.5	88	87.6	92.8	90.2	89.6	89.2	93.5	90.9	90.3	89.9	92.7	90.1	89.6	89.2
	60	91.1	88.5	88	87.6	92.8	90.2	89.6	89.2	93.5	90.9	90.3	89.9	92.7	90.1	89.6	89.2
	60	91.1	88.5	88	87.6	92.8	90.2	89.6	89.2	93.5	90.9	90.3	89.9	92.7	90.1	89.6	89.2
		91.1	88.5	88	87.6	92.8	90.2	89.6	89.2	93.5	90.9	90.3	89.9	92.7	90.1	89.6	89.2
		93.2	90.6	90.1	89.7	95	92.4	91.8	91.4	95.7	93.1	92.5	92.1	94.9	92.3	91.7	91.3
	120	93.2	90.6	90.1	89.7	95	92.4	91.8	91.4	95.7	93.1	92.5	92.1	94.9	92.3	91.7	91.3
		93.2	90.6	90.1	89.7	95	92.4	91.8	91.4	95.7	93.1	92.5	92.1	94.9	92.3	91.7	91.3
		93.2 89.1	90.6 86.6	90.1 86.1	89.7 85.7	95 90.8	92.4 88.3	91.8 87.7	91.4 87.3	95.7 91.5	93.1 89	92.5 88.4	92.1 88	94.9	92.3 88.2	91.7 87.7	91.3 87.3
		89.1	86.6	86.1	85.7	90.8	88.3	87.7	87.3	91.5	89	88.4	88	90.7	88.2	87.7	87.3
	5	89.1	86.6	86.1	85.7	90.8	88.3	87.7	87.3	91.5	89	88.4	88	90.7	88.2	87.7	87.3
		89.1	86.6	86.1	85.7	90.8		87.7	87.3	91.5	89	88.4	88	90.7	88.2	87.7	87.3
		90.8	88.2	87.7	87.3	92.5	89.9	89.3	88.9	93.2	90.6	90	89.6	92.4	89.8	89.3	88.9
	20	90.8	88.2	87.7	87.3	92.5	89.9	89.3	88.9	93.2	90.6	90	89.6	92.4	89.8	89.3	88.9
	30	90.8	88.2	87.7	87.3	92.5	89.9	89.3	88.9	93.2	90.6	90	89.6	92.4	89.8	89.3	88.9
75		90.8	88.2	87.7	87.3	92.5	89.9	89.3	88.9	93.2	90.6	90	89.6	92.4	89.8	89.3	88.9
/3		92.4	89.8	89.3	88.9	94.2	91.5	91	90.6	94.9	92.2	91.7	91.2	94.1	91.5	90.9	90.5
	60	92.4	89.8	89.3	88.9	94.2	91.5	91	90.6	94.9	92.2	91.7	91.2	94.1	91.5	90.9	90.5
		92.4	89.8	89.3	88.9	94.2	91.5	91	90.6	94.9	92.2	91.7	91.2	94.1	91.5	90.9	90.5
		92.4	89.8	89.3	88.9	94.2	91.5	91	90.6	94.9	92.2	91.7	91.2	94.1	91.5	90.9	90.5
		94.6 94.6	92 92	91.4 91.4	91 91	96.4 96.4	93.7	93.1	92.7 92.7	97.1 97.1	94.4	93.8	93.4	96.3 96.3	93.6	93	92.6
	120	94.6	92	91.4	91	96.4	93.7	93.1	92.7	97.1	94.4	93.8	93.4	96.3	93.6	93	92.6
		94.6	92	91.4	91	96.4	93.7	93.1	92.7	97.1	94.4	93.8	93.4	96.3	93.6	93	92.6
		90	87.5	87	86.6	91.8	89.2	88.6	88.2	92.5	89.9	89.3	88.9	91.7	89.1	88.6	88.2
	_	90	87.5	87	86.6	91.8	89.2	88.6	88.2	92.5	89.9	89.3	88.9	91.7	89.1	88.6	88.2
	5	90	87.5	87	86.6	91.8	89.2	88.6	88.2	92.5	89.9	89.3	88.9	91.7	89.1	88.6	88.2
		90	87.5	87	86.6	91.8	89.2	88.6	88.2	92.5	89.9	89.3	88.9	91.7	89.1	88.6	88.2
		91.7	89.2	88.6	88.2	93.4	90.9	90.3	89.9	94.2	91.5	91	90.6	93.4	90.8	90.2	89.8
	30	91.7	89.2	88.6	88.2			90.3	89.9		91.5	91		93.4	90.8		89.8
	30	91.7	89.2	88.6	88.2	93.4	90.9	90.3	89.9	94.2	91.5	91	90.6	93.4	90.8	90.2	89.8
100		91.7	89.2	88.6	88.2	93.4	90.9	90.3	89.9	94.2	91.5	91	90.6	93.4	90.8	90.2	89.8
		93.4 93.4	90.8	90.2	89.8 89.8	95.1 95.1	92.5	91.9 91.9	91.5 91.5	95.9 95.9	93.2	92.6	92.2	95.1 95.1	92.4	91.8 91.8	91.4
	60	93.4	90.8	90.2	89.8	95.1		91.9	91.5	95.9	93.2	92.6	92.2	95.1	92.4	91.8	91.4
		93.4	90.8	90.2	89.8	95.1		91.9	91.5		93.2	92.6	92.2	95.1	92.4	91.8	91.4
		95.6	92.9	92.3	91.9	97.4	94.7	94.1	93.7	98.1	95.4	94.8	94.4	97.3	94.6	94	93.6
	120	95.6	92.9	92.3	91.9	97.4	94.7	94.1	93.7	98.1	95.4	94.8	94.4	97.3	94.6	94	93.6
	120	95.6	92.9	92.3	91.9	97.4	94.7	94.1	93.7	98.1	95.4	94.8	94.4	97.3	94.6	94	93.6
		95.6	92.9	92.3	91.9	97.4	94.7	94.1	93.7	98.1	95.4	94.8	94.4	97.3	94.6	94	93.6
		90.6	88.1	87.5	87.1	92.3		89.2	88.8	93	90.5	89.9	89.5	92.3	89.7	89.1	88.7
	5	90.6	88.1	87.5	87.1	92.3		89.2	88.8	93	90.5	89.9	89.5	92.3	89.7	89.1	88.7
		90.6	88.1	87.5	87.1	92.3		89.2	88.8	93	90.5	89.9	89.5	92.3	89.7	89.1	88.7
		90.6	88.1	87.5	87.1	92.3	89.8	89.2	88.8	93	90.5	89.9	89.5	92.3	89.7	89.1	88.7
		92.3	89.7	89.1	88.7	94	91.4	90.8	90.4	94.7	92.1	91.5	91.1	94	91.3	90.8	90.4
	30	92.3 92.3	89.7 89.7	89.1 89.1	88.7 88.7	94	91.4 91.4	90.8 90.8	90.4	94.7	92.1 92.1	91.5 91.5	91.1 91.1	94	91.3 91.3	90.8	90.4
		92.3	89.7	89.1	88.7	94	91.4	90.8	90.4	94.7	92.1	91.5	91.1	94	91.3	90.8	90.4
125		93.9	91.3	90.8	90.4	95.7	93.1	92.5	92.1	96.5	93.8	93.2	92.8	95.7	93	92.4	92
		93.9	91.3	90.8	90.4	95.7	93.1	92.5	92.1		93.8	93.2		95.7	93	92.4	92
	60	93.9	91.3	90.8	90.4	95.7	93.1	92.5	92.1	96.5	93.8	93.2	92.8	95.7	93	92.4	92
		93.9	91.3	90.8	90.4	95.7	93.1	92.5	92.1	96.5	93.8	93.2	92.8	95.7	93	92.4	92
		96.2	93.5	92.9	92.5	98	95.3	94.7	94.3	98.8	96	95.4	95	97.9	95.2	94.6	94.2
	120	96.2	93.5	92.9	92.5	98	95.3	94.7	94.3	98.8	96	95.4	95	97.9	95.2	94.6	94.2
	120	96.2	93.5	92.9	92.5	98	95.3	94.7	94.3	98.8	96	95.4	95	97.9	95.2	94.6	94.2
		96.2	93.5	92.9	92.5	98	95.3	94.7	94.3	98.8	96	95.4	95	97.9	95.2	94.6	94.2

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Сульфидтеуші пиритті пайдалану арқылы техногенді үйінділердегі қиынбайытылатын тотыққан мыс рудаларын электрохимиялық поляризациялау

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Аңдатпа. Алғаш рет сульфидтеуші ретінде табиғи минерал пиритті пайдалану мүмкіндігі анықталды. Минерал пириттен күкірттің тотыққан мыс минералдарына өтіп жеңілфлотацияланатын мыс сульфидттерін түзу механизмі зерттелді. Қиынбайытылатын тотыққан мыс рудаларындағы күкірттің құрамын толықтыру үшін пирит (FeS₂) пайдаланылды,ол пульпаға жеңілфлотацияланатын мыс сульфидтері реакциясын түзу есебімен енгізілді. Мысты концентратқа бөліп алудың жоғары көрсеткіштеріне жету үшін және тоқ тығыздығының, күкірт қышқылының, сульфидтеуші пирит концентацияларының, электролит температурасының, электролиз ұзақтылығының тиімді параметрлерін анықтау үшін тотыққан Жезқазған мыс рудасын электрохимиялық сульфидтеуін экспериментті матиматикалық жоспарлау әдісімен өткіздік.

Кілт сөздер: тотыққан мыс рудасы, электролиз, ұнтақтау, катод, анод, сульфидтеуші, пирит табиғи минерал, тоқ тығыздығы, электролит, күкірт қышқылы, флотация, бөліп алу, сульфидті мыс концентраты.

Электрохимическая поляризация труднообогатимых техногенных отвальных окисленных медных руд с использованием сульфидизатора пирита

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Аннотация. Впервые определена возможность применения в качестве сульфидизатора природного минерала пирита. Изучен механизм перехода серы от минерала пирита на окисленные минералы с образованием легкофлотируемых сульфидов меди. Для дополнения содержания серы в труднообогатимой окисленной мед- 71

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ной руде использован пирит (FeS₂), который вводится в пульпу из расчета на реакцию образования легкофлотируемых сульфидов меди. С целью достижения максимальных значений по извлечению меди в концентрат и определения оптимальных параметров по плотности тока, концентрации серной кислоты, сульфидизатора — пирита, температуры электролита, продолжительности электролиза, исследования по электрохимическому сульфидированию Жезказганской окисленной медной руды проводили по методу математического планирования эксперимента.

Ключевые слова: окисленная медная руда, измельчение, электролиз, катод, анод, сульфидизатор, природный минерал пирит, плотность тока, электролит, серная кислота, флотация, извлечение, сульфидный медный концентрат.

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