

# Complex Oxidized Copper Ore Electrochemical Sulfidization Using Elemental Sulfur

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**Abstract.** A new method of electrochemical sulfidization of oxidized complex (refractory) copper ores was developed. Elemental sulfur was used as a sulfidizing agent to increase the dressing rates. Using the Seidel-Gauss method, the effect of the current density, the concentration of sulfuric acid, the sulfidizing agent, sulfur, the electrolyte temperature, the electrolysis duration on the degree of copper extraction into the flotation concentrate was studied. A mathematical model for the copper recovery into the concentrate was obtained and a nomogram was calculated. It follows that under electrolysis conditions at the optimal concentration of the sulfidizing agent, elemental sulfur, sulfuric acid, the current density, the electrolyte temperature, the electrolysis duration, the degree of copper extraction into the flotation concentrate reaches its maximum value  $\geq 95.0\%$ .

**Keywords:** oxidized copper ore, grinding, electrolysis, cathode, anode, sulfide dissipator, elemental sulfur, current density, electrolyte, sulfuric acid, flotation, recovery, sulfide copper concentrate.

## Introduction

In connection with the gradual depletion of reserves of copper sulfide deposits, more and more attention is being paid to technologies of dressing refractory oxidized copper ores, the composition of which consists mainly of copper minerals such as chrysocolla, malachite, azurite, turquoise, chalcocite, cuprite, etc., which are practically not amenable to the methods of flotation enrichment [1-5]. The Chemical-Metallurgical Institute carried out studies of developing new electrochemical methods of sulfiding such ores. These methods are based on studying the electrochemical behavior of oxides, carbonate, silicate and sulfides of monovalent and divalent copper, copper minerals malachite, chrysocolla, etc. These studies made it possible to establish the mechanisms of formation of compounds, contributed to determining the patterns of their transition from the oxidized phase to the sulfide one. The results of these works led to the development of fundamentally new methods of preparing refractory oxidized copper ores for flotation dressing [6, 7].

The purpose of this study is to reduce the cost of the electrochemical sulfiding process using elemental sulfur as a sulfidizer, which accumulates in a very large amount as a waste in the oil production of Western Kazakhstan.

## Experiment techniques

There were carried out studies with the aim of converting oxidized minerals into easily floatable sulfide on the ore of the Zhezkazgan deposit with the copper content of 1.2%, the degree of oxidation was 96%.

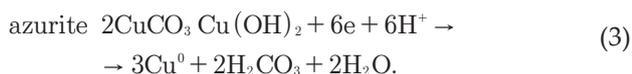
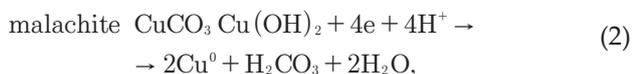
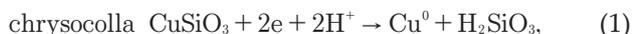
The process of electrochemical sulfiding is carried out in a temperature-controlled glass electrolyzer with the volume of 0.5 l, a titanium cathode and a lead anode are used as electrodes. The anode and cathode spaces are separated by an acid-resistant filter cloth. The crushed oxidized copper ore of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

The resulting pulp is loaded into the cathode space of the cell and subjected to electrical treatment under conditions of intensive mixing. After electrical treatment, the pulp is fed into the flotation machine. During flotation, the consumption of butyl xanthate is 120 g/l, foaming agent T 66 80 g/t. Flotation is carried out on a laboratory F-237 FL-A flotation machine.

The results of chemical analysis show that the studied ore, in addition to oxidized minerals malachite and azurite, mainly contains a finely disseminated difficult-to-dress mineral, chrysocolla, which is practically not amenable to flotation.

Elemental sulfur is used as a sulfidizing agent. It is introduced into the ore pulp based on the reaction of formation of easily floatable copper sulfides.

The essence of the process of electrochemical sulfiding consists in the fact that during the cathodic polarization of dispersed oxidized copper minerals [6], the latter is upon impact recovered to the elemental state according to the reactions:



Then, freshly formed very active copper, upon contact with elemental sulfur introduced as a sulfidizer, due to chemical affinity, forms easily floatable cuprous sulfide:



### Scientific results

Using the Seidel-Gauss method, there was studied the effect of the current density ( $i$ , 25-150 A/m<sup>2</sup>), sulfuric acid concentration ( $C_{\text{H}_2\text{SO}_4}$ , 3-24 g/l), the ratio of sulfur to copper contained in the ore (S:Cu, 0-1.5:1%), the electrolyte temperature ( $t$ , 25-65°C), the electrolysis duration ( $\tau$ , 20-120 min.), to recover copper into concentrate. Particular dependences were built (shown in Figure 1). The approximating functions were selected taking into account the physical meaning of the studied dependences.

Figure 1a shows that at a low cathode current density of 25 A/m<sup>2</sup>, the degree of copper recovery into the flotation concentrate is only 85.7%; this low recovery value is associated with the incomplete reduction of oxidized copper minerals in the ore to elemental one by reactions (1-3).

Relatively high copper recovery rates are achieved at current densities of 50 A/m<sup>2</sup> and higher.

The concentration of sulfuric acid (Figure 1, b) has

a significant impact on the extraction of copper. So, with increasing acidity of the solution (up to 24 g/l), partial dissolution occurs, i.e. opening the surface of resistant oxidized copper minerals, which contributes to the accelerated reaction (1-4) and increasing the copper recovery up to 98.56%.

The sulfidizer, elemental sulfur (Figure 1, c), has a significant effect on the copper recovery and tends to saturation; the most complete sulfidization of copper minerals occurs at the ratio of S:Cu equal to 1.5:1, probably at the indicated concentration the sulfur content is sufficient for sulfidation of all the oxidized copper minerals.

Increasing the temperature (Figure 1, d) leads to acceleration in the rate of the processes of reduction of oxidized minerals and their interaction with elemental sulfur to form easily floatable copper sulfides.

In the process duration interval of 60-120 min (Figure 1, e), the copper recovery naturally increases, which means that the reaction of the oxidized copper minerals reduction and the process of formation of copper sulfides proceeds completely.

For all the obtained partial dependences, the correlation coefficients and their significance were calculated, the values are given in Table 2.

The resulting equations for the copper recovery into the concentrate (Table 2), taking into account significant functions to describe the totality of acting factors, are generalized according to [8] in the form of their product normalized by the central experimental value to the power ( $n-1$ ), where  $n$  is number of particular functions. For conditions common to all the functions ( $i - 150\text{A/m}^2$ ,  $C_{\text{H}_2\text{SO}_4} - 24\text{ g/l}$ , S:Cu ratio - 1.5:1%,  $\tau - 60\text{ min}$ ,  $t - 65^\circ\text{C}$ ), the  $\epsilon_c$  value is 98.56%. In this case, the generalized equation will be expressed as follows:

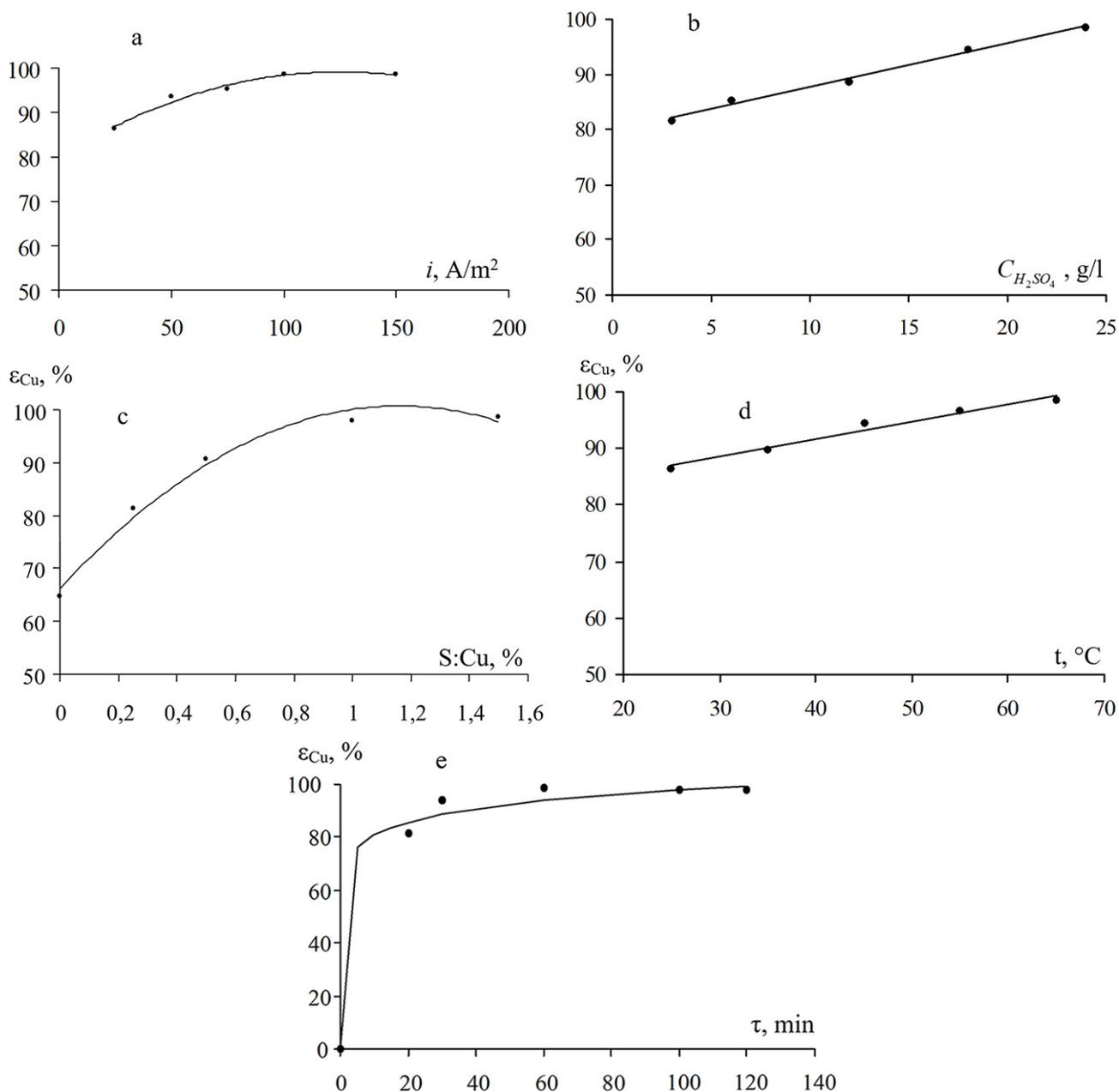
$$\epsilon_{\text{Cu}} = \frac{(-0,0012i^2 + 0,3089i + 79,943) \times (0,788C_{\text{H}_2\text{SO}_4} + 79,804) (0,3092t + 79,212)}{8873769594 [(-25,656(S:Cu))^2 + 59,512(S:Cu) + 66,193] (66,823\tau^{1/12})^{-1}} \quad (5)$$

Table 1 – Chemical composition (%) of oxidized copper ore

Cu <sub>tot</sub>	Cu <sub>oxid</sub>	S <sub>tot</sub>	Fe	Na <sub>2</sub> O	TiO <sub>2</sub>	SiO <sub>2</sub>	CaO	Al <sub>2</sub> O <sub>3</sub>	MgO
1.2	0.96	0.16	1.46	0.10	0.21	76.1	1.86	8.4	0.21

Table 2 – The correlation coefficient R and its significance t<sub>r</sub> for particular functions of copper recovery

Function	t <sub>r</sub>	R	Significance
$\epsilon = -0,0012i^2 + 0,3089i + 79,943$	8.10 > 2	0.966	significant
$\epsilon_{\text{Cu}} = 0,788C_{\text{H}_2\text{SO}_4} + 79,804$	167.4 > 2	0.994	significant
$\epsilon_{\text{Cu}} = -25,656(S:Cu)^2 + 59,512(S:Cu) + 66,193$	264.2 > 2	0.987	significant
$\epsilon_{\text{Cu}} = 0,3092t + 79,212$	53.7 > 2	0.984	significant
$\epsilon_{\text{Cu}} = 66,823\tau^{1/12}$	183.5 > 2	0.994	significant



a – on the current density; b – on the concentration of sulfuric acid; c – on addition of a sulfidizing agent, sulfur; d – on the temperature; e – on the duration of electrolysis

Copper recovery dependences on various factors

Comparison with then data calculated by direct substitution of experimentally determined values gives  $R=0.96$  and  $t_R=61.64 > 2$ ,  $\sigma_{abs}=0.32\%$ . The confidence interval calculated is determined in terms of  $t_R$  [9] using the formula:

$$\Delta = |99,4819 - 66,0389| \frac{1}{61,64} = \pm 0,55\%.$$

Sufficiently high accuracy of the obtained generalized equation makes it possible to carry out a kinetic analysis [10]. Since this requires taking a partial derivative with respect to duration ( $\tau$ ), i.e. the remaining variables are constant, this allows designating them in equation (5) together with the other coefficients by the total value

$$K = \frac{(-0,0012i^2 + 0,3089i + 79,943) \times 8873769594 [(-25,656(S:Cu))^2 + (0,788C_{H_2SO_4} + 79,804)(0,3092t + 79,212) + 59,512(S:Cu) + 66,193] (66,823\tau^{-1/2})^{-1}}$$

and the generalized equation will have the form:

$$\epsilon_{Cu} = K \cdot \tau^{-1/2}.$$

From here the process rate is equal to:

$$\frac{d\epsilon_{Cu}}{d\tau} = \frac{K}{12} \cdot \tau^{-3/2}. \quad (6)$$

By substituting  $\tau$  in the initial equation with  $\epsilon_{Cu}$ , there is obtained such an expression:

Table 3 – Nomogram of copper recovery. Extracted ≥ 95,0%

i, A/m <sup>2</sup>		25			50			75			150			200			
t, °C	$\frac{C_{H_2SO_4}}{\tau S:Cu}$	3	12	24	3	12	24	3	12	24	3	12	24	3	12	24	
25	20	0.2	36.3	39.5	43.7	37.9	41.2	45.6	38.9	42.3	46.8	40.6	44.1	48.8	41.4	44.9	49.7
		0.8	51.7	56.2	62.8	54.5	59.2	65.5	55.9	60.7	67.2	58.4	63.4	70.1	59.4	64.6	71.4
		1.5	54.7	59.5	64.4	55.9	60.8	67.2	57.4	62.3	68.9	59.9	65.0	71.9	60.9	66.2	73.3
	30	0.2	37.6	40.8	45.2	39.3	42.7	47.2	40.3	43.8	48.4	42.0	45.7	50.5	42.8	46.5	51.5
		0.8	53.5	58.2	64.9	56.5	61.3	67.8	57.9	62.9	69.6	60.4	65.6	72.6	61.5	66.8	73.9
		1.5	56.6	61.5	66.8	57.9	62.9	69.6	59.4	64.5	71.4	62.0	67.4	74.5	63.1	68.6	75.8
	70	0.2	39.9	43.4	48.0	41.7	45.3	50.1	42.7	46.5	51.4	44.6	48.5	53.6	45.4	49.4	54.6
		0.8	56.8	62.3	68.9	59.9	65.0	71.9	61.4	66.7	73.8	64.1	69.7	77.0	65.3	70.9	78.4
		1.5	60.1	63.9	70.7	61.4	66.7	73.8	63.0	68.5	75.7	65.8	71.5	79.0	66.9	72.7	80.5
	120	0.2	42.3	46.0	50.9	44.2	48.0	53.2	45.4	49.3	54.5	47.4	51.4	56.9	48.2	52.4	57.9
		0.8	60.3	66.1	73.1	63.5	69.0	76.4	65.2	70.8	78.3	68.0	73.9	81.7	69.2	75.2	83.2
		1.5	63.7	67.8	75.0	65.2	70.8	78.3	66.8	72.6	80.3	69.8	75.8	83.8	71.0	77.2	85.4
45	20	0.2	36.9	42.3	46.8	40.7	44.2	48.8	41.7	45.3	50.1	43.5	47.3	52.3	44.3	48.1	53.3
		0.8	55.4	60.8	67.2	58.4	63.5	70.2	59.9	65.0	71.9	62.5	67.9	75.1	63.7	69.2	76.5
		1.5	58.6	62.4	68.9	59.9	65.1	72.0	61.5	66.8	73.8	64.1	69.7	77.0	65.3	70.9	78.5
	30	0.2	40.3	43.8	48.5	42.1	45.7	50.6	43.2	46.9	51.9	45.0	48.9	54.1	45.9	49.8	55.1
		0.8	57.4	62.9	69.6	60.5	65.7	72.7	62.0	67.4	74.5	64.7	70.3	77.8	65.9	71.6	79.2
		1.5	60.7	64.6	71.4	62.0	67.4	74.5	63.6	69.1	76.4	66.4	72.1	79.8	67.6	73.4	81.2
	70	0.2	42.7	46.5	51.4	44.6	48.5	53.6	45.8	49.8	55.0	47.8	51.9	57.5	48.7	52.9	58.5
		0.8	60.8	66.8	73.8	64.2	69.7	77.0	65.8	71.5	79.0	68.7	74.6	82.5	69.9	75.9	84.0
		1.5	64.4	68.5	75.8	65.8	71.5	79.0	67.5	73.3	81.1	70.5	76.5	84.7	71.7	77.9	86.2
	120	0.2	45.3	49.3	54.5	47.4	51.5	56.9	48.6	52.8	58.4	50.7	55.1	60.9	51.6	56.1	62.0
		0.8	64.6	70.8	78.3	68.0	73.9	81.8	69.8	75.8	83.9	72.8	79.1	87.5	74.2	80.6	89.1
		1.5	68.3	72.7	80.4	69.8	75.8	83.9	71.6	77.8	86.0	74.7	81.2	89.8	76.1	82.7	91.4
55	20	0.2	40.2	43.7	48.4	42.0	45.6	50.5	43.0	46.8	51.8	44.9	48.8	54.0	45.8	49.7	55.0
		0.8	57.3	62.8	69.5	60.4	65.6	72.5	61.9	67.2	74.4	64.6	70.2	77.6	65.8	71.4	79.0
		1.5	60.6	64.4	71.3	61.9	67.3	74.4	63.5	68.9	76.3	66.3	72.0	79.6	67.5	73.3	81.1
	30	0.2	41.6	45.3	50.0	43.5	47.3	52.3	44.6	48.5	53.6	46.6	50.6	55.9	47.4	51.5	56.9
		0.8	59.3	65.0	71.9	62.5	67.9	75.0	64.0	69.6	76.9	66.9	72.7	80.4	68.0	73.9	81.8
		1.5	62.7	66.7	73.8	64.1	69.6	77.0	65.7	71.4	78.9	68.6	74.5	82.4	69.8	75.9	83.9
	70	0.2	44.2	48.0	53.1	46.1	50.1	55.5	47.3	51.5	56.8	49.4	53.7	59.4	50.3	54.6	60.4
		0.8	62.8	68.9	76.3	66.3	72.0	79.6	67.9	73.8	81.7	70.9	77.0	85.3	72.2	78.5	86.8
		1.5	66.5	70.8	78.3	68.0	73.8	81.8	69.7	75.7	83.8	72.8	79.0	87.5	74.1	80.5	89.0
	120	0.2	46.8	50.9	56.4	48.9	53.2	58.8	50.2	54.5	60.3	52.4	56.9	62.9	53.4	57.9	64.1
		0.8	66.7	73.2	80.9	70.3	76.4	84.5	72.1	78.3	86.6	75.3	81.8	90.5	76.6	83.3	92.1
		1.5	70.6	75.0	83.0	72.1	78.4	86.7	73.9	80.4	88.9	77.2	83.9	92.8	78.6	85.4	94.5
65	20	0.2	41.5	45.1	49.9	43.4	47.1	52.1	44.5	48.3	53.4	46.4	50.4	55.8	47.3	51.3	56.8
		0.8	59.0	64.8	71.7	62.3	67.7	74.8	63.8	69.4	76.7	66.7	72.5	80.1	67.9	73.7	81.6
		1.5	62.5	66.5	73.6	63.9	69.4	76.8	65.5	71.2	78.7	68.4	74.3	82.2	69.6	75.7	83.7
	30	0.2	42.9	46.7	51.7	44.9	48.8	53.9	46.0	50.0	55.3	48.0	52.2	57.7	48.9	53.2	58.8
		0.8	61.2	67.1	74.2	64.5	70.0	77.5	66.1	71.8	79.5	69.0	75.0	82.9	70.3	76.4	84.4
		1.5	64.7	68.8	76.1	66.1	71.9	79.5	67.8	73.7	81.5	70.8	76.9	85.1	72.1	78.3	86.6
	70	0.2	45.6	49.5	54.8	47.6	51.7	57.2	48.8	53.0	58.7	50.9	55.4	61.3	51.9	56.4	62.4
		0.8	64.9	71.2	78.7	68.4	74.3	82.2	70.1	76.2	84.3	73.2	79.6	88.0	74.5	81.0	89.6
		1.5	68.6	73.0	80.8	70.2	76.3	84.3	71.9	78.2	86.5	75.1	81.6	90.3	76.5	83.1	91.9
	120	0.2	48.3	52.6	58.2	50.5	54.9	60.7	51.8	56.3	62.3	54.1	58.8	65.0	55.0	59.8	66.2
		0.8	68.8	75.5	83.5	72.6	78.8	87.2	74.4	80.9	89.4	77.7	84.4	93.4	79.1	85.9	95.0
		1.5	72.8	77.5	85.7	74.5	80.9	89.5	76.4	82.9	91.7	79.7	86.6	95.8	81.1	88.2	97.5

$$\tau = \left( \frac{\varepsilon_{Cu}}{K} \right)^{12}, \quad (7)$$

that in addition to using kinetic analysis, provides the ability to calculate the duration of the process for any given value of copper recovery.

Substituting expression (7) into equation (6), there is obtained the velocity equation:

$$\frac{d\varepsilon_{Cu}}{d\tau} = \frac{K}{12} \left[ \left( \frac{\varepsilon_{Cu}}{K} \right)^{12} \right]^{\frac{11}{12}} = \frac{K}{12} \left( \frac{\varepsilon_{Cu}}{K} \right)^{11}. \quad (8)$$

The resulting process rate model can be processed for any combination of various factors, fixing all isotherms. We will limit for our purposes for the copper recovery equal to 52%.

The values of the process rate obtained from equation (8) for five temperatures (25, 35, 45, 55, 65 °C) will be converted into Arrhenius coordinates  $\ln \frac{d\varepsilon_{Cu}}{d\tau} - \frac{1}{T}$  with the construction of a straight-line dependence. From this dependence, we find the magnitude of the apparent activation energy. The latter amounted to 33.405 kJ/mol, which indicates the transition mode of the process, i.e. the diffusion rate and the reaction rate are approximately comparable.

Using the obtained model (5), a nomogram was constructed for the copper recovery into concentrate

(Table 3). With the help of the nomogram, other conditions for the copper recovery into the concentrate were also determined.

The kinetic parameters of the process were studied, and it was established that the formation process proceeded in the diffusion mode.

### Conclusion

Thus, a new method of electrochemical sulfidation of complex (refractory) oxidized copper ores was developed, where elemental sulfur was used as a sulfidizer. Using the Seidel-Gauss method, the effect of current density, the concentration of sulfuric acid, the sulfidizing agent, sulfur, the electrolyte temperature, the electrolysis duration on the degree of copper recovery into flotation concentrate was studied. A mathematical model of copper extraction was obtained and a nomogram was calculated, from which it followed that a given sufficiently high degree of copper extraction  $\geq 95.0\%$  can be achieved in the range of current densities of 25-150 A/m<sup>2</sup>, with the addition of a sulfidizing agent at the S:Cu ratio equal to 1.5:1 and the duration of 60-120 minutes.

The kinetic parameters of the process were studied, and it was established that the formation process proceeded in the diffusion mode.

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### Элементті күкіртпен тотыққан қиынбайытылатын мыс рудаларын электрхимиялық сульфидттеу

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**Аңдатпа.** Қиынбайытылатын тотыққан мыс рудаларын электрхимиялық сульфидтеудің жаңа әдісі жасалды. Байыту көрсеткіштерін жоғарылату үшін сульфидизатор ретінде элементті күкірт пайдаланылған. Зейдель-Гаусс әдісімен тоқ тығыздығының, күкірт қышқылы, сульфидтеуші элементті күкірт концентрацияларының, электролит температурасының, электролиз ұзақтылығының мыстың флотоконцентратқа бөліну деңгейіне әсері зерттелді. Жүргізілген зерттеулердің негізінде математикалық модель алынды және мысты флотоконцентратқа бөліп алу номограммасы есептелді, ол бойынша электролиз жағдайында тоқ тығыздығының, электролит температурасының, электролиз ұзақтылығының, сульфидтеуші элемент күкірттің, күкірт қышқылының тиімді концентрацияларында, мыстың флотоконцентратқа бөліну деңгейі ең биік көрсеткішке  $\geq 95,0\%$  жетеді.

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

#### **Электрохимическая сульфидизация окисленных труднообогатимых медных руд элементной серой**

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**Аннотация.** Разработан новый способ электрохимической сульфидизации окисленных труднообогатимых медных руд. Для увеличения показателей обогащения в качестве сульфидизатора применили элементную серу. Методом Зейделя-Гаусса изучены влияние плотности тока, концентрации серной кислоты, сульфидизатора – серы, температуры электролита, продолжительности электролиза на степень извлечения меди во флотоконцентрат. Получена математическая модель извлечения меди в концентрат и рассчитана номограмма, из которой следует, что в условиях электролиза при оптимальной концентрации сульфидизатора – элементной серы, серной кислоты, плотности тока, температуры электролита, продолжительности электролиза степень извлечения меди во флотоконцентрат достигает максимального значения не менее 95,0%.

**Ключевые слова:** окисленная медная руда, измельчение, электролиз, катод, анод, сульфидизатор, сера элементная, плотность тока, электролит, серная кислота, флотация, извлечение, сульфидный медный концентрат.

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