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Research article

Научная статья



## Processing of chalcopyrite concentrate by sulfating roasting

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**Abstract:** Chalcopyrite ( $\text{CuFeS}_2$ ) is one of the primary minerals processed on an industrial scale for copper production and often dominates copper concentrates sent for pyrometallurgical processing. This study demonstrates the efficient and selective extraction of copper from chalcopyrite concentrate through sulfating roasting, sulfuric acid leaching, and solvent extraction. At a roasting temperature of 700 °C for 1.5 h, chalcopyrite fully decomposes into hematite ( $\text{Fe}_2\text{O}_3$ ) and chalcantite ( $\text{CuSO}_4$ ). Leaching the calcine with a 0.02 M sulfuric acid solution transfers most of the copper to the aqueous phase, while iron concentrates in the solid residue. Additionally, precious metals concentrate in the residue after leaching of the calcine, with the following content in g/t: Pd – 41.61, Pt – 5.65, Ag – 96.22, Au – 4.81. The removal of iron from the leach solution using solvent extraction with di-2-ethylhexyl phosphoric acid was highly effective: with a 25 % extractant solution and an organic-to-aqueous ratio of 1:1 over two stages, the iron concentration in the aqueous phase dropped from 3.05 to 0.01 g/dm<sup>3</sup>, and with an organic-to-aqueous ratio of 1:2 over four stages, it decreased to 0.006 g/dm<sup>3</sup>. After iron purification and solution evaporation, copper sulfate was obtained with the following composition (%):  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  – 99.84 (equivalent to 25.42 % copper), Ni – 0.014, Al – 0.007, Fe – 0.0003, As – 0.0002.

**Keywords:** copper concentrate, chalcopyrite, roasting, copper, iron, extraction, precious metals.

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## Переработка халькопиритового концентрата с применением сульфатизирующего обжига

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**Аннотация:** Халькопирит ( $\text{CuFeS}_2$ ) является одним из основных минералов, перерабатываемых в промышленном масштабе для получения меди, который зачастую преобладает в медных концентратах, поступающих на последующую пирометаллургическую переработку. В работе показана возможность эффективного и селективного выделения меди из халькопиритового концентрата с применением сульфатизирующего обжига, сернокислотного выщелачивания и жидкостной экстракции. Установлено, что при температуре обжига 700 °C в течение 1,5 ч происходит полное разложение халькопирита с образованием гематита ( $\text{Fe}_2\text{O}_3$ )

и халькоцианита ( $\text{CuSO}_4$ ). В результате выщелачивания огарка раствором серной кислоты концентрацией 0,02 М в водную фазу переходит большая часть меди, в то время как железо концентрируется в твердом остатке. Кроме того, в результате выщелачивания огарка в остатке концентрируются и благородные металлы, содержание которых составляет, г/т: Pd – 41,61, Pt – 5,65, Ag – 96,22, Au – 4,81. Очистка раствора выщелачивания от железа посредством жидкостной экстракции ди-2-этилгексилфосфорной кислотой показала высокую эффективность: при использовании 25 %-ного раствора экстрагента при соотношении О : В = 1 : 1 на двух ступенях концентрация железа в водной фазе снижается с 3,05 до 0,01 г/дм<sup>3</sup>, а при О : В = 1 : 2 на четырех ступенях – до 0,006 г/дм<sup>3</sup>. После железоочистки и упаривания раствора получен медный купорос, содержащий, %:  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  – 99,84 (в пересчете на медь – 25,42), Ni – 0,014, Al – 0,007, Fe – 0,0003, As – 0,0002.

**Ключевые слова:** медный концентрат, халькопирит, обжиг, медь, железо, экстракция, благородные металлы.

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## Introduction

Chalcopyrite ( $\text{CuFeS}_2$ ) is one of the main industrial copper-bearing minerals, accounting for approximately 70 % of the world's copper reserves. The predominant method for extracting copper from chalcopyrite ores is froth flotation, which yields a copper-rich concentrate [1]. Most of the copper is produced using the metallurgical process of smelting—converting—refining. During the smelting of high-grade sulfide ores or concentrates, a matte containing up to 40 % Cu is obtained [2].

Despite the high efficiency of conventional pyrometallurgical methods for processing copper concentrates (CC) [3], alternative processing methods are currently being actively studied, although they have not yet found widespread industrial application. For example, various methods of leaching chalcopyrite-containing concentrates are being investigated to reduce sulfur dioxide emissions into the atmosphere [4]: pressure acid leaching [5], microwave acid leaching [6], chloride leaching [5], and leaching with mineral salt solutions combined with sulfuric acid [7], among others. Hydrometallurgical methods also help to prevent arsenic, which is often present in sulfide copper concentrates, from entering the gas phase, reducing the cost of copper production by eliminating the need for specialised dust and gas cleaning systems [8]. However, hydrometallurgical processing of chalcopyrite is challenging due to the formation of a passivating layer on the mineral surface, necessitating the use of strong oxidants such as hydrogen peroxide [9] or ozone [10], or its preliminary treatment, such as mechanoactivation [11], which significantly complicates its processing. The intensification of hydrometallurgical processing of sulfide CCs can also be achieved through the application of pressure leaching [12; 13].

Bacterial leaching of chalcopyrite-containing materials is developing rapidly [13–15], but bioleaching is characterised by a slow reaction rate and the need to

maintain optimal conditions for bacterial activity, which limits the practical application of this method.

In addition to the methods mentioned above, sulfating roasting can be used to process sulfide and chalcopyrite CCs by converting copper and iron from sulfides to sulfates [16–19]. The advantage of this method is the use of lower temperatures compared to conventional oxidative roasting [20]. However, when such a concentrate is leached, both metals are transferred to the aqueous phase, leading to the challenge of separating iron and copper by hydrometallurgical means. According to literature data, chalcopyrite decomposition into iron and copper sulfides occurs at roasting temperatures of 330–357 °C, and these sulfides are converted into the corresponding sulfates between 357–555 °C [21]. As the roasting temperature increases to 555–613 °C, iron sulfate decomposes into iron and sulfur oxides, while a higher temperature is required for copper sulfate decomposition [21], indicating the potential for selective copper extraction from chalcopyrite concentrate during the leaching stage. Additionally, the behaviour of precious metals, often present in sulfide CCs, during roasting and leaching of chalcopyrite concentrate is of interest. Therefore, the aim of this study is to investigate the behaviour of copper, iron, and precious metals during the roasting of chalcopyrite concentrate and leaching of the calcine, followed by iron removal to obtain copper sulfate of the required quality.

## Experimental technique

The study used chalcopyrite copper concentrate obtained from the beneficiation of copper-sulfide ore from the Chineyskoe deposit, with the following composition (%): Cu – 20.35, Fe – 23.07, S – 22.91, Ni – 0.47, Ca – 0.75, Al – 0.87, Co – 0.05, and trace elements (g/t): As – 138.95, Se – 98.31, Te – 15.74,

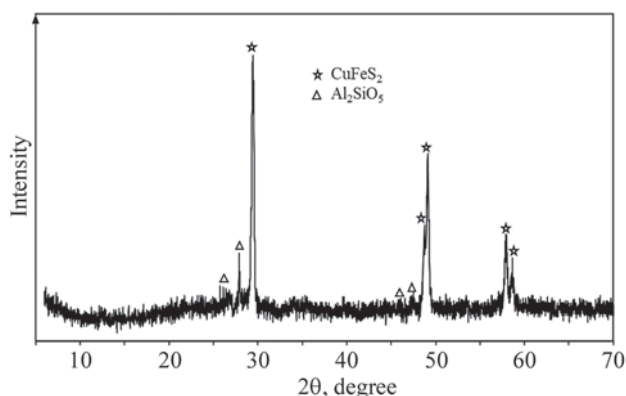


Fig. 1. Phase composition of the initial CC

Рис. 1. Фазовый состав исходного МК

V — 128.12, Mo — 249.18, Pd — 25.95, Pt — 2.84, Au — 2.97, Ag — 60.58. The low arsenic content in the concentrate allows for the use of pyrometallurgical processing methods. The phase composition of the initial concentrate is primarily chalcocyanite with a kyanite ( $\text{Al}_2\text{SiO}_5$ ) admixture (Fig. 1).

The concentrate was ground to a size of  $<74\ \mu\text{m}$  and held in a laboratory electric furnace (SNOL 7.2/1300, Umega Group, Lithuania) for 0.25 to 2.0 h at temperatures ranging from 650 to 900 °C. The heating rate to the required temperature was 10 °C/min, and the sample mass was 50 g. The resulting calcine was leached with sulfuric acid solutions of various concentrations. Iron (III) extraction from the leach solution was carried out using di-2-ethylhexyl phosphoric acid (D2EHPA) (Volgogradpromproject, Russia) in an inert aliphatic diluent RJ-3 (Volganeftchem, Russia) for 5 minutes at different organic-to-aqueous ratios and temperatures. The concentrations of non-ferrous metals and iron were determined using atomic absorption spectrometry (“AAnalyst 400”, Perkin Elmer, USA), while concentrations of precious metals and trace elements were measured by inductively coupled plasma mass spectrometry (ICP-MS) on an ELAN 9000 DRC-e instrument (Perkin Elmer, USA). The phase composition of the samples was assessed by X-ray phase analysis (XRD) using an XRD-6000 diffractometer (Shimadzu, Japan).

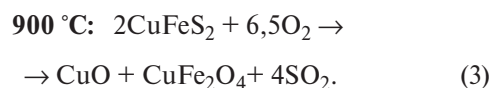
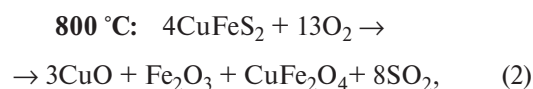
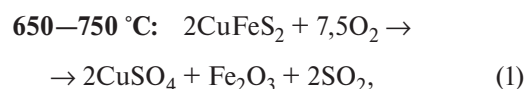
## Results and discussion

### 1. Thermal treatment of the copper concentrate

The roasting of the copper concentrate was conducted for 120 min at temperatures ranging from 650 to 900 °C. X-ray diffraction (XRD) patterns of the con-

centrates roasted at different temperatures are shown in Fig. 2. It was found that at lower roasting temperatures (650–750 °C), the main iron-bearing phase is hematite ( $\text{Fe}_2\text{O}_3$ ), while the copper-bearing phase is chalcocyanite ( $\text{CuSO}_4$ ). However, at 800 °C, copper oxide ( $\text{CuO}$ ) and copper ferrite ( $\text{CuFe}_2\text{O}_4$ ), begin to form, which is associated with the decomposition of copper sulfate and the initial interaction between copper oxide and iron oxide. This interaction is confirmed by the XRD pattern of the sample roasted at 900 °C, where characteristic peaks of hematite are absent, and copper ferrite becomes the primary iron-containing phase. Additionally, copper is present in the form of oxide.

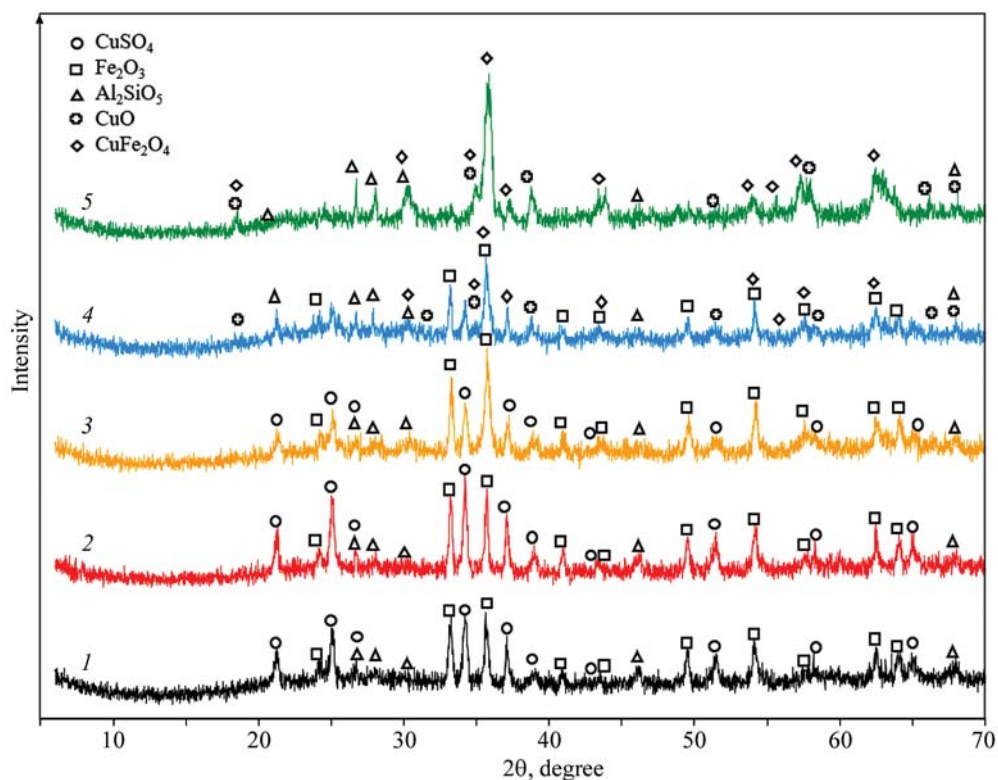
Thus, the processes occurring at different roasting temperatures can be summarised in the following equations:



The changes in the phase composition of the calcine over time were studied at a temperature of 700 °C. It was found that chalcocyanite ( $\text{CuFeS}_2$ ) could no longer be identified after 90 min of roasting, with copper and iron present only as chalcocyanite and hematite (Fig. 3), indicating the potential for effective separation of iron and copper from the roasted concentrate during leaching.

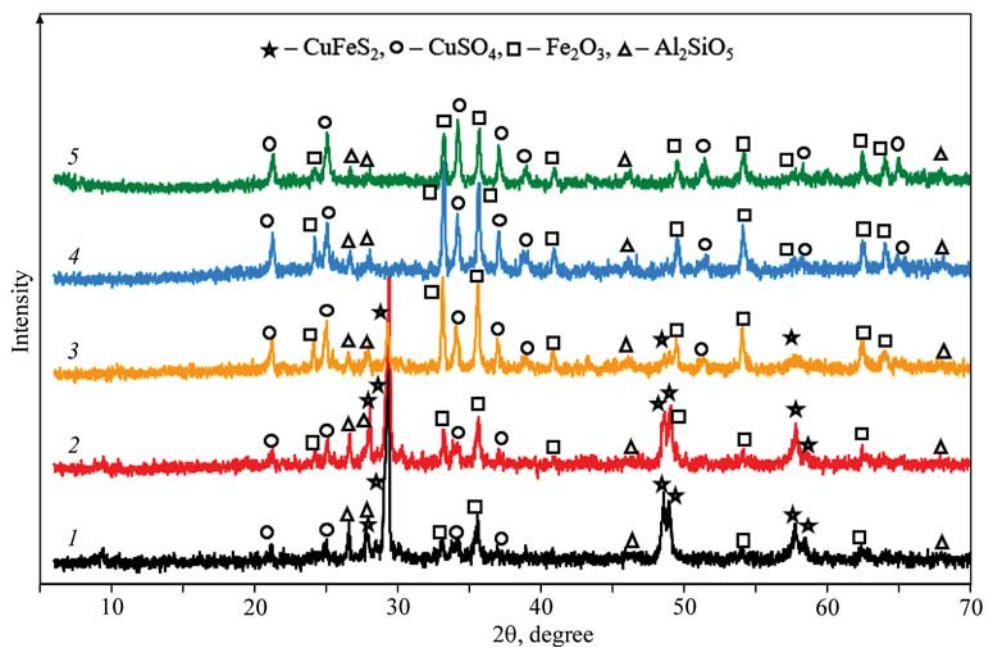
### 2. Sulfuric acid leaching of the roasted concentrate

During the study of the leaching process of the calcine obtained after roasting at 700 °C, using sulfuric acid solutions of various concentrations, it was found that leaching with a solution containing 0.02 M  $\text{H}_2\text{SO}_4$ , resulted in copper extraction of  $E(\text{Cu}) = 83\%$  and iron extraction of  $E(\text{Fe}) = 4,6\%$  (Fig. 4). As the acid concentration increased to 1.5 M, copper and iron extraction increased to 93 % and 14 %, respectively. These results are consistent with those shown in Figs. 2 and 3: during roasting, copper transitions into water-soluble chalcocyanite, while iron remains as insoluble hematite. As the acid concentration increases, hematite reacts with sulfuric acid to form soluble iron (III) sulfate, leading to an increase in  $\text{Fe(III)}$  concentration in the leach solu-



**Fig. 2.** Phase composition of the calcine after roasting for 120 min at various temperatures  
 $t$ , °C: 1 – 650, 2 – 700, 3 – 750, 4 – 800, 5 – 900

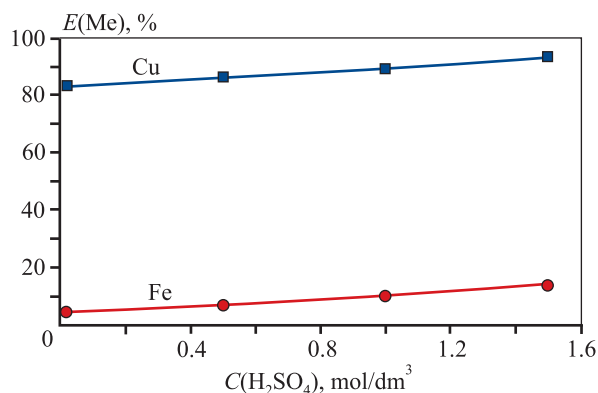
**Рис. 2.** Фазовый состав огарка после обжига в течение 120 мин при различных температурах  
 $t$ , °C: 1 – 650, 2 – 700, 3 – 750, 4 – 800, 5 – 900



**Fig. 3.** Phase composition of the calcine at different roasting times at 700 °C  
 $\tau$ , min: 1 – 15, 2 – 30, 3 – 60, 4 – 90, 5 – 120

**Рис. 3.** Фазовый состав огарка в зависимости от времени обжига при  $t = 700$  °C  
 $\tau$ , мин: 1 – 15, 2 – 30, 3 – 60, 4 – 90, 5 – 120





**Fig. 4.** Effect of  $H_2SO_4$  concentration on the leaching of copper concentrate calcine

$t = 60^\circ C$ ,  $\tau = 1$  h,  $S : L = 1 : 5$

**Рис. 4.** Влияние концентрации  $H_2SO_4$  на выщелачивание огарка МК

$t = 60^\circ C$ ,  $\tau = 1$  ч,  $T : Ж = 1 : 5$

tion. The increase in copper concentration in the solution may be related to the presence of copper oxide in the calcine, which is not detectable by XRD but reacts with sulfuric acid during leaching, resulting in higher copper concentrations in the solution. Although higher acid concentrations improve copper extraction, the use of a less concentrated leaching solution is preferable, as increasing the acid concentration complicates the subsequent separation of iron and copper.

In addition to the behavior of the major components during sulfuric acid leaching of the calcine, the distribution of precious metals and other impurities in the concentrate is also of significant interest. Analysis of the composition of the leaching residues showed that approximately 60 % of nickel is transferred into the solution, while precious metals almost entirely remain in the solid residue (Table 1). Other trace elements, such as arsenic, selenium, tellurium, vanadium, and molybdenum, are barely leached by 0.02 M  $H_2SO_4$ , concentrating in the leach residue. The content of trace elements in the residue after leaching with 1.5 M  $H_2SO_4$  is lower than after leaching with 0.02 M  $H_2SO_4$ , especially noticeable for arsenic and molybdenum. Thus, with increasing acid concentration, more of these components are extracted into the solution, reducing the selectivity of the leaching process. Based on the data in Fig. 4 and Table 2, 0.02 M  $H_2SO_4$  was chosen as the leaching solution for the roasted concentrate.

A study of the leaching kinetics of the calcine showed that copper extraction into the solution in the first 15 min reaches 72.4 %, gradually increasing to 83.5 % after 60 min (Fig. 5). In contrast, iron extraction in the first 15 min is only 0.9 %, but after 60 min, it increases

to 4.6 %. Extending the leaching time does not significantly enhance the extraction of either copper or iron, so subsequent leaching processes were conducted for 60 min to maximize copper extraction.

An investigation of the effect of the solid-to-liquid ratio on concentrate leaching showed that copper extraction decreases slightly: at an  $S : L$  ratio of 1 : 10, copper extraction is 85.9 %, while at  $S : L = 1 : 2$ , it is 74.6 %, corresponding to copper concentrations of 17.46 and 75.96 g/dm<sup>3</sup>, respectively (Fig. 6). Throughout the  $S : L$  range, iron extraction does not exceed 5.6 %, which is observed at  $S : L = 1 : 10$ , and its maximum concen-

**Table 1. Metals distribution during leaching of copper concentrate calcine ( $S : L = 1 : 10$ ,  $t = 60^\circ C$ ,  $\tau = 1$  h)**

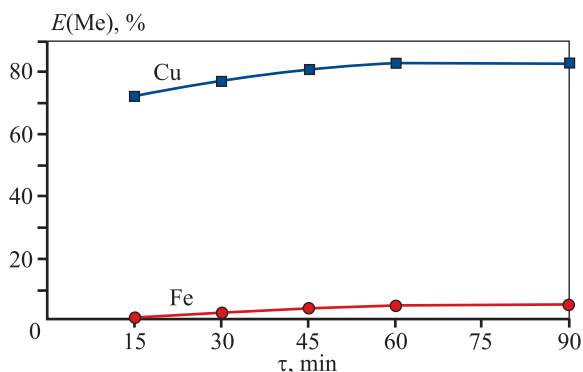
Таблица 1. Распределение металлов при выщелачивании огарка МК ( $T : Ж = 1 : 10$ ,  $t = 60^\circ C$ ,  $\tau = 1$  ч)

Me	Unit	$C(Me)_{initial}$	Composition of leaching residues of the calcine	
			$C(H_2SO_4) = 0.02$ M	$C(H_2SO_4) = 1.5$ M
Ni	%	0.47	0.31	0.30
Cu		20.35	2.0	1.6
Ca		0.75	0.70	0.70
Co		0.05	0.02	0.01
Al		0.87	1.30	1.62
Fe		23.07	40.8	40.1
As	г/т	138.95	257.80	134.20
Se		98.31	128.30	97.30
Te		15.74	22.63	25.96
Ag		60.58	96.22	101.49
Pd		25.95	41.61	45.44
Au		2.97	4.81	5.59
Pt		2.84	5.65	5.06
V		128.12	208.3	192.9
Mo		249.18	349.8	217.1

**Table 2. Effect of temperature on the leaching of copper concentrate calcine ( $S : L = 1 : 5$ ,  $C(H_2SO_4) = 0.02$  M,  $\tau = 1$  h)**

Таблица 2. Влияние температуры на выщелачивание огарка МК ( $T : Ж = 1 : 5$ ,  $C(H_2SO_4) = 0,02$  М,  $\tau = 1$  ч)

$t$ , $^\circ C$	$E(Cu)$ , %	$E(Fe)$ , %
20	81.5	4.5
40	82.3	4.6
60	83.2	4.6

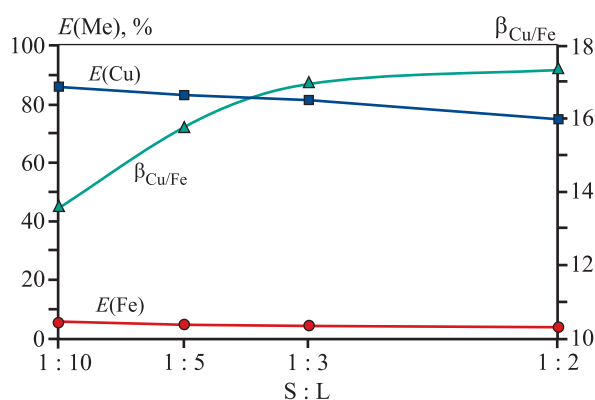


**Fig. 5.** Kinetics of calcine leaching

$S : L = 1 : 5$ ,  $C(H_2SO_4) = 0.02$  M,  $t = 60$  °C

**Рис. 5.** Кинетика выщелачивания огарка

$T : Ж = 1 : 5$ ,  $C(H_2SO_4) = 0.02$  M,  $t = 60$  °C



**Fig. 6.** Effect of  $S : L$  on the extraction and separation of Fe and Cu during leaching

$C(H_2SO_4) = 0.02$  M,  $\tau = 1$  h,  $t = 60$  °C

**Рис. 6.** Влияние  $T : Ж$  на извлечение и разделение Fe и Cu при выщелачивании

$C(H_2SO_4) = 0.02$  M,  $\tau = 1$  ч,  $t = 60$  °C

tration is  $4.38 \text{ g/dm}^3$  at  $S : L = 1 : 2$ . Notably, copper extraction at  $S : L = 1 : (3+5)$  is 81.2 % and 83.1 %, respectively, indicating a more efficient process than leaching at  $S : L = 1 : 2$ . However, at  $S : L = 1 : 10$ , the copper concentration in the solution ( $17.46 \text{ g/dm}^3$ ) is insufficient for further processing.

The  $S : L$  ratio also plays a crucial role in the separation of copper and iron during leaching. While the initial  $Cu/Fe$  ratio in the concentrate is 0.88, during leaching, it increases in the solution to 13.5 and 17.3 at  $S : L = 1 : 10$  and  $1 : 2$ , respectively, with a non-linear dependence (see Fig. 6). The lack of direct proportionality can be explained by the increased copper concentration in the solution at lower  $S : L$  ratios, which likely reduces the activity of sulfuric acid, leading to less interaction with hematite. Consequently, the optimal  $S : L$  ratio

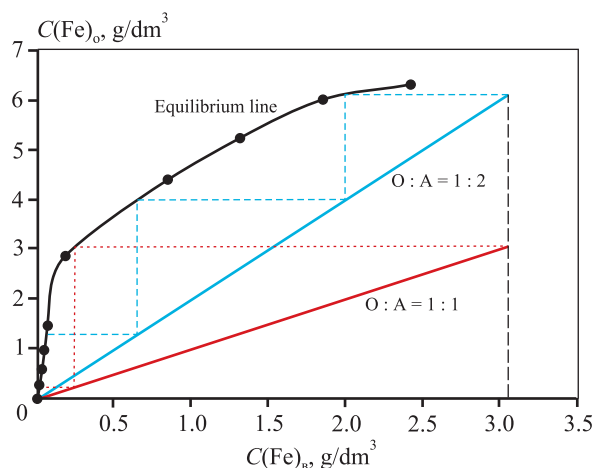
range is  $1 : (5+3)$ , where copper and iron concentrations in the solution are  $33.8\text{--}55.0 \text{ g/dm}^3$  and  $2.15\text{--}3.25 \text{ g/dm}^3$ , respectively, with a separation coefficient ( $\beta_{Cu/Fe}$ ) of 15.7–16.9.

It was found that increasing the leaching temperature slightly improves copper extraction into the solution and has little effect on iron extraction (Table 2). The minimal effect of temperature on copper extraction is due to the lack of a chemical reaction during copper leaching, as the process is based on the dissolution of chalcocyanite in the liquid phase. The lack of temperature influence on iron extraction is explained by the low acid concentration in the leaching solution.

### 3. Iron solvent extraction from the leach solution

To separate iron (III) and copper (II) in sulfate solutions, the hydrolytic method of precipitating iron as an insoluble hydroxide (iron cake) is often used [22]. However, some copper co-precipitates with the cake, leading to copper losses in the waste. Therefore, it was proposed to separate iron (III) and copper (II) by extracting Fe(III) from the copper solution using extractants based on D2EHPA, which has proven effective for extracting iron (III) from a copper electrolyte [23]. A 25 % solution of technical-grade D2EHPA in an inert aliphatic diluent was used for extraction. Based on the McCabe-Thiele plot for Fe(III) solvent extraction from the leach solution, it was found that iron is nearly quantitatively extracted in two stages at  $O : A = 1 : 1$  and in four stages at  $O : A = 1 : 2$ , with residual Fe(III) concentrations in the raffinate of  $0.01$  and  $0.006 \text{ g/dm}^3$ , respectively (Fig. 7), corresponding to extraction rates of 99.7 % and 99.8 %. Notably, copper (II) extraction does not exceed 2.7 %, as under the studied conditions, D2EHPA has a higher affinity for iron (III) than for copper (II) [24], ensuring more efficient Fe(III) extraction. As a result, the organic phase is saturated with iron (III), which prevents the co-extraction of copper (II).

The organic phase saturated with iron (III) can be regenerated by stripping using hydrochloric [25] or oxalic [26] acids, allowing the extractant to be recycled back into the extraction stage. After thorough iron extraction, the obtained raffinate was evaporated and cooled, leading to the precipitation of copper sulfate, which contained (%):  $CuSO_4 \cdot 5H_2O$  — 99.84 (equivalent to 25.42% copper), Ni — 0.014, Al — 0.007, Fe — 0.0003, As — 0.0002. The resulting mother liquor can be either evaporated further or used for leaching roasted concentrate. If nickel impurities concentrate in the solution during further processing, the filtrate can be treated for effec-



**Fig. 7.** McCabe-Thiele plot for Fe(III) solvent extraction by D2EHPA

$C(\text{Cu}) = 35 \text{ g/dm}^3$ ,  $C(\text{Fe}^{3+}) = 3.05 \text{ g/dm}^3$ ,  $C(\text{H}_2\text{SO}_4) = 0.02 \text{ M}$ ,  $t = 20^\circ\text{C}$ ,  $\tau = 5 \text{ min}$

**Рис. 7.** Изотерма экстракции Fe(III) Д2ЭГФК

$C(\text{Cu}) = 35 \text{ г/дм}^3$ ,  $C(\text{Fe}^{3+}) = 3,05 \text{ г/дм}^3$ ,  $C(\text{H}_2\text{SO}_4) = 0,02 \text{ M}$ ,  $t = 20^\circ\text{C}$ ,  $\tau = 5 \text{ мин}$

tive copper and nickel separation, for example, by solvent extraction [27].

Thus, sulfating roasting allows for not only the effective separation of copper from iron and precious metals during leaching but also the production of copper sulfate with less than 0.16 % impurities after iron solvent extraction from the copper sulfate solution.

## Conclusion

The method for processing chalcopirite copper concentrate presented in this study allows for avoiding one of the main challenges in non-ferrous hydrometallurgy — the stage of iron removal from solutions via hydrolytic means. The difference in the thermal stability of iron and copper sulfates within the temperature range of 650–750 °C enables the conversion of most of the iron into oxide, while copper remains in the form of sulfate after roasting. As a result, during sulfuric acid leaching, copper transitions into the aqueous phase, and iron remains in the insoluble residue. It was established that leaching is best performed with a 0.02 M sulfuric acid solution for 1 hour at a solid-to-liquid ratio of 1 : (3+5) and a temperature of 20 °C. The possibility of separating copper and precious metals, which concentrate in the solid phase along with iron during leaching, is also significant. The low content of iron and sulfuric acid in the leach solution enables iron removal via selective cation exchange solvent extraction. Using a 25 % D2EHPA solution in an inert

aliphatic diluent at O : A = 1 : 1 over two stages results in 99.7 % iron extraction. The obtained raffinate can be further used for copper sulfate production or for copper electrodeposition.

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**A.Yu. Sokolov** — defined the objectives of the study, conducted the experiments, contributed to the discussion of the results, and wrote the article.

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**А.Ю. Соколов** — определение цели работы, проведение экспериментов, участие в обсуждении результатов, подготовка статьи.

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