

DOI: 10.31643/2025/6445.39

Metallurgy



Effect of amino acids on the extraction of copper from sub-conditional raw materials

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	ABSTRACT The decrease in the quality of mineral raw materials processed by industry, as well as the increase in requirements for environmental protection, necessitate the development of new directions in the technology of their processing. Biogeotechnology refers to one of the modern areas of
Received: May 28, 2024 Peer-reviewed: June 28, 2024 Accepted: September 13, 2024	scientific and technological progress in the field of processing mineral raw materials. Biologically active additives are increasingly used along with the use of microorganisms in the extraction processes for non-ferrous and precious metals, in particular amino acids which are an integral part of the culture liquid of microorganisms. The processes of sulfuric acid leaching of copper from low-grade ore in the presence of amino acids of different structures and their effect on copper electrolysis were studied. Low-grade ore from one of the Kazakhstan deposits was used as the starting raw material. The copper content in the ore is 0.39%. The diffusion nature of the restrictions was established during the study of the kinetics of the process of sulfuric acid leaching of copper, and the rate constants and value of the effective activation energy were calculated, which amounted to 1.817 kJ/mol. The effect of amino acids with different structures on the leaching process was studied. The positive effect was increased in the glycine - leucine - cysteine histidine - asparagine series. Depending on the structure of the amino acid, the degree of copper extraction into the solution increases in the range of 1-15%. The effect of aminoacetic acid as a biologically active additive on the process of electrolysis of copper from poor solutions was studied. It was established that the process is inhibited by the reduction of monovalent copper to the metallic state in the presence of glycine.
	Keywords: amino acids, copper, leaching, mineral raw materials, microorganisms.
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Introduction

Currently, SX-EW technology (leaching - liquid extraction - electrochemical reduction) is widely introduced into the world industry to process lowgrade copper ores. It involves sulfuric acid leaching of the original ore [1]. The copper extraction degree into solution depends primarily on the composition of the original ore. This method is most suitable for oxidized ores. Up to 90% copper can be extracted into solution from oxidized ores during sulfuric acid leaching [2]. Biooxidation is used along with sulfuric acid leaching for ores with more complex mineralogical composition, in particular for sulfide and mixed ores. Biooxidation is an attractive alternative to traditional physical and chemical methods of ore beneficiation, due to the reduced resource intensity of the technology and less harmful effects on the environment [[3], [4]]. Biotechnology for the extraction of copper from lowgrade ore was first introduced on an industrial scale in 1982 at the Lo Aguirre copper mine of Sociedad Minera Pudahuel (Chile). Then a similar process was introduced at 17 enterprises, including 10 mines in Chile, as well as in the USA, Peru, and Australia [5].

Such metals as copper, uranium, nickel and gold are obtained with the use of biohydrometallurgy [[6], [7], [8]]. The basis of biohydrometallurgical technologies are the microbial oxidation processes for hard-to-open minerals by acidophilic microorganisms [9]. The use of microorganisms in the processes of extraction of non-ferrous and precious metals can significantly increase the complexity of the use of raw materials and ensure effective environmental protection.

At the same time, some works dedicated to the leaching of low-grade copper-containing ores in the presence of amino acids, which are an integral part of the culture fluid during bioleaching, appeared recently.

The authors studied the leaching of chalcopyrite concentrate from the Sarcheshmeh copper mine (Kerman, Iran) in a solution of aminoacetic acid (glycine) in the presence of oxygen [[10], [11]]. The effect of such parameters as glycine concentration (0.4–2 M), temperature (30–90 °C), stirring speed (250–750 rpm), pH (9–12), oxygen consumption (0.5–2 l/min) and pulp density (1–20%) was studied. Research was performed to extract copper from chalcopyrite concentrate. The results show that an increase in temperature from 30 to 60 °C increases copper recovery, while at temperatures above 60 °C, there is a decrease in copper recovery, probably as a

result of the conversion of glycine to glycinate and a decrease in oxygen solubility. Moreover, long leaching times at higher pH levels (10.5 and 12) result in decreased copper recovery, primarily due to the chemical precipitation of copper sulfide and crystallization of copper glycinate. When pulp density increases from 1 to 20%, copper recovery decreases significantly. According to the results obtained, the authors recommend using glycine leaching under optimal conditions in the presence of oxygen for the processing of low-grade chalcopyrite concentrates and ores, as well as tailings shows the efficiency of leaching copper contained in low-grade ores in a solution of 0.1 mol/dm³ sodium chloride, in the presence of 0.05 mol/dm³ glycine or aspartic acid at a temperature of 80 °C for 15 hours [12]. The copper extraction degree achieved was 90%. The object of study was ore crushed to a particle size of 2–3 mm with a copper content of 1.0%. Good results were also obtained with the use of phenylalanine hydrochloride, serine hydrochloride and protein hydrolysates. Baker's yeast was used with a crude protein content of 52% and nucleic acids of 5.8% to obtain protein hydrolysates. Copper recovery was at least 92%. It was established that the ability of protein hydrolyzate to interact with copper ions is due to its amino acid composition, i.e. the high content of aspartic acid and glycine.

Thus, it follows from the above sources that bioleaching is increasingly introduced into the technological process; however, bioprocesses are quite lengthy. Besides, they require adaptation of microorganisms to environmental conditions, and therefore, copper leaching study with the use of substandard ores with sulfuric acid in the presence of amino acids, and the effect of their structure on the leaching process characteristics, is of great interest.

The final stage of SX–EW technology is copper electrolysis. The process conditions continuously change during its electrolytic separation from dilute solutions in a periodic mode: with a decrease in copper concentration, the limiting current density continuously decreases, and it increasingly exceeds the limit while maintaining a constant electrolysis current density, and the evolution of hydrogen becomes more and more intense [13]. It is especially noticeable during the extraction of copper from waste electrolytes and poor solutions. In addition to reducing process performance, there is a negative impact of released gases (sulfur dioxide and hydrogen in large quantities) on the environment. At the same time, describes new methods for the

formation of electrolytes using amino acids as buffering, complexing and surfactant additives for nickel [14]. They help to reduce the negative burden on the environment without compromising the quality of the final product.

In connection with the above, the purpose of this work is to study the influence of amino acids, in particular, their structure on the degree of leaching of copper with sulfuric acid from substandard ores, as well as on its electrolytic reduction from sulfuric acid solutions.

Experimental part

Research methods. Test experiments intended to leach the original copper ore were performed to determine the optimal conditions for its leaching. A unit including a reactor with a mechanical stirrer of the PE 8399 brand from Ekros was used to leach the original ore. Optimal conditions were established. They are sulfuric acid concentration $-40~\text{g/dm}^3$, room temperature, process duration -4~hours, S:L ratio = 1:4.

The leaching process kinetics was studied by transfer of a solution of sulfuric acid in the amount of 0.1 dm³ (acid concentration was 40 g/dm³) and ore in a ratio S:L = 1:4, as well as the specified amount of amino acid, in 0.25 dm³ conical flasks. The concentration of aminoacetic acid (glycine) was 0.05, 0.1 and 0.5 M/dm³, and that of leucine, asparagine, histidine and cysteine was 0.5 M/dm³. The flasks were hermetically sealed, and their contents were intensively mixed on an orbital shaker. The number of flasks was equal to the number of samples taken during the sorption process and corresponded to the number of points on the graph. All experiments were performed in duplicate. The result was equal to the average value.

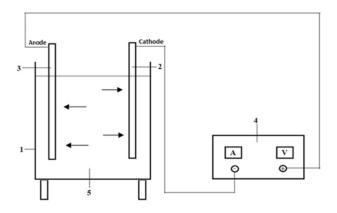


Figure 1 – Unit for copper electrolysis 1 - electrolysis bath; 2- cathode; 3 – anode; 4 – rectifier; 5 – electrolyte

Copper electrolysis was performed in a unit schematically shown in Figure 1. Stainless steel was used as the anode, and lead was used as the anode. The concentration of copper sulfate was 1 g/dm³ and glycine - 0.05-0.5 M/dm³.

Analysis methods

The quantitative content of copper was determined in leaching solutions of the original ore, as well as during the electrolysis process with the use of an Optima 8000DV inductively coupled plasma (ICP) atomic emission spectrometer.

X-ray diffraction data of the ore were obtained with a D8 Advance diffractometer (Bruker AXS GmbH) with a cobalt anode, a - Cu radiation, and X-ray fluorescence data - with a Venus 200 "PANalytical" wave-dispersive spectrometer. Diffraction patterns were interpreted, and interplanar distances were calculated with EVA software. The samples were interpreted, and phases were searched with the use of the "Search/match" program with the ASTM card database.

The method of scanning electron microscopy (SEM) and X-ray spectral microanalysis (XMA) was performed on a JEOL device - JXA-8230.

Substandard ore was used during the research. It was crushed to a particle size of 0.071 mm. Comprehensive studies of this ore were previously performed using X-ray fluorescence, chemical, and X-ray phase analysis, as well as scanning electron microscopy (SEM) and X-ray spectral microanalysis (XMA). The main component of the ore is quartz according to phase analysis (Table 1), i.e. the ores are dominated by silicate rocks, with smaller quantities containing clinochlore, muscovite, albite and microcline.

Table 1 - Phase composition of ore from the Bayskoye deposit

Name	Formula	Content, %
Quartz, syn	SiO2	39.4%
Albite	Na(AlSi3O8)	21.5%
Microcline, intermediate	KAISi3O8	16.8%
Clinochlore-1MIIb, ferroan	(Mg,Fe)6(Si,Al)4O10(OH)8	11.4%
Muscovite-2M1	KAI2(AISi3O10)(OH)2	10.8%

The elemental composition of the ore is presented in Table 2. It follows from there that the main elements are creamium, potassium, calcium, sodium, magnesium and iron.

Table 2 - Results of X-ray fluorescence analysis of the initial ore of the Bayskoye deposit

0	Na	Mg	Al	Si	Р	S	Cl	K	Ca	Ti
56. 142	1.4 13	1.3 85	7.0 77	25. 813	0.0 78	1.1 42	0.0 27	1.8 37	0.7 53	0.6 14
Mn	Fe	Cu	Zn	As	Rb	Sr	Zr	Ni	Р	b
0.0 18	3.0 06	0.6 26	0.0 4	0.0 14	0.0 11	0.0 11	0.0 12	0.1 75	0.0	07

The ore, along with copper, also contains zinc and lead according to scanning electron microscopy and X-ray microanalysis (Fig. 2), i.e. the ore is classified as polymetallic, and the host rock, along with silicates, includes chlorides and antimony compounds. The copper content in the ore sample was 0.39% according to chemical analysis.

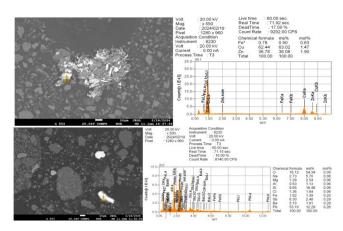


Figure 2 – Results of scanning electron microscopy and X-ray microanalysis

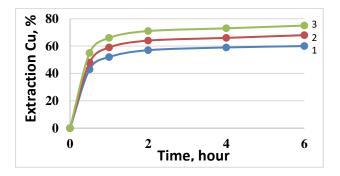


Figure 3 – Kinetic curves of copper leaching with sulfuric acid solution, temperature, $^{\circ}$ C: 1 - 20; 1 – 40; 3 – 60.

The kinetics of the copper leaching process from this raw material with sulfuric acid was studied in the process of studying the physical and chemical patterns of dissolution of non-ferrous metals. Figure 3 shows the kinetic curves of copper leaching at temperatures of 20, 40, and 60 °C.

Based on the results obtained with the use of the Arrhenius equation, the copper leaching rate constants presented in Table 3 and the effective activation energy were calculated. It was 1.817 kJ/mol.

Table 1.2.3 – Values of copper leaching rate constants at different temperatures and their logarithms

Leaching rate	Т, К		
constant	293.0	313.0	333.0
K · 10 ⁻⁴ , sec ⁻¹	4.61	5.18	5.66
lgK	-3.337	-3.285	-3.247

The calculated value of the effective activation energy (1.817 kJ/mol) indicates that the limiting stage of the leaching of copper compounds is diffusion.

In practice, there are several methods intended to remove diffusion inhibition. It is an increase in temperature, the speed of mixing the solution, and the use of surfactants.

It can be assumed that amino acids will also prove to be an additive that stimulates the process because the formation of copper complex compounds with amino acids is possible according to the literature. It can help to increase the degree of copper extraction into solution [[15], [16]].

The effect of amino acid concentration, using glycine as an example, on the kinetics of copper leaching was studied during the research. The glycine concentration was 0.05 and 0.5 M/dm³. The results obtained are presented in Table 4. It follows from Table 4 that the main amount of copper is leached during the first two hours. The copper extraction degree increases as the concentration of amino acids increases. When the glycine concentration increases from 0.05 to 0.5 M/dm³, the degree of copper extraction increases from 64.8 to 75.1%.

Table 4 - Dependence of the degree of copper extraction on the concentration of glycine in the solution.

Extraction rate, %				
Time of	Glyd	cine concen	tration, M/	dm³
	0	0 0.05 0.1 0.5		
30	39.9	42.1	45.5	48.1
60	50.3	53.3	57.8	62.8
120	55.4	57.6	66.4	70.1
240	60.2	62.2	69.5	73.9
360	61.9	64.8	70.9	75.1

Studies were performed for the leaching process of this ore in the presence of the following amino acids: cysteine, histidine, asparagine and leucine, to identify the effect of the amino acid structure on the degree of copper extraction. The results obtained are presented in Figure 4, and it follows from it that the copper extraction degree during the leaching process increases in the presence of amino acids located in the line of glycine - leucine — cysteine - histidine - asparagine, i.e., the opposite relationship is observed in comparison with the gold leaching in the presence of amino acids [[17], [18], [19], [20]].

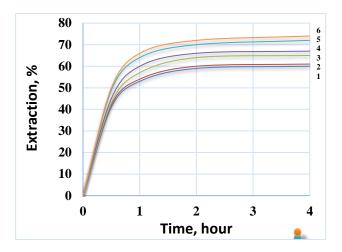


Figure 4 - Effect of amino acid structure on the degree of sulfuric acid leaching of copper.

1-blank; 2-asparagine; 3- histidine; 4-cysteine;

5- leucine; 6-glycine

It can be assumed based on the results obtained that if the effect of the amino group in the resulting complexes predominates during cyanide leaching of gold then there is the effect of the carboxyl group, on the contrary, during leaching copper with sulfuric acid, i.e. the higher the contribution of ionic bonding to the formation of copper complexes with amino acids, the higher the positive effect of the amino acid on the copper extraction degree during sulfuric acid leaching. Consequently, aliphatic amino acids have the strongest effect on copper leaching.

The effect of aminoacetic acid (glycine) on the electrolytic reduction of copper was studied with the use of model solutions as an example. We focused on the copper content in poor electrolytes. The concentration of copper and sulfuric acid was 1.0 and 100 g/dm³, respectively. $CuSO_4 \cdot 5 H_2O$ was used to prepare the model solution.

Electrolysis was performed on the unit shown in Figure 1. In the first stage, the optimal electrolysis time was determined. The duration of the experiments was sequentially 0.5; 1.0; 2.0; 4.0 and

6.0 hours, current density 0.2 A/m². The resulting kinetic curve is presented in Figure 5.

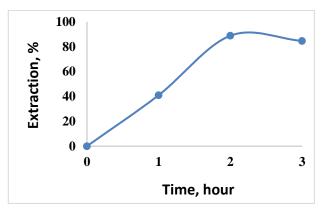


Figure 5 – Kinetic curve of copper electrolysis
It follows from the figure that the optimal duration of
the process is 2 hours

Study of the sediment after 2 hours of electrolysis by the X-ray phase method showed that metallic copper was its basis. Besides, it contains a small amount of cuprous oxide and copper sulfate pentahydrate (Table 5). This composition of the sediment may indicate a staged reduction of copper.

The divalent copper ions are reduced first to the monovalent state, and then to metallic copper:

$$Cu^{+2} + e = Cu^{+}$$

 $Cu^{+} + e = Cu^{0}$
 $Cu^{+2} + 2e = Cu^{0}$

Partial adsorption of the original electrolyte takes place in addition.

After 4 hours, the sediment also contains lead, along with copper. This fact indicates that by this time there is a partial dissolution of the anode - lead, which forms lead sulfate and is partially deposited on the cathode (Table 6). The data obtained once again confirm the fact that the optimal duration of copper electrolysis should be considered to be 2 hours at a given current density.

Table 5 - Composition of the cathode deposit after 2 hours of electrolysis

Name	Formula	Content, %
Copper	Cu	88.9
Cuprite, syn	Cu₂O	7.4
Chalcanthite, syn	CuSO ₄ ·5H ₂ O	3.7

Table 6 - Composition of the cathode deposit after 4 hours of electrolysis

Name	Formula	Content, %
Cuprite, syn	Cu	72.5
Anglesite, syn	Pb(SO ₄)	13.4
Copper	Cu ₂ O	11.1
Kobyashevite	CuSO ₄ ·5H ₂ O	3.0

Copper electrolysis was performed at the next stage under the same conditions in the presence of aminoacetic acid - glycine. The glycine concentration was 0.1 and 0.5 M. The results obtained are presented in Figure 6.

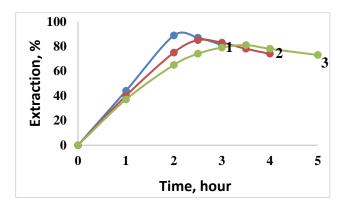


Figure 6 - Kinetic dependence of copper electrolysis in the presence of glycine
Amino acid concentration:
1- in the absence of an amino acid; 2 - 0.1M; 3 - 0.5M

As it follows from the figure, the presence of glycine in the solution increases the recovery time of copper, i.e. glycine in this case plays the role of a depressant. It is confirmed by X-ray phase analysis data (Table 7, 8), according to which the process is inhibited by the reduction of monovalent copper to the metallic state.

Table 7 - Results of X-ray phase analysis of the cathode product (glycine concentration 0.5 M)

Name	Formula	Content, %
Copper(I) oxide Copper Oxide	Cu₂O	57.2%
Copper	Cu	33.5%
Chalcanthite, syn	Cu ₂ SO ₄ ·5H ₂ O	9.3%

Table 8 - Results of X-ray phase analysis of the cathode product (glycine concentration 0.1 M)

Name	Formula	Content, %
Copper(I) oxide Copper Oxide	Cu ₂ O	31.1%
Copper	Cu	59.1%
Chalcanthite, syn	Cu ₂ SO ₄ ·5H ₂ O	8.8%

Thus, amino acids as bio-additives play a positive role in the leaching of copper from low-grade ore, and they are depressants of the copper reduction process during electrolysis. An increase in the concentration of amino acids increases the inhibition of the discharge of copper ions.

Conclusions

Biohydrometallurgical technologies are widely used for the production of non-ferrous and precious metals. The use of microorganisms can significantly increase the complexity of the use of raw materials and ensure effective environmental protection. Recently, bio-additives, in particular amino acids, which are an integral part of the culture liquid of microorganisms, are increasingly used in the production cycle, along with microorganisms.

The effect of amino acids on the leaching and subsequent electrolysis of copper from low-grade copper-containing ore, which includes quartz, albite, microcline, clinochlore, and muscovite, was studied. The copper content in the ore is 0.39%. The kinetics of the ore-leaching process with sulfuric acid was studied. The diffusion nature of the restrictions was established with the use of the Arrhenius equation, and the rate constants were calculated.

The effect of amino acids with different structures on the leaching process was studied. It was shown that the concentration of the amino acid, as well as its structure, affects the degree of copper extraction that decreases in the line of glycine - leucine - cysteine - histidine - asparagine.

The final stage of hydrometallurgical copper processing is electrolysis. It was found based on a study of the use of aminoacetic acid - glycine as an additive to the electrolyte during the electrolysis process that its presence inhibits the process of reduction of monovalent copper to the metallic state.

Thus, amino acids, predominantly of the aliphatic series, play a positive role as biological

additives in the leaching of copper from low-grade ore, and they act as depressants for the copper reduction process during electrolysis, and an increase in the concentration of amino acids increases the inhibition of the discharge of copper ions.

Conflicts of interest. On behalf of all authors, the corresponding author states that there is no conflict of interest.

CRediT author statement: R. Abdulvaliev: Conceptualization, Methodology, Software. T. Surkova: Data curation, Writing draft preparation. D.Yessimova, K. Smailov: Visualization, Investigation. Zh.Baltabekova, A. Berkinbayeva: Supervision. M. Stachowicz: Software, Validation. Z. Dossymbayeva: Reviewing and Editing.

Acknowledgements. This work was supported by the Ministry of Science and Education of the Republic of Kazakhstan [BR18574018].

Cite this article as: Abdulvaliev RA, Surkova TYu, Baltabekova ZhA, Yessimova DM, Stachowicz M, Smailov KM, Dossymbayeva ZD, Berkinbayeva AN. Effect of amino acids on the extraction of copper from sub-conditional raw materials. Kompleksnoe Ispolzovanie Mineralnogo Syra = Complex Use of Mineral Resources. 2025; 335(4):50-58. https://doi.org/10.31643/2025/6445.39

Амин қышқылдарының сапасы төмен шикізаттардан мыс алуға әсері

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	ТҮЙІНДЕМЕ
	Өнеркәсіптік жолмен өңделетін минералды шикізат сапасының төмендеуі, сонымен қатар
	қоршаған ортаны қорғау талаптарының артуы оларды өңдеу технологиясының жаңа
	бағыттарын дамытуды қажет етеді. Биогеотехнология минералдық шикізатты өңдеу
	саласындағы ғылыми-техникалық прогрестің қазіргі заманғы бағыттарының біріне жатады.
	Түсті және асыл металдарды алу процестерінде микроорганизмдерді қолданумен қатар,
	биологиялық белсенді қоспалар, атап айтқанда микроорганизмдердің сұйықтығының
	құрамдас бөлігі болып табылатын аминқышқылдары көбірек қолданылады. Әртүрлі
Мақала келді: <i>28 мамыр 2024</i>	құрылымдағы амин қышқылдарының қатысында төмен сапалы кеннен мысты күкірт
Сараптамадан өтті: 28 маусым 2024	қышқылымен шаймалау процестері, сондай-ақ олардың мыс электролизіне әсері зерттелді.
Қабылданды: <i>13 қыркүйек 2024</i>	Бастапқы шикізат ретінде қазақстандық кен орындарының бірінің төмен сапалы кені
	пайдаланылды. Кендегі мыс мөлшері 0,39% құрайды. Шектеулердің диффузиялық сипаты
	мысты күкірт қышқылымен шаймалау процесінің кинетикасын зерттеу кезінде белгіленіп,
	жылдамдық константалары мен тиімді белсендендіру энергиясы есептелді, ол 1,817
	кДж/моль болды. Құрылымы әртүрлі аминқышқылдарының шаймалау процесіне әсері
	зерттелді. Оң әсердің жоғарылауы глицин – лейцин – цистеин – гистедин – аспарагин
	қатарында көрсетілген. Амин қышқылының құрылымына байланысты ерітіндіге мыстың
	алыну дәрежесі 1-15% аралығында артады. Кедей ерітінділерден мысты электролиздеу
	процесіне биологиялық белсенді қоспа ретінде аминсірке қышқылының әсері зерттелді.
	Глициннің қатысуымен бір валентті мысты металдық күйге келтіру арқылы процесс
	тежелетіні анықталды.
	Түйін сөздер: мыс, шаймалау, минералды шикізат,, амин қышқылдары, микроорганизмдер.
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Влияние аминокислот на извлечение меди из некондиционного сырья

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	РИПРИ В В В В В В В В В В В В В В В В В В В
	Снижение качества перерабатываемого промышленностью минерального сырья, а также
	увеличение требований к охране окружающей среды обуславливают необходимость
	разработки новых направлений в технологии их переработки. Биогеотехнология относится к
	одному из современных направлений научно-технического прогресса в области
	переработки минерального сырья. Наряду с применением микроорганизмов в процессах
	извлечения цветных и благородных металлов все шире используют биологически активные
F 20 2024	
Поступила: <i>28 мая 2024</i> Рецензирование: <i>28 июня 2024</i>	добавки, в частности, аминокислоты, которые являются составной частью культуральной
Принята в печать: <i>13 сентября 2024</i>	жидкости микроорганизмов. Нами рассмотрены процессы сернокислотного выщелачивания
припита в печать. 13 сентиори 2024	меди из низкосортной руды в присутствии аминокислот разной структуры, а также влияние
	их на электролиз меди. В качестве исходного сырья использовали низкосортную руду одного
	из Казахстанских месторождений. Содержание меди в руде -0,39%. Исследованием
	кинетики процесса сернокислотного выщелачивания меди установлен диффузионный
	характер ограничений, рассчитаны константы скорости и значение эффективной энергии
	активации, которое составило 1,817 кДж/моль. Изучено влияние аминокислот разной
	структуры на процесс выщелачивания. Показано увеличение положительного влияния в
	ряду: глицин — лейцин — цистеин - гистедин, - аспарагин. В зависимости от структуры
	аминокислоты степень извлечения меди в раствор увеличивается в пределах 1-15%. Изучено
	влияние аминоуксусной кислоты в качестве биологически активной добавки на процесс
	электролиза меди из бедных растворов. Установлено, что в присутствии глицина процесс
	тормозится восстановлением одновалентной меди до металлического состояния.
	Ключевые слова: медь, выщелачивание, минеральное сырье, аминокислоты,
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