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Electrochemical Sulphidization of the Refractory Copper Mineral Malachite from the Composition of Oxidized Waste Ores of Zhezkazgan

[1,2]***JASHIBEKOVA Anel,*** *Master's Degree, anelyadzhashi@gmail.com,*
[1]***DOSPAYEV Murat,*** *Dr. of Tech. Sc., Head of Laboratory, manten.mur@mail.ru,*
[1]****FIGURINENE Irina,*** *Cand. of Chem. Sc., Leading Researcher, electrochimik@mail.ru,*
[3]***KAKENOV Kairat,*** *Cand. of Tech. Sc., Chief of Department, sattu55@mail.ru,*
[3]***YESSENBAYEVA Gulmira,*** *Cand. of Tech. Sc., Senior Researcher, esenbaeva_keu@mail.ru,*
[1]*Zh. Abishev Chemical-Metallurgical Institute, 63 Ermekov Street, Karaganda, Kazakhstan,*
[2]*NPJSC «Abylkas Saginov Karaganda Technical University», 56 N. Nazarbayev Avenue, Karaganda, Kazakhstan,*
[3]*Karaganda University of Kazpotrebsoyuz, 9 Akademicheskaya Street, Karaganda, Kazakhstan,*

**corresponding author.*

Abstract. *The aim of the research is the studying the electrochemical reduction of the refractory copper mineral malachite from oxidized ores in the presence of sodium sulfite, a sulphidizing agent, and formation of easily floatable copper (I) sulfide under voltammetric polarization conditions. The studies were conducted using a special electrode assembly design for voltammetric studies and microelectrolysis at a controlled potential of both dispersed ore monominerals and depolarizer solutions. Electrode mechanisms for the monovalent copper sulfide formation during combined cathodic reduction of malachite and sodium sulfite were established. A sulfite ion is easily reduced in an acidic medium to highly active colloidal elemental sulfur, making it an effective sulphidizer for malachite. Copper sulfide was synthesized from malachite under microelectrolysis conditions, and the sulphidizing effect of sulfite was confirmed. The effect of the electrode material on the cathodic decomposition of the synthesized* Cu_2S *was observed.*

Keywords: *oxidized copper ore, mineral malachite, sodium sulfite, voltammetry, electrochemical sulphidizing.*

Introduction

Currently, the Republic of Kazakhstan is experiencing a gradual depletion of high-quality mineral reserves which necessitates the processing of refractory oxidized man-made waste ores and tailings. Non-ferrous metal deposits from the upper horizons are oxidized, while those from the middle horizons are mixed and characterized by a low sulfur content and a high silicon oxide content. Refractory oxidized ores contain 0.5-1% non-ferrous metals, and the oxidation state reaches 90-95%. Refractory oxidized copper waste ores also contain predominantly oxidized forms of copper reaching 90%. The mineralogical composition of oxidized ores is primarily represented by such minerals as chrysocolla, malachite, azurite, tenorite, and cuprite, which prevent copper recovery using traditional flotation beneficiation methods. The presence of refractory minerals in ores: oxides, silicates, basic sulfates and carbonates, significantly complicates their processing and in some cases gives unsatisfactory results during flotation enrichment.

To achieve high copper recovery from refractory oxidized ores, thermal, biological, and electrochemical processing methods [1-6] are used worldwide. These methods allow for targeted modification of the surface properties of refractory minerals. The most promising method of the comprehensive and deep processing of refractory oxidized copper ores is electrochemical polarization of ore pulp, which facilitates the efficient conversion of oxidized minerals to an easily floatable sulfide form [7-9]. Since this method is performed at low temperatures (~25-70°C) and energy consumption is significantly reduced, multiple regeneration of electrolyte and reagents is possible, and harmful toxic gas emissions are absent, unlike those associated with pyrometallurgy.

The aim of this research is to study electrochemical reduction of the refractory copper mineral malachite from oxidized ores in the presence of sodium sulfite, a sulphidizing agent, and formation of easily floatable copper (I) sulfide under voltammetric polarization conditions.

Research methods

The natural copper oxide monomineral malachite was used for voltammetric studies. Voltammetric measurements were conducted using a special electrode [9]. This special electrode assembly (Figure 1) is used for voltammetric studies of ore monominerals. It ensures contact between solid particles and the substrate metal surface and allows for easy changes in the working electrode material and surface area.

For polarization, a small sample of the dispersed monomineral is placed on the working surface of metal substrate, covered with filter paper, and pressed on top with cap (4). The working surface area of the metal substrate plate is 8.6 x 10^{-6} m^2 with the washer (3) internal diameter of 1.65 mm. Surface preparation of the metal substrate consists of mandatory polishing with sandpaper, rinsing with distilled water, and degreasing with ethyl alcohol. The potential scan rate is 10 mV/s. The potential of the reference silver/silver chloride electrode is +0.203 V. All the potential values in the voltammetric curves are given relative to the normal hydrogen electrode.

Scientific results

A titanium electrode that is inert in both acidic and alkaline environments, was chosen as the substrate electrode for the voltammetric measurements. Figure 2 shows that the polarization curve for malachite reduction in an acidic environment exhibits a single, distinct wave at the potential of approximately +0.4 V (curve 2). The surface of the titanium electrode at the point of contact with the test sample is coated with a red precipitate of newly formed elemental copper when recording the voltammetric curve.

According to [10], reduction of solid metal salts or oxides occurs through the following electrode processes:

1 – direct discharge of the solid particle;

2 – the electron carrier is an intermediate substance formed at the cathode;

3 – the solid particles undergo decomposition into cations and anions, which are then reduced at the electrode.

From this, it follows that the cathodic reduction of the oxidized mineral malachite ($CuCO_3{\cdot}Cu(OH)_2$) can occur either through the reduction of Cu^{2+} ions formed during partial dissolution of this mineral or through direct electron discharge of the solid particles. That is, the reduction process of this mineral proceeds in parallel via mechanism I:

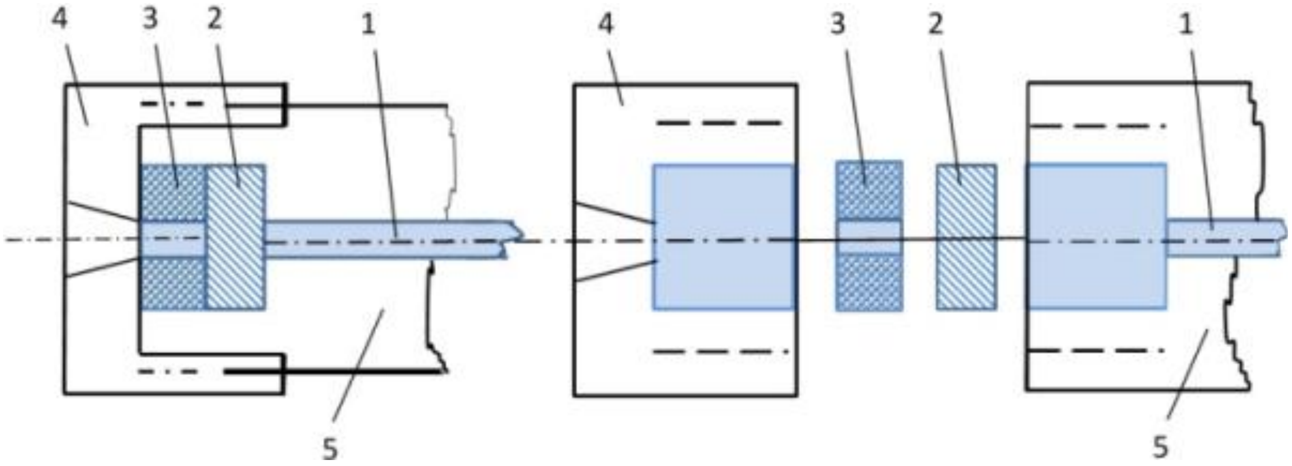
$$CuCO_3 \cdot Cu(OH)_2 + 4\bar{e} + 4H^+ \rightarrow 2Cu^0 + \\ + H_2CO_3 + 2H_2O, \quad E^0 = +0.508B \tag{1}$$

and via mechanism III:

$$CuCO_3 \cdot Cu(OH)_2 + 2H_2SO_4 \rightarrow \\ \rightarrow 2CuSO_4 + H_2CO_3 + 2H_2O, \tag{2}$$

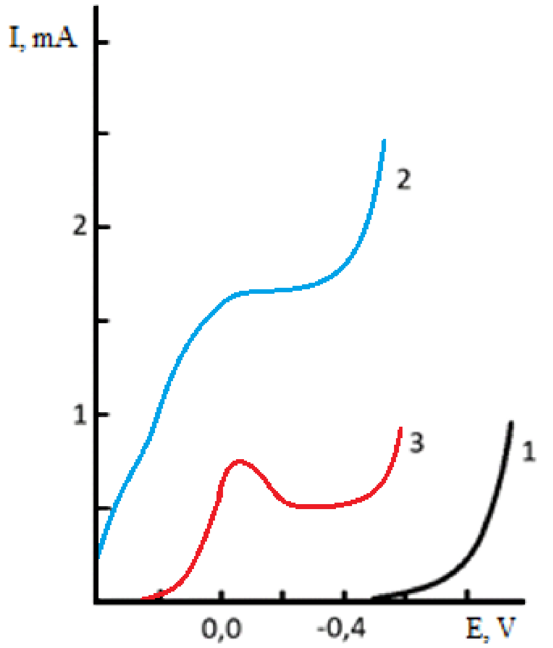
$$Cu^{2+} + 2\bar{e} \rightarrow Cu^0. \tag{3}$$

To elucidate the nature of this wave (Figure 2, curve 2), a cathodic polarization curve was recorded for the solution containing only divalent copper ions. At the potential of E≈ +0.05 V, the limiting current for the reduction of copper (II) ions (Figure 2, curve 3) accord-



1 – current lead; 2 – working electrode; 3 – washer; 4 – cap; 5 – electrode assembly corpus

Figure 1 – A special electrode assembly design



1 – background; 2 – background + a sample of malachite mineral; 3 – background + solution of Cu(II) ions 12 g/L

Figure 2 – Cathode voltammetric curves of a titanium electrode in a 100 g/L H_2SO_4 solution

ing to reaction (3) was recorded. The product of the electrode reaction is a compact, reddish precipitate of elemental copper. The maximum current (Figure 2, curve 3) indicates that the solid-phase direct discharge of particles plays the key role in the reduction of the oxidized mineral malachite.

Next, the cathodic behavior of sodium sulfite that was chosen as a sulphidizing agent stable in an acidic environment, was studied. On a titanium electrode (Figure 3, a), sulfite reduction occurs with the formation of hydrogen sulfide and elemental sulfur:

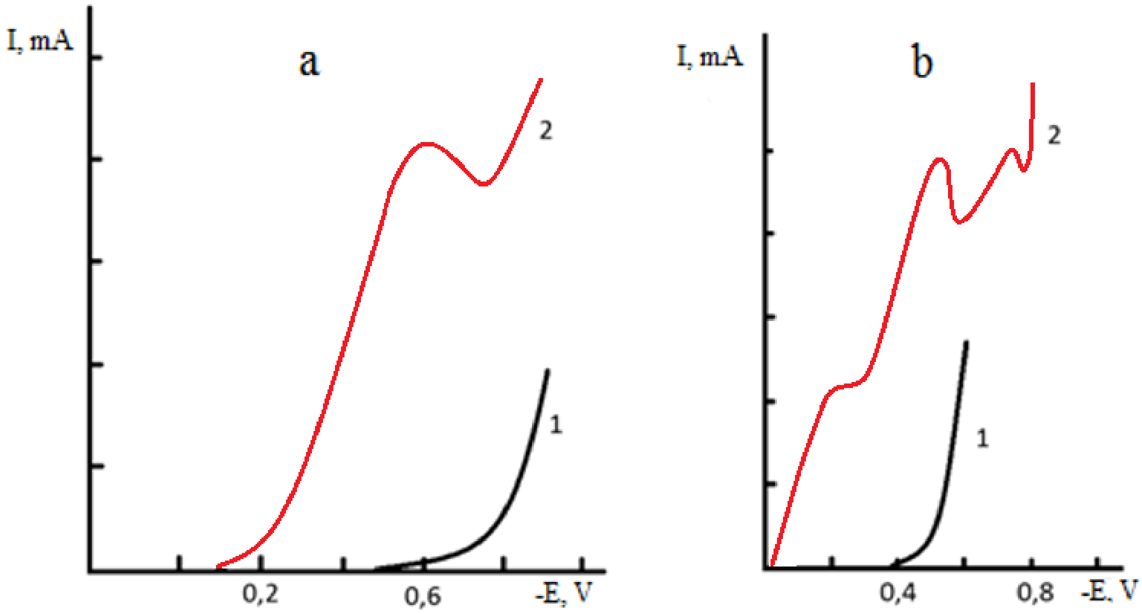
$$SO_3^{-2} + 4\overline{e} + 6H^+ \rightarrow S^0 + 3H_2O, \quad E^0 = +0.585B. \qquad (4)$$

On the copper electrode, the reduction of sulfite ion occurs via a more complex mechanism, as the voltammetric curve exhibits several current peaks (Figure 3, b). The first wave corresponds to the formation of elemental sulfur via reaction (4), after which sulfur, due to its high affinity, instantly reacts with the surface of the copper electrode to form sulfide:

$$2Cu + S \rightarrow Cu_2S. \qquad (5)$$

This is also confirmed by the fact that at the first wave potential, the copper electrode surface becomes coated with a black Cu_2S film. The second and third waves are associated with the further reduction of the formed sulfide. Thus, in an acidic medium, a sulfite ion is easily reduced to highly active colloidal elemental sulfur, and can therefore serve as an effective sulphidizer for the mineral malachite.

The combined cathodic reduction of malachite and a sulfide sulphidizer on a titanium electrode was studied (Figure 4, curve 2). The



1 – background solution (100 g/L H_2SO_4); 2 – background + 15 g/L Na_2SO_3

Figure 3 – Cathode voltammetric curves of sulfite ion reduction on titanium (a) and copper (b) electrodes

first maximum corresponds to the reduction of malachite particles to elemental copper, while the second corresponds to the reduction of sulfite to colloidal sulfur, with the electrode surface becoming coated with a loose black precipitate.

An increase in malachite sample weight leads to an increase in the height of the first maximum, while with increasing sulfite concentration, the height of the second maximum on the voltammetric curve increased (Figure 4, curve 2). Thus, by analyzing the obtained voltammetric curves, it can be concluded that on a titanium electrode in the presence of malachite and sulfite ions, malachite particles are first reduced via reactions (1-3) followed by sulfite ions forming elemental sulfur via reaction (4). Then. the newly formed active elemental copper and sulfur react directly chemically to form copper sulfide via reaction (5). As mentioned above, the black precipitate becomes visually visible on the cathode surface.

During electrochemical sulphidization of the oxidized copper mineral, the primary process of converting malachite into easily floatable copper (I) sulfide will occur during the calculated electrolysis duration. If the permissible time interval is exceeded, an undesirable process of decomposition of the resulting product Cu_2S can occur. In this regard, the cathodic behavior of electrochemically synthesized copper (I) sulfide on titanium, copper, and steel electrodes was studied. Electrosynthesis was performed by microelectrolysis at a controlled potential. We found that the reduction of the synthesized copper (I) sulfide depends on the electrode material. The equilibrium potential of copper and stainless steel in a background H_2SO_4 solution is +0.2 V, while that of titanium is ~0.0 V. The equilibrium potential of all studied electrode materials with a Cu_2S sample is the same and is ~+0.4 V. In the sulfide powder–electrode–solution system, the electrode potential is significantly more positive than in the electrode–solution system. So, the sulfide powder "imposes" a potential on the substrate metal, the magnitude of which depends on the $Cu^{2+}/Cu+$ ratio. On the cathodic volt-ampere curves (Figure 5, curves 1 and 2), taken on copper and titanium electrodes, two waves are observed at potentials of ~ +0.4 and 0.0 V. The authors assume that the first wave corresponds to the reduction of Cu^{2+} ions partially formed when an electrode with a weighed sample of Cu_2S is immersed in a solution of sulfuric acid, to Cu+ ions. The second wave corresponds to the Cu_2S reduction:

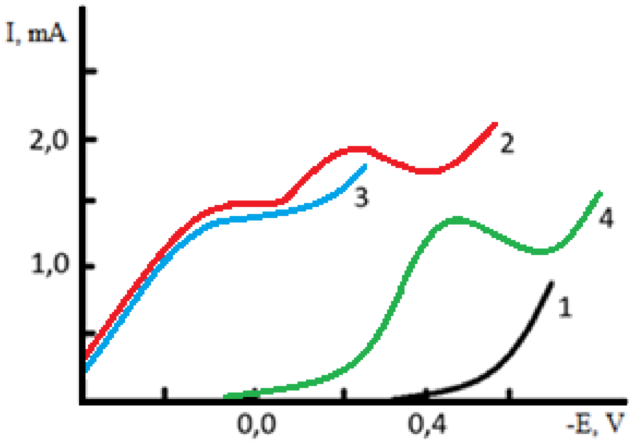
$$Cu_2S + 2H^+ + 2\bar{e} \rightarrow Cu^0 + H_2S_{solution}, \qquad (6)$$

$$E^0 = -0.305B.$$

The electrode surface at the point of contact with the sample is coated with elemental copper.

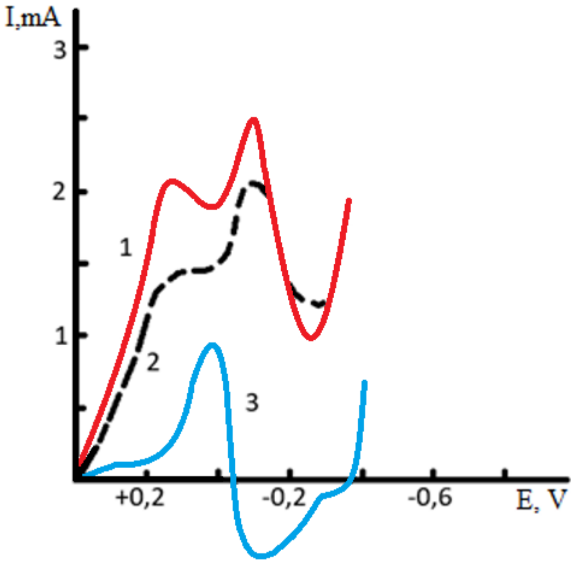
In the cathode curve of a stainless steel electrode (Figure 5, curve 3), the first current maximum is observed at a potential of ~+0.1 V. This is due to the fact that, as we noted earlier, partial dissolution of the sample occurs, with the release of Cu^{2+} ions into the solution that undergo a carburization reaction with the electrode material:

$$Cu^{2+} + Fe^0 \rightarrow Cu^0 + Fe^{2+}, \qquad (7)$$



1 – background (100 g/L H_2SO_4); 2 – background + sample of malachite mineral + 15 g/L Na_2SO_3; 3 – background + sample of malachite mineral; 4 – background + 15 g/L Na_2SO_3

Figure 4 – Cathode voltammetric curves of a titanium electrode



1 – copper; 2 – titanium; 3 – stainless steel

Figure 5 – Cathode voltammetric curves of copper sulfide on solid electrodes in a sulfuric acid solution, 100 g/L

and form elemental copper. Therefore, the reduction waves of Cu^{2+} ions are not observed on the polarization curve. The first current maximum is associated with the direct discharge of copper sulfide via the electron mechanism (reaction 6). The potential of this wave coincides with the potential of the second peak, corresponding to the discharge of Cu_2S, on the cathodic polarization curves of copper and titanium (Figure 5, curves 1 and 2). Freshly formed elemental copper is very active and dissolves in a sulfuric acid solution to the divalent state. This process is recorded as an anodic wave. With further polarization, Cu2+ ions in the near-cathode layer are reduced to elemental copper, which corresponds to the third wave at a potential of ~ -0.2 V on the voltammetric curve. This is followed by a wave of hydrogen sulfide evolution along with hydrogen.

Conclusion

Electrochemical reduction of the refractory copper mineral malachite from refractory oxidized ores in the presence of sodium sulfite as a sulphidizer and formation of readily floatable copper (I) sulfide under voltammetric polarization conditions were studied. The studies were conducted using a special electrode assembly design for voltammetric studies and microelectrolysis at a controlled potential of both dispersed ore monominerals and depolarizer solutions. Electrode mechanisms for the formation of monovalent copper sulfide during combined cathodic reduction of malachite and sodium sulfite were established. A sulfite ion is easily reduced in an acidic medium to highly active colloidal elemental sulfur making it an effective sulphidizer for malachite. Copper sulfide was synthesized from malachite under microelectrolysis conditions, and the sulphidizing effect of sulfite was confirmed. The effect of the electrode material on the cathodic decomposition of the synthesized Cu_2S was observed. Thus, targeted preliminary electrochemical polarization of oxidized copper mineral particles with altered surface properties enables their conversion to the sulfide form with the selection of an effective sulphidizing agent. The obtained results are of practical significance and serve as a prerequisite for the electrolytic conversion of refractory oxidized copper minerals to the easily floatable sulfide form.

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Жезқазған тотыққан үйінді кендерінің құрамына кіретін қиынбайытылатын малахит мыс минералын электрохимиялық жолымен сульфидтендіру

[1,2]**ДЖАШИБЕКОВА Анель Нурлановна,** *магистрант, anelyadzhashi@gmail.com,*
[1]**ДОСПАЕВ Мурат Мантенович,** *т.ғ.д., зертхана меңгерушісі, manten.mur@mail.ru,*
[1]***ФИГУРИНЕНЕ Ирина Владиславовна,** *х.ғ.к., жетекші ғылыми қызметкер, electrochimik@mail.ru,*
[3]**КАКЕНОВ Кайрат Саттуевич,** *т.ғ.к., бөлім бастығы, sattu55@mail.ru,*
[3]**ЕСЕНБАЕВА Гульмира Ахмадиевна,** *т.ғ.к., аға ғылыми қызметкер, esenbaeva_keu@mail.ru,*
[1]Ж. Әбішев атындағы Химия-металлургия институты, Ермеков көшесі, 63, Қарағанды, Қазақстан,
[2]«Әбілқас Сағынов атындағы Қарағанды техникалық университеті» КеАҚ, Н. Назарбаев даңғылы, 56, Қарағанды, Қазақстан,
[3]Қазтұтынуодағы Қарағанды университеті, Академическая көшесі, 9, Қарағанды, Қазақстан,
**автор-корреспондент.*

Аңдатпа. *Жұмыстың мақсаты – тотыққан кендер құрамына кіретін қиын байытылатын малахит мыс минералының электрохимиялық тотықсыздануын және сульфидтеуші натрий сульфитінің қатысуымен вольтамперлік поляризациялау жағдайында жеңіл флотацияланатын мыс сульфиді (I) түзілуін зерттеу. Зерттеулер өте ұсақ кен минералдарды, ерітіндідегі деполяризаторларды вольтамперлік поляризациялау және олардың микроэлектролизын арнайы конструкцияланған электродта потенциалды бақылау жағдайында өткізілді. Малахит минералының және натрий сульфитімен бірлесіп катодтық тотықсыздану кезінде бір валентті мыс сульфиді түзілунің электродтық механизмі анықталды. Сульфит-ионы қышқыл ортада белсенділігі жоғары коллоидты элементті күкіртке дейін жеңіл тотықсызданады, сондықтан малахит минералын сульфидтейтін тиімді сульфидтеуші болып саналады. Микроэлектролиз жағдайында малахит минералынан және дәлелденген натрий сульфитінің сульфидтеуші әсерінің негізінде мыс сульфиді синтезделді. Синтезделген* Cu_2S*-тің катодтық ыдырауына электрод материалының әсері анықталды.*

Кілт сөздер: *тотыққан мыс кені, малахит минералы, натрий сульфиті, вольтамперометрлік, электрохимиялық сульфидтендіру.*

Электрохимическая сульфидизация труднообогатимого минерала меди малахита из состава окисленных отвальных руд Жезказгана

[1,2]**ДЖАШИБЕКОВА Анель Нурлановна,** *магистрант, anelyadzhashi@gmail.com,*
[1]**ДОСПАЕВ Мурат Мантенович,** *д.т.н., зав. лабораторией, manten.mur@mail.ru,*
[1]***ФИГУРИНЕНЕ Ирина Владиславовна,** *к.х.н., ведущий научный сотрудник, electrochimik@mail.ru,*
[3]**КАКЕНОВ Кайрат Саттуевич,** *к.т.н., начальник отдела, sattu55@mail.ru,*
[3]**ЕСЕНБАЕВА Гульмира Ахмадиевна,** *к.т.н., старший научный сотрудник, esenbaeva_keu@mail.ru,*
[1]Химико-металлургический институт имени Ж. Абишева, ул. Ермекова, 63, Караганда, Казахстан,
[2]НАО «Карагандинский технический университет имени Абылкаса Сагинова», пр. Н. Назарбаева, 56, Караганда, Казахстан,
[3]Карагандинский университет Казпотребсоюза, ул. Академическая, 9, Караганда, Казахстан,
**автор-корреспондент.*

Аннотация. *Целью исследований является изучение процесса электрохимического восстановления труднообогатимого минерала меди малахита из состава окисленных руд в присутствии сульфидизатора сульфита натрия и формирования легкофлотируемого сульфида меди (I) в условиях вольтамперной поляризации. Исследования проведены с помощью специально сконструированного электрода для вольтамперных исследований и проведения микроэлектролиза при контролируемом потенциале как дисперсных рудных мономинералов, так и растворов деполяризаторов. Установлены электродные механизмы образования сульфида одновалентной меди при совместном катодном восстановлении минерала малахита и сульфита натрия. Сульфит-ион легко восстанавливается в кислотной среде до высокоактивной коллоидной элементной серы, поэтому является эффективным сульфидизатором минерала малахита. В условиях микроэлектролиза был синтезирован сульфид меди из минерала малахита и подтверждено сульфидирующее действие сульфита. Обнаружено влияние материала электрода на катодное разложение синтезированного* Cu_2S.

Ключевые слова: *окисленная медная руда, минерал малахит, сульфит натрия, вольтамперометрия, электрохимическое сульфидирование.*

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