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Қ. И. Сәтпаев атындағы Қазақ ұлттық техникалық зерттеу университеті

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ГЕОЛОГИЯ ЖӘНЕ ТЕХНИКАЛЫҚ ҒЫЛЫМДАР СЕРИЯСЫ



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NAS RK is pleased to announce that News of NAS RK. Series of geology and technical sciences scientific journal has been accepted for indexing in the Emerging Sources Citation Index, a new edition of Web of Science. Content in this index is under consideration by Clarivate Analytics to be accepted in the Science Citation Index Expanded, the Social Sciences Citation Index, and the Arts & Humanities Citation Index. The quality and depth of content Web of Science offers to researchers, authors, publishers, and institutions sets it apart from other research databases. The inclusion of News of NAS RK. Series of geology and technical sciences in the Emerging Sources Citation Index demonstrates our dedication to providing the most relevant and influential content of geology and engineering sciences to our community.

Қазақстан Республикасы Ұлттық ғылым академиясы "ҚР ҰҒА Хабарлары. Геология және техникалық ғылымдар сериясы" ғылыми журналының Web of Science-тің жаңаланған нұсқасы Emerging Sources Citation Index-те индекстелуге қабылданғанын хабарлайды. Бұл индекстелу барысында Clarivate Analytics компаниясы журналды одан әрі the Science Citation Index Expanded, the Social Sciences Citation Index және the Arts & Humanities Citation Index-ке қабылдау мәселесін қарастыруда. Web of Science зерттеушілер, авторлар, баспашылар мен мекемелерге контент тереңдігі мен сапасын ұсынады. ҚР ҰҒА Хабарлары. Геология және техникалық ғылымдар сериясы Emerging Sources Citation Index-ке енуі біздің қоғамдастық үшін ең өзекті және беделді геология және техникалық ғылымдар бойынша контентке адалдығымызды білдіреді.

НАН РК сообщает, что научный журнал «Известия НАН РК. Серия геологии и технических наук» был принят для индексирования в Emerging Sources Citation Index, обновленной версии Web of Science. Содержание в этом индексировании находится в стадии рассмотрения компанией Clarivate Analytics для дальнейшего принятия журнала в the Science Citation Index Expanded, the Social Sciences Citation Index и the Arts & Humanities Citation Index. Web of Science предлагает качество и глубину контента для исследователей, авторов, издателей и учреждений. Включение Известия НАН РК. Серия геологии и технических наук в Emerging Sources Citation Index демонстрирует нашу приверженность к наиболее актуальному и влиятельному контенту по геологии и техническим наукам для нашего сообщества.

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**M. M. Dospaev¹, A. Bayeshov², M. Z. Zhurinov², A. S. Zhumakanova²,
A. K. Bayeshova³, B. B. Syzdykova¹, D. M. Dospaev⁴, K. S. Kakenov⁵**

¹Chemical and Metallurgical Institute named after Zh. Abishev, Karagandy, Kazakhstan,

²Sokolsky Institute of Fuel, Catalysis and Electrochemistry, Almaty, Kazakhstan,

³Al-Faraby Kazakh National University, Almaty, Kazakhstan,

⁴Karaganda State Technical University, Karagandy, Kazakhstan,

⁵Karaganda Economic University of Kazpotrebooyuz, Karagandy, Kazakhstan.

E-mail: manten.mur@mail.ru, elhimproc@mail.ru

**PHYSICAL-AND-CHEMICAL REGULARITIES
OF FORMING CHRYSOCOLLA MINERAL
IN METASILICATE SOLUTIONS**

Abstract. The chrysocolla mineral takes a unique place among the minerals found in the complex oxidized copper ores. For developing the newest efficient methods of processing such ores it is necessary study comprehensively the properties of the chrysocolla mineral. The difficulty is in distinguishing between monomineral fractions from the ore. In literature there are no data of obtaining chrysocolla artificially, there are only different authors' suppositions regarding the mineral formation in the earth crust by means of copper adsorption from solutions by silica. In this connection we for the first time by the method of voltammetry have established the mechanism of forming the chrysocolla mineral during anodic polarization of the electrode in the sodium metasilicate solution. There has been shown a possibility to obtain chrysocolla by means of electrolysis from the water solution of sodium metasilicate. There has been studied the effect of the current density, the concentration of sodium metasilicate, the solution temperature and the duration of electrolysis on the chrysocolla current yield. In optimal conditions the current yield is 78.5 %. There has been carried out a comparative analysis of the obtained by electrolysis chrysocolla with its natural analogue. There have been used methods of IR spectroscopy, differential-thermal and chemical analyses that confirm the identification of the electrolysis chrysocolla with chrysocolla taken as the reference.

Keywords: chrysocolla mineral, voltammetry, electrolysis, sodium metasilicate.

Introduction. The involvement in poor anthropogenic dump oxidized complex copper ores could make a real prognosis for a significant copper production and improvements in the ecological environment in the Republic of Kazakhstan.

Copper mineral chrysocolla ($\text{CuSiO}_3 \cdot n\text{H}_2\text{O}$) alongside with malachite and azurite make the basis of the oxidized ore and is not practically subject to flotation methods of dressing [1-5]. The first task in the development of efficient methods of such ores processing is studying physical-and-chemical properties of chrysocolla. The difficulty consists in separating monomineral fractions of the specified mineral from the oxidized copper ore [6]. It should be noted that there are no information of artificial obtaining chrysocolla in literature, there are only different authors' suppositions regarding the formation of chrysocolla but their opinions vary. For example, academician S. S. Smirnov connects chrysocolla forming with the phenomenon of copper absorption from silica solutions [7], others indicate the possibility of the mineral occurrence be means of processing with the bicarbonate-phosphate copper solution with sodium silicate at certain values of the solution pH [8].

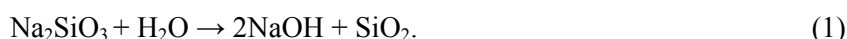
For the first time we have studied the electrochemical mechanism of chrysocolla formation and shown the possibility of the mineral electrolytic synthesis. On the mineral obtained there has been carried out a comparative analysis with its natural analog.

Experiment methodology. The studies have been carried out by the method of taking polarization curves in sodium metasilicate solution. Polarization curves have been taken using a special clamping electrodes of the specific design [9]. Voltammetric measurements have been made at the temperatures of 25-750 °C, the potential sweep rate of 10 mV/s and the concentration of electrolyte of 0.5-7.5 g/l.

The electrolysis has been carried out in a thermostated electrolysis cell of 500 mL capacity, the electrodes have been made of cathodic copper. For preparing the electrolyte there has been used distilled water and sodium metasilicate solution (Na₂SiO₃). The duration of the experiments has been 15-180 minutes. When electrolyzed, chrysocolla precipitate have been washed with distilled water, filtered and dried in the atmospheric air. The copper content have been determined by a chemical analysis [10]. The synthesized product has also been investigated by the IR spectroscopy and differential-thermal analysis methods.

Discussing results. It is known that when dissolved in water sodium metasilicate is hydrolyzed and its solution possesses an alkali reaction [11]:

On the anodic polarization curve of the copper electrode there are observed two waves.



The first weakly pronounced wave corresponds to the process of forming cuprous copper oxide [12]:



The second wave corresponds to the process of cuprous copper active transition into copper hydroxide [13]:



Copper hydroxide formed [14] reacts with sodium silicate and forms a new phase: the chrysocolla mineral



Based on the voltammetric studies carried out for the first time has been shown the possibility of obtaining the chrysocolla mineral by the electrolytic method [15].

There has been studied the current density, the concentration of sodium metasilicate, the temperature of the solution and the duration of electrolysis effect on the chrysocolla yield.

The essence of the process consists in the following:

- firstly, in the process of electrolysis on the cathode there is separated goes hydrogen



- on the anode there is observed the copper electrode dissolution



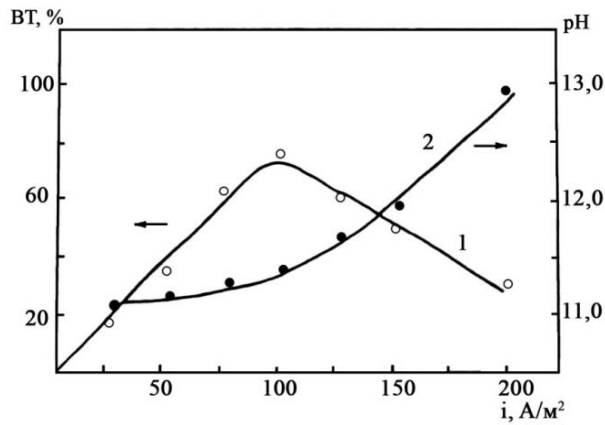
The cupric copper ions formed in the electrolyte capacity interact with the present in the solution silicate-ions (SiO₃²⁻) and form chrysocolla of bright-blue color in the reaction:



The experimental data show (figure 1, curve 1) that with the gradual increasing the current density in the range of 25 to 100 A/m², there is observed the current yield growth from 18 to 78.5 %, respectively. The current density increasing over 100 A/m² leads to a significant decreasing the current yield of the chrysocolla formation.

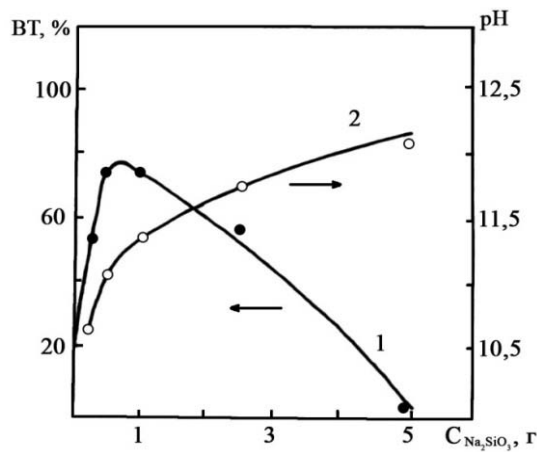
A significant effect on the product current yield is made by the initial concentration of sodium metasilicate. As it is seen in figure 2, chrysocolla is formed only in a restricted area with concentrations ranging from 0.5 to 5 g/l. The maximum yield (78.5 %) has already been reached at the 0.5 g/l concentration. The concentration increasing up to 5 g/l will result in a sharp stopping of chrysocolla formation, the current yield will decrease up to zero.

The solution temperature effect has been studied in the range of 20-800 °C; with the temperature increasing the chrysocolla current yield gradually decreases (figure 3, curve 1).



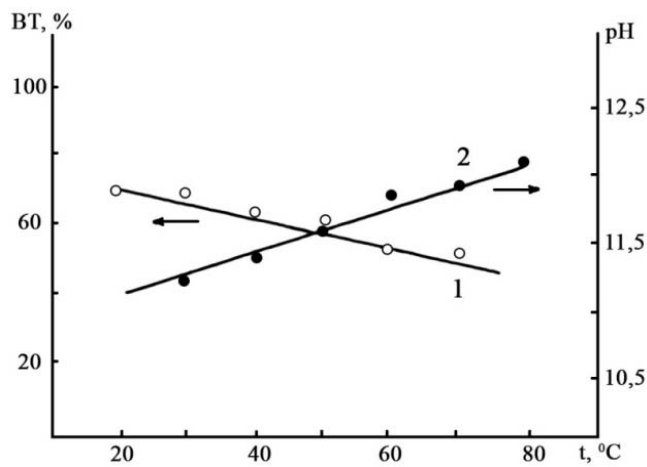
1 – current yield curve; 2 – pH changing curve

Figure 1 – Current density effect on the chrysocolla current yield: sodium metasilicate concentration 0.5 g/l, temperature 20 °C, electrolysis duration 60 min.



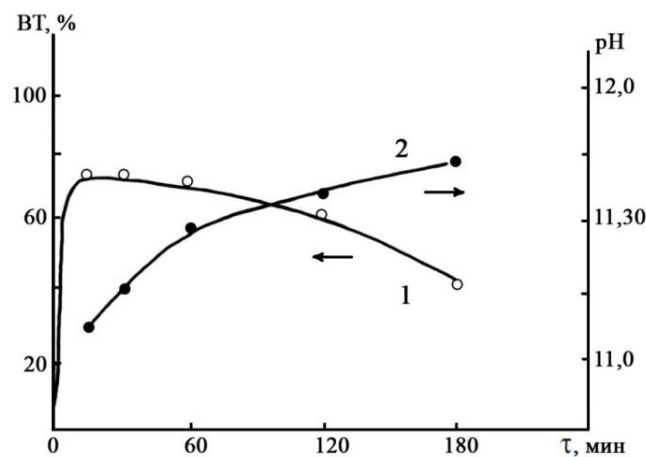
1 – current yield curve; 2 – pH changing curve

Figure 2 – Current density effect on the chrysocolla current yield: current density 100 A/m², temperature 20 °C, electrolysis duration 60 min.



1 – current yield curve; 2 – pH changing curve

Figure 3 – Temperature effect on the chrysocolla current yield: current density 100 A/m², sodium metasilicate concentration 0.5 g/l, electrolysis duration 60 min.



1 – current yield curve; 2 – pH changing curve

Figure 4 – Electrolysis duration effect on the chrysocolla current yield: current density 100 A/m², sodium silicate concentration 0.5 g/l, temperature 20 °C

With increasing the duration of electrolysis (figure 4), the chrysocolla current yield reaches the maximal value (78.5 %). The further increasing of the duration leads to decreasing the current yield.

To ensure the purity of the synthesized chrysocolla during electrolysis, the pH value of the electrolyte solution must be maintained in the range of 11.5...12.0

The precipitate of the chrysocolla obtained has been analyzed by the chemical, IR-spectroscopy and differential-thermal analysis methods.

The data of the chemical composition of chrysocolla in literature are very scarce, which is explained by the difficulty of obtaining sufficient quantities of a homogeneous material that does not contain impurities. The chemical composition of chrysocolla that does not contain aluminum can be expressed by the formula of copper metasilicate $\text{CuSiO}_3 \cdot n\text{H}_2\text{O}$ [6]. According to [17], the color of pure chrysocolla is predominantly blue, it is easily decomposed by acids and ammonia. F. V. Chukhrov [6] published the analysis of a light blue chrysocolla from the Zlatoust area of the Zhezkazgan deposit. When viewing this chrysocolla under the microscope it turns out that it is microscopically homogeneous and can be treated as monomineral corresponding to the formula $\text{CuO} \cdot \text{SiO}_2 \cdot 1,5\text{H}_2\text{O}$.

The aggregates of chrysocolla obtained have a light blue color and are decomposed by acids and ammonia.

The chemical analysis of the synthesized chrysocolla has been carried out for copper and silica according to the well-known techniques [18]. According to the results of the analysis, the copper content in the precipitate is 44.81 %, and silicon 20.13 %; when converted to chrysocolla $\text{CuSiO}_3 \cdot n\text{H}_2\text{O}$, there has been established compliance with the formula $\text{CuSiO}_3 \cdot 3,6\text{H}_2\text{O}$.

The IR spectra of the resulting light blue precipitate have been recorded in the UR-20 type spectrometer in the 500-1600 cm⁻¹ region.

Figure 5 shows the IR spectra of monomineral chrysocolla adopted as the reference [19] and chrysocolla synthesized by electrolysis. As it can be seen in the Figure, the absorption bands and intensities of chrysocolla obtained by electrolysis are identical with the corresponding values of the reference chrysocolla.

Figure 6 shows the curves of heating chrysocolla from Zhezkazgan (a) and synthesized by us (b). The differential-thermal analysis of the studied product has been carried out in a derivatograph of Q-1000 design at a rate of 10 deg/min to 1000 °C. On the curves of heating chrysocolla from Zhezkazgan (a) there are several distinctly pronounced thermal effects, in the author's opinion [20] caused by the separation of weakly bound water (120-140 °C) from the mineral, the lattice decay (450-530 °C), the formation of a new crystalline phase (690-700 °C) and sintering the substance (1040 °C). If we compare the curves of heating synthesized chrysocolla (b) with the DTA mineral curves, their similarity is generally noticeable, but it should be taken into account that the electrochemical synthesis is carried out in an aqueous medium. Therefore, in this case the effects are somewhat blurred, which is in good agreement with the theoretical

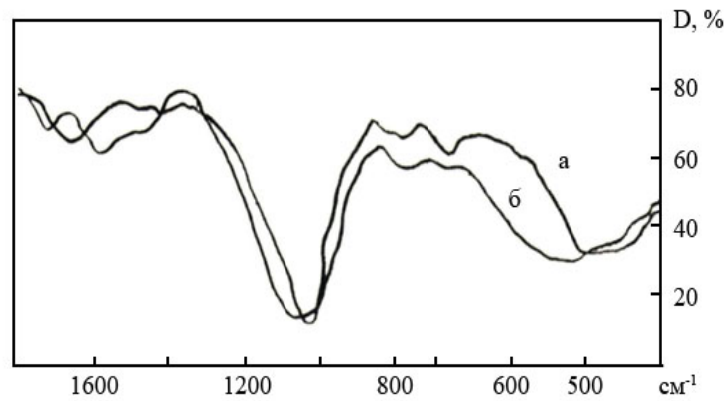


Figure 5 – Chrysocolla IR spectra:
a – chrysocolla taken as the reference [19]; b – chrysocolla synthesized by electrolysis

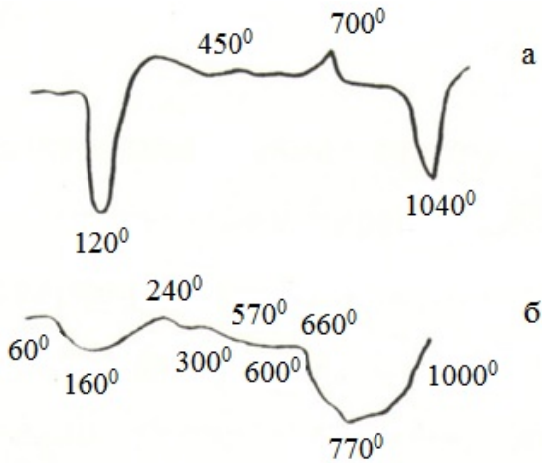


Figure 6 – DTA curves for chrysocolla:
a – chrysocolla of the Zhezkazgan deposit;
b – chrysocolla obtained by electrolysis

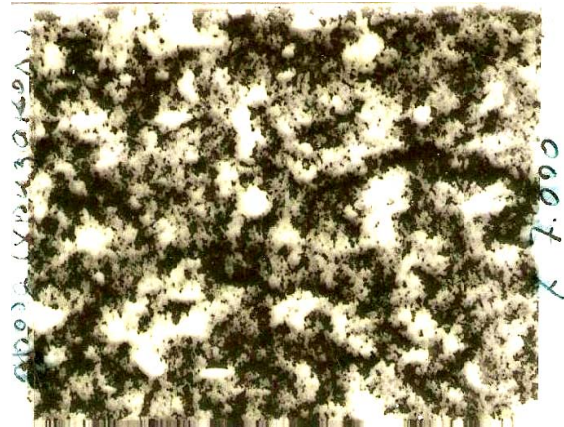


Figure 7 – Photomicrograph
of the chrysocolla mineral obtained by electrolysis
(x 7000)

provisions of DTA. On the heating curves of synthetic chrysocolla the effects at 60-160 °C and at 240-300 °C are similar in nature to the effects of the mineral. At the temperatures of 570-600 °C and 660-770 °C there is observed superposition of the exoeffect with the endoeffect, which is explained by forming the crystalline phase and its thermal transformation, respectively. A small number of effects and their minor values on the DTA curves of synthetic chrysocolla testifies to its purity, which agrees well with the conclusions of [20].

Conclusions. By the method of taking voltammetric polarization curves it has been established that two waves are observed on the anodic polarization curve of the copper electrode in the solution of sodium metasilicate. The first weakly expressed wave is referred to the process of forming cuprous oxide, the second wave corresponds to the process of transition of cuprous oxide to copper hydroxide, the formed copper hydroxide reacts with sodium metasilicate and forms the chrysocolla mineral.

For the first time there has been shown the possibility of obtaining chrysocolla by electrolysis from the aqueous solution of sodium metasilicate and it has been established that the current yield of the mineral depends on the condition of carrying out electrolysis, i.e. on the solution initial pH, the current density, the concentration of sodium metasilicate, the temperature and the duration of electrolysis. A comparative analysis of electrochemically synthesized chrysocolla with its natural analogue has been carried out using IR spectroscopy and chemical and differential analysis methods.

The results of analytical studies confirm the identity of electrolyzed chrysocolla with that taken as the reference.

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**М. М. Доспаев¹, А. Баешов², М. Ж. Журинов², А. С. Жумаканова²,
А. К. Баешова³, Б. Б. Сыздыкова¹, Д. М. Доспаев⁴, К. С. Какенов⁵**

¹Ж. Әбішев атындағы Химия-металлургия институты, Қарағанды, Қазақстан,

²Д. В. Сокольский атындағы Жанармай, катализ және электрохимия институты, Алматы, Қазақстан,

³Аль-Фараби атындағы Қазақ ұлттық университеті, Алматы, Қазақстан,

⁴Қарағанды мемлекеттік техникалық университеті, Қарағанды, Қазақстан,

⁵Қазтұтынуодағы Қарағанды экономикалық университет, Қарағанды, Қазақстан.

**МЕТАСИЛИКАТ ЕРТІНДІЛЕРІНДЕ ХРИЗОКОЛЛА МИНЕРАЛЫНЫҢ
ҚАЛЫПТАСУЫНЫҢ ФИЗИКА-ХИМИЯЛЫҚ ЗАҢДЫЛЫҚТАРЫ**

Аннотация. Қиынбайытылатын тотыққан мыс кенінің құрамына кіретін минералдардың ішінде хризоколла минералы ерекше орын алады. Осындай кендерді өндеудің жана тиімді әдістерін жасау үшін хризоколла минералының қасиеттерін жанжақты зерттеу қажет. Бұл жағдайда ең негізгі қиындық ол кеннен минералдың мономинералдық фракцияларын бөліп алу. Ғылыми әдебиеттерде хризоколланы жасанды жолымен алу туралы мәлімет жоқ, тек әртүрлі авторлардың ол минералдың жер қыртысында кремнезем ертінділерінен адсорбция арқылы қалыптасу туралы жорамалдары бар. Осыған байланысты біз алғаш рет вольтамперометрия әдісімен калий метасиликаты ертіндісінде мыс электродың анодтық поляризациялау кезінде хризоколла минералының түзілу механизмін анықтыдық. Калий метасиликаты сулы ертіндісінен электролиз арқылы хризоколланы алу мүмкіндігі көрсетілді. Хризоколланың тоқ шығымына тоқ тығыздығының, калий

метасиликаты концентрациясының, ертінді температурасының және электролиз узақтығының әсері зерттелді. Электролиздің тиімді жағдайында тоқ шығымы 78,5%-ті құрайды. Электролизбен алынған хризоколланы оның табиғи аналогімен салыстыру анализі өткізілді. Пайдаланылған ИК-спектроскопия, дифференциалды-термиялық және химиялық анализ әдістері, электролизбен алынған хризоколланың эталон ретінде қабылданған хризоколламен ұқсастығын дәлелдеді.

Түйін сөздер: хризоколла минералы, вольтамперометрия, электролиз, кремнийқышқылды натрий.

**М. М. Доспаев¹, А. Баяшов², М. Ж. Журинов², А. С. Жумаканова²,
А. К. Баяшова³, Б. Б. Сыздыкова¹, Д. М. Доспаев⁴, К. С. Какенов⁵**

¹Химико-металлургический институт им. Ж. Абишева, Караганда, Казахстан,

²Институт топлива, катализа и электрохимии им. Д. В. Сокольского, Алматы, Казахстан,

³Казахский национальный университет им. аль-Фараби, Алматы, Казахстан,

⁴Карагандинский государственный технический университет, Караганда, Казахстан,

⁵Карагандинский экономический университет казпотребсоюза, Караганда, Казахстан.

ФИЗИКО-ХИМИЧЕСКИЕ ЗАКОНОМЕРНОСТИ ФОРМИРОВАНИЯ МИНЕРАЛА ХРИЗОКОЛЛЫ В МЕТАСИЛИКАТНЫХ РАСТВОРАХ

Аннотация. Минерал хризоколла занимает особое место среди минералов, входящих в состав труднообогатимых окисленных медных руд. Для создания новых эффективных методов переработки таких руд необходимо всесторонне изучить свойства минерала хризоколлы. Трудность при этом заключается в выделении из руды мономинеральных фракций. Сведений о получении хризоколлы искусственным путем в литературе нет, лишь встречаются предположения различных авторов о формировании минерала в земной коре путем адсорбции меди из растворов кремнеземом. В связи с этим нами впервые методом вольтамперометрии установлен механизм образования минерала хризоколлы при анодной поляризации медного электрода в растворе метасиликата калия. Показана возможность получения хризоколлы электролизом из водного раствора метасиликата калия. Изучено влияние плотности тока, концентрации метасиликата калия, температуры раствора и продолжительности электролиза на выход по току хризоколлы. В оптимальных условиях электролиза выход по току составляет 78,5%. Проведен сопоставительный анализ полученной электролизом хризоколлы с ее природным аналогом. Использованы методы ИК-спектроскопии, дифференциально-термического и химического анализов, которые подтверждают идентичность полученной электролизом хризоколлы с хризоколлой, принятой в качестве эталона.

Ключевые слова: минерал хризоколла, вольтамперометрия, электролиз, кремнекислый натрий.

Information of the authors:

Dospaev M. M. – Doctor of Engineering, Head of the Laboratory of Electrochemical Processes of the Chemistry and Metallurgical Institute named after Zh. Abishev; manten.mur@mail.ru; ORCID ID: 0000-0001-9602-8099

Bayeshov A. – academic NAS RK, Doctor of Chemistry, D. V. Sokolsky Institute of Fuel, Catalysis and Electrochemistry; bayeshov@mail.ru; ORCID ID: 0000-0003-0745-039X

Журинов М. Ж. – academic NAS RK, Doctor of Chemistry, professor, Director of D.V. Sokolskiy Institute of Fuel, Catalysis and Electrochemistry; m.zhurinov@ifce.kz; ORCID ID: 0000-0001-5314-1219

Zhumakanova A. S. – Candidate of Technical Sciences, Sokolsky Institute of Fuel, Catalysis and Electrochemistry; zhumakanova62@mail.ru; ORCID ID: 0000-0003-4983-4199

Bayeshova A. K. – Doctor of Engineering, professor, 3Al-Faraby Kazakh National University; azhar_b@bk.ru; ORCID ID: 0000-0002-9076-8130

Syzdykova B. B. – Master of Engineering, junior researcher of the Chemical and Metallurgical Institute named after Zh. Abishev; syzdykova.b@gmail.com; ORCID ID: 0000-0003-4497-6366

Dospaev D. M. – Master of Engineering, Leading Engineer of the International Material Science Center of Karaganda State Technical University; dospaev_dar@mail.ru; ORCID ID: 0000-0002-8276-093X

Kakenov K. S. – Candidate of Technical Sciences, Head of the Certification and Standardization of Consumer Goods Department of Karaganda Economic University of Kazpotreboyuz; sattu55@mail.ru; ORCID ID: 0000-0003-2314-4595

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