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ИЗВЕСТИЯ

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РЕСПУБЛИКИ КАЗАХСТАН

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OF THE ACADEMY OF SCIENCES
OF THE REPUBLIC OF KAZAKHSTAN

ХИМИЯ ЖӘНЕ ТЕХНОЛОГИЯ СЕРИЯСЫ

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NAS RK is pleased to announce that News of NAS RK. Series of chemistry and technologies 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 chemistry and technologies in the Emerging Sources Citation Index demonstrates our dedication to providing the most relevant and influential content of chemical sciences to our community.

Қазақстан Республикасы Ұлттық ғылым академиясы "ҚР ҰҒА Хабарлары. Химия және технология 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. Webof Science зерттеушілер, авторлар, баспашылар мен мекемелерге контент тереңдігі мен сапасын ұсынады. ҚР ҰҒА Хабарлары. Химия және технология сериясы Emerging Sources Citation Index-ке енуі біздің қоғамдастық үшін ең өзекті және беделді химиялық ғылымдар бойынша контентке адалдығымызды білдіреді.

НАН РК сообщает, что научный журнал «Известия НАН РК. Серия химии и технологий» 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 в Emerging Sources Citation Index демонстрирует нашу приверженность к наиболее актуальному и влиятельному контенту по химическим наукам для нашего сообщества.

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PRODUCTION OF COPPER BROMIDE AT POLARIZATION BY AN ALTERNATING CURRENT

Abstract. The electrochemical method for the production of copper (I) bromide at polarization by an alternating current at 50 Hz frequency in the potassium bromide solution was developed for the first time. The effects of basic electrochemical parameters on the copper bromide formation were studied. The electrode pairs, “copper-copper” and “titanium-steel” electrodes in the two electrolysis, were connected to the electrochemical circuit and impulse current was obtained. It was shown that the copper bromide formation depends on the current density of the copper electrode in the first electrolysiscell and the current density of the titanium electrode in the second electrolysiscell. At the current density 800 A/m^{2}on the copper electrode and 60 kA/m^{2}onthe titanium electrode, the current efficiency of the copper bromide formation showed the highest value and accounted for 81% respectively. It was observed that when the concentration of potassium bromide solution is increased up to 2 mol/l, the current efficiency of the copper bromide formation increases, and it is decreased due to electrode surface passivation at higher concentrations. Increasing the solution temperature leads to the redissolution of the formed copper bromide, thereby to the decrease of the current efficiency value. It has been shown that as the alternating current increased, the current efficiency of the copper-bromide formation decreased, when the current frequency was 300 Hz, it showed the lowest value. Optimum values of electrolysis parameters were determined and the obtained copper (I) bromide content was identified by the X-ray phase analysis.

Keywords: copper bromide, alternating current, copper electrode, titanium electrode, current efficiency, electrolysis.

At present, alternating current (AC) electrolysis is of interest to many researchers in the field of electrochemistry due to the rapid development of electrochemical processes. Various forms of using AC allow to eliminate the electrode passivation and to intensify synthesis of many compounds [1].

Copper (I) bromide is a strong reductant that is widely used in chemical production, especially in the synthesis of organic matters. It acts as a catalyst in many organic reactions [2]. Brominating reactions and polymerization reactions of aromatic cycle compounds are carried out based on copper bromide [3-7].

Works [8-10] show that copper bromide forms coordination compounds with organic compounds and [11] presents the first results of the visualization of nanoparticles synthesis process at high speed by using the CuBr-laser.

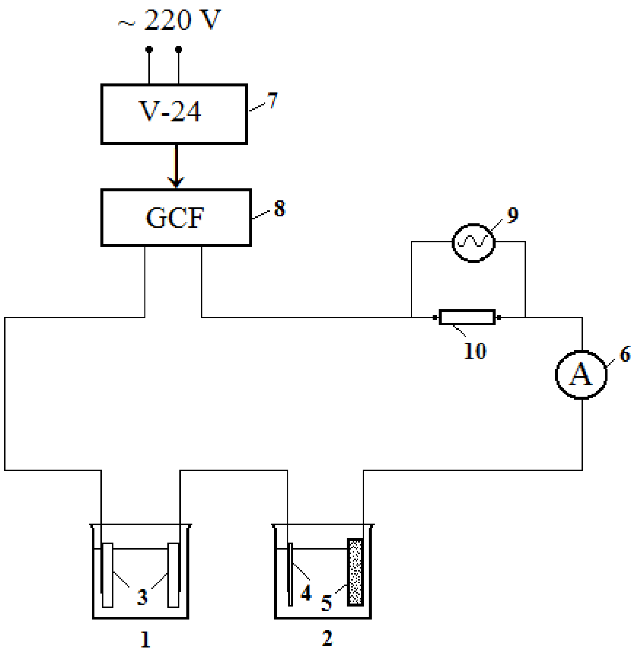
Russian scientists I.P.Chernobayev, L.A.Kasatkina and V.G.Kolesnikova obtained copper bromide by electrolysis in the presence of the anodic current. They carried out their study based on the copper and graphite electrode in the presence of aqueous solution of the copper vitriol, potassium bromide and sulfuric acid. The disadvantage of this study is that the SO_2gas released as a result of electrolysis which is poisonous and causes discomfort for the study from the sanitary and hygienic point of view [12].

As previously reported, the electrochemical properties of copper in chloride, iodide, sulphate aquatic media were investigated and an electrochemical method of obtaining various copper compounds was developed [13-16],and the electrochemical methods of synthesis of copper and zinc inorganic compounds were presented [17].

In our previous study we investigated the formation of copper bromide by polarizing copper electrode with anodic current and established the optimum conditions of electrolysis [18]. The current efficiency of the copper bromide formation reached 72%.

In this work, electrochemical production of copper bromide was carried out in the presence of the alternating current with a frequency of 50 Hz. The experiments for obtaining copper bromide were carried out in 100 ml thermostatic glass electrolysis. The second electrolysis cell was connected to the electrochemical circuit for impulse current transmission from the circuit. In the first electrolysis cell, two flat copper plates (S_{Cu}=0.0006 m^2) were placed and 2M KBr solution was used as an electrolyte. In the second electrolysis cell, a small titanium wire (S_{Ti}= 0.000006 m^2) and a large stainless steel (S_{SS} = 0.0015 m^2) were used as electrodes and 5% NaOH solution as electrolyte. It is known from literature [1] that a small titanium electrode is used as an additional electrode to produce impulse current of such frequency from the industrial alternating current of 50 Hz frequency. This method is typically used for synthesis of compounds by dissolving metals at polarization by an alternating current. It should be noted that the results of the preliminary study revealed that the copper electrode was dissolved to form copper bromide when performing the electrolysis by using the copper electrode and the titanium wire in 2M KBr solution with an alternating current, but at that time the titanium wire was agitated and caused difficulties in performing the electrolysis. In this regard, to avoid titanium agitation, the main copper electrodes in the first electrolysis cell were immersed in KBr solution, while the titanium wire in the second electrolysis were immersed in NaOH solution and connected to an AC source (Figure 1). After the electrolysis, the copper (I) bromide formed in the first electrolysis was filtered, rinsed with distilled water and absolute alcohol, dried and weighted by the weight method. The current efficiency value was calculated by the anodic half-cycle period of AC.

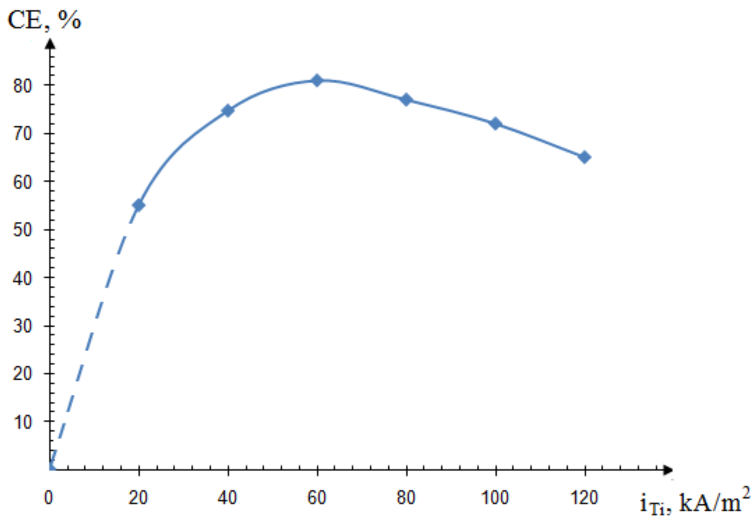
Preliminary studies have shown that the amount of the copper bromide formed in the first electrolysis cell directly depends on the current density of the copper electrode in this electrolysis cell and the current density of the titanium electrode in the second electrolysis cell. Therefore, the effect of the current density in these electrodes on the current efficiency of the copper bromide formation was comprehensively studied.



1 - first electrolysis cell; 2 - second electrolysis cell; 3 - copper electrodes; 4 - titanium electrode; 5 - stainless steel electrode; 6 - ammeter; 7 - alternating current source(V-24); 8 - AC frequency generator(GCF); 9 - oscillograph; 10 - resistance (1 Om).

Figure 1 - The principal scheme for the installation used for obtaining copper bromide by impulse current polarization

The effect of the current density on the titanium electrode polarized by an alternating current in the second electrolysis cell on the current efficiency of the copper (I) bromide formation was studied at the range of 20-120 kA/m^2. At this time, the current density of the copper electrodes polarized by an alternating current in the first electrolysis cell was kept constant (800 A/m^2). As a result of increasing the current density of the titanium electrode, the current efficiency of the copper bromide formation in the first electrolysis cell can firstly be increased and then decreased. At the current density in the titanium electrode 60 kA/m^2, the current efficiency reached the maximum value (81%). This can be explained by the formation of an oxide layer with semiconductor properties on the surface of the titanium electrode in anodic half-cycle period. This is due to the current density increase that the oxide layer on the surface of the titanium electrode is loose, its valve properties become weaker, and the alternating current is poorly formed in the circuit (Figure 2).



i_{Cu} = 800 A/m^2, [KBr] = 2 M, τ = 0.5 h, t = 20^0C, ν = 50 Hz.

Figure 2 - The effect of the current density in the titanium electrode on the dissolution of the copper electrode forming copper bromide at impulse current polarization

When the copper electrode is polarized by the production frequency alternating current, the following electrochemical reactions can take place during its anodic half-cycle period:

$$Cu^0 - e^- \leftrightarrow Cu^+ \qquad E^0 = +0.52\ V \qquad (1)$$

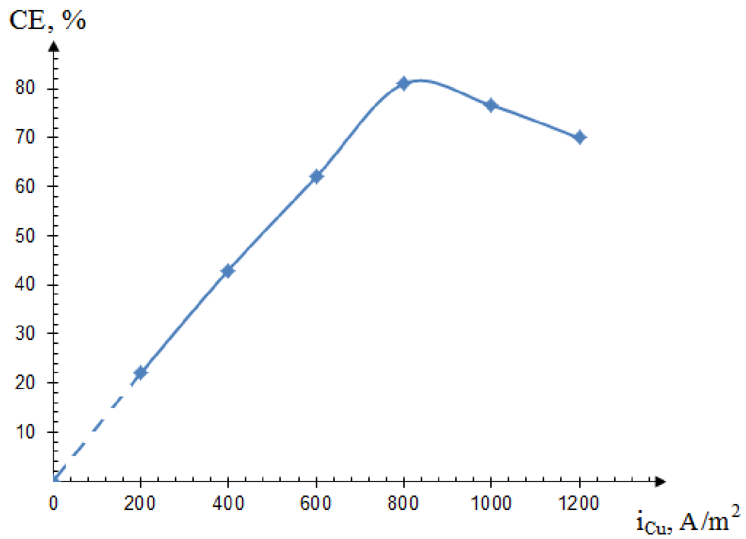
$$Cu^+ - e^- \leftrightarrow Cu^{2+} \qquad E^0 = +0.15\ V \qquad (2)$$

The results of the study have shown that single valence copper ions are stable in bromide solutions [19].

It is known that the solubility of copper (I) bromide is low (SP = $5.3 \cdot 10^{-9}$) [20, 21], so the formed Cu (I) ions are interconnected with Br^- ions and copper (I) bromide is formed. It can be observed by the formation of orange sediment formed at the bottom of the solution:

$Cu^+ + Br^- \rightarrow CuBr\downarrow$ (3)

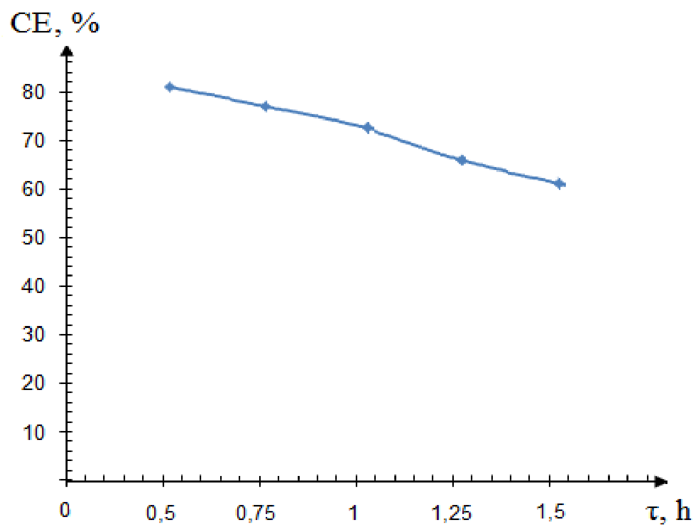
The effect of the current density in the copper electrode polarized by an alternating current on the current efficiency of the copper bromide formation was studied. The electrolysis of the copper electrode was performed at the current density of 200-1200 A/m^2; the current density increased up to 800 A/$m^2$and showed the highest value (81%). Further increase in the current density decreases the current efficiency. This is explained by the increase of the current density, the increase in the percentage of additional reactions and by the gradual passivation of the electrode after the formation of thin film of the copper bromide on the surface of the copper electrode (Figure 3).



i_{Ti} = 60 кA/m^2, [KBr] = 2 M, τ = 0,5 h, t = 20^{0}C, ν = 50 Hz.

Figure 3 - The effect of the current density in the copper electrode on the formation of the copper bromide polarized by AC

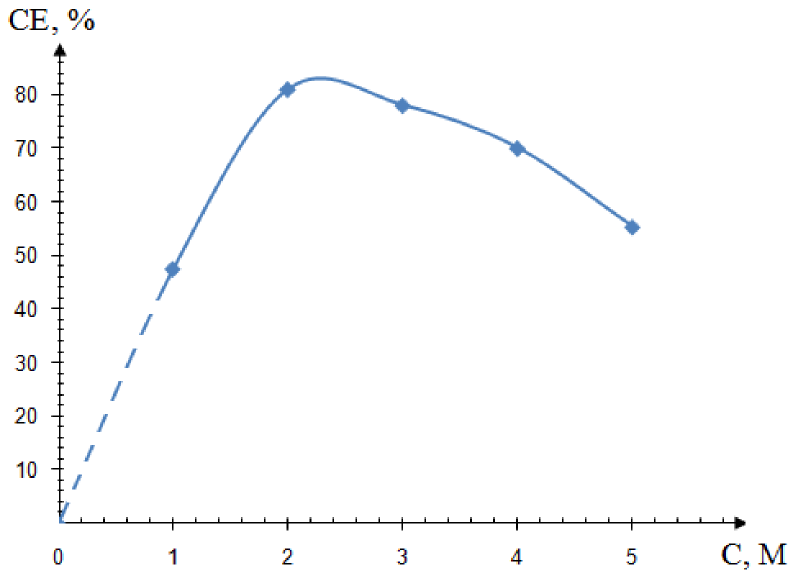
During the polarization of copper electrode by an alternating current, the effect of the electrolysis duration on the current efficiency of the copper (I) bromide formation was considered. The electrolysis was carried out for 0.5-1.5 hours. As the duration of electrolysis increased, a decrease in the current efficiency of the copper bromide formation was observed. This can be explained by the increased number of additional reactions in electrodes due to increased reaction time as well as decreased concentration of bromide ions in the solution and the passivation of the copper electrode surfaces with electrolysis products (Figure 4).



i_{Cu} = 800 A/m^2, i_{Ti} = 60 кA/m^2, [KBr] = 2 M, t^0 =20^{0}C, ν = 50 Hz.

Figure 4 - Effect of the electrolysis duration on the dissolution of copper electrode forming copper bromide at polarization by AC

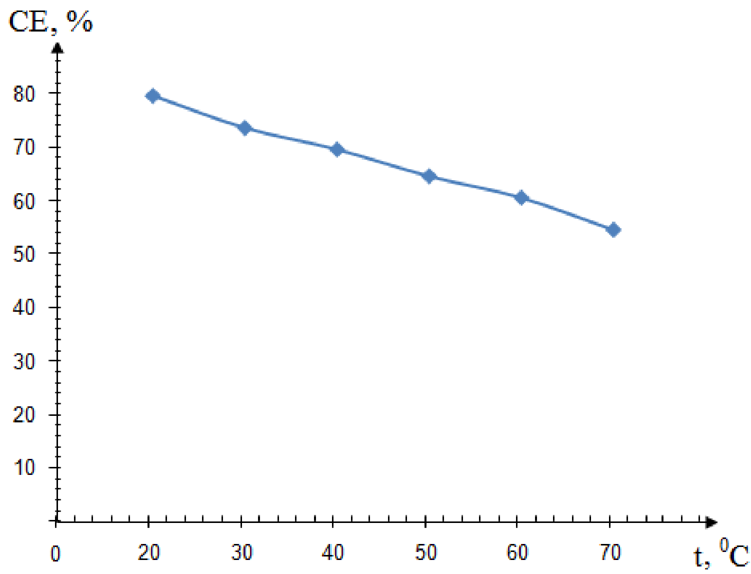
The current efficiency of CuBr formation at polarization by AC was investigated in solutions of 1-5 M potassium bromide and results are shown in Figure 5. When the electrolyte concentration exceeded 2 M, the current efficiency was 81% of maximum value. As a result of the further increase of the solution concentration, a gradual decrease can be observed in the current efficiency. At first, the interaction of the bromide ions with the copper (I) ions in the solution increases due to the increased electrolyte concentration. At high concentrations of the bromide ions, the copper electrode is covered with its bromide film and the process of passivation take place (Figure 5).



i_{Cu} = 800 A/m^2, i_{Ti} = 60 кA/m^2, τ = 0.5 h, t^0 = 20^{0}C, ν = 50 Hz;

Figure 5 - The effect of the potassium bromide concentration on the copper bromide formation at polarization of AC

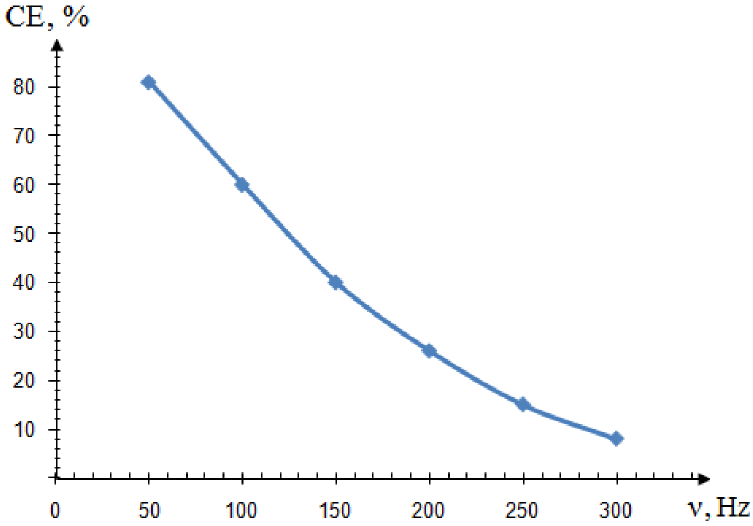
The influence of the solution temperature on the current efficiency of the copper bromide formation at polarization of AC in the copper potassium bromide solution was investigated. The electrolysis was performed between the temperature intervals 20-70^{0}C. As the solution temperature increases, it is possible to observe that the current efficiency has gradually decreased. This phenomenon can be presumed by the increase of the oxygen gas release, an additional reaction, due to the increase of the solution temperature or the dissolution of the formed copper (I) bromide (see Figure 6).



i_{Cu} = 800 A/m^2, i_{Ti} = 60 кA/m^2, [KBr] = 2 M, τ = 0.5 сағ, ν = 50 Hz.

Figure 6 - Effect of the solution temperature on the dissolution
of the copper electrode forming copper bromide at polarization by AC

The effect of the alternating current frequency on the current efficiency of the copper bromide formation at polarization by AC was investigated at 50-300 Hz (Figure 7). Since the AC frequency increases, the current efficiency of the copper bromide formation decreases, and the lowest dissolution of the copper electrode was observed at the 300 Hz current frequency. This phenomenon can be assumed that the periods which rapidly change at high current frequencies cannot fully provide the duration of the anodic half-cycle period required for the formation of the copper (I) ions, and copper ions, which have already been formed in the anodic half-cycle period, do not manage to be fully diffused into the solution volume while the metal ions in a dual and diffusive layer can participate in the reverse oxidation reaction. This phenomenon is repeated cyclically. As a result, the current efficiency of the copper dissolution decreases.



i_{Cu} = 800 A/m^2, i_{Ti} = 60 кA/m^2, [KBr] = 2 M, τ = 0.5 h, t = 20^{0}C.

Figure 7 - The effect of AC frequency on the dissolution of copper electrode forming copper bromide at polarization by AC

Thus, the effect of basic electrochemical parameters (current density in the copper and titanium electrode, electrolyte concentration, solution temperature, electrolysis duration, and AC frequency) on the copper (I) bromide formation by pairing the copper electrode with the titanium electrode in the potassium bromide aqueous solution at polarization by alternating impulse current was investigated and favourable conditions for the formation of copper (I) bromide were established: i_{Cu}=800 A/m^2, i_{Ti}=60 кA/m^2, [KBr]=2 M, t=20^{0}C, τ=0.5 h, ν=50 Hz. In these favourable conditions, the current efficiency of the copper (I) bromide formation reached 81%. The obtained copper (I) bromide content was identified by the X-ray phase analysis. The results of the study have shown that they can be used in production copper bromide.

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АЙНЫМАЛЫ ТОКПЕН ПОЛЯРИЗАЦИЯЛАУ АРҚЫЛЫ МЫС БРОМИДІН АЛУ

Аннотация. Электродтарды калий бромиді ерітіндісінде жиілігі 50 Гц импульсті айнымалы токпен поляризациялау арқылы мыс (I) бромидін алудың электрохимиялық тәсілі алғаш рет жасалды. Мыс бромидінің түзілуіне негізгі электрохимиялық параметрлердің әсерлері зерттелді. Екі электролизерде орналасқан «мыс-мыс» және «титан-болат» электродтар жұптары электрохимиялық тізбекке тізбектеле жалғанып, импульсті ток алынды. Мыс бромидінің түзілуі бірінші электролизердегі мыс электродындағы ток тығыздығына және екінші электролизердегі титан электродындағы ток тығыздығына тікелей тәуелді екендігі көрсетілді. Мыс электродындағы ток тығыздығы 800 А/м^2 мәнінде және титан электродындағы ток тығыздығы 60 кА/м^2-ге тең болғанда, мыс бромидінің түзілуінің ток бойынша шығымы (ТШ) ең жоғары мәнді көрсетіп, сәйкесінше 81% құрады. Калий бромиді ерітіндісінің концентрациясын 2 моль/л-ге дейін жоғарылатқанда, мыс бромидінің түзілуінің ток бойынша шығымы артады, ал одан жоғары концентрацияларда электрод бетінің пассивтелуіне байланысты бұл көрсеткіштің төмендейтіні анықталды. Ерітінді температурасын жоғарылату түзілген мыс бромидінің қайта еруіне әкелетіні, соның салдарынан ТШ мәнінің төмендейтіндігі анықталды. Айнымалы ток жиілігін жоғарылатқан сайын мыс бромидінің түзілуінің ток бойынша шығымы төмендеп, ток жиілігі 300 Гц болғанда, ең төмен мәнге ие болатыны көрсетілді. Электролиз параметрлерінің оңтайлы мәндері анықталып, алынған мыс (I) бромидінің құрамы рентгенофазалық анализ әдісімен идентификацияланды.

Түйін сөздер: мыс бромиді, импульсті айнымалы ток, мыс электроды, титан электроды, ток бойынша шығым, электролиз.

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ПОЛУЧЕНИЕ БРОМИДА МЕДИ ПРИ ПОЛЯРИЗАЦИИ ПЕРЕМЕННЫМ ТОКОМ

Аннотация. Впервые разработан электрохимический способ получения бромида меди (I) при поляризации электродов импульсным переменным током с частотой 50 Гц в растворе бромида калия. Исследовано влияние основных электрохимических параметров на процесс образования бромида меди. Импульсный ток был получен вследствие погружения двух пар электродов «медь-медь» и «титан-сталь» в электролизеры, соединенные в электрохимическую цепь последовательно. Показано, что образование бромида меди зависит от плотности тока на медном электроде в первом электролизере и плотности тока на титановом электроде во втором электролизере. При плотности тока, равной 800 А/м^{2}на медном электроде и 60 кА/м^2 на титановом электроде, выход по току (ВТ) образования бромида меди достигает максимального значения и составляет 81%. Установлено, что при увеличении концентрации раствора бромида калия до 2 моль/л выход по току образования бромида меди повышается, а при более высоких значениях концентрации вследствие пассивации поверхности электрода данный показатель уменьшается. Выявлено, что повышение температуры раствора приводит к растворению образовавшегося бромида меди и уменьшению ВТ. Показано, что с повышением частоты тока уменьшается выход по току образования бромида меди и самое низкое значение его наблюдается при частоте тока, равной 300 Гц. Установлены оптимальные параметры электролиза, состав полученного бромида меди (I) идентифицирован рентгенофазовым анализом.

Ключевые слова: бромид меди, импульсный переменный ток, медный электрод, титановый электрод, выход по току, электролиз.

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