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Д.В. Сокольский атындағы
«Жанаармай, катализ және электрохимия институты» АҚ

ХАБАРЛАРЫ

ИЗВЕСТИЯ

НАЦИОНАЛЬНОЙ АКАДЕМИИ НАУК
РЕСПУБЛИКИ КАЗАХСТАН
АО «Институт топлива, катализа и
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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 Үлттық 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. Бул индекстегі барысында Clarivate Analytics компаниясы журналды одан өрі the Science Citation Index Expanded, the Social Sciences Citation Index және the Arts & Humanities Citation Index-ке қабылдау мәселесін қарастыруды. Web of Science зерттеушілер, авторлар, баспашилар мен мекемелерге контент тереңдігі мен сапасын үсінады. ҚР ҰҒА Хабарлары. Химия және технология сериясы Emerging Sources Citation Index-ке енүі біздің қогамдастық үшін ең өзекті және беделді химиялық ғылымдар бойынша контентке адалдығымызды білдіреді.

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**STUDY OF KINETICS OF COPPER OXIDATION BY ELECTROLYSIS UNDER
NON-STATIONARY CONDITIONS**

Abstract. Data on the study of the kinetics and mechanism of copper electro-oxidation -reduction processes in alkaline and neutral solutions were obtained in this work. Patterns of the electrochemical oxidation of copper using alternating current were established. Based on the data of polarisation measurements kinetic parameters were calculated: the heterogeneous rate constant, the effective activation energy of copper electro-oxidation, which allow to establish the course of the metal oxidation process in alkaline and neutral environments and its characteristics. Potentiodynamic measurements in alkaline solutions at a copper anode indicate the formation of Cu₂O, CuO и Cu(OH)₂. Anodic dissolution of copper in neutral salt solutions includes two main stages: the formation of Cu(I) and Cu(II) ions. It was found that the product of oxidation at high potentials is the Cu(II) ion. The nature of the anion electrolyte affects the rate of copper anodic oxidation, while the process occurs more efficiently in solutions of sulfate and sodium chloride and is accompanied by significant polarisation. Based on the study of kinetics the possibility of obtaining powders of metal oxide was shown, which further allowed to determine the parameters of the electrolysis process in non-stationary conditions in order to obtain highly dispersed materials based on copper oxides with specific physico-chemical properties.

Key words: copper electro-oxidation, copper (I) oxide, copper (II) oxide, unsteady electrolysis, electrolyte, polarisation curves.

Introduction.

In recent years increasing attention has been drawn to studies on the synthesis of ultrafine metal powders with given structure and dispersion, due to which they can be used to create new effective materials of various purposes. A large number of works is focused on the study of electrochemical methods of production of finely dispersed metal powders at direct current, requiring the use of additional equipment for rectifying alternating current, the need to use chemically resistant electrodes in various environments, and in some cases, use of membranes to separate the electrode spaces, thereby complicating the process [1-3]. Current method is also distinguished by a complex electrolyte composition, which contains salts of metals under consideration, various additives, including organic compounds [4-6]. It should be noted that increased current intensity leads to passivation of the anodes and formation of impurities in the main product. Replacing direct current allows synthesising various compounds through the use of soluble electrodes.

The research to date states that the transition from direct current to alternating current of various forms

(pulsating, asymmetric, pulsed), including those with an adjustable component, favourably affects the synthesis of new compounds [7-10].

When selecting the optimal conditions, the following advantages of alternating current can be highlighted: firstly, the synthesis of compounds that cannot be obtained with a stationary current, secondly, high productivity of installations, and thirdly, a relatively low power consumption. However, the advantages of non-stationary electrolysis are not fully utilized both due to insufficient knowledge and the need to replace the direct current source with special electronic equipment [11-13].

Principles of the processes conducted using alternating current require further study. Use of non-stationary modes expands the possibilities of studying electrode processes, as well as inquiries of an applied nature.

Using alternating current for electrochemical processes presents a unique opportunity for the synthesis of nanodispersed materials based on metal oxides. Given that, there is no information in literature on the production of such materials by this method; therefore, further research in the field of

electrochemical synthesis of copper oxide using an alternating current of industrial frequency appears to be relevant.

The aim of this work is to study the kinetic principles of copper electro-oxidation-reduction in alkaline and neutral solutions.

Methods.

The electrochemical oxidation-reduction of copper in neutral aqueous solutions was studied by plotting potentiodynamic polarisation curves and electrolysis under galvanostatic conditions.

Potentiodynamic polarisation curves were recorded on an SVA-1BM potentiostat using a three-electrode thermostated glass cell YaES-2.

Thermostatting was conducted with an ITZh-0-03 thermostat. Current-potential curves were registered with an H307/1 flatbed two-coordinate recorder at a potential sweep rate of 5-100 mV/s. A copper rod ($S=0.5 \text{ cm}^2$) was used as a working electrode. A silver chloride electrode and a platinum electrode were used as a reference and auxiliary electrodes, respectively. All values of the working electrode potential were given in relation to the silver chloride electrode ($E_0 = \text{«plus» } 0.222\text{V}$).

To characterise the mechanics of the electro-oxidation - reduction process of copper, the following kinetic parameters were calculated: charge transfer coefficients (α), diffusion coefficients (D), heterogeneous rate constants of the electrode process (k_s) and the effective activation energy of the process (E_a) [14,15].

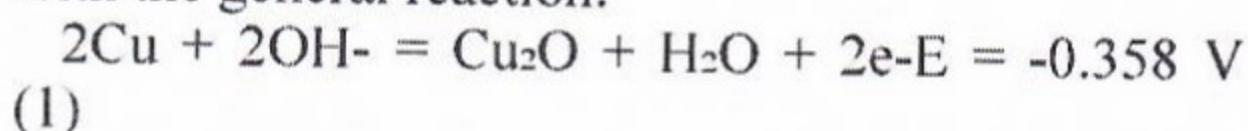
Results and discussions.

Kinetics of the electrode processes of copper oxidation-reduction in alkaline solutions were investigated in the potentiodynamic conditions. Anode-cathodic and cathode-anodic cyclic polarisation curves were recorded to establish the mechanism of electrode processes occurring on a copper electrode during polarisation with alternating current.

Polarisation measurements on a copper electrode were taken in sodium hydroxide electrolyte solutions in the concentration range of 0.4-4 mol/l, at temperatures of 20-50°C and potential sweep rates of 5-100 mV/s.

Two anodic and corresponding cathodic peaks were observed on the cyclic voltammogram (figure 1), which indicate the formation and reduction of Cu_2O and $\text{Cu}(\text{OH})_2$ or CuO .

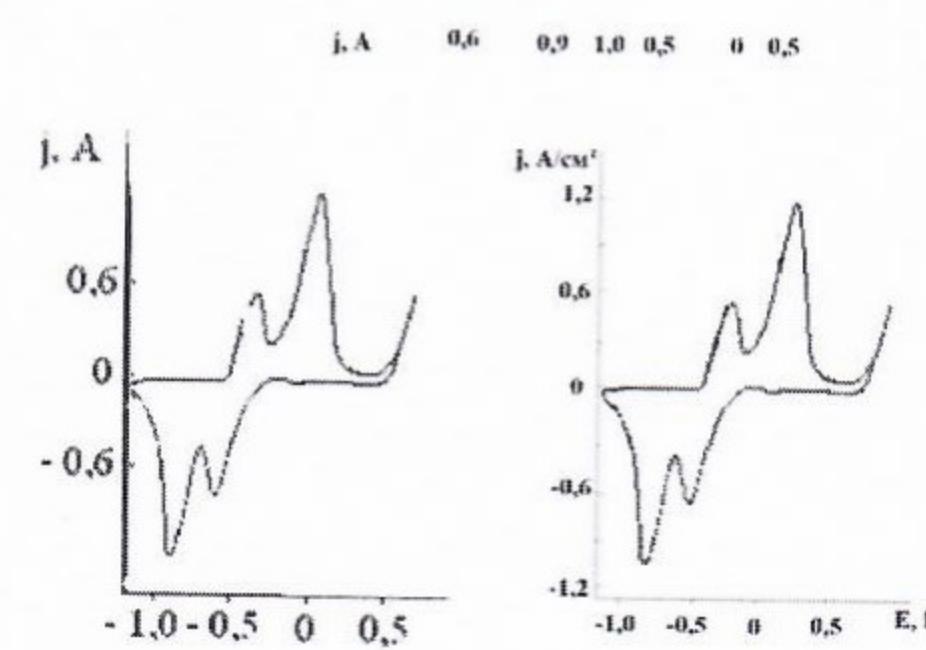
Potentiodynamic measurements in alkaline solutions on a copper anode indicate the formation of Cu_2O , CuO and $\text{Cu}(\text{OH})_2$. It is likely that copper (I) oxide first forms on the copper anode, in accordance with the general reaction:



According to the data in the studied literature, copper(II) oxide CuO and bivalent copper hydroxide $\text{Cu}(\text{OH})_2$ form during anodic oxidation, on the other hand, a number of publications [16-18] confirm the

anodic oxidation of copper electrodes, commencing with initial oxidation, namely, the formation of Cu_2O .

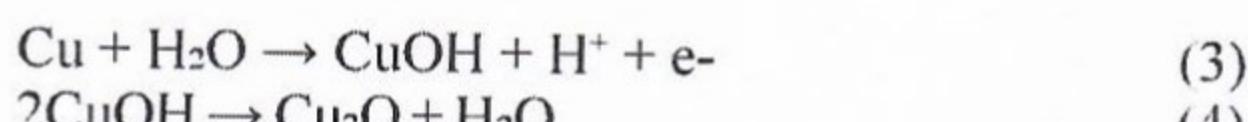
Formation of Cu_2O in an aqueous solution of sodium hydroxide can be governed both by the above electrochemical reaction (1) and the chemical reaction given in (2):



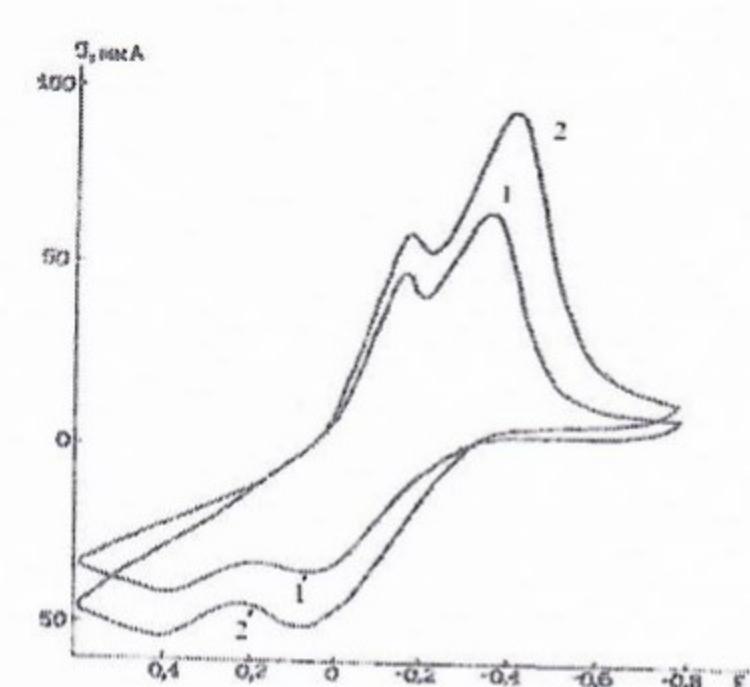
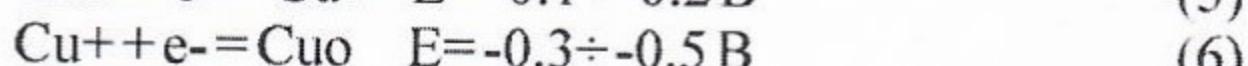
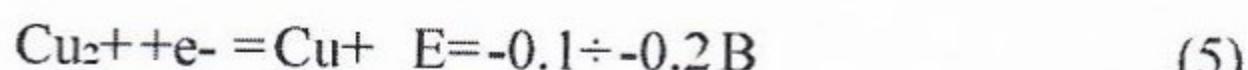
$$v = 50 \text{ mV/c}, t = 20^\circ\text{C}$$

Figure 1 – Cyclic voltammogram on a copper electrode in 0.4 mol/l sodium hydroxide solution

Monovalent copper(I) hydroxide formed by reaction (3) with further dehydration can also be considered as a product of anodic oxidation along with the formed copper(I) oxide:



The electrochemical behaviour of a copper electrode in solutions of neutral sodium salts - Na_2SO_4 , NaNO_3 and NaCl in the concentration range of 0.4-4 mol/l, at temperatures 20-50°C and potential sweep rates of 5-100 mV/s was investigated. At 25°C cyclic voltammograms present curves with two clearly pronounced current maximums (figure 2), which is indicative of a two-step copper ionisation-discharge process, schematically given as:



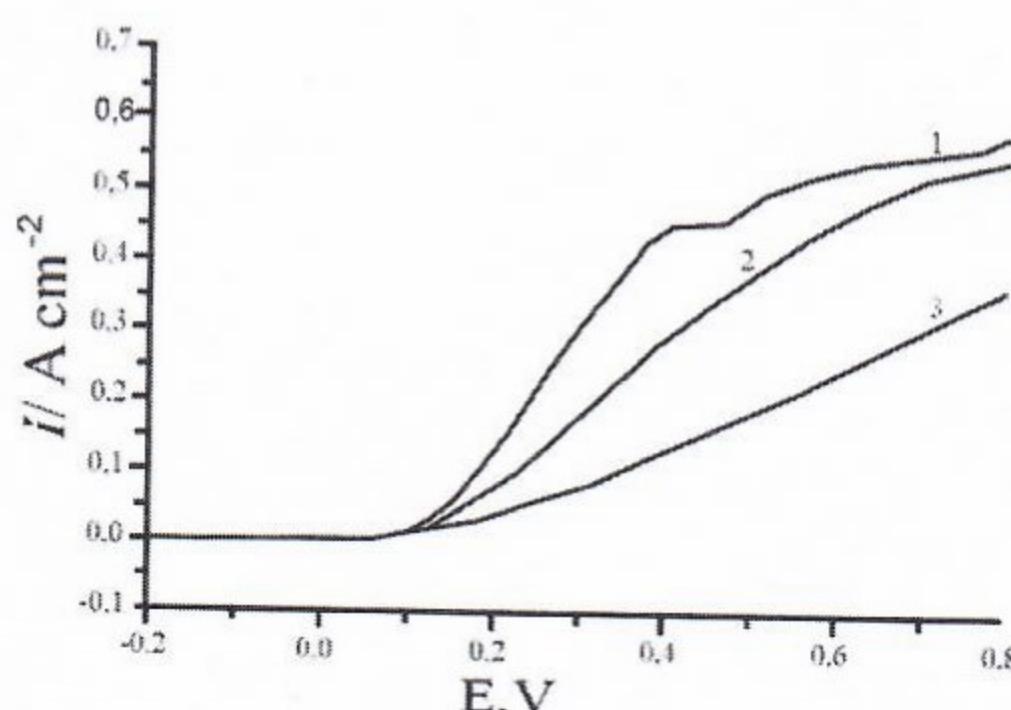
$$v = 50 \text{ mV/c}, t = 20^\circ\text{C}$$

Figure 1 – Cyclic voltammogram on a copper electrode in 0.4 mol/l sodium hydroxide solution

In order to study the nature of the electrolyte

anion, anodic potentiodynamic curves were recorded in solutions of 0.4 mol/l of Na_2SO_4 , NaNO_3 and NaCl .

Anodic process of copper dissolution in a neutral solution shown in Figure 3 can be explained as follows: at the initial stage of dissolution Cu_2O is formed on the electrode surface, and then, with an increase in the applied potential Cu^+ ions are oxidised to Cu_2^+ ions, which subsequently accumulate near the electrode surface.



1 - Na_2SO_4 , 2 - NaCl , 3 - NaNO_3

Figure 3 - Polarisation curves of a copper electrode in neutral salt solutions with a concentration of 0.4 mol/l and a sweep rate of 10 mV/s

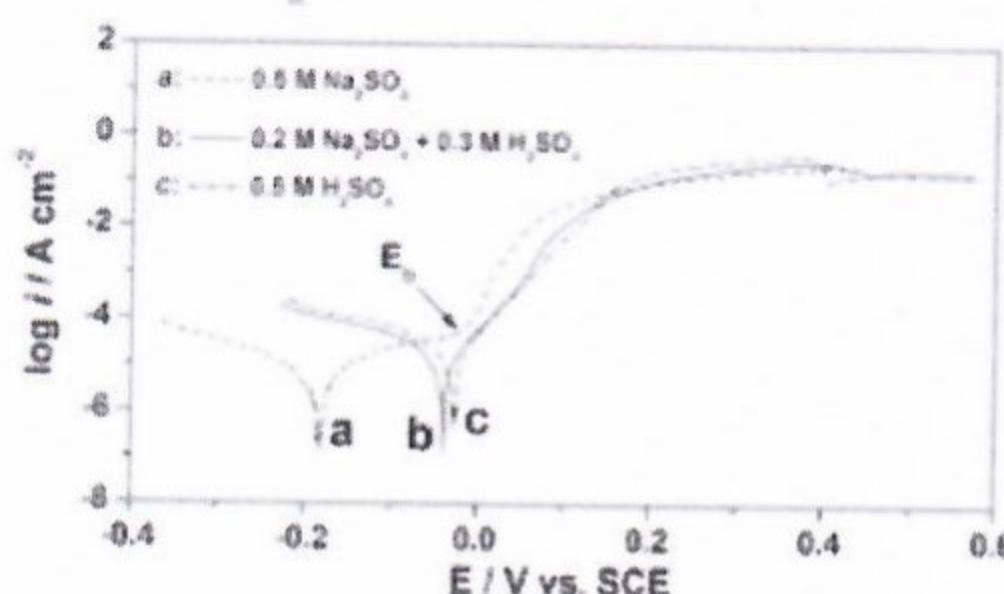
Anodic dissolution of copper in neutral salt solutions includes two main stages: formation of Cu(I) and Cu(II) . It is generally recognised that the product of oxidation at high potentials is Cu(II) . The rate of anodic oxidation of copper is affected by the nature of the electrolyte, while the process is more efficient in sulfate and sodium chloride solutions, accompanied by significant polarisation.

Values of the potentials were converted from the silver chloride electrode to the standard hydrogen scale for interpreting the polarisation measurements. Current density values were plotted on a logarithmic scale in order to construct polarisation curves in Tafel coordinates (E , mV - $\log |i|$) as shown in Figure 4.

The curves show the sections corresponding to the Tafel equation [19]:

$$\eta = a + b \cdot \lg i, \quad (7)$$

where: η – electrochemical overvoltage; a и b – coefficient of the Tafel equation; i – current density.



1 - Na_2SO_4 , 2 - NaCl , 3 - NaNO_3

Figure 4 - Tafel curves of a copper electrode in neutral salt solutions with a concentration of 0.4 mol/l and a sweep rate of 10mV/s

Ions transport and diffusion coefficients and heterogeneous rate constants of electrochemical processes were determined from the polarisation curves. Using the Matsuda and Ayabe equation the values of the electron transfer coefficients (α) for anodic processes were calculated from the difference between the peak and half-peak voltammograms (Table 1).

Table 1 - Kinetic parameters of metallic copper oxidation

Environment	α	D, cm^2/s	$k_s, \text{cm/s}$
$\text{CuO} - e^- \rightarrow \text{Cu}^+$ (first stage)			
alkaline	0.34	$1.53 \cdot 10^{-4}$	$3.02 \cdot 10^{-3}$
neutral	0.37	$1.38 \cdot 10^{-4}$	$3.84 \cdot 10^{-3}$
$\text{Cu}^+ - e^- \rightarrow \text{Cu}_2^+$ (second stage)			
alkaline	0.25	$1.27 \cdot 10^{-4}$	$2.71 \cdot 10^{-3}$
neutral	0.28	$1.19 \cdot 10^{-4}$	$3.36 \cdot 10^{-3}$

The value of the effective activation energy E_a calculated from the temperature-kinetic relations for the process in an alkaline environment, in the range of 20-60°C, for the first and second stages of copper discharge-ionisation were equal to 17.56 kJ/mol and 19.44 kJ/mol, respectively. In a neutral environment for the first and second stages of copper oxidation were 23.31 kJ/mol and 26.89 kJ/mol, respectively. Obtained activation energy values are indicative of diffusion controlled electrochemical process, suggesting that the second stage of copper(I) ions oxidation to copper(II) ions is rate-limiting.

Conclusion. Kinetics of electrode processes of copper oxidation-reduction in alkaline solutions were investigated in the potentiodynamic mode.

Electrochemical behaviour of a copper electrode in neutral sodium salts - Na_2SO_4 , NaNO_3 and NaCl was investigated in the concentration range of 0.4-4 mol/L, at temperatures of 20-50oC and potential sweep rates of 5-100 mV/s. It was established that during anodic copper oxidation Cu_2O is the primary product of the electrode reaction.

Based on the data of polarisation measurements kinetic parameters were calculated: the heterogeneous rate constant (k_s), effective activation energy (A_{eff}) of copper and zinc electro-oxidation process in alkaline and neutral environments, which allow to define the course of oxidation of metals in alkaline and neutral environments and its characteristics. It was demonstrated that powders of metal oxides (zinc and copper) could be obtained by electrolysis in non-stationary conditions.

Based on the reaction kinetics the possibility of obtaining zinc and copper oxides was shown, from which the required parameters of the electrolysis process were determined, in order to obtain dispersed materials based on metal oxides with specified physicochemical properties.

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СТАЦИОНАРЛЫҚ ЕМЕС ЖАҒДАЙЛАРДА ЭЛЕКТРОЛИЗБЕН МЫСТЫҢ ТОТЫГУНЫҢ КИНЕТИКАЛЫҚ ЗАНДЫЛЫҚТАРЫН ЗЕРТТЕУ

Аннотация. Жұмыста сілтілік және бейтарап ерітінділерде мыстың электр тотығу-тотықсыздандыру кинетика мен механизмі бойынша зерттеу нәтижелері алынған. Айнымалы тоқты пайдалану кезінде мыстың электрохимиялық тотығу заңдылықтары анықталды. Мыстың электр тотығу-тотықсыздану процесінің механизмін сипаттау үшін келесі кинетикалық параметрлер есептеледі: зарядтың тасымалдау коэффициенттері, диффузия коэффициенттері, электродты процесінің гетерогенді жылдамдық константалары және процестің тиімді активтену энергиясы. Потенциодинамикалық өлшеулер мыс анодында сілтілі ерітінділерде Cu_2O , CuO және $\text{Cu}(\text{OH})_2$ түзілуін көрсетеді. Мыстың бейтарап тұз ерітінділерінде анодтың еруі екі негізгі кезеңді қамтиды: Cu (I) және Cu (II) иондарының түзілуі. Жоғары потенциалдардағы тотығу өнімі Cu (II) ионы екендігі анықталды. Электролит анионының табигаты мыстың анодты тотығу жылдамдығына әсер етеді, ал процесс натрий сульфаты мен натрий хлориді ерітінділерінде тиімдірек және айтарлықтай поляризациямен жүреді. Кинетикалық заңдылықтарды зерттеу негізінде мыс оксидтерін алу мүмкіндігі дәлелденді, бұл берілген физика-химиялық қасиеттеріне ие металл оксидтері негізінде дисперсті материалдар алу үшін электролиз процесінің параметрлерін анықтауға мүмкіндік берді.

Түйін сөздер: мыстың электр тотығуы, мыс (I) оксиді, мыс (II) оксиді, стационарлық емес электролиз, электролит, поляризациялық қисықтары.

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ИССЛЕДОВАНИЕ КИНЕТИЧЕСКИХ ЗАКОНОМЕРНОСТЕЙ ОКИСЛЕНИЯ МЕДИ ЭЛЕКТРОЛИЗОМ В НЕСТАЦИОНАРНЫХ УСЛОВИЯХ

Аннотация. В работе получены данные по исследованию кинетики и механизма электроокисления-восстановления меди в щелочных и нейтральных растворах. Выяснены закономерности электрохимического окисления меди с использованием переменного тока. По данным поляризационных измерений рассчитаны кинетические параметры: гетерогенная константа скорости, эффективная энергия активации процесса электроокисления меди, позволяющие установить протекание процесса окисления металла в щелочной и нейтральной средах и его характеристику. Потенциодинамические измерения в щелочных растворах на медном аноде свидетельствуют об образовании Cu_2O , CuO и $\text{Cu}(\text{OH})_2$. Анодное растворение меди в нейтральных растворах солей включает две основные стадии: образование ионов Cu (I) и Cu (II). Установлено, что продуктом окисления при высоких потенциалах является ион Cu (II). Природа аниона электролита влияет на скорость анодного окисления меди, а процесс протекает эффективнее в растворах сульфата и хлорида натрия и сопровождается значительной поляризацией. На основании изучения кинетических закономерностей показана возможность получения порошков оксидов металлов, что позволило определить параметры проведения процесса электролиза в нестационарных условиях с целью получения высокодисперсных материалов на основе оксидов меди с заданными физико-химическими свойствами.

Ключевые слова: электроокисление меди, оксид меди (I), оксид меди (II), нестационарный электролиз, электролит, поляризационные кривые.

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RESEARCH OF MALTING PROPERTIES OF KAZAKHSTAN TRITIKALE GRAIN VARIETIES FOR USE IN THE BEVERAGE INDUSTRY

Abstract. Today the beverage industry occupies an important place in the processing industry of the Republic of Kazakhstan and is one of the most attractive investment sectors of the economy.

However, this industry is still not provided in sufficient quantities with its own, domestic high-quality raw materials - rye and / or barley malt. For example, fermented malt is used as a source of colors and aromas. It is the main raw material in the preparation of kvass, kvass wort concentrate and kvass bread, the rate of application of this type of malt is from 40 to 60%, depending on the method of production of this type of product.

The processing of cereals with a high protein content (above 12%) and a low starch content and extractiveness is economically disadvantageous, and undesirable from a quality point of view.

The most important directions in solving this problem should be recognized as the improvement and development of new resource-saving technologies of malt using non-traditional types of raw materials.

Currently, the use of triticale grain in the fermentation industry in the production of malt and the further replacement of traditional types of malt for the preparation of alcohol, beer, kvass is promising. In recent years, new varieties of triticale have been obtained in the Republic of Kazakhstan, which are distinguished by high technological properties, which are included in the State Register.

In this regard, it is obvious that the performance of work related to theoretical and experimental research aimed at the development of new technological modes of preparation of malt from triticale is one of the urgent tasks, the decision of which, to create an assortment of drinks. The purpose of the research work is to study the malting properties of Kazakhstani varieties of triticale grain.

Key words: Malt, barley, triticale, fermentation industry, kvass, kvass wort concentrate, ethyl alcohol, extract, variety, germination ability.

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