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

ХАБАРЛАРЫ

ИЗВЕСТИЯ

НАЦИОНАЛЬНОЙ АКАДЕМИИ НАУК
РЕСПУБЛИКИ КАЗАХСТАН
АО «Институт топлива, катализа и
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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.

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PHYSICAL-AND-CHEMICAL FEATURES AND MECHANISM OF COPPER OXIDE (I) FORMATION IN ALKALINE CHLORIDE SOLUTIONS

Abstract. The most promising ways to obtain highly dispersed copper oxides are electrochemical methods. This method of transferring copper into a solution allows finding accurately the formation modes of the required valence ion and obtaining high chemical purity compounds. The methods of producing copper oxide (I) are based on the anodic oxidation of metallic copper in an alkaline chloride-containing solution. The main advantage of the electrochemical method is the possibility of obtaining high quality powder with a smaller particle size not containing metallic copper and bivalent copper oxide. The results of studying anodic corrosion of copper in weakly alkaline and acidic chloride solutions show that, depending on the content of chloride ions in the solution and the specified potential value, there can be formed complex copper chloride compounds that in the process of hydrolysis form copper oxide (I) powder. The region of thermodynamically stable existing of monovalent copper chloride complexes at low potential values and pH increases.

In this work there has been for the first time made the calculation and a diagram has been constructed that allows determining only the most probable pH, pCl^- and the electrode region, in which copper oxide (I) can be obtained.

Key words: copper oxide (I), complex copper chloride, equilibrium potentials, thermodynamics, diagram of equilibrium systems.

Summary. To predict the reactions occurring under specific conditions for producing copper oxide (I), and to establish the regions of preferential existence of a particular phase, we constructed the E –pH diagram for the $Cu – Cu^- – H_2O$ system. The concentration of chloride ions is assumed to be 4.25 $mol.dm^{-3}$, the activity coefficient is 0.78, the pH varies from 0 to 14, the temperature is 298 K. The work presents a diagram of the stable existence of the Cu_2O and CuCl compounds depending on the solution pH and pCl^-. It shows the equilibrium conditions in the $Cu – Cu^- – H_2O$ system at pH equal to 13 depending on pCl^-. The potential range at which Cu_2O is stable is very limited and its width is within 0.2 V under the conditions studied for the synthesis of copper oxide (I).

The diagrams calculated by us allow determining only the most probable region of pH, pCl^- and the electrode potential, in which copper oxide (I) can be obtained.

Introduction. Powdered copper oxide (I) is used to prepare solder paste, copper-containing additives in friction and anti-friction couples and a number of other industries. The most large-scale consumer of copper oxide (I) has recently become the chemical industry, using this reagent to prepare biocidal paints that protect the underwater part of ships and structures from fouling by microorganisms [1].

However, copper oxide (I) currently being produced does not fully meet the requirements of mechanical engineering, electronic and chemical industries in terms of the particle size, chemical composition and scale of production. Thus, in manufacturing raw materials for semiconductors of the electronics industry [2], materials with high anti-wear tribological properties [3-5], antifouling paints, there is required dispersed nano-sized copper oxide (I) with a high content of the main component. Copper oxide (I) of nanoscale is almost impossible to be obtained by traditional pyrometallurgical, autoclave and chemical methods.

Experiment methodology. One of present day methods of obtaining high dispersion oxides of metals is an electrochemical method. The advantage of this method of preparation is formation of nanoscale powder particles in the electrode layer. The lack of knowledge of the electrochemical processes occurring on the electrodes, the mechanism of powder formation, the reasons for the poor performance of the electrolysis process formed by limited anode current densities impede the industrial development of the electrochemical method of producing copper oxide (I) by direct current electrolysis. Experimental data on the destruction of metals in alkaline solutions are given in works [6–9]. In electrolysis using various forms of current other than direct current, it becomes possible to carry out a number of electrochemical processes with the best performance. In works [10-17] it was shown that during the electrolysis of copper under the conditions of non-stationary electrolysis in aqueous electrolytes, its intensive destruction with the formation of copper oxides was observed.

Discussing results. The results of studying anodic corrosion of copper in weakly alkaline and acidic chloride solutions show that depending on the content of chloride ions in the solution and the value of the specified potential, chloride complexes of copper (I) $\mathrm{CuCl_m^{-(m-1)}}$ or $\mathrm{CuCl_{тв}}$ ions can be formed. Increase the flow velocity contributes to increasing the limiting anodic current density values. Copper oxide (I) is formed as a result of the hydrolysis reaction of copper (I) chloride complexes.

The electrochemical method of converting metals into a solution allows finding accurately the ion formation modes of the required valence and obtaining a compound of high chemical purity for the main component.

The reactions have the following values of standard potentials (E°):

$$\mathrm{Cu - 2e \leftrightarrow Cu^{+2}}\ E° = 0.3377 \pm 0.0007\mathrm{B} \tag{1}$$

$$\mathrm{Cu - e \leftrightarrow Cu^{+}}\ E° = 0.5072 \pm 0.0015\mathrm{B} \tag{2}$$

Judging by the values of standard potentials in aqueous solutions that do not contain complexing ligands, with anodic polarization there is more likely running reaction (1) [3].

In the presence of chloride ions, the picture changes: so for the reaction

$$\mathrm{Cu + Cl^{-} \leftrightarrow CuCl_{тв} + e,} \tag{3}$$

the E° value is equal to 0.137 B, that is much more negative than the potential of reaction (1) [4].

Besides, in chloride solutions there can be copper oxidation to complex ions in the reaction:

$$\mathrm{Cu + mCl^{-} \leftrightarrow CuCl_m^{-(m-1)} + e} \tag{4}$$

It should be noted that at a high concentration of chloride ions in the solution, the composition of copper (1) is as follows: $\mathrm{CuCl_4^{-3}}$ and $\mathrm{CuCl_3^{-2}}$, at this there is mainly formed $\mathrm{CuCl_4^{-3}}$.

Let us calculate the standard potential according to m=4 ($E°\mathrm{CuCl_4^{-3}}$/Cu). The dissociation constant (Kg)$\mathrm{CuCl_4^{-3}}$ makes $2.5 \cdot 10^{-6}$ [3, 4]. The copper electrode equilibrium potential for reaction (2), according to the Nernst equation is equal to: $\mathrm{Cu^{+}/Cu}$

$$E = E^0_{\mathrm{Cu^+/Cu}} + \frac{RT}{nF}\lg \mathrm{Cl_{Cu^+}}.$$

In the solution containing chloride ions and copper in the form of $\mathrm{CuCl_4^{-3}}$, the standard potential value makes:

$$E_{\mathrm{CuCl_4^{-3}/Cu}} = E^0_{\mathrm{Cu^+/Cu}} + 0.05914\lg Kg + 0.05914\lg\frac{[\mathrm{CuCl_4^{-3}}]}{[\mathrm{Cl^-}]^4}. \tag{6}$$

Then:

$$E^0_{CuCl_4^{-3}/Cu} = 0.5072 + 0.05914\lg(2{,}5\cdot10^{-6}) = 0.176B\ . \qquad (7)$$

This calculation allows drawing a conclusion that the chloride medium is thermodynamically most favorable for copper oxidation with the formation of Cu (I) ions at the anode, whereas in solutions that do not contain a complexing ligand, copper is more likely to dissolve with forming copper (II) by reaction (1).

The calculation has been performed for the system at 298 K due to the absence of literature and reference data for the dissociation constants of copper (I) chloride complexes at elevated temperatures. Since relative changes in potentials do not change with increasing temperatures, the results obtained are useful in analyzing reactions carried out at temperatures higher than 298 K.

To predict the reactions occurring under specific conditions for the production of copper oxide (I), and to establish the regions of the predominant existence of a particular phase, we have constructed the E –pH diagram for the $Cu – Cu^+ – H_2O$ system (Figure 1). The concentration of chloride ions is assumed to be 4.25 $mol.dm^{-3}$, the activity coefficient is 0.78, the pH value varies from 0 to 14, the temperature is 298 K. Figure 2 shows the diagram of the stable existence of Cu_2O and CuCl compounds depending on the solution pH and pCl^-, and Figure 3 shows the equilibrium conditions in the $Cu – Cu^+ – H_2O$ system at pH equal to 13 depending on pCl^-.

Electrochemical and chemical reactions, as well as such expressions for equilibrium potentials are given in Table 1. The values of standard potentials and constants are taken on the basis of works [18, 21].

Table 1 – Reactions equations and standard potentials values

No. of reactions equation	Equilibrium potentials and pH
Electrochemical reactions	
1. $2Cu + H_2O \leftrightarrow Cu_2O + 2H^+ + 2e$	$E=0.471 – 0.0591pH$
2. $Cu + H_2O \leftrightarrow CuO + 2H^+ + 2e$	$E=0.569 – 0.0591pH$
3. $Cu + 2H_2O \leftrightarrow Cu(OH)_2 + 2H^+ + 2e$	$E=0.609 – 0.0591pH$
4. $Cu_2O + H_2O \leftrightarrow 2CuO + 2H^+ + 2e$	$E=0.669 – 0.0591pH$
5. $Cu_2O + 3H_2O \leftrightarrow 2\ Cu(OH)_2 + 2H^+ + 2e$	$E=0.747 – 0.0591pH$
6. $Cu \leftrightarrow Cu^{+2} + 2e$	$E=0.337 + 0.0295\ \lg\alpha_{Cu^{+2}}$
7. $Cu^+ \leftrightarrow Cu^{+2} + e$	$E=0.153(\alpha_{Cu^+} = \alpha_{Cu^{+2}})$
8. $Cu + Cl^- \leftrightarrow CuCl_{\text{тв}} + e$	$E=0.124 – 0.0591\ \lg\alpha_{Cl^-}$
9. $Cu + 2Cl^- \leftrightarrow CuCl_2^- + e$	$E= 0.189+0.0591\lg\alpha_{CuCl_2^-} – 0.1182\lg\alpha_{Cl^-}$
10.$Cu + 3Cl^- \leftrightarrow CuCl_3^{-2} + e$	$E=0.229+0.0591\lg\alpha_{CuCl_3^{-2}} – 0.1773\lg\alpha_{Cl^-}$
11.$Cu + 4Cl^- \leftrightarrow CuCl_4^{-3} + e$	$E=0.176+0.0591\lg\alpha_{CuCl_4^{-3}} – 0.2364\lg\alpha_{Cl^-}$
12.$Cu_2O+2H_2O+Cl^- \leftrightarrow Cu_2(OH)_3Cl+H^++2e$	$E=0.451+0.0295pH – 0.00295\ \lg\alpha_{Cl^-}$
13. $2CuCl + 3H_2O \leftrightarrow Cu_2(OH)_3Cl + Cl^- +3H^++2e$	$E=0.785 – 0.0882pH – 0.0295\lg\alpha_{Cl^-}$
14. $CuCl + 2H_2O \leftrightarrow Cu(OH)_2 + Cl^- + 2H^+ +e$	$E=0.777 – 0.0591pH – 0.0591\lg\alpha_{Cl^-}$
15. $Cu^+ + H_2O \leftrightarrow HCuO_2^- +3H^+ +e$	$E=1.733 – 2.232pH$
Chemical reactions	
16. $Cu_2O+ 2H^++2Cl^- \leftrightarrow 2CuCl + H_2O$	$pH = 5.66 + \lg\alpha_{Cl^-}$
17. $2CuO + H^+ + Cl^- + H_2O \leftrightarrow Cu_2(OH)_3Cl$	$pH = 7.4 + \lg\alpha_{Cl^-}$
18. $2CuCl_2^- + H_2O \leftrightarrow Cu_2O + 4Cl^- +H^+$	$pH = 4.63 + \lg\alpha_{Cl^-} – \lg\alpha_{CuCl^-}$

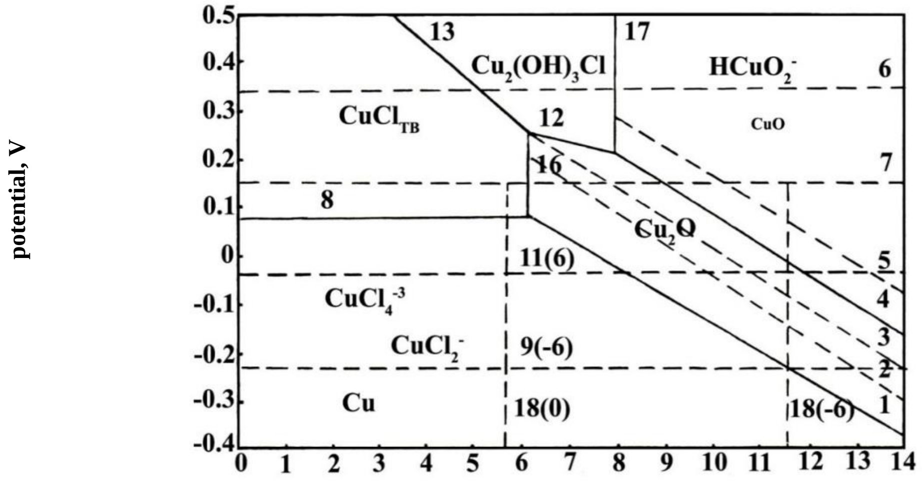

Figure 1 - E−pH diagram for the Cu – Cu$^+$ – H_2O system
C_{NaCl} = 4.25 mol.dm^{-3}
T = 298 K

According to the diagram (Figure 1), the region of thermodynamically stable existence of CuCl decreases with the values of potential smaller than +0.09 V and with increasing pH. At potentials corresponding to the region above line (13), dicupritrioxychloride may be formed, and at pH exceeding 6.18 copper (I) chloride may hydrolyze to form copper oxide (I).

It should be noted that at high pH the equilibrium potential of reaction (I) is more negative than the potential of the copper oxidation to copper (I) chloride and is comparable with the potential for the formation of complex copper ions: $CuCl_2^-$,$CuCl_3^{-2}$, $CuCl_4^{-3}$ (reactions 9, 10, II). Therefore, during electrochemical oxidation of copper in the test solution with pH =13 and with E lower than 0.09 V, the formation of a solid CuCl phase on the anode is thermodynamically unlikely.

Attention is drawn to the wide range of pH values and at the same time a narrow range of potential values at which the existence of copper oxide (I) is likely. At potentials above lines (1–5), copper oxide (I) is oxidized to copper (II) oxide and copper (II) hydroxide.

Straight line 16 (Figure 2) is the hydrolysis equilibrium line (according to equation (16). Below line (16) copper chloride is stable above, its hydrolysis occurs with the formation of copper oxide (I). [22]

The regions of stable existence of metallic copper, copper (I) chloride and copper oxide (I) at pH 13, depending on pCl$^-$ are presented in Figure 3. Straight lines 1, 8 and 16, corresponding to the above equations, are the lines of the equilibrium state of the systems. In region 1 the metallic phase is stable. In region II chloride copper compounds are stable that can hydrolyze to form Cu_2O at pCl$^-$ that is higher than 6, 18. In region III Cu_2O is stable.

The range of potentials at which Cu_2O is stable is very limited and its width is within 0.2 - x V under the conditions studied for the synthesis of copper oxide (I).

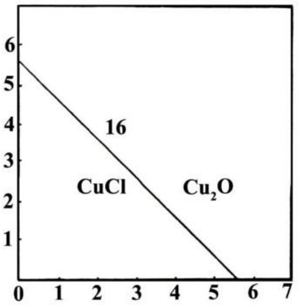

Figure 2 – Diagram of the CuCl– Cu_2O system equilibrium

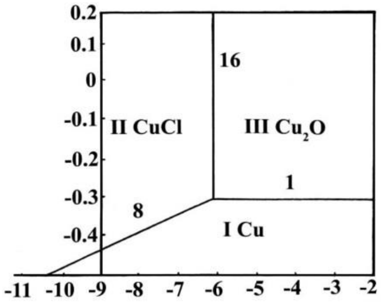

Figure 3 – Diagram of the Cu – CuCl– Cu_2O system equilibrium (pH = 13)

Conclusions. Thus, the diagrams calculated by us allow determining only the most probable region of pH, pCl^- and the electrode potential, in which it is possible to obtain copper oxide (I).

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СІЛТІЛІ ХЛОРИДТІ ЕРІТІНДІЛЕРДЕ МЫС ОКСИДІНІҢ (І) ТҮЗІЛУ МЕХАНИЗМІ МЕН ФИЗИКА-ХИМИЯЛЫҚ ЕРЕКШЕЛІКТЕРІ

Аннотация. Мыстың жоғары дисперсті оксидтерін алудың ең тиімді тәсілдері электрохимиялық әдістер болып табылады. Мыс ерітіндісіне ауыстырудың көрсетілген әдісі талап етілетін валенттілік ионының түзілу режимін дәл табуға және жоғары химиялық тазалықты қосылыстарды алуға мүмкіндік береді. Мыс оксидін(І) алу әдістері сілтілі хлорлы ерітіндіде металл мыстың анодты тотығуына негізделеді. Электрохимиялық әдістің негізгі артықшылығы металл мысы және екі валентті мыс оксиді жоқ бөлшектерінің мөлшері аз, жоғары сапалы ұнтақты алу мүмкіндігі болып табылады. Әлсіз сілтілі және қышқыл хлоридті ерітінділердегі мыстың анодты коррозиясын зерттеу нәтижелері ерітіндідегі хлорид-иондардың құрамына және берілетін әлеуеттің көлеміне байланысты гидролиз процесінде мыс тотығы (І) ұнтағын қалыптастыратын мыстың хлоридті кешендері пайда болуы мүмкін екенін көрсетті. Бір валентті мыстың хлоридті кешендерінің термодинамикалық тұрақты өмір сүру аймағы әлеуеттің аз мәндері және pH өсуі кезінде қысқарады.

Бұл жұмыста бірінші рет есептеу жүргізілді және мыс оксидін (І) алу мүмкін болатын электрод потенциалын **Тірек сөздер:** мыс тотығы (І), мыстың хлоридтік комплекстер, тепе-тендік потенциалдар, термодинамика, тепе-тендік құрылымдар диаграммасы

ың PH, pCl– ықтимал аймағын ғана анықтауға мүмкіндік беретін диаграмма жасалды.

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ФИЗИКО-ХИМИЧЕСКИЕ ОСОБЕННОСТИ И МЕХАНИЗМ ОБРАЗОВАНИЯ ОКСИДА МЕДИ (I) В ЩЕЛОЧНЫХ ХЛОРИДНЫХ РАСТВОРАХ

Аннотация. Наиболее перспективными способами получения высокодисперсных оксидов меди являются электрохимические методы. Указанный метод перевода в раствор меди позволяет достаточно точно найти режимы образования иона требуемой валентности и получить соединения высокой химической чистоты. Методы получения оксида меди(I) базируются на анодном окислении металлической меди в щелочном хлоридсодержащем растворе. Основным преимуществом электрохимического метода является возможность получения порошка высокого качества с меньшим размером частиц, не содержащих металлическую медь и оксид двухвалентной меди. Результаты исследований анодной коррозии меди в слабощелочных и кислых хлоридных растворах показали, что в зависимости от содержания хлорид-ионов в растворе и величины задаваемого потенциала могут образовываться хлоридные комплексы меди, которые в процессе гидролиза формируют порошок оксида меди (I). Область термодинамически устойчивого существования хлоридных комплексов одновалентной меди при малых значениях потенциала и росте pH сокращается.

В данной работе впервые произведен расчет и построена диаграмма, которая позволяет определить только наиболее вероятную область pH, pCl^- и потенциала электрода, в которой возможно получение оксида меди (I).

Ключевые слова: оксид меди (I), хлоридные комплексы меди, равновесные потенциалы, термодинамика. диаграмма равновесных систем**.**

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