

HYDROGEN PRODUCTION IN A COMBINED ELECTROCHEMICAL SYSTEM: ANODE PROCESS

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Abstract. Various methods of hydrogen production are known: traditional (for example, electrolysis of water and conversion of hydrocarbons) and combined thermochemical methods. The method of obtaining hydrogen by electrolysis of aqueous solutions of hydroxides of alkali metals is the most energy-intensive one, though considered one of the most promising in the European Union. The purpose of this work is the scientific substantiation of the electrochemical production of hydrogen with reduced energy consumption in a combined, open mass transfer system, the composition of the catholyte, and the concentration of its components to ensure the conditions for reducing the energy consumption for the hydrogen release.

To reduce the energy consumption for hydrogen production in the combined electrochemical method, the anode on which oxygen is released in an acidic medium is replaced by a soluble anode with an equilibrium potential more negative than the potential of oxygen release. Such a soluble anode can be iron with a standard potential of -0.44 V. At the same time, the decomposition voltage in this system was equal to 0.41 V compared to 1.23 V in the case of traditional electrolysis of water. The overvoltage of iron dissolution in a chloride medium is several tens of millivolts, and the potential difference between the anode and the cathode when hydrogen is released can be much smaller than during the usual decomposition of water. The Pourbaix diagram and possible products of the electrochemical dissolution of iron were considered.

The process of iron dissolution was studied in a $1 \text{ mol} \cdot \text{L}^{-1}$ solution based on Na_2SO_4 in the addition of NaCl with concentrations up to about $50 \text{ g} \cdot \text{L}^{-1}$. The cathode was platinum, the anode was the St3 iron electrode. According to the data of cyclic voltammetry, it was established that the maximum current density of iron dissolution increases with an increase in the concentration of sodium chloride in the electrolyte. The dynamics of changes in the potential values of the onset passivation (the Flade potential) and complete passivation (activation potential) depending on the concentration of sodium chloride were also established.

It is established that with an increase in NaCl concentration up to $50 \text{ g} \cdot \text{L}^{-1}$, the Flade potential is shifted towards anode by 0.8 V. At average chlorine concentrations of $\sim 10 \text{ g} \cdot \text{L}^{-1}$, intense current fluctuations are observed instead of passivation. The maximum of dissolution iron anode current density $700 \text{ mA} \cdot \text{cm}^{-2}$ was achieved in the Na_2SO_4 solution with the addition of NaCl in the amount of $50 \text{ g} \cdot \text{L}^{-1}$.

Keywords: hydrogen, electrolysis, cathode, soluble iron anode, hydrogen energy.

1. Introduction

Hydrogen is commercially produced from fossil hydrocarbons or by electrolysis of water. These processes occur at high temperatures and/or at high voltages, which requires significant energy costs. As a result, the obtained hydrogen is expensive, which limits its use as an environmentally friendly fuel.

We proposed a method for producing hydrogen in a combined electrochemical system open in terms of mass transfer, in which the anode process of oxygen release is replaced by the process of dissolving an anode made of a metal with an electronegative potential [1]. The principal technological scheme of this method of hydrogen production is presented in the first part of the article [2].

The requirement for the anode material is that this metal should be widespread, available, and cheap. Moreover, it may be a waste of another production. The anode dissolution products should not form a solid phase in the electrolyzer, which would change the hydrodynamics of the circulating electrolyte. They should be used either as an independent product or be a raw material for another production. Such a materi-

al can be iron, the most common structural material, the waste of which in the form of chips or scrap is widely distributed and available.

Note that the cathodic process of hydrogen release and the anodic process of iron dissolution are inextricably linked by the electric current flowing through the system and should be considered simultaneously.

The catholyte and the anolyte can have different composition and concentration. To prevent the mixing of solutions, the cathode and anode spaces are separated by a membrane [2]. In this case, the electrode processes in the anode and cathode space can be considered separately.

The thermodynamics and kinetics of the processes occurring in the system can be illustrated by the diagram shown in Fig. 1. The kinetics of hydrogen release is described by the polarization curve (1) - the dependence of the current density (reaction rate) on the electrode potential.

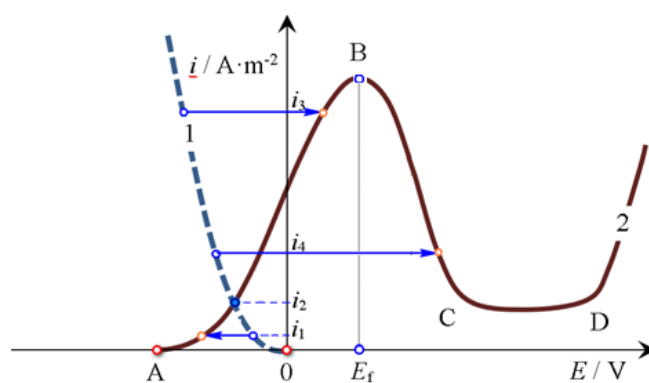


Figure 1 – Schematic position of the polarization curves of hydrogen release (1) and iron anode dissolution (2) in a combined electrochemical system open for mass transfer

The first part of this work [2] substantiated the composition of the electrolyte, the pH of which was ~ 1.5 . By the Nernst equation, the dependence of the hydrogen electrode potential on pH is determined by the equation:

$$E = E_0 - 0.058(V) \cdot pH = 0V - 0.087V \cdot 1.5 = -0.087V, \quad (1)$$

that is, the equilibrium potential of hydrogen is slightly different from zero. When current is applied, it shifts toward negative potentials (cathodic process). The equilibrium potential of iron is more negative. The standard potential of iron is -0.44 V and shifts to the negative side when the concentration (activity) of iron ions decreases. Since the optimal concentration of iron ions during the long-term operation of the system has not yet been determined by us, it can be assumed that it will be in the range $C = 0.01 \div 1.00 \text{ mol} \cdot \text{L}^{-1}$. The equilibrium potential according to the Nernst equation is equal to $-(0.44 \div 0.5)$ V at different concentrations of ions (point A, Fig. 1).

Process decomposition stress is calculated as:

$$U_0 = E_+^0 - E_-^0 = -0.087 - (-0.5 B) B = 0.41 B, \quad (2)$$

where: E_+ is the potential of the more positive electrode; E_- is the potential of the more negative electrode.

This voltage is much less than the water decomposition voltage of 1.23 V.

Noteworthy is zone 3 between polarization curves 1 and 2. Spontaneous dissolution of iron is possible in this zone without current being supplied from outside:



However, the speed of this process is relatively low, and to increase it, it is necessary to supply current from any external source.

At the same time, the polarity of the potential difference changes to the opposite (indicated by arrows in Fig. 1). Anode changes the sign from minus to plus and cathode - to minus. After this, the system stops working as a current source and starts working as an electrolyzer. At certain potentials called the Flade potentials or the onset passivation potentials E_p (point B, Fig. 1), the electrode begins to be covered by the corresponding oxides. As a result, the current decreases sharply.

At the same time, the hydrogen release current flowing through the cathode will decrease, since the same amount of electricity necessarily passes through the electrodes. At point C, the current reaches a minimum. The potential of this point is marked as E_a , and from this value the activation of dissolution begins with further cathode polarization.

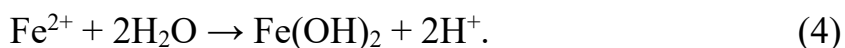
Further polarization of the electrodes will lead to the achievement of point D, the beginning of oxygen release, which is categorically unacceptable in this system. In this case, the purpose of this study is to determine the conditions under which the anodic process proceeds at the maximum possible rate (current density) and the limiting potential of the passivation start (Flade potential, point B). To prevent the passivation of iron, chloride ions are usually introduced into the solution [3].

During the electrochemical dissolution of the iron anode, the reaction proceeds



In the concentration range 10^{-6} - $1 \text{ mol} \cdot \text{L}^{-1}$ and at a $\text{pH} < 6.5$, by Pourbaix diagram for the Fe-H₂O system iron in the solution is in the divalent ions form (Fig. 2 [4]).

In a neutral medium, ferrous iron hydrolyzes with the formation of an insoluble precipitate of iron hydroxide



That is, to prevent the formation of sediment in the electrolyzer, the pH of the anolyte should be less than 6.

With an increase in pH (in the area $6.5 < \text{pH} < 9$), the solubility of $\text{Fe}(\text{OH})_2$ sharply decreases from 1 to $10^{-6} \text{ mol} \cdot \text{L}^{-1}$ (see the Pourbaix diagram for the Fe-H₂O

system, Fig. 2 [4]), which leads to precipitation of iron hydroxide crystals. The sizes of the formed crystals depend on the pH. In neutral and weakly alkaline solutions, when the solubility of the hydroxide is high, relatively large crystals are formed. At $\text{pH} > 9$, a suspension with finely dispersed crystals is formed. The occurrence of this process in the electrolyzer is undesirable, therefore it is advisable to acidify the anolyte to a pH of about 3. As can be seen from the Pourbaix diagram, under these conditions, ferrous iron ions are not hydrolyzed. Thus, the optimal pH value is in the range of 3 ÷ 6.

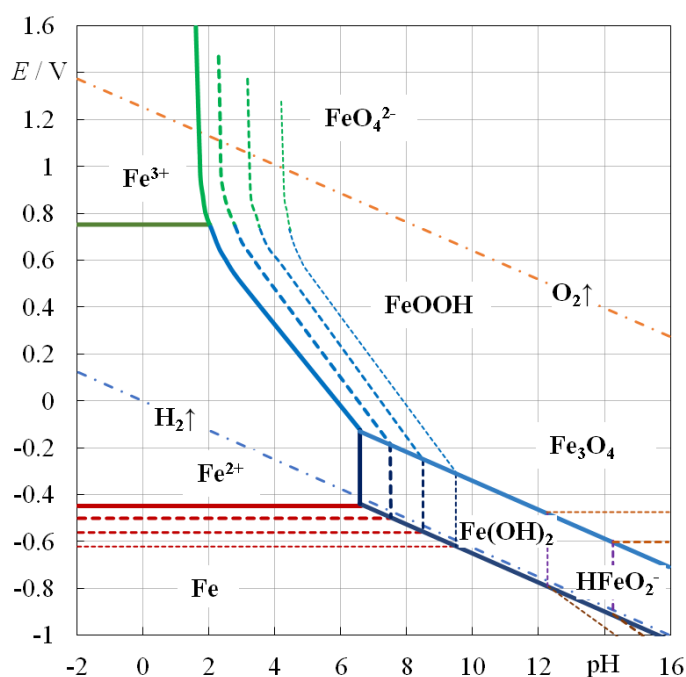


Figure 2 – Pourbaix diagram for the Fe-H₂O system. The lines correspond to the equilibrium of reactions (3), (4) at different concentrations of the corresponding Fe^{2+} : 1 (solid line), 10^{-2} , 10^{-4} , 10^{-6} mol·L⁻¹ (thin dotted line). In contrast to [4], the lines of the equilibrium potential for the release of hydrogen and oxygen are given for a pressure of 0.1 MPa

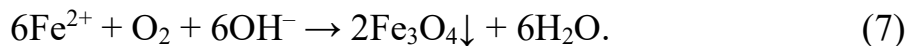
As noted in the first part of the article [2], the electrolyte in the cathode circulation circuit is a mixture of 1M NaCl and HCl at a pH of about 1.5. Such an electrolyte is quite electrically conductive, relatively non-aggressive, and in the case of diffusion through the membrane into the anode space does not significantly change the pH anolyte.

The latter is especially important since the iron ions accumulated in the anolyte should not be hydrolyzed in the electrolyzer with the formation of a solid phase. On the other hand, to remove excess iron, its oxidation with air oxygen in a saturator at a pH of at least 3 is assumed, followed by the $\text{Fe}(\text{OH})_3$ hydroxide precipitation in a sediment tank and separation in filter presses [1]. That is, the anolyte pH must be maintained in the range from 3 to 6. It is difficult to assume the participation of anodic reaction products in cathodic processes, since the electrodes are separated by a membrane, and the potential for reducing iron ions on platinum in an acidic medium is not reached.

The solution from the anode space of the electrolyzer enters the saturator, where divalent irons are oxidized by atmospheric oxygen to the trivalent state. Ferric ions at pH value 3 to 6 are hydrolyzed, forming a water-insoluble solid phase [5]:



or



At these pH values and a relatively low temperature ($< 40 - 50^\circ\text{C}$), as well as the concentration ratio $[\text{OH}^-]/[\text{Fe}^{2+}] < 2$, phase-pure $\gamma\text{-FeOOH}$ is formed [5, 6]. It can be seen from the Pourbaix diagram that in the area of $\text{pH} > 6.5$ at temperatures of about 18°C , it is possible to obtain mixtures of intermediate compounds - Fe(II)-Fe(III) hydroxo salts, as well as nanocrystalline $\alpha\text{-FeOOH}$ [7, 8]. These processes proceed at a high speed, but in our case, they are unsuitable since the return of the electrolyte after the iron separation in the electrolysis process requires its acidification with a significant consumption of expensive acids.

From the saturator, the resulting colloidal solution enters the sediment tank, where the flakes are coarsened, and then to the filter press, in which the solid phase is separated from the solution. The solution is returned to the electrolyzer, and the iron hydroxide can be used for the production of coagulants, or, after heat treatment, go to metallurgical enterprises. The content of iron in the resulting precipitate is close to 65 %, which makes it a valuable raw material. In this case, the use of a soluble iron anode fully meets the above requirements.

The purpose of this work:

- establishing the optimal composition and concentration of the electrolyte of the anode space to ensure the anode dissolution at the maximum possible speed; to create conditions for the iron release in the form of hydroxides;
- determining the passivation onset potential (the Flade potential) in solutions of different compositions.

2. Methods

The research on iron anode dissolution was carried out by the method of cyclic voltammetry in various electrolytes based on a base solution of Na_2SO_4 with a concentration of $1 \text{ mol}\cdot\text{L}^{-1}$. Different amounts of NaCl were added to the base electrolyte to study the effect of chlorine. For each electrolyte composition, four cycles were recorded in the range of potentials $-1.0 - +1.0 \text{ V}$ with a potential sweep speed of $10 \text{ mV}\cdot\text{s}^{-1}$. Polarization of electrode began in the direction of anode. During the experiment, at the same time as the current and potential were recorded on the electrochemical cell, additional parameters were recorded, namely the total voltage between the steel and platinum electrodes. Potentiostat TIONiT-2.08 was used to record potentiodynamic characteristics. The equipment was controlled using the MultiCycle program with the recording of measurement results on a computer.

The research was carried out at a room temperature of 18°C . In the process of electrochemical experiments, the temperature of the solution was controlled. A slight

increase in temperature was observed, approximately by 1 °C per cycle, i.e. within 18÷22 °C.

3. Results and discussion

Based on the registered potentiodynamic characteristics (Fig. 3), the effect of adding NaCl on the dissolution process of the iron anode was analyzed. The obtained cyclic curves allow studying the process of the anode dissolution and passivation, namely, to determine the passivation onset potentials (the Flade potential, E_p) and complete passivation potentials (activation onset potential). The Flade potential is determined by the intersection of the activation and passivation branches of the polarization curve, i.e., point B in Fig. 1. The complete passivation potential corresponds to point C. The process of the onset of activation E_a is determined by the reverse course of the polarization curve. It is close to the complete passivation potential (point C).

In our case, the greatest attention is paid to determining the passivation onset potential, since it corresponds to the maximum value of the current density, and, accordingly, the speed of not only the anode dissolution but also the hydrogen release.

Dependencies of the E_p value and the maximum current density on the concentration of sodium chloride are presented in Fig. 4, 5.

In the absence of chlorine (Fig. 3a), the dissolution peak reaches a maximum in the first cycle at the value of the current density $i_p=i_{max}=39 \text{ mA}\cdot\text{cm}^{-2}$ ($E_p=E_{max}=-0.24 \text{ V}$).

When adding NaCl solution to the basic sodium sulfate solution, an increase in the maximum value of the current density (1 maximum) is observed with increasing concentration (Fig. 3b–e). This dependence is shown in more detail in Fig. 5. At the same time, at low concentrations of chlorine, for example, 2.5 g of NaCl per one liter of 1M Na_2SO_4 solution (Fig. 3b), after the first cycle, the surface passivation increases, and the maximum value of the dissolution current decreases in the potential range from 0.3 V (and more). When the amount of NaCl is $5.0 \text{ g}\cdot\text{L}^{-1}$ Na_2SO_4 solution (Fig. 3c), the value of the first maximum of the current density increases significantly.

Figure 4 shows the change in the maximum current density at points B(i_p) and C(i_a) depending on the Cl ions concentration in the working solution. A similar dependence for the potentials E_p and E_a , which was determined by the differential curves of the change in current density from polarization, is shown in Fig. 5.

Fig. 6 schematically shows the change in the course of the polarization curves of cathodic (1) and anodic processes (2) in different electrolytes. The figure also shows the polarization curve of oxygen release.

The analysis of cathode polarization curves 1_0 and 1_1 was carried out in the first part of this work [2]. In a sodium chloride solution with a concentration of $1 \text{ mol}\cdot\text{L}^{-1}$, the hydrogen release overvoltage at the platinum electrode is significant (about 1 V), and the polarization curve 1_0 is flat. In the NaCl + HCl mixture at a pH of 1.5, the polarization decreases significantly, so curve 1_1 has a smaller angle of inclination.

The course of anode polarization curves strongly depends on sodium chloride content. In a pure Na_2SO_4 solution, the iron anode is quickly passivated, and the max-

imum current density does not exceed $50 \text{ mA} \cdot \text{cm}^{-2}$ (curve 2₀). In the electrolyte composition $\text{Na}_2\text{SO}_4 + \text{NaCl}$ $50 \text{ g} \cdot \text{L}^{-1}$ at a pH of ~ 5.5 , the maximum current density increases 30 times and reaches $700 \text{ mA} \cdot \text{cm}^{-2}$ (curve 2₁). This provides a fundamental opportunity to significantly intensify the process of hydrogen production, subject to additional research and the use of an appropriate catalyst.

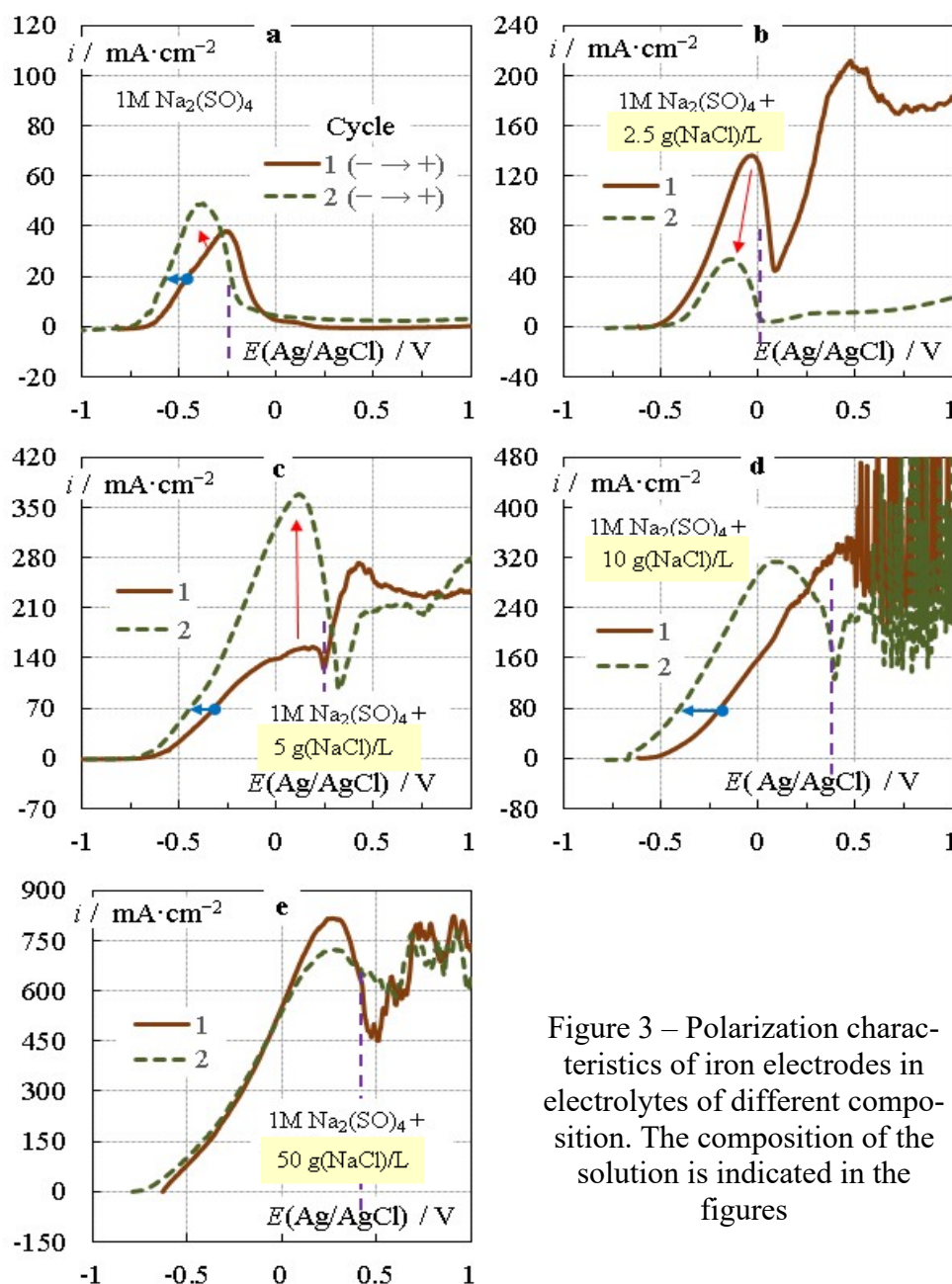


Figure 3 – Polarization characteristics of iron electrodes in electrolytes of different composition. The composition of the solution is indicated in the figures

In classical electrolysis of water, the breakdown voltage is 1.23 V and does not depend on the composition and concentration of the electrolyte and the material of the electrodes. At the current densities used in industry, the voltage during electrolysis increases due to overcoming polarization and ohmic resistances. At operating values of the current density of $150 \div 200 \text{ mA} \cdot \text{cm}^{-2}$, the voltage is in the range of 1.8–2.2 V, depending on the design of the electrolyzer, the catalyst material, and the composition of the electrolyte.

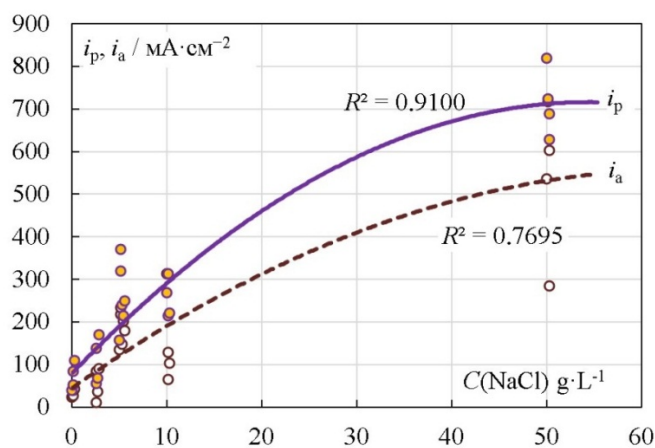


Figure 4 – Dependence of the first maximum of the current density (i_p) (at the passivation onset potential) and i_a on the chlorine concentration in the working solution

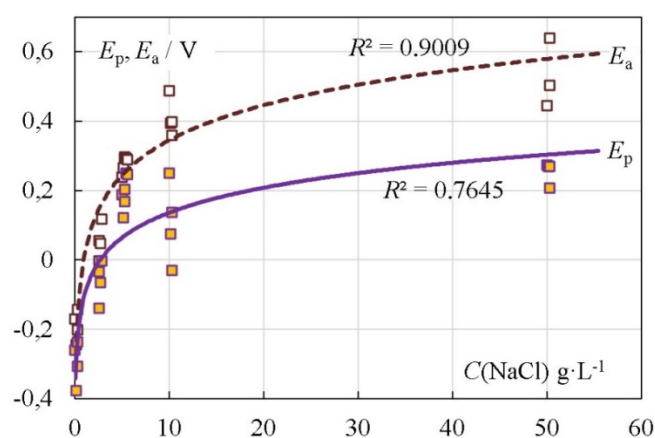
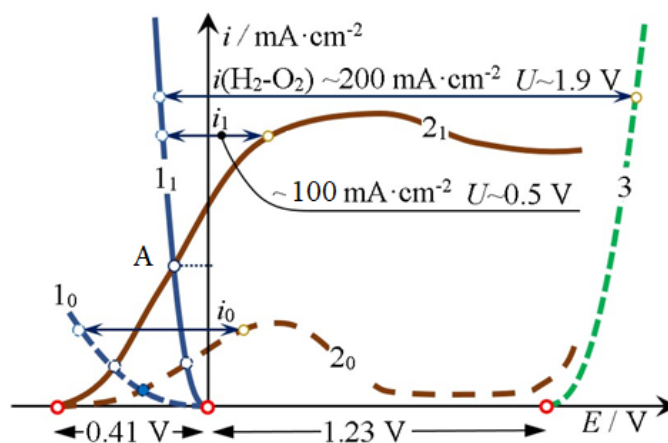


Figure 5 – Dependence of the Flade potential E_p and the potential E_a on the chlorine concentration in the working solution



1₀ – hydrogen release in NaCl solution on a platinum electrode; 1₁ – hydrogen release in a NaCl+HCl solution on a platinum electrode; 2 – dissolution of iron in solutions: 2₀ – 1M Na₂SO₄; 2₁ – 1M Na₂SO₄+5 g·L⁻¹ NaCl; 3 – oxygen release

Figure 6 – Schematic representation of polarization curves of hydrogen release (1) and iron anode dissolution (2) in electrolytes of different compositions and concentration. The green curve is the polarization curve of oxygen release

In our system, spontaneous flow of the process of hydrogen release is possible with the simultaneous iron dissolution. The electromotive force of this process is ~ 0.41 V. As the current density gradually increases, the potential difference decreases (the gap between the anode and cathode curves, Fig. 6) and at point A becomes equal to 0 (short-circuited element). At the same time, the current densities are relatively low. To increase them, it is necessary to set the voltage from the outside. In this case, the system stops working as a chemical source of current and starts working as an electrolyzer. At a current density of $100 \text{ mA} \cdot \text{cm}^{-2}$, the decomposition voltage in our laboratory model was exactly 0.55 V. This is approximately 3–4 times less than in traditional water electrolysis. Thus, our proposed hydrogen production scheme is more energy efficient. It does not require high temperatures, or the use of expensive materials and reagents, but ensures the production of electrolytically pure hydrogen.

4. Conclusions

1. The anodic process of iron dissolution in electrolytes of different compositions and concentrations in a combined electrochemical system open for mass transfer was researched.

2. The dissolution of iron in a neutral solution of 1M Na_2SO_4 is accompanied by anode passivation and oxygen release, which is unacceptable in this system. The addition of chlorine ions to the solution leads to a decrease in the anode passivation.

3. The maximum current density of the iron anode dissolution increases with an increase in the content of chlorine ions and reaches $700 \text{ mA} \cdot \text{cm}^{-2}$ at a concentration of 50 g of NaCl in a liter of 1M Na_2SO_4 solution.

4. The values of the onset potentials (the Flade potential) and complete passivation of the anode was determined depending on the sodium chloride concentration. When the concentration of chlorine ions in the solution increases to $50 \text{ g} \cdot \text{L}^{-1}$, the Flade potential shifts towards anode (that is, to higher potentials) by approximately 0.6–0.8 V.

6. The energy characteristics of electrolysis with hydrogen production in the proposed combined system was analyzed. It is shown that the voltage in this case is 3–4 times lower (0.55 V) than in classical electrolysis of water (1.8–2.2 V). In this case, the possibility of hydrogen production of electrolytic purity with reduced energy consumption is substantiated.

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ОТРИМАННЯ ВОДНЮ В КОМБІНОВАНІЙ ЕЛЕКТРОХІМІЧНІЙ СИСТЕМІ: АНОДНИЙ ПРОЦЕС

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Анотація. Відомі різні способи виробництва водню: традиційні (наприклад, електроліз води і конверсія вуглеводнів) та комбіновані термохімічні методи. Спосіб отримання водню електролізом водних розчинів гідроксидів лужних металів - це найбільш енергоємний спосіб, але розглядається в Європейському Союзі як один з найперспективніших. Метою цієї роботи є наукове обґрунтування електрохімічного отримання водню із зниженими енерговитратами в комбінованій, відкритій за масопереносом системі, складу католіту та концентрації його компонентів для забезпечення умов зменшення енерговитрат на виділення водню.

Для зменшення енерговитрат на виробництво водню при комбінованому електрохімічному способі, анод, на якому виділяється кисень у кислому середовищі, замінюють на розчинний анод з рівноважним потенціалом більш негативним за потенціал виділення кисню. Таким розчинним анодом може бути залізо із стандартним потенціалом $-0,44$ В. При цьому напруга розкладання в даній системі дорівнює $-0,44$ В проти $1,23$ В при традиційному електролізі води. Перенапруга розчинення заліза в хлоридному середовищі становить кілька десятків мілівольт, а різниця потенціалів анода і катода при виділенні водню може бути значно меншою ніж при звичайному розкладанні води. Було розглянуто діаграму Пурбе та можливі продукти електрохімічного розчинення заліза.

Процес розчинення заліза досліджували при циклуванні у розчині на основі Na_2SO_4 з концентрацією 1 моль/л з добавками NaCl концентрацією приблизно до 50 г/л. Катод для проведення процесу електролізу – платина, анод – залізний електрод Ст3. За даними циклічної вольтамперометрії встановлено, що максимальна густина струму розчинення заліза зростає із збільшенням концентрації хлориду натрію в електроліті. Також була встановлена динаміка зміни значень потенціалів початку пасивації (потенціал Фладе) і повної пасивації (потенціал активації) в залежності від концентрації хлориду натрію.

Встановлено, що при збільшенні концентрації NaCl до 50 г/л потенціал Фладе зміщується в анодний бік на $0,8$ В. При середніх концентраціях хлору ~ 10 г/л замість зони пасивації спостерігається зона інтенсивних коливань струму. Максимальна густина струму розчинення залізного аноду 700 mA/cm^2 була досягнута в розчині Na_2SO_4 при додаванні NaCl в кількості 50 г/л.

Ключові слова: водень, електроліз, катод, розчинний залізний анод, воднева енергетика.