Selection of electrode materials for membrane less fuel cells based on immiscible liquids

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Abstract. The possibility of using various materials as electrodes for membraneless fuel cells based on immiscible liquids, both from the standpoint of the thermodynamic possibility of the process and from the point of view of their corrosion resistance, is considered. The corrosion resistance of materials from alloys based on iron, as well as carbon was investigated for use as electrodes in membraneless fuel cells based on immiscible liquids. The possibility of using them instead of electrodes based on ruthenium and platinum in the studied alkaline aqueous ethanol system was shown. It was found that the highest electromotive force in the system occurs when using an activated carbon anode in the top ethanol phase and a carbon steel 10 cathodes in the bottom aqueous phase. The possibility of using activated carbon instead of a ruthenium-coated titanium electrode and platinum-coated titanium electrode, at almost equal exchange currents, will significantly reduce the cost of manufacturing a fuel cell. Thus under certain conditions, this type of fuel cell can compete with membrane fuel cells that have received industrial implementation.

1. Introduction
Fuel cells for direct energy conversion, excluding inefficient combustion processes, have recently been of great interest around the world due to the increased demand for energy and resource-saving processes.

Currently, there are several types of fuel cells: alkaline [1], membrane [2], membraneless co-laminar fuel cells [3], membraneless fuel cells based on immiscible liquids [4, 5].

Now membraneless fuel cells based on immiscible liquids have been of particular interest, since they don’t have membranes, as well as many of the disadvantages inherent in other types of fuel cells. Membraneless fuel cells based on immiscible liquids are simple in design, reliable, and under certain conditions can compete with membrane fuel cells that have received industrial implementation.

Previously, according to the results of the literature search for membraneless fuel cells based on immiscible liquids, we selected an aqueous ethanol system: EtOH + KOH + H₂O [6, 7]. For this system, we experimentally plotting a binodal using the cloud point method proposed in [8]. According to the obtained binodal, the system components concentrations were selected (EtOH – 30% m/m; KOH – 40% m/m; H₂O – 30% m/m), at which the system is layered into two immiscible phases. The composition of the ethanol (top) phase: EtOH – 57% m/m, KOH – 28% m/m, H₂O – 15% m/m, pH 13.1. The composition of the aqueous (bottom phase): EtOH ~ 0% m/m, KOH – 50% m/m, H₂O – 50% m/m, pH 13.2.
It was also revealed that the use of platinum and other noble metals and alloys based on them as catalysts limits their use in most fuel cells due to the high cost [8-14]. However, for alkaline fuel cells, as the cell proposed by us, the use of noble metals and alloys based on them as cathode and anode catalysts is not entirely justified. In an alkaline electrolyte, the energy barrier to the oxygen reaction path is reduced compared to the proton-conducting electrolyte, and even materials with relatively low oxygen adsorption energy can be sufficiently active catalysts for its reduction [15]. This expands the range of possible catalytic systems, including those based on base metals. Since the possibilities of using various materials in alkaline fuel cell have not yet been studied, it is of scientific and practical interest to study new catalysts in the aqueous ethanol system EtOH + KOH + H₂O, which this article is devoted to.

2. Methods
The fuel cell is a rectangular box with walls of 4 mm in thickness made of Plexiglas XT 20070. Internal dimensions are 150 × 100 × 50 mm, and a nominal capacity is 750 mL. Anodes and cathodes were changeable.

The following anodes are used:
- platinum-coated titanium electrode (thin layer of platinum on a perforated titanium net of 150 × 100 mm in dimensions);
- ruthenium-coated titanium electrode (thin layer of ruthenium on a perforated titanium net of 150 × 100 mm in dimensions);
- activated carbon electrode (in a wire basket made of stainless steel 18Cr-9Ni), the basket is 150 × 95 × 30 mm in dimensions, the wire is 0.3 mm in diameter, meshes are 0.5 × 0.5 mm in dimensions, the mass of the loaded carbon is 107 g;
- stainless steel (18Cr-9Ni) wire net electrode of 150 × 100 mm in dimensions, the wire is 0.3 mm in diameter, meshes are 0.5 × 0.5 mm in dimensions.

The following cathodes are used:
- stainless steel (18Cr-9Ni) wire net electrode of 150 × 100 mm in dimensions, the wire is 0.3 mm in diameter, meshes are 0.5 × 0.5 mm in dimensions.
- low carbon steel (0.1% C) wire net electrode of 150 × 100 mm in dimensions, the wire is 0.25 mm in diameter, meshes are 1 × 1 mm in dimensions.

A two-phase system (EtOH – 30% m/m, KOH – 40% m/m, H₂O – 30% m/m) was used in all experiments. The composition of the ethanol (top) phase: EtOH – 57% m/m, KOH – 28% m/m, H₂O – 15% m/m, pH 13.1. The composition of the aqueous (bottom) phase: EtOH ~ 0% m/m, KOH – 50% m/m, H₂O – 50% m/m, pH 13.2.

The volume of the two-phase system was 700 ml, including the bottom phase 250 ml, the top phase 450 ml.

Potassium hydroxide (containing 90.0 % of the main substance) and ethanol (containing 4.0 % of water) were used. The cell was purged with oxygen before the experiment.

Potential and current in the fuel cell were measured by the Elins P-30JM potentiostat-galvanostat (software ver. 3.90).

Potentiodynamic polarization was carried out in a three-electrode cell, in which the metal under study was the working electrode, the saturated silver chloride electrode (SSCE) was the reference electrode, and the platinum electrode was the auxiliary electrode. The potential sweep rate was 0.19 mV/s. A solution of EtOH ~ 0% m/m, KOH – 50% m/m, H₂O – 50% m/m, pH 13.2 was used as an electrolyte for the polarization of low carbon steel 10 and stainless steel of 18Cr-9Ni type. A solution of EtOH – 57% m/m, KOH – 28% m/m, H₂O – 15% m/m, pH 13.1 was used as an electrolyte for the polarization of platinum-coated titanium electrode, ruthenium-coated titanium electrode, and activated carbon electrode in a wire basket of stainless steel of 18Cr-9Ni type. The cathodic and anodic polarization was carried out from the steady-state potential in the system, from the potential of corrosion.

The experiments were carried out at room temperature 20 ÷ 22°C.
3. Results and Discussion

The thermodynamic stability of chemical compounds is determined by the sign and value of the change in the isobaric potential during their formation from elementary substances. The rate of oxidation of a material is not directly related to a change in its isobaric potential but depends on the nature of the medium containing the oxidant. An analysis of the composition of the top and bottom phases of the studied system EtOH + KOH + H₂O shows that they are electrically conductive, and metals are oxidized according to the electrochemical mechanism:

\[ \text{Me} = \text{Me}^{z+} + ze \]

\[ n\text{Ox}+ze = m\text{Red}, \]

where Ox is oxidant, Red is its reduced form.

Each of these reactions proceeds conjugated and obeys its kinetic laws.

The equilibrium that is established when a metal is immersed in a salt solution containing ions of this metal is an equilibrium state between the metal and ion phases. The value of the equilibrium electrode potential depends on the nature of the metal and solvent, the temperature and activity of metal ions in the electrolyte, and can be calculated by the Nernst equation. However, in our case, two electrodes placed in two different electrolytes that do not contain their ions take part in the operation of the fuel cell. Therefore, the electrode potentials of the electrodes are non-equilibrium and can only be determined empirically, since the Nernst equation is not applicable to them.

Analysis of standard electrode potentials of metals in aqueous solutions at 25°C shows that it increases in the next order: Al/Al³⁺ (-1.660 V), Ti/Ti²⁺ (-1.630 V), Ti/Ti³⁺ (-1.210 V), VN⁵⁺ (-0.876 V), Zn/Zn²⁺ (-0.762 B), Cr/Cr³⁺ (-0.740 V), Fe/Fe²⁺ (-0.440 V), Ni/Ni²⁺ (-0.250 V), Fe/Fe³⁺ (-0.037 V), Cu/Cu²⁺ (+0.337 V), Cu/Cu⁺ (+0.521 V), PV/PV⁴⁺ (+0.784 V), Ag/Ag⁺ (+0.799 V), Pd/Pd²⁺ (+0.987 V), Pt/Pt²⁺ (+1.190 V), Au/Au⁺ (+1.500 V), Au/Au³⁺ (+1.690 V) [16].

It can be assumed that the greatest electromotive force occurs between the metal having the most negative standard electrode potential and the metal having the most positive standard electrode potential, i.e. between aluminum and gold. However, this order only indicates the thermodynamic possibility of the oxidation process, does not characterize its rate, and does not allow to unambiguously judge the possibility of using a galvanic pair under certain conditions.

But aluminum in media having pH>12 is unstable and dissolves at high speed with hydrogen depolarization [16]. The oxide film on aluminum dissolves by the reaction:

\[ \text{Al}_2\text{O}_3 + 2 \text{KOH} = 2\text{KAlO}_2 + \text{H}_2\text{O}. \]

Based on this the possibility of using aluminum in the EtOH + KOH + H₂O system was not considered. This does not exclude the possibility of its use in other systems, e.g., in EtOH + K₂CO₃ + H₂O. A preliminary analysis of its chemical resistance showed that aluminum oxidation proceeds less intensely in solutions of potassium carbonate.

Titanium has negative standard potentials Ti/Ti²⁺ (-1.63 V), Ti/Ti³⁺ (-1.21 V), and refers to thermodynamically unstable metals. Due to its ability to self-passivation even in the absence of oxidants, titanium is highly resistant in many media. However, in concentrated solutions of alkalis, titanium is strongly corroded with the formation of soluble titanates [17]. This excludes the possibility of its use in the studied system EtOH + KOH + H₂O.

Vanadium having a standard electrode potential VN⁵⁺ (-0.876 V) was also excluded from consideration due to its deficiency.

Zinc with a standard electrode potential Zn/Zn²⁺ (- 0.762 V) is thermodynamically unstable and can be oxidized with the reduction of both hydrogen cations and oxygen [17]. In alkaline solutions at pH > 12 zinc forms soluble zincates with alkali which leads to a high rate of its destruction. Since in the studied system EtOH + KOH + H₂O pH = 13.2 in the bottom phase, and pH = 13.1 in the top phase, this metal was also not considered.
Chromium is close to zinc in the value of a standard electrode potential \( \text{Cr/Cr}^{3+} (-0.740 \, \text{V}) \). Due to the ease of transition to a passive state, its corrosion resistance far exceeds that of zinc in various media. However, in concentrated alkalis soluble chromites \( \text{Cr(OH)}_6^{3-} \) are formed due to amphotericity of \( \text{Cr}_2\text{O}_3 \) and the metal becomes unstable, which also excludes the possibility of its use in the studied system \( \text{EtOH + KOH + H}_2\text{O} \) [15].

Iron is next to chromium in terms of standard electrode potential. Standard equilibrium electrode potentials of iron are \( \text{Fe/Fe}^{2+} (-0.440 \, \text{V}) \) and \( \text{Fe/Fe}^{3+} (-0.037 \, \text{V}) \); therefore, iron usually dissolves with the formation of \( \text{Fe}^{2+} \) ions. The corrosion resistance of iron in media is determined by its thermodynamic instability, amphotericity, and the ability to passivate and to form films of secondary insoluble products. From a thermodynamic point of view, it can be oxidized in neutral and alkaline media only when oxygen is reduced. At \( pH = 4.0-9.5 \) iron corrosion is practically independent of \( pH \) (Fig. 1) [17, 18].

![Figure 1. Effect of pH on the corrosion rate of iron [17].](image)

When increasing \( pH > 9.5 \) the corrosion rate decreases as a result of the formation of a dense layer of iron hydroxide sparingly soluble in alkali on the metal surface. Due to the amphotericity of iron at \( pH > 14 \) the corrosion rate increases sharply with the formation of soluble hypoferrite compounds [17]. Thus, iron, due to its standard electrode potential and high resistance in concentrated alkalis, can be considered as the electrode material in the \( \text{EtOH + KOH + H}_2\text{O} \) system in \( pH \) range 13.1\text{–}13.2.

Analysis of the equilibrium diagram of \( \text{Fe-H}_2\text{O} \) shows that in the range \( pH = 13 \) to 14 the following iron compounds pass into solution (Fig. 2) [19]:

\[
2\text{Fe}_3\text{O}_4 + \text{H}_2\text{O} = 3\text{Fe}_2\text{O}_3 + 2\text{H}^+ + 2e
\]

\[
\text{Fe} + 4\text{H}_2\text{O} = \text{Fe}_3\text{O}_4 + 8\text{H}^+ + 8e
\]

In practice iron is not used as a structural material due to low strength properties, carbon and alloy steels are more widely used. The increase in carbon content in carbon steels practically does not affect their corrosion rate in neutral and alkaline solutions. In general, the corrosion behavior of carbon steels in these media is similar to the behavior of iron [17]. Therefore, low carbon steels, as well as iron, can be used as an electrode in the \( \text{EtOH + KOH + H}_2\text{O} \) system. Steels alloyed with 17\text{–}20\% \( \text{Cr} \) have good corrosion resistance in aggressive media. Additionally their alloying with 8\text{–}12\% \( \text{Ni} \) facilitates the onset of a passive state, including in media that do not have strong oxidizing properties. Currently, stainless steels of 18\text{Cr}-10\text{Ni} type are the most common type of steels used in various media, including alkali solutions with a concentration up to 50\% and a temperature up to 120°C [17].
Based on this, it can be concluded that materials such as iron, carbon steel, and stainless steels of 18Cr-10Ni type can be used in an alkaline fuel cell as a cathode material, in particular, in the considered bottom phase containing 50% KOH and 50% H₂O.

The choice of anode material for the top phase of the fuel cell was made on the basis of the following considerations. First, the anode material should have the most positive value of the electrode potential relative to the potential of the cathode metal in the bottom phase of the system. Secondly, it should not be corroded. Based on the values of the standard electrode potential, platinum Pt/Pt²⁺ (+1.190 V) fully meets these conditions. The composition of the top phase of the considered fuel cell consists of 57% ethanol, 28% potassium hydroxide, 15% water. According to literature data, platinum is resistant to caustic alkali without oxidants, in ethanol solutions of any concentration up to a boiling point [17, 20]. It can be assumed that in the top phase of the considered fuel cell platinum will also have high chemical resistance.

However, due to its high cost and deficit, and the required large area of the electrode, platinum sheets or meshes may be used. Platinum-coated titanium electrodes and ruthenium-coated titanium electrodes may be used too. Ruthenium is one of the platinum analogs. Ruthenium coatings deposited on the surface of various metals significantly increase their service life. Ruthenium can exist in various valence states, and it has not yet been adequately studied. Therefore, the study of the behavior of this material in the fuel system in question has a certain scientific interest.

Carbon and materials based on it may be other promising materials for an electrode in membraneless fuel cells with immiscible liquids [15]. Carbon materials have high chemical resistance in various media, also, they are commercially available, chemically and electrochemically stable, and have high electrical conductivity comparable to that of metals. In this regard, it was decided to investigate the possibility of using activated carbon instead of platinum electrodes.
Thus as a result of preliminary analysis, the number of materials was determined for use as electrode materials in the considered membraneless fuel cell with immiscible liquids in the $\text{EtOH} + \text{KOH} + \text{H}_2\text{O}$ system.

As the cathode material for the bottom phase, iron, carbon steels, and stainless steels of 18Cr-10Ni type were chosen. As the anode material for the top phase, platinum-coated titanium electrodes, ruthenium-coated titanium electrodes, and activated carbon were chosen.

An analysis of the cathodic and anodic polarization curves of ruthenium-coated titanium electrode in the medium $\text{EtOH} – 57\% \text{ m/m, KOH} – 28\% \text{ m/m, H}_2\text{O} – 15\% \text{ m/m, pH 13.1}$ shows that the electrode material is in the passivity zone, the current at the corrosion potential is small and about 100 $\mu$A (Fig. 3).

![Figure 3. Anodic (red) and cathodic (blue) polarization curves of ruthenium-coated titanium electrode in a solution of $\text{EtOH} – 57\% \text{ m/m, KOH} – 28\% \text{ m/m, H}_2\text{O} – 15\% \text{ m/m, pH 13.1}$.](image)

The behavior of the ruthenium-coated titanium electrode in the potential region -450 mV to -270 mV remains to be investigated, since this type of electrode has not been previously studied, and the obtained results are of scientific interest. It can be assumed that the decrease in the potential region -400 mV to -260 mV observed on the anodic polarization curve is associated with a change in ruthenium valence (its valence can be up to 8) [21]. From the anodic and cathodic polarization curves of the platinum-coated titanium electrode in the medium $\text{EtOH} – 57\% \text{ m/m, KOH} – 28\% \text{ m/m, H}_2\text{O} – 15\% \text{ m/m, pH 13.1}$ (Fig. 4) it can be seen that the corrosion potential is -550 mV. This material is also in the passivity zone and the current at the corrosion potential is slightly more than 100 $\mu$A.

![Figure 4. Anodic (red) and cathodic (blue) polarization curves of](image)
platinum-coated titanium electrode in a solution of EtOH – 57% m/m, KOH – 28% m/m, H₂O – 15% m/m, pH 13.1.

An analysis of the cathodic and anodic polarization curves of the electrode of activated carbon in a wire basket of stainless steel of 18Cr-9Ni type (Fig. 5) shows that the steady-state oxidation potential in this system is -450 mV, and the exchange currents at this potential are about 100 μA.

Figure 5. Anodic (red) and cathodic (blue) polarization curves of electrode of activated carbon in a wire basket of stainless steel of 18Cr-9Ni type in a solution of EtOH – 57% m/m, KOH – 28% m/m, H₂O – 15% m/m, pH 13.1.

The studies of the oxidation of the electrode material in the bottom phase of the system consisting of EtOH ~ 0% m/m, KOH – 50% m/m, H₂O – 50% m/m, pH 13.2 showed the following. The initial potential of steel 10 is -1140 mV, the steel is passivated starting from a potential of -1000 mV, repassivation begins at a potential of 160 mV (Fig. 6).

Figure 6. Anodic polarization curve of steel 10 in a solution of EtOH ~ 0% m/m, KOH – 50% m/m, H₂O – 50% m/m, pH 13.2.
The current at the corrosion potential for steel 10 is about 100 μA (Fig. 7).

Figure 7. Anodic (red) and cathodic (blue) polarization curves of steel 10 in a solution of EtOH ~ 0% m/m, KOH – 50% m/m, H₂O – 50% m/m, pH 13.2.

The corrosion potential of stainless steel of 18Cr-9Ni type in this medium is -300 mV. The steel is in a passivity state up to a potential of 180 mV, at which repassivation begins (Fig. 8). An analysis of the cathodic and anodic polarization curves shows that the current at the corrosion potential, as in previous cases, for stainless steel of 18Cr-9Ni type is about 100 μA (Fig. 9).

Figure 8. Anodic polarization curve of stainless steel of 18Cr-9Ni type in a solution of EtOH ~ 0% m/m, KOH – 50% m/m, H₂O – 50% m/m, pH 13.2.
4. Conclusions
Based on the belief that the greatest potential difference between the anode and cathode is necessary for the greatest electromotive force in the system, an anode of activated carbon in a wire basket of stainless steel of 18Cr-9Ni type in the top ethanol phase, and a cathode of carbon steel 10 in the bottom aqueous phase meet these conditions to the greatest degree.

The possibility of using activated carbon instead of a ruthenium-coated titanium electrode and platinum-coated titanium electrode, at almost equal exchange currents, will significantly reduce the cost of manufacturing a fuel cell. The results have been obtained for the first time. It is necessary to continue research in this direction for choosing new materials of electrodes and finding additional ways to intensify the processes occurring in the fuel cell from the point of view of both thermodynamics and kinetics of the processes themselves.

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