Characterization and use of anionic membranes for alkaline fuel cells

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Abstract

The aim of this work was to check the possibility of using an anionic membrane or a solid polymer electrolyte (SPE) in an alkaline fuel cell. In first, the membrane was electrochemically characterized by measuring ionic conductivity (potentiometric method and electrochemical impedance) and transport numbers were measured (Henderson's and Hittorf's method). The ion-exchange capacity was also determined as well as the water content of the membrane. Then, an alkaline laboratory fuel cell using the SPE was assembled and showed the feasibility of the system. Finally, the performances were widely improved while using an interfacial solution between the electrodes and the membrane. © 2001 Elsevier Science B.V. All rights reserved.

Keywords: Membrane characterization; Alkaline fuel cell; Solid polymer electrolyte; Electrode-membrane interface

1. Introduction

Fuel cells have received an increasing interest for many years. Several kinds of fuel cell exists, but actually the most important is the PEMFC. This fuel cell uses an acidic membrane as electrolyte and gives excellent results, but the price of this membrane is too expensive and limits the development of this kind of cell. So, many systems have been considered to substitute it. In this work, the aim was to check the possibility of using an anionic-exchange membrane or an alkaline solid polymer electrolyte (SPE) in a fuel cell.

SPE can be included in two separate classes [1] for which the main difference is based on the ionic conduction model within the polymer structure.

Ion-solvating polymer or polymer–salt complexes are part of this first class of ionic conducting polymer materials. The polymer contains electronegative heteroatoms (such as oxygen, nitrogen, sulfur or phosphorus) which interact with the cations of the ionic salt by a donor–acceptor link. The principle of ionic conduction within the structure is based on heteroatom–cation interactions, and on the mobility of amorphous polymer chains. SPE has been extensively developed for applications in lithium batteries, particularly by Armand et al. [2,3] in the late 1970s by using polymer based on polyethylene oxide. The laboratory of industrial

electrochemistry (CNAM) developed alkaline SPE by dissolving KOH in PEO [4,5] and then in another copolymer (ethylene oxide and epichlorhydrin). These SPE exhibited interesting ionic conductivities (10^{-3} S cm^{-1}) and had been used in Ni–Zn, Ni–Cd, Ni–MH, Zn–air and MH–air batteries [6–9]. SPE based on epichlorhydrin and lithium salts have often been studied [10,11], particularly in lithium battery applications [12,13].

The other class of SPE developed extensively since 1970 are polyelectrolytes. This kind of polymers are ionomers: ionic sites (type sulfonate or quaternary ammoniums) are grafted on the skeleton of the polymer chain. A mobile counter-ion is associated to each ionic function in order to preserve the polymer electroneutrality. The most important example of polyelectrolyte is the Nafion membrane which is extensively used in numerous industrial processes, and particularly in PEMFC. A new kind of polyelectrolyte was recently developed in the laboratory [14]. The objective of this work was to use the SPE developed in an alkaline fuel cell.

Alkaline fuel cells were particularly used on the NASA space flights throughout the 1960s and the 1970s, and is now used in the space shuttles [25]. Recently, several zero emission vehicles were performed using this kind of fuel cells [26]. However, the problems always encountered are due to the liquid alkaline electrolyte responsible for the carbonation of the system sensitive to CO_2, and for the electrodes flooding. Using a SPE instead of a conventional liquid electrolyte in a fuel cell (like PEMFC) allows for increasing the volumetric energy density, avoiding problems

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of leakage and preventing the gas electrode flooding mainly responsible for the death of the fuel cells.

Thus, it would be interesting to use in an alkaline fuel cell, a cheap anion-exchange membrane with good electrochemical properties and also good mechanical and chemical stability with time. In this work, a particular interest was devoted to the measurements of the membranes ionic conductivity in order to use them in alkaline SPE fuel cells, and to the measurements of the anionic transport number in order to limit the carbonation problems. The general objective of this work was to reach performances as close as possible as the one obtained with a classical alkaline fuel cell or with a PEMFC. However, knowing that the diffusion coefficient of H^+ ions is usually 4 times higher than for OH^- ions, in order to get the same results, it would be necessary to have a concentration of OH^- ions in the alkaline membrane 4 times more important than in the acidic one [16].

2. Experimental

2.1. Studied membranes

Membranes preparation was described in the literature [14]. Two kinds of membranes were characterized and used in alkaline fuel cells

- The first one was only quaternized by diazabicycloctan (Dabco). Its composition was: H55:Dabco (5:1);[1]
- the second one was quaternized both by Dabco and triethylamin (TEA). Its composition was: H55:Dabco:-TEA (5:1:1) (see Footnote 1).

2.2. Characterization

2.2.1. Ion-exchange capacity (IEC)

This method [22] gives the membrane ion-exchange capacity when the counter-ions are OH^-. After dipping in pure water for 1 day, a membrane sample was immersed for 2 days in 100 ml of an aqueous solution (1 M KOH), to convert the membrane into the OH-form. The membrane was then washed several times with distilled water, then equilibrated in this medium to remove traces of KOH solution. The membrane was then immersed for 24 h in 100 ml of 10^{-2} M HCl solution, and the anion-exchange capacity was determined from the decrease in acidity, which was obtained by back titration after neutralization of the membrane. The ion-exchange capacity was determined from the following relation:

$$IEC = \frac{(n_{HCl,0} - n_{HCl,af.\,neut.})}{m}$$

where $n_{HCl,0}$ is the HCl quantity in the initial solution (10^{-2} M, 100 ml); $n_{HCl,\,af.neut.}$ the HCl quantity in the solution after neutralization of the membrane; m the dry membrane mass.

2.2.2. Water content (WC)

The water content was first measured when the membrane was into its Cl-form, and second when it was into its OH-form.

2.2.2.1. Water content of the Cl-form.
After synthesis, the membrane was washed with distilled water and then immersed in this media for 4 h. It was then dried with Joseph paper and weighed: the weight of the humidified membrane m_h was determined. The membrane was then dried at 60°C for 2 h and weighed again: the dry weight of the membrane m_d was obtained.

The water content defines the percentage of absorbed water in the humidified membrane

$$WC = \frac{(m_h - m_d)}{m_h}$$

2.2.2.2. Water content of the OH-form.
The same operative protocol was used, but first an ionic exchange ($Cl^- \rightarrow OH^-$) was performed by placing the membrane for 24 h in distilled water, and for 48 h in a 1 M KOH solution.

2.2.3. Ionic conductivity

2.2.3.1. Potentiometric method.
The experimental cell (Fig. 1) used to measure ionic conductivity and transport numbers of the membrane was composed of two compartments. Each one had two openings to use one platinized platinum electrode and one Hg–HgO reference electrode. Water tightness between each compartment and the membrane was obtained using two gaskets.
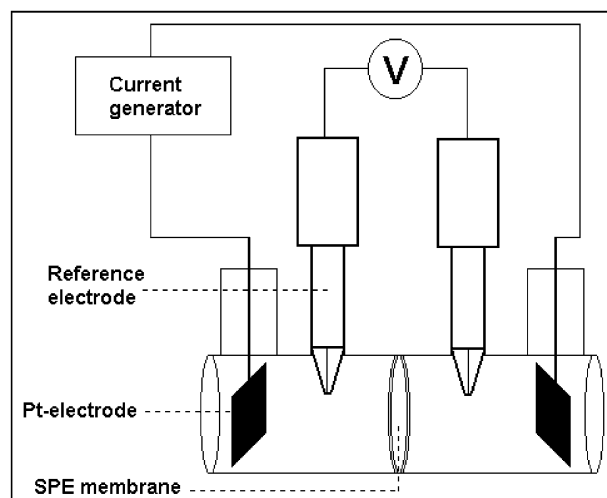

Fig. 1. Schematic representation of the experimental cell.

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[1] The parenthetical numbers indicated the proportion of the different products.

Electrical current can circulate between platinum electrodes and therefore through the membrane. The measurements necessary to determine the ionic conductivity and the transport numbers of the membrane were performed between two reference electrodes.

The membrane ionic conductivity was determined by two successive measurements of potential difference between the reference electrodes; the first without membrane, and the second with the membrane between the two compartments. The KOH solution concentration in the experimental cell was varied between 0.1 and 7.2 M.

Without membrane, the measured resistance was one of the KOH aqueous solution between the two reference electrodes: $R = R_{KOH}$; with a membrane, the total resistance was the sum of the electrolyte resistance (KOH) and of the membrane one: $R = R_{KOH} + R_{Membrane}$

The membrane resistance was determined from the difference of these two resistances. Ionic conductivity, σ (S cm^{-1}) of the membrane was determined owing to the following relation:

$$\sigma = \frac{l}{R_{Membrane}S}$$

where l is the membrane thickness (cm); S is the membrane surface exposed to the electric field (cm^2); $R_{Membrane}$ the membrane resistance (Ω). These measurements gave the ionic conductivity of the membrane in a KOH solution.

2.2.3.2. Electrochemical impedance. Electrochemical impedance completed the previous measurements by giving the intrinsic conductivity of the membrane (i.e. conductivity induced by counter-ions associated to ionic positive sites of the ionomer, and favored by water molecules).

A cell test was used in which the polymer electrolyte is sandwiched between a pair of contacting electrodes. These electrodes were blocking ones, made of platinized platinum. An alternating potential was imposed between the two electrodes and the measurements were carried out at different frequencies (65 kHz–1 Hz).

Since the test cell contained capacitive (and perhaps also inductive) components as well as resistances, an impedance Z rather than a resistance R was obtained. In test cells involving polymeric electrolyte, the inductive components could be ignored [28]. The obtained data were represented in the form of a plot of real against imaginary terms. The real contributions arised from the resistances and the imaginary terms from the capacitances. In the case of polymer electrolyte, the plot obtained experimentally was often compared to the one produced by an equivalent circuit made of a capacitor in series with a capacitor and a resistance in derivation (a semi-circle followed by a spike). The electrolyte resistance was obtained at the intersection point between the spike and the x-axis. The experimental response was relatively different from the theoretical one, but it allowed to determine the intrinsic resistance of the membrane.

2.2.4. Transport number

2.2.4.1. Henderson's method. This static method derived from Henderson's equation determines the transport numbers under diffusion.

The membrane separated the cell in two compartments. Each one contained a KOH solution of different concentration (0.1 and 1 M). A concentration battery of electromotive force $\Delta\varphi$ was then created. The potential difference measured between the reference electrodes was the sum of the electromotive force, $\Delta\varphi$ and of the membrane potential $\Delta\varphi_M$.

In Henderson's equation (case of a binary electrolyte 1:1), the membrane potential is related to anionic and cationic transport numbers. As $(t^+ + t^-) = 1$, anionic and cationic transport numbers were determined from the following relation [1,23]:

$$t^- = \frac{1}{2}\left[1 + \frac{F}{RT}\frac{\Delta\varphi_M}{\ln(a_{KOH,1\,M}/a_{KOH,0.1\,M})}\right]$$

2.2.4.2. Hittorf's method. This dynamic method allows to determine transport numbers under migration and therefore in conditions of an operating fuel cell. A controlled electrolysis was achieved and implied a current flow between the two platinum electrodes. In the test cell, the membrane separated two compartments full of the same KOH solution. Electrochemical reactions produced hydrogen and OH$^-$ ions on Pt-cathode, and oxygen on Pt-anode while consuming hydroxyl ions. OH$^-$ and K$^+$ ions of potash were submitted to an electric field and migrated through the membrane in order to preserve the total electroneutrality of solutions. Molar balance on OH$^-$ ions in each compartment led to transport numbers after a known electricity quantity was passed through the membrane. The cationic transport numbers obtained in the cathodic and the anionic compartment were deduced from the following equations:

$$t^+ = \frac{zF}{it}(c^0v^0 - c^+v^+) \quad \text{and} \quad t^+ = \frac{zF}{it}(c^-v^- - c^0v^0)$$

where z is the charge of the ions OH$^-$, F the Faraday's number, i the applied current during electrolysis, t the electrolysis time, c^0, c^+, c^- the OH$^-$ concentration at the beginning and at the end of the electrolysis in the anodic and in the cathodic compartment, v^0 is the initial volume in each compartment, and v^+ and v^- are the final volume in the anodic and in the cathodic compartment, respectively.

3. Results and discussions

3.1. Chemical stability

Membranes were steady in KOH solutions up to concentrations of 1 M. Over this concentration, a red color could be observed on the membrane meaning that its structure was

Table 1
IEC of the two anionic membranes ((meq. g^{-1}) of dry material)

	H55:Dabco (5:1) (meq. g^{-1})	H55:Dabco:TEA (5:1:1) (meq. g^{-1})
First test	0.53	0.51
Second test	0.63	0.61
Third test	0.54	0.49

Table 2
IEC of the H55:Dabco (5:1) membrane as a function of the ion-exchange time in a 1 M KOH solution ((meq. g^{-1}) of dry material)

Ion-exchange time	2 days	7 days	15 days
H55:Dabco (5:1) (meq. g^{-1})	0.53	0.53	0.57

Table 3
Membranes water content as a function of the counter-ion

	H55:Dabco (5:1)	H55:Dabco:TEA (5:1:1)
Cl-form (%)	13–18	19–22
OH-form (%)	29–41	31–45

deteriorated. At 80°C, this degradation was faster in KOH concentrated solutions.

3.2. Ion-exchange capacity (IEC)

Results of the membranes IEC into their OH-form are reported in Table 1. Values of IEC are in the range of 0.5–0.6 meq. g^{-1} and are lower than the anionic commercial membrane ones [17].

The IEC was then studied as a function of the ion-exchange time in KOH, 1 M solution (2, 7 and 15 days) with the membrane H55:Dabco (5:1). The results presented in Table 2 show that the IEC changes only slightly according to the ion-exchange time.

These results were confirmed by an other experiment, while achieving an ion-exchange by electrodialysis for 8 h in KOH solution: the IEC was again 0.56 meq. g^{-1}.

3.3. Water content (WC)

These measurements allowed to determine the water content of two membranes when Cl^{-} was the counter-ion in the ionomer, then when it was OH^{-} (Table 3).

WC into OH-form are larger than WC into Cl-form. The presence of the OH^{-} ions as counter-ions in the polymer structure seems to make the membrane more absorbent.

3.4. Ionic conductivity

Ionic conductivity achieved at 25°C according to the potentiometric method showed that the membrane

conductivity depended on the concentration of KOH solution in which measurements were done (Fig. 2). Ionic conductivity increased when the KOH concentration increased. It reached a maximum value when the KOH solution concentration was 3.5 M. Values of ionic conductivity obtained for the two membrane types could be considered as identical since the experimental error was relatively important (10–20%). Ionic conductivities were generally superior to 10^{-2} S cm^{-1} for KOH concentrations comprised between 0.5 and 6 M (4×10^{-2} S cm^{-1} was obtained in KOH 3.5 M at 25°C for H55:Dabco:TEA (5:1:1) membrane).

These results were rather encouraging, but remained nevertheless lower than those obtained with the protonic Nafion membrane for which the intrinsic ionic conductivity measured by electrochemical impedance can reach 10^{-1} S cm^{-1} [15]. The intrinsic ionic conductivity of the anionic membrane in water achieved by the same method gives values ranging from 5×10^{-5} to 10^{-4} S cm^{-1}. These values could be explained by the low quantity of OH^{-} ions in the membrane when dipped in water.

Results show that two kinds of ionic conduction must be considered within the membrane. The first one was relative to the experimentation achieved by electrochemical impedance. It concerns the intrinsic ionic conduction of the membrane by the OH^{-} ions which were counter-ions
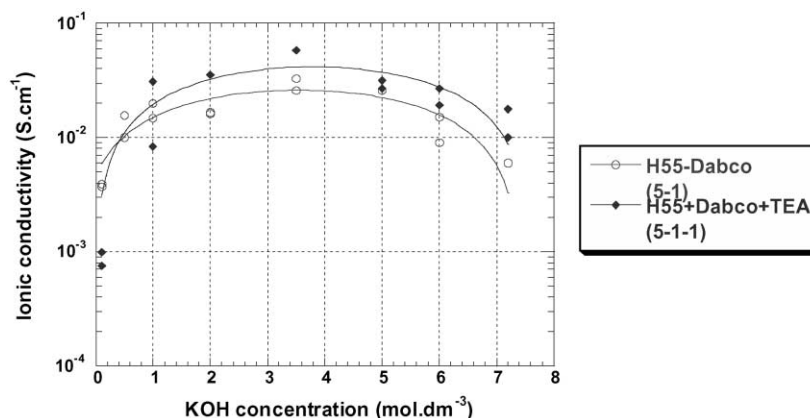

Fig. 2. Ionic conductivity of the two membranes studied as a function of KOH concentration at 25°C.

directly associated to positive ionic sites of the ionomer. The second kind of conduction implies OH^- ions present in the potash phase, absorbed by the membrane. When the KOH concentration was high, the quantity of OH^- ions absorbed by the membrane can be very important. That is why the membrane ionic conductivity in these conditions reached values over $10^{-2}\,S\,cm^{-1}$. A model of ionic conduction taking into account these two kinds of ionic conduction was already proposed by Zabolotsky et al. [18] and Zabolotsky and Nikonenko [19]. It was recently used by Gavach and coworkers [20,21] to give account of this phenomenon.

3.5. Transport numbers

3.5.1. Henderson's method

Henderson's transport numbers were measured under concentration gradient of KOH solutions (0.1 and 1 M). The values are reported in Table 4.

Transport numbers under diffusion obtained for the two membranes were of about 0.95. Then one can suppose that ionic conduction within the membrane is essentially of anionic type. But these results does not give any clue to select the best membrane.

3.5.2. Hittorf's method

Anionic transport numbers measured under potential gradient are reported in Table 5.

Anionic transport numbers were measured for the two membranes as a function of the current density and the KOH solution concentration. All the values were in the range of 0.99–1. The nature of the membrane, the concentration of the solution and the current density cannot change anything

to these results although it was known and observed [20] that the transport numbers decreased with increasing concentration. Perhaps the current density was not high enough to observe this phenomenon. However, these results were better than those obtained with Henderson's method perhaps because the Hittorf's method was more accurate. These results were encouraging because with such a selectivity, the membrane would not get carbonated. The anionic transport number measured by Hittorf's method for an alkaline gel was close to 0.6.

4. Application

Electrochemical characterization of the two membranes studied led to encouraging and satisfactory results for application in an alkaline fuel cell (AFC).

4.1. AFCs using anionic membranes

Gas diffusion electrodes used in the fuel cells were prepared in-house. They were used as anode for electrochemical oxidation of hydrogen and as cathode for electrochemical reduction of oxygen. They were made up of a nickel foam current collector (Nitech). A diffusion layer (carbon black Vulcan XC72 + PTFE) was added to the support by a roller pressing process. An active layer of PTFE, dissolved polymer electrolyte and platinized carbon (E-Tek) was prepared directly by spray on the diffusion layer.

Polarization curves obtained with these electrodes in aqueous KOH 7.2 M electrolyte were slightly better than those obtained with commercial electrodes having Pt-quantity 4 times higher (0.13 versus 0.50 mg Pt cm^{-2} for commercial electrodes [24]).

The electrode–membrane–electrode assembly was achieved while roller pressing the membrane directly between the two gas diffusion electrodes. There was no interfacial solution between the electrodes and the membrane.

These polarization curves (Fig. 3) showed that the AFC using the H55:Dabco:TEA (5:1:1) membrane was less polarized than the other. The membranes thickness being of 150 μm, it was possible that the resistance of the H55:Dabco:TEA (5:1:1) membrane could be lower than the one of the H55:Dabco (5:1) membrane. But this purpose was not clearly showed during ionic conductivity measurements. The experimental error could also explain the differences between the two polarization curves.

Nevertheless, these first results showing the system feasibility were rather interesting, even though current densities only reached 60 mA cm^{-2}. The maximum power density developed by the fuel cell was 20 mW cm^{-2} with the H55:Dabco:TEA (5:1:1) membrane. However, these experiments were achieved at ambient temperature, under atmospheric pressure, without optimization of gas diffusion electrodes and moreover without interfacial solution. By

Table 4
Henderson's transport numbers for the two membranes studied

Membrane	H55:Dabco (5:1)	H55:Dabco:TEA (5:1:1)
Test 1	0.93	0.95
Test 2	0.94	0.95
Test 3	0.98	0.95
Test 4	0.93	0.96

Table 5
Transport numbers obtained for the two membranes studied with the Hittorf's method as a function of the KOH concentration and of the current density

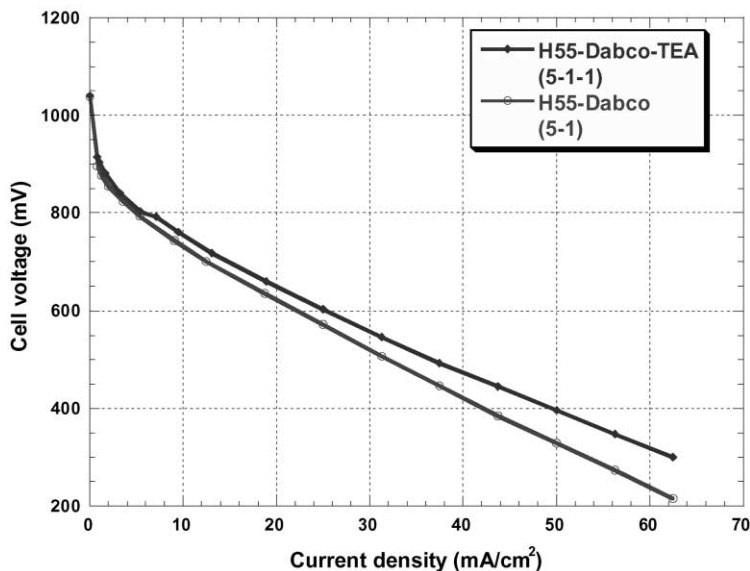| Solutions	Current density (mA cm^{-2})	H55:Dabco (5:1) (t^-)	H55:Dabco:TEA (5:1:1) (t^-)
10^{-2} M KOH	20	–	0.99
10^{-1} M KOH	20	0.99	0.99
	40	0.99	1
	60	–	1
	100	1	–
1 M KOH	60	1	–

Fig. 3. Polarization curves of AFCs using the studied membranes. $P(O_2) = P(H_2) = 1$ atm, $T = 25°C$

using an interfacial solution, one should increase active surfaces and the quantity of OH^- ions and water molecules at the interface of electrode–membrane This could contribute to decrease the interfacial resistance. A way of improvement of the interface is proposed and described in the following paragraph.

4.2. Use of an interfacial solution between the electrodes and the membrane

The fuel cell was assembled with the same gas diffusion electrodes (0.13 mg Pt cm^{-2}), and the H55:Dabco:TEA (5:1:1) membrane. An interfacial solution based on polyacrylic acid and KOH 1 M was used between the electrodes and the membrane. It made up the ionic continuity at the interface electrode–membrane.

This experiment was aimed at decreasing the interfacial resistance and at improving the SPE fuel cell performances. The electrochemical system was tested in the same conditions (25°C, under atmospheric pressure of H_2 and O_2). The polarization curve obtained is presented in Fig. 4, and is compared first to the one obtained without interfacial solution, and second to a Union Carbide fuel cell [27] using an alkaline liquid electrolyte (KOH).
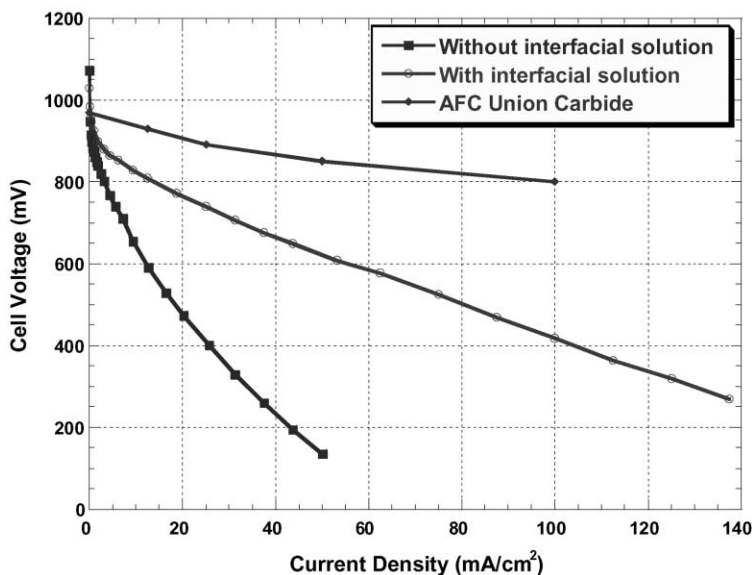

Fig. 4. Influence of the interfacial solution at the electrode–membrane interface on the polarization curves of an AFC using a H55:Dabco:TEA (5:1:1) membrane, $P(O_2) = P(H_2) = 1$ atm, $T = 25°C$. Polarization curve of Union Carbide fuel cell: $P(O_2) = P(H_2) = 1$ atm, $T = 60°C$ [27].
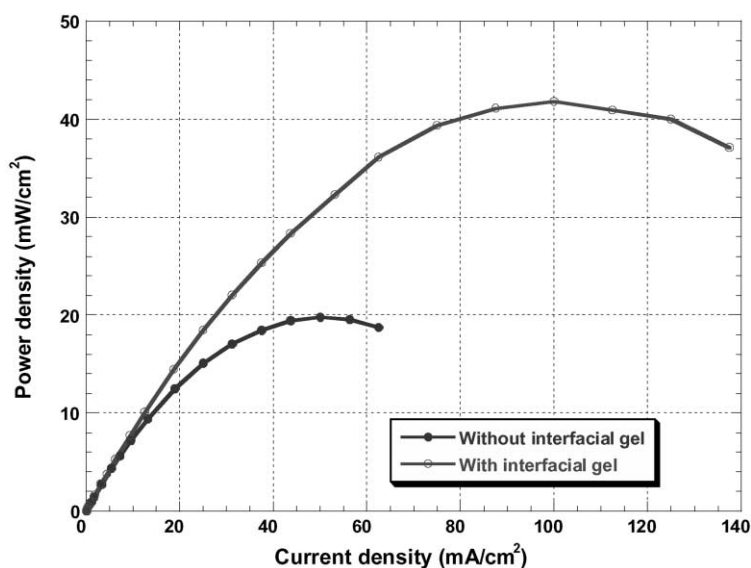
Fig. 5. Influence of the interfacial solution at the electrode–membrane interface on the power density of an AFC using the SPE membrane H55:Dabco:TEA (5:1:1), $P(O_2) = P(H_2) = 1$ atm, $T = 25°C$.

In the same way, Fig. 5 compares the power density obtained from these polarization curves and shows the role of an interfacial gel at the electrode–membrane interface.

The use of an interfacial solution between the electrodes and the membrane improved directly the performances of the SPE AFC. Current densities of $140\,\text{mA cm}^{-2}$ were obtained, and the maximum power density delivered by the fuel cell ($43\,\text{mW cm}^{-2}$) was twice higher than the one obtained without interfacial solution. However, the polarization curves of the SPE fuel cell was worse than the one obtained in alkaline liquid electrolyte. These results could be explained first by the electrolyte ionic conductivity (the ionic conductivity reaches $0.6\,\text{S cm}^{-1}$ for the KOH electrolyte and is limited to $10^{-2}\,\text{S cm}^{-1}$ for the SPE at $25°C$). Moreover, the operating temperature is about $60°C$ for the Union Carbide fuel cell, and only $25°C$ for the SPE system.

Of course the SPE fuel cells operating with the KOH gel can induce carbonation problems as K^+ are free in this media. But this experiment was preliminary. An interfacial solution based on the modified polymer in which the transport number approximately equal to 1 should limit the carbonation, can be considered in the future.

5. Conclusion

Two anionic-exchange membranes were characterized. They showed good electrochemical properties: ionic conductivity superior to $10^{-2}\,\text{S cm}^{-1}$ in KOH solutions, and anionic transport numbers between 0.95 and 0.99. The ion-exchange capacity into the OH-form was low compared to commercially available membranes and constituted a good way of improvement for the intrinsic properties of the laboratory membranes.

These interesting results allowed us to assemble and characterize a laboratory alkaline fuel cell using a SPE. This study showed first the feasibility of an alkaline fuel cell using a SPE. Then the use of an interfacial gel between the electrodes and the membrane increased the power density to $43\,\text{mW cm}^{-2}$ at $25°C$ under atmospheric pressure of H_2 and O_2. Although, the performances with this system were lower than those obtained with conventional AFC and PEMFC, these last results clearly show the necessity to develop an ionic continuum between the electrolyte and the reaction sites. It was very important that the SPE penetrated in the porous structure of the active layer in order to increase the reaction surfaces. The next objective will be to make an interfacial solution from the modified polymer in order to avoid the carbonation of the fuel cell.

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