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Effect of the catalytic ink preparation method on the performance of high temperature polymer electrolyte membrane fuel cells

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Abstract

Two methods of preparation of the membrane-electrode-assemblies based on polybenzimidazole membranes have been studied for high temperatures PEMFCs. One is called the “colloidal method” (using acetone as solvent), and the other is the “solution method” (using dimethylacetamide as solvent). Physical property studies (SEM micrographs and pore size distribution) and electrochemical analyses in half-cell (Electrochemical Impedance Spectroscopy, Polarization Curves for Oxygen Reduction and Cyclic Voltammetry) were carried out to characterise the structural and electrochemical behaviour of both methods. Finally, a cell performance investigation, using electrodes prepared by both methods was carried out at three different temperatures (125, 150, and 175 °C), in a single PEMFC setup. A better behaviour was obtained for the “solution method” at the two highest temperatures at intermediate current densities, whereas at 125 °C the best results were obtained with the “colloidal method” in all the current densities ranges. A discussion of the behaviours observed with the different characterisation techniques is made.

Keywords: Polybenzimidazole; Catalyst ink; Gas diffusion electrodes; Structural characterisation; Electrochemical characterisation; Cell performance

1. Introduction

The proton exchange membrane fuel cells (PEMFCs) have been considered as a suitable alternative to internal combustion and diesel engines because of their high power density, high-energy conversion efficiency and low emission level. However, one of the major issues limiting the introduction of PEMFCs for their applications is the poisoning of platinum-based anode catalysts by the trace amount of CO, inevitably present in the reformed fuel. In order to ameliorate the problem of CO poisoning and improve the power density of the cell, it would be interesting to work at temperatures above 100 °C [1–4].

Among several parts in the fuel cell, MEA (Membrane Electrode Assembly) is one of the most important components. Nafion®-type perfluorosulfonated polymers have been used for a fuel cell membrane and a catalyst-binding material for electrode layers in MEA. However, the conductivity of Nafion®-type polymers is not high enough to be used for fuel cell operations at high temperature (>90 °C) and atmospheric pressure because they dehydrate under these conditions [5]. This dehydration also results in membrane shrinkage and consequent poor contact and proton conduction between membrane and the electrodes. Therefore, dehydration at elevated temperatures can potentially offset any performance benefit arising from high CO tolerances.

In the emerging family of polybenzimidazoles, it is phosphoric acid that impregnates the polymers and is responsible for their proton transport, so water is not required to keep a high conductivity and the thermal stability is much higher. Among polybenzimidazoles, the most widely used acid-doped polymer is poly[2,2-(*m*-phenylene)-5,5 bibenzimidazole], generically referred to as polybenzimidazole (PBI). In the last few years, several research groups have

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been working on this new membrane [6–10]. However, so far, almost all the efforts in the field of the high temperature PEMFC has been focused upon the membrane, either improving the own membrane or modifying it. Little effort has been carried out in the area of the gas diffusion electrodes, compared with phosphoric acid fuel cells (PAFC) or Nafion®-PEMFC. Only in the article published by Wang et al. [11], attention is paid to the manner of preparation of the gas diffusion electrodes. In the rest of the information available in the literature, either commercial electrodes impregnated with PBI or even non-impregnated [10–14], or home-made electrodes with the same polymer treatment have been used [15,16]. It is expected that optimisation of high temperature gas diffusion electrodes will cause improvements on the cell performance [17].

The aim is to study the effect of the solvent used to prepare the catalyst ink on the electrode performance in High Temperature PEM fuel cells. Two different solvents will be employed, one of them leading to the polymer to be dissolved in the solvent ("solution method") and the other forcing the polymer to form colloids ("colloidal method"). The evaluation of these electrodes will be carried out through single cell performance, electrochemical and structural analysis.

Previous works regarding to this topic were carried out in Nafion® PEMFC [18,19]. In these, the elections of the solvents depended on the dielectric constant ε, from whose value Nafion® can remains in the catalyst ink totally dispersed or can precipitate in a colloid-like form. Polybenzimidazole has been reported to be a very insoluble polymer and it can only interact with a few polar solvents. Among them, the most widely used is *N*,*N*′-dimethylacetamide, but its high boiling point and toxicity does not make it an appropriate solvent to be used for preparing air-brushing electrodes. In sight of this, acetone was proposed as an alternative solvent because of its lower toxicity, boiling point, and mainly, its capacity of dispersing polybenzimidazole as a colloidal-like form when the mixture is immersed in an ultrasonic bath as opposed to other traditional solvents used in air-brushing low temperature electrodes, such as water or 2-propanol [20].

In summary, this work pretends to be a continuation of the work carried out by Shin et al. [18] and Kim et al. [19], but using polybenzimidazole as polymer electrolyte. Similar techniques and methodologies are applied in this work, which, at the same time, it is considered to be a new step in the optimisation challenge of high temperature polymer electrolyte membranes fuel cells systems.

2. Experimental

The way of preparation of the membrane-electrode-assemblies (MEAs) is described below. On top of a 3.2 cm × 3.2 cm carbon supporting layer (Toray graphite paper, TGPH-090, 20% wet-proofed, Etek Inc.), a micro-porous layer, compounded by 1 mg cm^{-2} Ketjen Black 300 carbon (Azko Nobel, UK) and Teflon (40 wt.%) as binder, was deposited by air-brushing using isopropanol as solvent to make this layer hydrophobic and avoid the penetration of catalyst particles into the carbon paper.

Catalyst inks were prepared with Pt/C powder (20% Pt on Vulcan XC-72, Etek Inc.), polybenzimidazole (PBI) solution (5wt.% in *N*,*N*-dimethylacetamide, DMAc) and two different solvents depending on the method used to prepare the ink. In the "colloidal method", the necessary amount of PBI was dripped in acetone, and the mixture was treated in an ultrasonic bath to force the dispersion of the PBI in the acetone. After 1 h, the catalyst powder was added to the mixture and ultrasonic treatment was kept for another hour. The platinum and polymer loadings were 0.5 and 0.7 mg cm^{-2}, respectively. In the "solution method", the procedure is the same but the polymer is dispersed in DMAc, where remains in a soluble state. The inks were sprayed onto the micro-porous layer by air-brushing. Then, the electrodes were dried and sintered at 190 °C for an hour inside an inert ventilated oven. Once this time elapsed, all the electrodes were doped with 2 M phosphoric acid and left to dry up overnight. Assemblies were made with the electrodes and the acid doped membranes (11 M phosphoric acid for more than 2 weeks) by hot pressing at 150 °C, 0.1 t cm^{-2} and 10 min. The MEA preparation procedure is shown in Fig. 1.

The anode and the cathode of the MEA were in contact with high-density graphite blocks, impregnated with phenolic resin, into which were cut gas parallel flow channel. The ridges between the channels provided the electrical contact to the carbon electrodes and the total machined geometrical area of 9 cm^2. Electrical heater were mounted at the rear of the graphite blocks to maintain the desired cell temperature which was controlled through a temperature controller and monitored by thermocouples imbedded in the graphite blocks. Gold-plated metallic bolts were screwed into the blocks to allow electrical contact. Gases were fed to the cell at a flow rate of 0.4 l min^{-1} for hydrogen and 0.6 l min^{-1} for oxygen without any pre-treatment.

Electrochemical characterisation of the electrodes (cyclic voltammetry, polarization curves for oxygen reduction, and electrochemical impedance spectroscopy) was carried out in a half-cell setup shown in Fig. 2. It consisted of a jacketed glass body with a Teflon holder where the working electrode was inserted. The counter electrode was a platinised mesh (50 mm × 50 mm, Alfa Aesar) and the reference electrode was a Hg/Hg_2SO_4 (MMS) electrode immersed in the electrolyte used in the measurement, connected to the cell by a Luggin capillary. The cell was sealed by using a glass lid. The working electrode was pressed to the Teflon holder by screw-cap lid. A platinum o-ring was located at the back of the electrode to provide the electrical contact along with a Pt wire and two Viton® o-rings were used to avoid any damage of the working electrode and seal the head of the holder.

Electrochemical impedance spectroscopy (EIS) was performed at an amplitude of 5 mV in the frequency range of 10 KHz–0.1 Hz and a potential of −0.4 V (0.7 V versus standard hydrogen electrode) with respect to the open

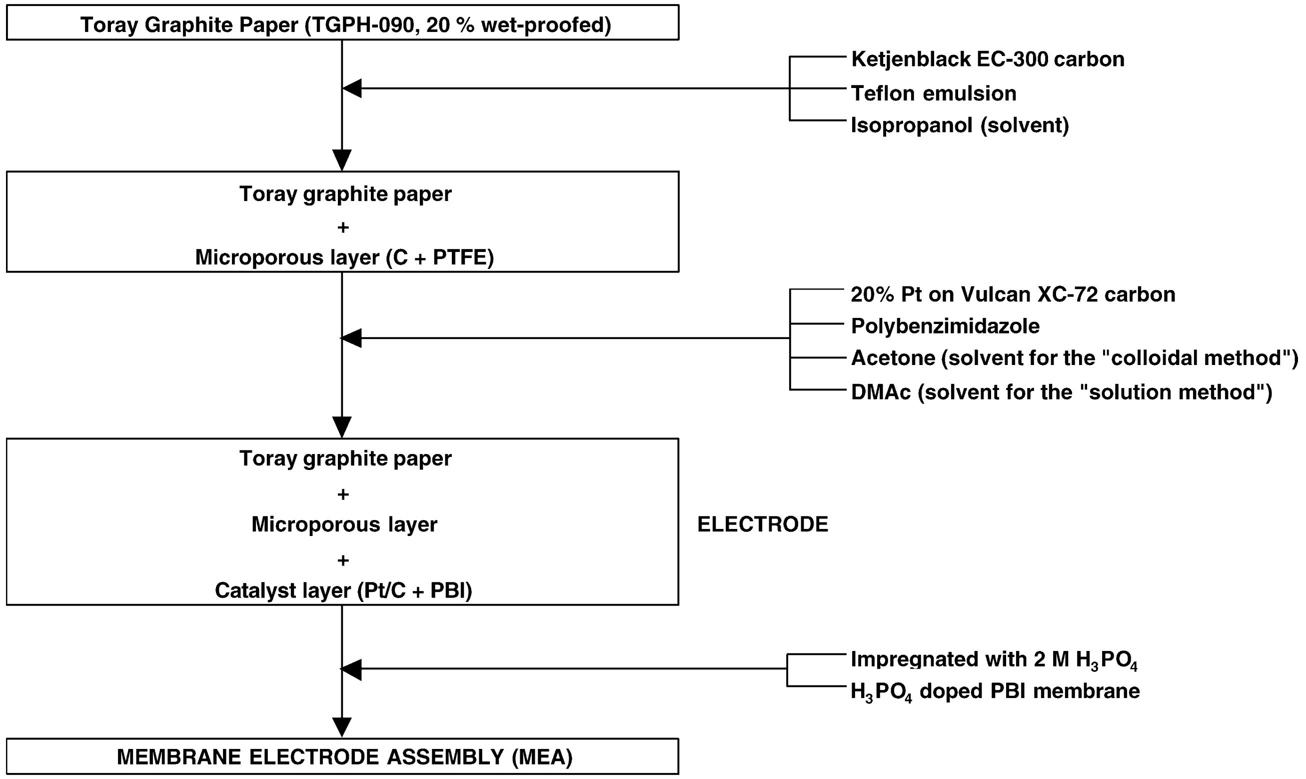


Fig. 1. Scheme of the procedure of the MEA fabrication.

circuit potential of the half-cell which contained 85% *ortho*-phosphoric acid. Oxygen reduction reaction (ORR) studies were carried out in the same system between 1.2 and 0 V versus standard hydrogen electrode (SHE). In the case of cyclic voltammetry (CV), the potential was swept between 0 and 1.4 V versus SHE and the electrolyte was changed for 1 M sulfuric acid, in order to estimate the electrochemical active area of the electrode and the platinum utilisation. Data were

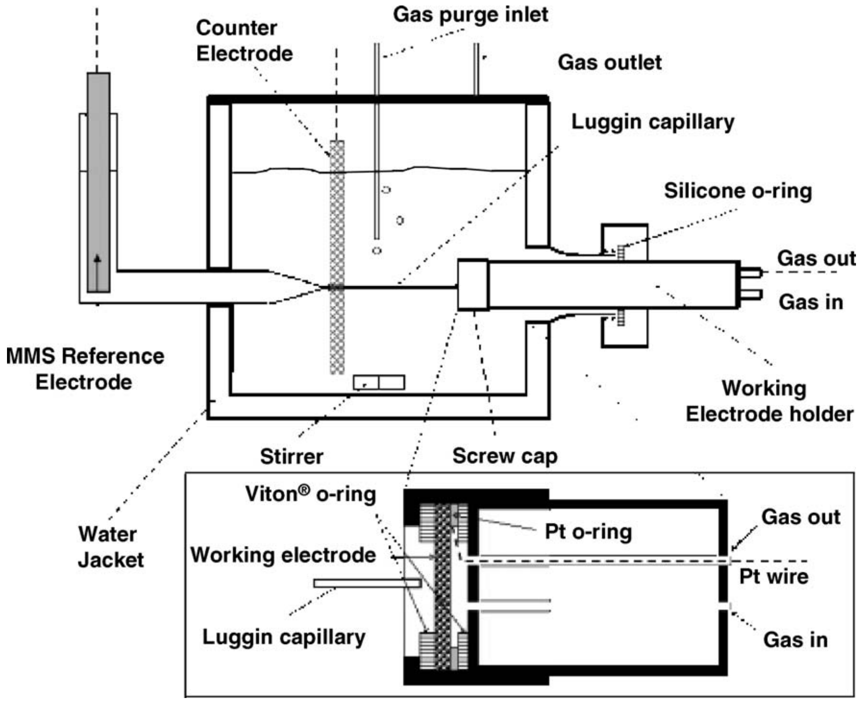


Fig. 2. Schematic diagram of the half-cell used for electrochemical measurements.

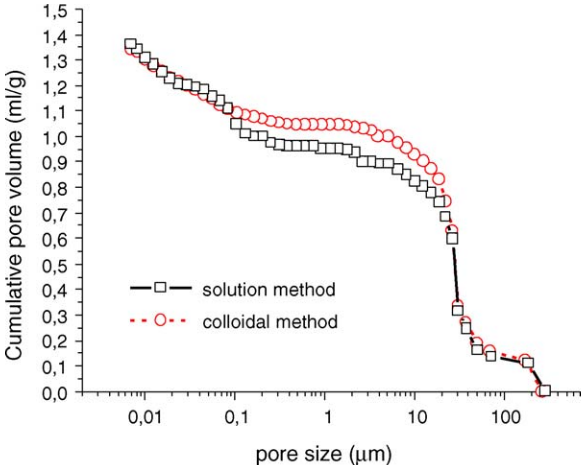


Fig. 3. Cumulative pore volume distribution of the electrodes.

collected using a computer controlled Gill ac potentiostat (ACM Instruments Ltd., UK), loaded with V3 Single Page Stat software.

Pore size distribution was determined by using mercury porosimeter analysis (Micromeritics Autopore III. S.9400) and the porous microstructure was studied by scanning electron microscopy (SEM) with a Philips XL30-CPDX4i.

3. Results and discussion

3.1. Structural analysis

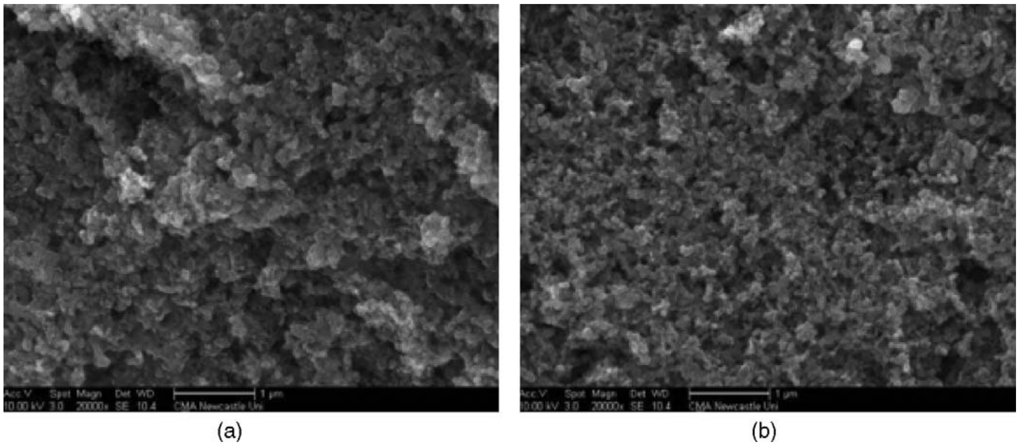
Pore size distribution of the electrodes according to the preparation procedure is showed in Fig. 3. As it can be seen, both curves are similar except in the zone ranged between 0.1 and 20 μm, where the specific volume of the pores is greater in the "colloidal method" electrodes as evaluated from the area. According to the literature [18,19,21–23], this zone corresponds to the secondary pores one, defined as the space existing between the agglomerates (formed by catalyst particle/PBI) formed by the catalyst particles and the ionomer (polymer electrolyte).

In order to have a better understanding of this result, it is worth looking at the SEM micrographs of Fig. 4. From them, it can be discerned that the electrodes prepared by the "solution method" show a more uniform and denser structure when compared to the electrode made by the "colloidal method". Besides, it is noteworthy that the size of the agglomerates is larger in the "colloidal method" electrodes. When PBI is dripped into the acetone, it forms a precipitate dispersed in an ultrasonic bath until becoming a colloid-like solid, which would justify the larger volume of the agglomerates. If PBI is dripped in DMAc, the polymer remains soluble in the ink, and therefore, the formation of big agglomerates is less likely. These differences may explain the greater compactness of the "solution method" electrodes and their lower secondary pores volume, which should impair the mass transfer through them, as it will be confirmed by the polarization curve, showed later on. In Fig. 5, it can be seen a schematic representation of the possible structure of both electrodes. Pores beneath 0.1 μm are called primary pores [18,19,21–23], and correspond to the space between the particles which form the agglomerates. In both cases the specific volume of the primary pores is the same, which implies that the hypothetic structure of the particles which form the agglomerate is independent on the size of these, and therefore, on the solvent used to prepare the catalyst ink.

3.2. Electrochemical analysis

In order to study the effect of the preparation procedure of the catalyst ink on the electrochemical behaviour of the electrodes, three different studies were carried out: cyclic voltammetry (CV), polarization curves for the oxygen reduction reaction (ORR), and electrochemical impedance spectroscopy (EIS).

In order to evaluate the electrochemical surface area (ESA) and platinum utilisation of the electrodes prepared by both methods, CV of the electrodes in 1 M H_2SO_4 were carried



(a)

(b)

Fig. 4. Surface structure of the electrodes for the different ink preparation method: (a) colloidal method and (b) solution method.

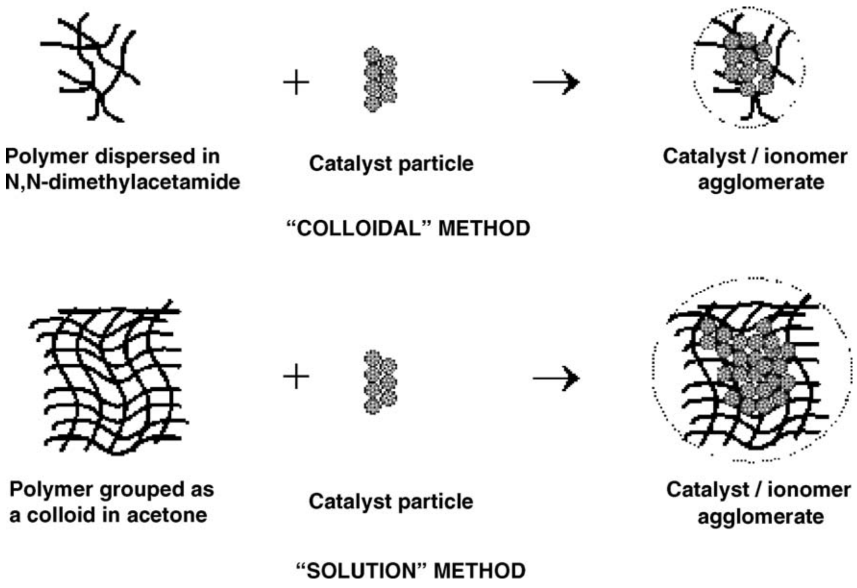


Fig. 5. Representation of the electrodes structure according to the catalyst ink preparation.

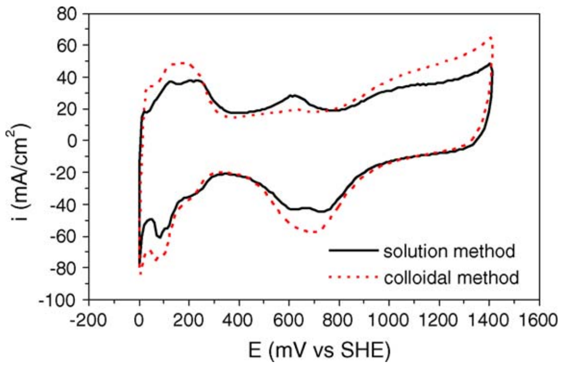


Fig. 6. Cyclic voltammograms for the different ink preparation method.

out. Fig. 6 shows the voltammograms obtained and Table 1 collects the values of the electrochemical surface area and platinum utilisation. The calculation of the ESA (cm^2 of active platinum cm^{-2} electrode) was carried out by Eq. (1):

$$\mathrm{ESA} = \frac{A}{SQ} \tag{1}$$

where A (mV mA cm^{-2}) is the area under the hydrogen desorption peak for the platinum catalyst after subtracting the double layer charge, S is the scanning rate (mV s^{-1}) and Q (mC cm^{-2}) is the charge associated to the oxidation of atomic hydrogen on a smooth platinum surface, 0.22 mC cm^{-2}.

Table 1
Electrodes characteristics depending upon the ink preparation procedure

	Ink preparation method	
	Colloidal method	Solution method
Pt loading (mg Pt cm^{-2})	0.5	0.5
ESA (cm^2 Pt cm^{-2})	641	389
Pt utilisation (%)	20	12.1

In order to calculate the Pt utilisation (cm^2 of active platinum cm^{-2} of available platinum), it is necessary to know the total surface of platinum in the electrode, S_{Pt} (cm^2 of available platinum cm^{-2} electrode). For it, Eq. (2) must be applied:

$$S_{\mathrm{Pt}} = \frac{L_{\mathrm{Pt}} S_{\mathrm{A}}}{C_{\mathrm{Pt}}} \tag{2}$$

where L_{Pt} (mg Pt cm^{-2}) is the platinum loading in the electrode, C_{Pt} is the platinum loading in the catalyst powder (mg Pt mg^{-1} powder) and S_{A} (m^2 Pt g^{-1} catalyst) is the active surface of the catalyst (data obtained from the catalyst supplier).

The ratio between ESA and S_{Pt} is the platinum utilisation:

$$\mathrm{Pt\ utilisation} = \frac{\mathrm{ESA}}{S_{\mathrm{Pt}}} \tag{3}$$

As it can be seen in Table 1, there is a significant increase in the ESA (and therefore, platinum utilisation) when the "colloidal method" is used to prepare the catalyst ink. In the "solution method", since each polymer chain can move freely, it would be more possible for the ionomers to surround the carbon particles, blocking the electron conduction, and decreasing therefore, the platinum utilisation, since an ionomer is an electronic insulator [18].

Fig. 7 shows the quasi steady-state polarization curve for oxygen reduction. The electrode prepared following the "colloidal method" shows a better performance in all the range of potential. This can be explained by the higher ESA and less mass transfer problems of the electrode made by the "colloidal method" because of the larger pore size of the agglomerates formed in this type of electrodes.

Table 2 collects the values corresponding to the Tafel slope (corresponding to the first linear region) and onset potential of

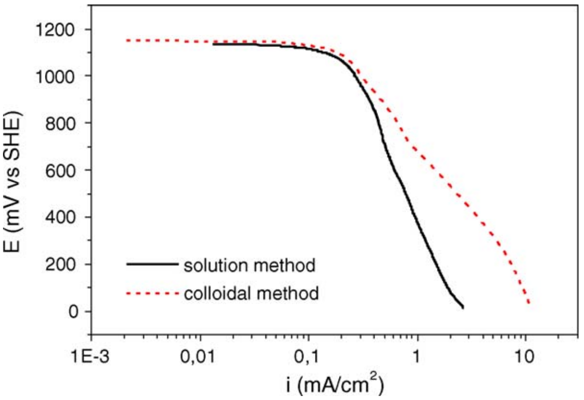


Fig. 7. Linear sweep of oxygen reduction for the different ink preparation method.

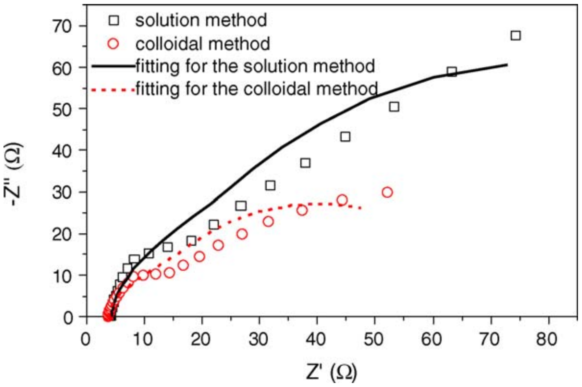


Fig. 8. Nyquist plots of electrodes at 0.7 V for the different catalyst ink preparation method. Lines represent the best fits of the equivalent circuit to data points.

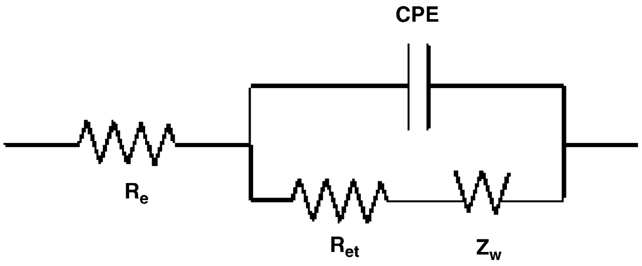


Fig. 9. Equivalent circuit used to represent the electrochemical impedance for the oxygen reduction reaction at Pt/PBI interface.

the electrodes. Values for the Tafel slope in both electrodes are much superior to those reported in literature for the oxygen reduction reaction, corresponding to the Damjanovic model [24,25]. According to this model, the Tafel slope changes from 60 to 120 mV dec^{-1} depending on the applied potential. This variation is owing to the different degree of coverage of the platinum surface [26]. Similar values of the Tafel slope have been reported in *ortho*-phosphoric acid for a smooth and pure platinum surface [27]. Besides, in this reference, it was observed a higher Tafel slope (about 200 mV dec^{-1}) for hydrogen peroxide reduction on Pt. Therefore, it is possible that the oxygen reduction follows the called "peroxide generating pathway" mechanism, which is more likely in the presence of adsorbed "impurities" (as it might be considered the polymer electrolyte). The almost double value of the Tafel slope for the "solution method" could be due to the increase of the path length for the oxygen diffusion, according to the consideration of the thin-film-flooded-agglomerate model [28].

Since the purpose of the study was to compare the activity of both electrodes for the oxygen reduction process, which plays an important role in the efficiency of fuel cell, it can be clearly discerned that the electrode made by the "colloidal method" presents a more intensive activity for all the potential range swept.

Impedance plots for both electrodes are shown in Fig. 8. An equivalent circuit (EC) for this system is the Randles–Ershler EC shown in Fig. 9. R_e, R_{ct} and Z_W represent the uncompensated ohmic resistance, charge transfer resistance, and finite-length Warburg impedance, respectively. CPE represents a constant phase element to account for non-uniform diffusion. Fits of the experimental values to the equivalent circuit are also represented in Fig. 8. Values of charge transfer resistance and Warburg impedance (corresponding to a frequency of zero) obtained from the fitted data are collected in Table 3.

When both curves are compared, two important differences between them can be observed. First of all, the charge transfer arc has got smaller radius in the case of the "colloidal method", and therefore, a smaller R_{ct}, indicative of a faster kinetic process. This behaviour is associated with an extension of the three-phase interface (gas/electrolyte/electrode), reached with the "colloidal method", which is consistent with the results obtained by cyclic voltammetry. Secondly, at low frequency, the electrode made by the "solution method" presents a linear section, with a slope of approximately 1, which corresponds with typical semi-infinite Warburg impedance, caused by mass transport limitation of reactant species. In the case of the electrode made by the "colloidal method", an uncompleted semicircle is visualised and could be associated to finite-length Warburg impedance, due again

Table 2
Tafel slope and onset potential for the different ink preparation method

	Ink preparation method	
	Colloidal method	Solution method
Tafel slope (mV dec^{-1})	221.9	390.7
Onset potential (mV)	1121.3	1102.7

Table 3
Charge transfer resistance and Warburg impedance for equivalent circuit shown in Fig. 7

	Ink preparation method	
	Colloidal method	Solution method
Charge transfer resistance (Ω)	10.7	20.9
Warburg impedance (Ω)	67.5	142.3

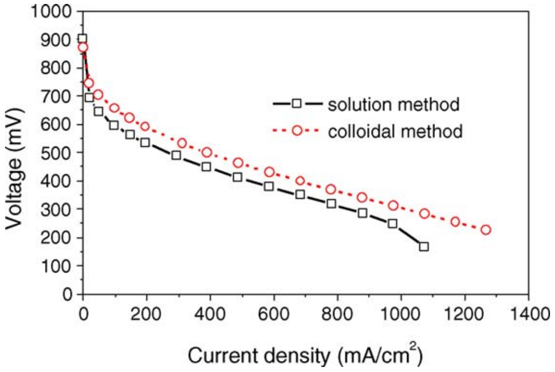


Fig. 10. Effect of the catalyst ink preparation on fuel cell performance. $T = 125\,^\circ C$; $H_2/O_2 = 1/1$ atm. No pre-humidification.

to mass transfer limitations [29]. The higher value of the Warburg impedance in the case of the "solution method" confirms the worse characteristics of these electrodes for mass transfer, because of the different pore size distribution arisen from the preparation method used for each kind of electrodes.

In summary, all the studies carry out in half-cell confirm the better electrochemical properties of the electrodes made by the "colloidal method", and make clear the influence of the electrodes preparation method on their properties. Hence, it should be expected the electrodes made by the "colloidal method" to show a higher performance in the actual fuel cell.

3.3. Cell performance

Different experiments were carried out in an actual fuel cell at three temperatures (125, 150, and 175 °C), with electrodes prepared by both methods in order to study their performances.

Figs. 10–12 show the single cell performance according to the different catalyst ink preparation for the three different temperatures. All the results are the average of five consecutive measurements with a temporal space between them of 60 min, keeping the cell at open circuit potential during that period. As it has been reported [15,16], an increase of the temperature causes an improvement in the cell performance because of the higher conductivity of the membrane and the higher catalytic activity. Thus, for a current density of $1\ A\,cm^{-2}$, for the "colloidal method", a voltage of 305, 362, and 398 mV are reached at 125, 150, and 175 °C, respectively.

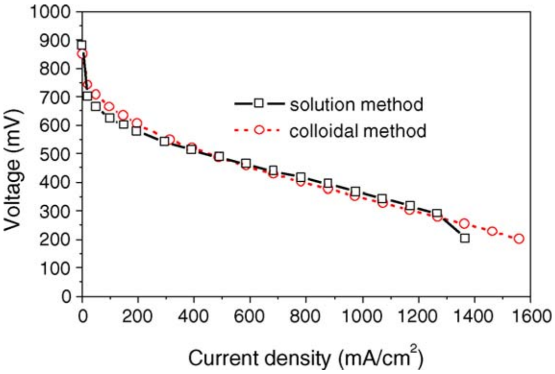


Fig. 11. Effect of the catalyst ink preparation on fuel cell performance. $T = 150\,^\circ C$; $H_2/O_2 = 1/1$ atm. No pre-humidification.

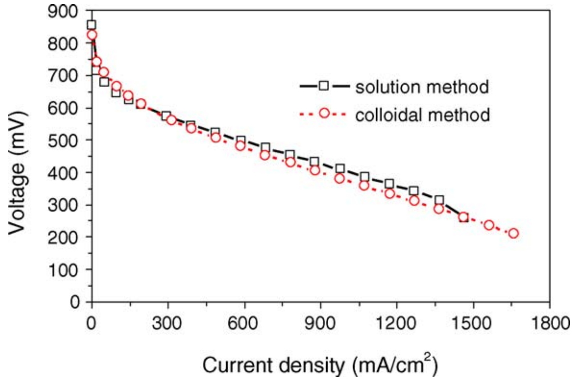


Fig. 12. Effect of the catalyst ink preparation on fuel cell performance. $T = 175\,^\circ C$; $H_2/O_2 = 1/1$ atm. No pre-humidification.

Focusing on the comparison between the performances obtained with the different manner of catalyst ink preparation, it is worth it to divide the cell performance in three regions. The first one, corresponding to low current densities (up to $0.3\ A\,cm^{-2}$), is associated to electrochemical kinetics of oxygen reduction; the second one, corresponding to intermediate current densities (up to 0.9, 1.2, $1.35\ A\,cm^{-2}$ for 125, 150, and 175 °C, respectively), is associated to electric resistance of the fuel cell, and finally, for high current densities, the voltage drops are associated to mass transfer limitation, due to the lack of reactant reaching the catalyst sites [30].

In region one, it can be seen, for the three temperatures, that the performance obtained by the "colloidal method" electrodes is superior to the performance by the "solution method" electrodes. This result was expected because of the better electrochemical properties of the electrodes made by the "colloidal method". At the highest current densities, "colloidal method" electrodes also give better results than "solution method" electrodes, since the large pore size of the electrode made by the "colloidal method" facilitates the diffusion of the reactant towards the catalyst sites. These better diffusion properties showed by the "colloidal method" electrodes were previously demonstrated by the EIS, and predicted with the pore size distribution graph and SEM micrographs.

Table 4 collects the value of the ohmic resistance from the slope of the polarization curve and the corresponding membrane resistance in region two, assuming that this is the main ohmic loss source. It is well known that the membrane conductivity depends on relative humidity, temperature, and doping level [15,16,31]. Considering that for all the couples of experiments, doping level and temperature are the same, the only difference is the relative humidity. As it is known for this system, this variable has got a strong influence on the membrane conductivity [31]. In Fig. 3, it was seen that the electrode made by the "colloidal method" presented an

Table 4
Ohmic resistance and corresponding membrane conductivity obtained from the polarisation curves

	Ink preparation method					
	Colloidal method			Solution method		
	125 °C	150 °C	175 °C	125 °C	150 °C	175 °C
Slope of the polarization curve ($\Omega\,cm^2$)	0.22	0.19	0.17	0.22	0.17	0.16
Corresponding membrane conductivity ($S\,cm^{-1}$)	0.023	0.026	0.029	0.023	0.029	0.031

opener structure than the electrode made by the "solution method". Although this fact is favourable for mass transfer of reactants, it may not be so favourable in the actual fuel cell conditions at intermediate current densities. It must be considered that the only source to keep the membrane humid is the steam produced in the cathode. Since the porosity of the electrodes made by the "solution method" is lower and the particles are more finely dispersed, the steam may find more difficulties to escape from the catalyst layer towards the bipolar plate channels, and therefore, the membrane would have more chances to absorb water, increasing its conductivity. This could not be so noticeable at the lowest temperature because the vapour pressure is smaller, which would cause a drop in the drying process rate of the membrane.

The results obtained in the actual fuel cell show that in the first and third regions of the polarization curves the electrodes prepared by the "colloidal method" have a better performance than those prepared by the "solution method", as it was expected according to the results obtained by electrochemical characterisation. Nevertheless, in the second region, the best results were obtained by the "solution method" electrodes. This could be due to the new elements presents in an actual fuel cell, such as the membrane and the different operational conditions such as working in gas phase at higher temperature, which could give rise to changes in the trends obtained by the half-cell studies.

Furthermore, it must pointed out that these results have been obtained without any optimisation of other parameters, such PBI loading in the catalyst layer, influence of the amount of phosphoric acid in the electrodes, influence of the amount of PTFE in both catalyst and micro-porous layer, etc. Further optimisation of these and other parameters should come accompanied of new improvements in the fuel cell performance and will be presented in following papers.

4. Conclusions

From the work carried out, it has been demonstrated the influence of the manner of catalyst ink preparation on the structure, electrochemical properties (EAS, Tafel slope, charge transfer resistance, and Warburg impedance), and fuel cell performance of electrodes prepared by either "colloidal method" or "solution method". The electrodes made by the "colloidal method" presented an opener structure, due to secondary pore enlargement, than the ones made by the "solution method", facilitating the gas access towards the catalyst sites, and therefore, reducing the mass transfer limitation. Moreover, this larger size of the agglomerates formed by the "colloidal method" reduced the probability of electron insulation caused by the ionomers increasing the gas/electrolyte/electrode interface and, therefore, the platinum utilisation. All these issues predicted a superior behaviour of the electrodes made by the "colloidal method".

Actual fuel cell polarization curve were carried out at three different temperatures in a fuel cell device. The MEA prepared with the "colloidal method" showed the best performance at the lowest temperature as it was expected, according to the studies in the half cell and the structural analyses. However, an inversion of the trend at intermediate current densities occurred at the two highest temperatures. This might be due to the denser structure of the electrode prepared by the "solution method" which would raise the possibilities of the membrane for capturing water from the steam produced at the cathode.

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