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# Mesoporous carbon as Pt support for PEM fuel cell

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ABSTRACT

A mesoporous carbon (MP) supported Pt nanocatalyst was evaluated as anode and cathode catalyst for PEM fuel cell. Kinetics study of the oxygen reduction reaction were characterized by using the rotating disk electrode (RDE) and rotating ring disk electrode (RRDE) techniques in acid media. Membrane electrode assemblies (MEAs) were prepared using Pt supported on MP as anodic and cathodic catalysts and the fuel cell performance evaluated. Polarization and power curves show a similar performance as cathode catalyst when compared to commercial catalyst while there is an 8% improvement when used as anode catalyst.



## 1. Introduction

The advantages of Polymer Exchange Membrane Fuel Cells (PEMFC) as portable and stationary power source have been widely described [1,2]. Nevertheless, PEMFC still struggle to position themselves in the market due that their efficiencies are not as high as the theoretical values and also due to manufacturing cost [3,4]. No catalyst other than Pt has been found yet to present a better catalytic efficiency, for the anode and cathode reactions, which, due to its low natural abundance, possesses a high cost.

In relation with the previous statement, the support used for the metal catalyst nanoparticles plays a major role in the catalytic activity. It has been shown that the nature and morphology of the carbon support and the interaction between support and metal particles influences the nanoparticle dispersion on the support, the metal particles morphology, and the stability of the final catalyst [5–8], thus affecting the catalytic activity of the metal particles [9,10].

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Several ordered mesoporous carbons (OMC) with different synthesis conditions of drying, xerogels, cryogels and aerogels, and/or different casting conditions, hard and soft template, have been used as catalysts support for fuel cells [11,12]. These OMC possesses morphological properties such as surface area, pore sizes, surface groups, etc that have shown to improve the catalytic effect by an adequate metal nanoparticle size, nanoparticles distribution across the surface, anchoring of the nanoparticles to the support, and improvement of the reactant mass transport [13–15].

Recently our group has work in the preparation and characterization of metal nanoparticles catalyst on mesoporous carbon (MC) as fuel cell catalyst [15–18]. Pt catalyst supported on MC was used as cathode catalyst in direct methanol fuel cell (DMFC) showing improved results when compared against Pt supported on carbon Vulcan [18].

In the present work the Pt catalyst supported on MC, prepared by an impregnation and reduction method, was characterized by the rotating disk electrode (RDE) technique for the oxygen reduction reaction (ORR) and was employed as anode catalyst for the $H_2$ oxidation and cathode catalyst for the $O_2$ reduction. Fuel cell measurements were performed on 5 $cm^2$ active area single MEA cell and its performance compared against a commercial cataltyst.

## 2. Experimental

### 2.1. Mesoporous carbon and catalyst supported preparation

Mesoporous carbon support was obtained using the method previously described [19]. Briefly, a precursor was prepared by polymerization of resorcinol (Fluka) and formaldehyde (Cicarelli, 37 wt %). Sodium Acetate (Cicarelli) was used as catalyst and a cationic polyelectrolyte (Poly-diallyldimethylammonium chloride, PDADMAC, Sigma–Aldrich) was used as a structuring agent. The carbonized material at 1000 °C was grinded and passed through a mesh with a pore size of 40 μm. The mesoporous carbon has a surface area of 580 $m^2\ g^{-1}$ as obtained by BET, and the pore size distribution shows a peak at 20 nm [19].

The preparation and characterization of Pt nanoparticles on MC was described elsewhere [17,18]. Briefly, adequate amounts of solutions of the metal precursors $H_2PtCl_6{\cdot}6H_2O$ (Tetrahedron) (Aldrich) was added to a slurry of the carbon support while stirring. The pH was adjusted to 8 with 1 M NaOH (Pro Analysis, Merck) aqueous solution and heated to 80 °C. $NaBH_4$ (granular 98%, Sigma Aldrich) was added in a molar ratio of 3:1 ($NaBH_4$ to metal salt) to the suspension while heating for 2 h, followed by stirring for 12 h at room temperature. The liquid was centrifuged and the solid was separated, washed and dried on a vacuum oven at 60 °C overnight. The obtained Pt/MC had a 35% metal loading with 3.1 mean particle diameter while for Pt/C the values were 38% and 4.2 nm, respectively, as obtained by thermogravimetric analysis (TG), X-ray diffraction (XRD) and transmission electron microscopy TEM [17].

### 2.2. Oxygen reduction analysis by RDE and RRDE

Oxygen reduction experiments were performed employing an Autolab PGSTAT302N potentiostat (Echochemie, Netherlands). For the rotating experiments the Autolab potentiostat was coupled to a Rotating Ring-Disk Electrode (RRDE, Pine Research Inst.; Raleigh, NC). A gold disk electrode (0.196 $cm^2$) with platinum ring electrode (Pine Research Inst.) was used as the working electrode. A Saturated Calomel Electrode (SCE) was used as reference electrode and all potentials were referred to the Reference Hydrogen Electrode (RHE). The counter electrode was a large area rolled platinum wire (0.5 mm in diameter, 30 cm length). A three electrodes electrochemical cell with a jacket was employed, and its temperature was controlled by circulating a thermostatized liquid using a Techne temperature controller. RDE and RRDE measurements were performed at 25 °C in 0.5 M $H_2SO_4$ aqueous media. Linear sweep Voltammograms (LSV) were recorded for the ORR at different rotation speed ($\omega = 100$–2500 rpm). The potential was scanned between 1.0 and 2.0 V (vs. NHE) at 5 mV $s^{-1}$. The calibration of the RRDE was carried out by measuring the disk and ring currents in a 0.005 M $K_3Fe(CN)_6$ + 0.1 M $K_2SO_4$ electrolyte. The disk potential was cycled between 0.2 and 1.0 V at 5 mV $s^{-1}$ while the ring potential was fixed at 1.4 V in order to oxidize the $Fe^{+3}$ generated in the disk. This procedure was repeated at different rotating speed. The RRDE collection efficiency ($N$) was determined from the slope of disc current ($I_D$) vs. ring current ($I_R$) plots [20–22].

### 2.3. MEA preparation and fuel cell testing

MEAs were prepared with Pt/MC on one electrode and commercial Pt/C 40% (E-TEK) on the other electrode. These MEAs were tested using the Pt/MC as anode and cathode catalyst. For comparison an MEA with commercial catalyst on both sides was also prepared. The catalyst was applied onto the membrane by spray method with and active area of 5 $cm^2$. The catalyst suspension was prepared by mixing the catalyst (0.5 mg Pt $cm^{-2}$), with different amounts of Nafion solution (5% vol. Ion Power) and isopropanol. The Proton Exchange Membrane used was Nafion 117 (Ion Power) which was placed between two gas diffusion layers (Carbon Cloth 10% PTFE from ElectroChem Inc.) and pressed for 1.5 min at 120 °C with 100 kg $cm^{-2}$. The Nafion membrane was previously treated by boiling in $H_2O_2$ 3% ($H_2O_2$ 30%, Biopack) followed by $H_2SO_4$ 3% (95–97%, Merck). The MEAs were mounted in standard single cell housing with a serpentine flow fields. Proper Teflon gasket films (50–150 μm) were inserted and the cell uniformly bolted with a torque of 2.3 N m. $O_2$ and $H_2$ were humidified at 100% RH and fed to the cell, the humidifying bottles and the cell temperatures were controlled individually. After assembly of the cell, the MEA was subject to the following conditioning procedure. First, the temperatures were increased to values of 60 °C. Then the potential of the cell was maintained constant at 0.4 V for 25–30 min until the current stabilizes. The potential was then set back to open circuit voltage (OCV) until it remains constant and the polarization curve was registered. Those steps were repeated until no more change in the maximum power output was observed. Galvanodynamic polarization tests were performed at three different cell

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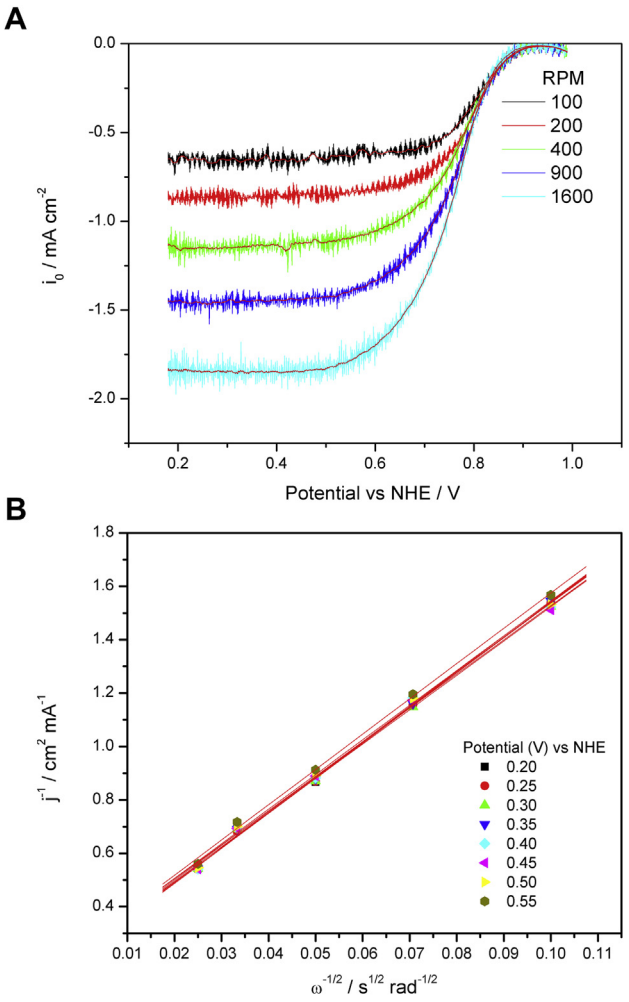


**Fig. 1 – A) Linear sweep voltammetry curves obtained in 0.5 M $H_2SO_4$ for ORR for Pt/MC at different rotation rates. B) Koutecky–Levich plots showing the linear relation between $j^{-1}$ and $\omega^{-1/2}$.**

temperatures, 40, 60 and 80 °C with humidified $H_2$ and $O_2$ at a flow of 200 ml min$^{-1}$. Experiments were also conducted with back pressure control (30 psi) at the exit of the anode and cathode.

All fuel cell tests were performed with a Scribner fuel cell test station and in all cases the humidifier temperature was set 5 °C above the cell temperature.

## 3. Results and discussion

### 3.1. ORR analysis by RDE

The overall measured current density, $j$, of the oxygen reduction can be expressed in terms of the kinetic current density, $j_k$, and the diffusion limited current density, $j_d$, by the Koutecky–Levich (KL) equation [23]:

$$\frac{1}{j} = \frac{1}{j_k} + \frac{1}{j_d} = \frac{1}{j_k} + \frac{1}{B\omega^{1/2}} \quad (1)$$

$B$ being,

$$B = 0.2nFC_oD_o^{2/3}\nu^{-1/6} \quad (2)$$

where 0.2 is a constant used when the rotation speed, $\omega$, is expressed as rpm, $n$ is the number of electrons transferred per molecule of $O_2$ reduced, $F$ the Faraday constant, $C_O$ is the concentration of oxygen dissolved ($1.1 \times 10^{-6}$ mol cm$^{-3}$), $D_o$ is the diffusion coefficient of oxygen in the solution ($1.4 \times 10^{-5}$ cm$^2$ s$^{-1}$) [24], and $\nu$ the kinematic viscosity of the 0.5 M $H_2SO_4$ solution ($1.0 \times 10^{-2}$ cm$^2$ s$^{-1}$) [24] all of them at 25 °C. It is possible to calculate the theoretical slope of the KL plot (log $j$ vs. $\omega^{-1/2}$) considering a four electrons process, i.e., a complete reduction of $O_2$ to $H_2O$. The dependences of the oxygen concentration and diffusion coefficient with temperature were taken into account for corrections of the theoretical 4 electrons KL plots using the values presented in literature [25]. Fig. 1A shows a set of LSV obtained in an RDE configuration in $O_2$ saturated 0.5 M $H_2SO_4$ at 25 °C, which exhibit a well defined charge-transfer kinetic control, mixed kinetic-diffusion, and diffusion-limited currents. Fig. 1B shows the slopes of the KL plot from which the number of electrons involved in the ORR was calculated. The theoretical slope (considering $n = 4$) was 9.42 mA$^{-1}$ cm$^2$ rpm$^{1/2}$ at 25 °C, while the experimental slope observed for Pt/MC was 12.9 mA$^{-1}$ cm$^2$ rpm$^{1/2}$. The measured KL slope yields $n = 3$ (considering a transfer coefficient $\alpha = 0.5$). Moreover, the determination of the number of electrons by KL for the Pt/C yield a value of 3.2 leading to the conclusion that the ORR follows the same mechanism for Pt/MC as for the commercial one. Tafel analysis was also performed at 25 °C in order to obtain information of the reduction mechanism in both, Pt/C and Pt/MC catalysts through the Tafel slope ($b$) and exchange current density ($j_0$). Usually, Tafel plots for the ORR on platinum electrodes show different well-defined slopes, at low and high current densities. In general, the slope at low current densities (related to the transfer of 2 electrons) is constant with temperature, and Tafel slopes are analyzed at high current densities (related to the transfer of 4 electrons). Fig. 2 shows the Tafel curves for Pt/MC and Pt/C. From the figure the values of $b$ obtained were 119 mV dec$^{-1}$ for Pt/MC and 105 mV dec$^{-1}$ for Pt/C indicating that the rate determining step

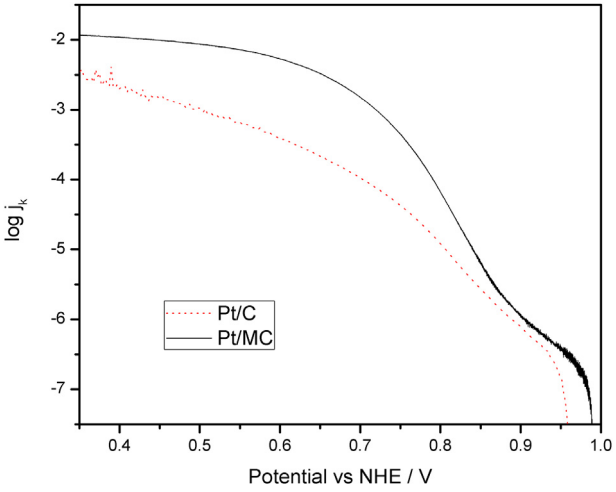


**Fig. 2 – Tafel plots (log $j$ vs potential) for Pt/C and Pt/MC.**

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(rds) for the ORR on both catalyst is the transfer of the first electron to the $O_2$ adsorbed onto the catalyst surface. This demonstrates that the ORR proceeds by the same mechanism on both catalysts as expected due that the carbon support should not modified the mechanism. From Fig. 2 can also be observed that the equilibrium potential ($E_{eq}$) for Pt/C is lower than for Pt/MC indicating that the overpotential needed in order to obtain a given current density would be lower for the last one. The exchange current, was evaluated taking into consideration the reversible oxygen electrode potential ($E_r$) at 25 °C [20,21]. The obtained values of $j_0$ for Pt/MC was $4.37 \times 10^{-5}$ mA cm$^{-2}$ and $5.79 \times 10^{-6}$ mA cm$^{-2}$ for Pt/C showing that Pt/MC presents higher catalytic activity than Pt/C, which is consistent with the observed difference in the $E_{eq}$.

### 3.2. ORR analysis by RRDE

The RRDE analysis allows us to calculate the $H_2O_2$ percentage generated during the $O_2$ reduction using the equation [26]:

$$\%H_2O_2 = \frac{200I_R/N}{I_D + I_R/N} \quad (3)$$

where $N$ is the experimental collection efficiency which corresponds to the $I_R/I_D$ ratio ($N = 0.18$). Percentages of $H_2O_2$ *vs.* cell potential at different rotation speeds were calculated using Eqn. (3), and they are shown in Fig. 3. For Pt/MC a maximum value of 0.25% at 200 rpm and 0.9 at 900 rpm were obtained. This result indicates that, at 25 °C, the ORR proceeds mostly to water, with a yield of ~99%, following preferentially a 4 electron transfer reaction mechanism. However, although the production of $H_2O_2$ is low, is not negligible and presents a dependence on the applied potential. On the other hand, the percentage of $H_2O_2$ for Pt/C are 1.25% (200 rpm) and 2% (900 rpm) being the amount of $H_2O_2$ formed 2 to 3 times higher than those obtained for Pt/MC.

### 3.3. Fuel cell test

Polarization measurements at the three different temperatures for the Pt/MC as anode and cathode catalyst with a backpressure of 30 PSI are shown in Fig. 4(A, B). As anode catalyst, Pt/MC shows high limiting current density ($j_{lim}$) and peak power densities ($P_{peak}$), attaining ca. 300 mW cm$^{-2}$ at 80 °C. As cathode catalyst, the Pt/MC shows a good overall performance only at 80 °C presenting high $j_{lim}$ and a $P_{peak}$ of 260 mA cm$^{-2}$.

Fig. 5 shows the comparison of Pt/MC as anode and cathode catalyst against an MEA prepared with commercial Pt/C on both electrodes at 80 °C and 30 PSI back pressure. From Fig. 5 can be observed that the $j$ values in the polarization are higher for the commercial catalyst up to a value of 400 mA cm$^{-2}$. Above that value, $j$ decreases sharply than for the Pt/MC as anode and cathode catalyst. The limiting current is 200 mA cm$^{-2}$ and 300 mA cm$^{-2}$ higher for Pt/MC as cathode and anode respectively than for Pt/C. The $P_{peak}$ density is ca 5% lower for Pt/MC that for Pt/C. On the other hand, $P_{peak}$ for Pt/MC as anode catalyst is 8% higher than Pt/C. A closer look at the polarization curves indicates that the mass transport is better on the MEA that uses Pt/MC as catalyst when compared to the one prepared with commercial ones. This is observed at the high current density zone of the polarization plot, where the mass transport overpotential controls the polarization. As indicated above the polarization decreases sharply above a

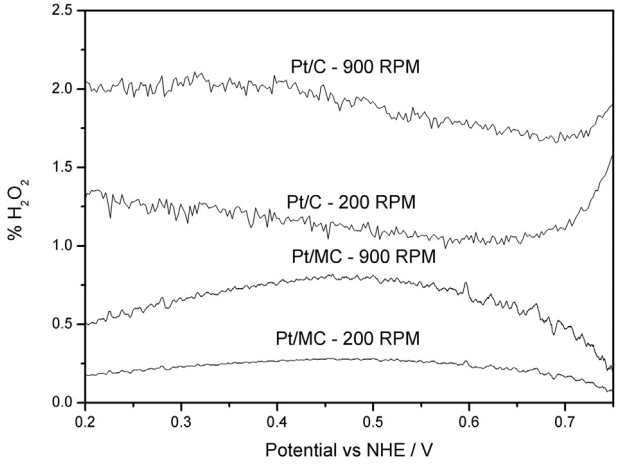


**Fig. 3 – Percentage of $H_2O_2$ produced as function of the disk potential at different rotating rates at 25 °C.**

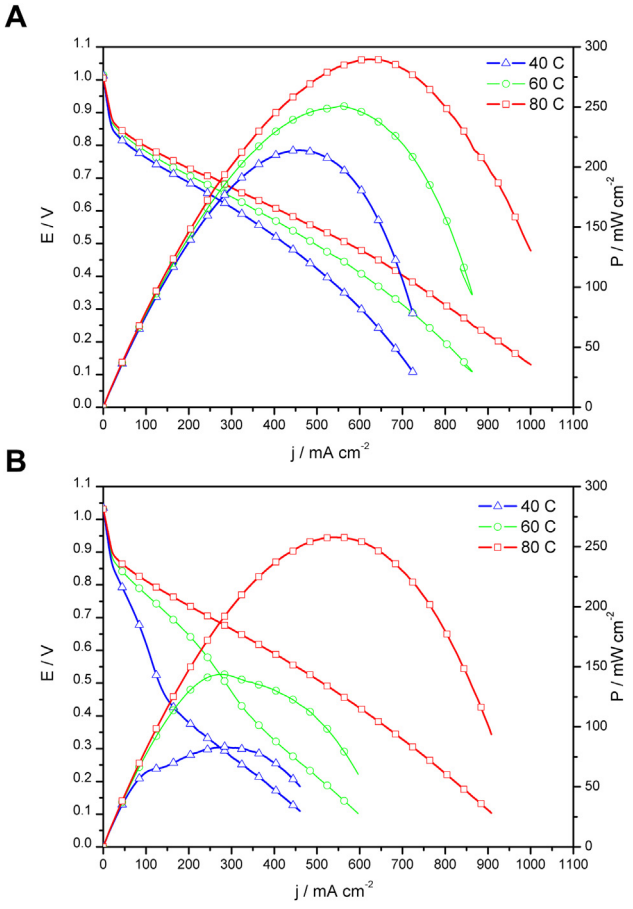


**Fig. 4 – Polarization and power plots at different temperatures for MEAs prepared with A) Pt/MC as anode catalyst. B) Pt/MC as cathode catalyst. In all cases commercial Pt/C was used in opposite electrode and 30 PSI back pressure at the anode and cathode.**

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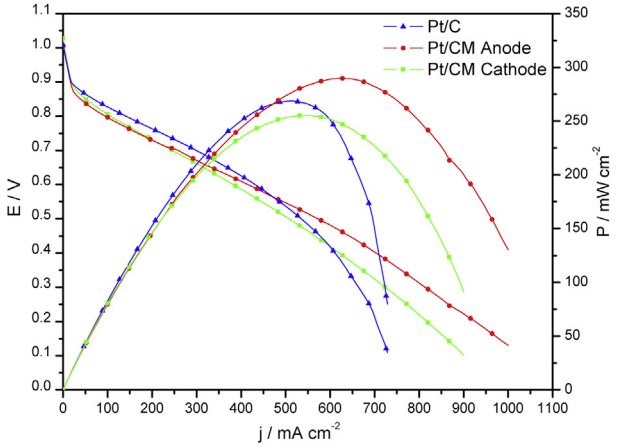


**Fig. 5 – Polarization and power plots for Pt/MC as anode and as cathode catalyst vs the commercial catalyst at 80 C and 30 PSI back pressure.**

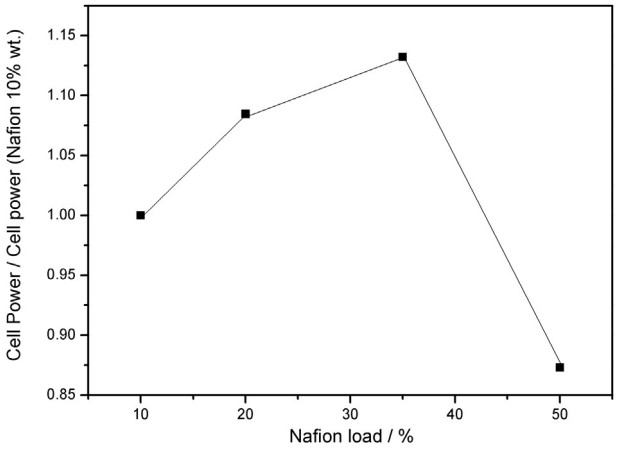


**Fig. 6 – $P_{peak}$ for the different amounts of Nafion used in the final catalyst for Pt/MC used as cathode catalyst.**

current density of 400 mA $cm^{-2}$ for Pt/C while that is not the case for Pt/MC as anode or cathode catalyst. Similar results were observed in previous works where PtRu was deposited on MC and used as anode catalyst for DMFC [15,17], and also as support for $PdNi_2$ catalyst for cathodes of DMFC [16]. In those reports, it was shown that the MC not only provide a suitable substrate that resulted in a highly dispersed catalyst with a narrow particle size distribution, but also the mass transport showed improvements when compared to the catalyst supported on Vulcan carbon.

In Sections 3.1 and 3.2 was shown that Pt/MC catalyst presents a better catalytic activity towards ORR that Pt/C. However, the results described above showed a lower power for Pt/MC as cathode catalyst than for Pt/C even though the mass transport controlled region is better. Different factors affect the overall performance of the fuel cell besides the catalyst catalytic activity, such as catalyst deposition method, catalyst ink formulation and pressing conditions to name a few. Moreover, the hydrophilicity/hydrophobicity in the anode and cathode might be different depending on the material composition and test conditions. In order to further evaluate the role of the MC, MEAs with different formulation in terms of the amount of Nafion binder used in the ink of the cathode side were evaluated. The different MEAs with an amount of Nafion binder, by weight of the final catalyst deposited on the gas diffusion layer, from 10% to 50% were measured at 80 °C and 30 PSI BP. Polarization of those MEAs rendered curves similar to the one showed in Fig. 5B for 80 °C. Fig. 6 shows the normalized $P_{peak}$ obtained with the different concentration of Nafion binder in the cathode. Can be observed from the figure, that with 35% w/w of Nafion there is a maximum of the $P_{peak}$ obtained. Rao et al. [27] demonstrated that the material porosity have a crucial effect in the catalyst participation on the triple phase boundary. Nafion penetration in small pores (<20 nm) is hindered, reducing therefore the formation of the triple phase boundary. Thus, the results might be explained by the improvement of the triple phase boundary by improving the ionic contact with the catalyst surface [28].

## 4. Conclusions

The effect of a mesoporous carbon support on the catalytic performance of Pt nanoparticles was analyzed. The catalyst prepared was evaluated for hydrogen oxidation reaction and oxygen reduction reaction. Mesoporous carbon with a pore distribution of about 20 nm and a pore volume of 0.99 $cm^3$ $g^{-1}$ was chosen as support. The Pt nanoparticles were obtained by the impregnation method by sodium borohydride reduction in aqueous media.

The supported catalyst was characterized by RDE and RRDE for ORR. The catalyst support on MC showed a lower overpotential than Pt supported on Vulcan to achieve similar current density in Tafel plot. Moreover, Pt/MC has lesser peroxide generation performance. However, the mesoporous support although presents a positive effect in the mass transports does not shows a significant positive effect on the power density. The Pt/MC showed better cell performance than Pt/C as anode catalyst. Pt/MC catalyst in the anode section showed a $P_{peak}$ 8% higher than Pt/C. Furthermore, at high current densities the Pt/MC showed better mass transport, demonstrating a positive effect of the mesopority of carbonous support in the performance of catalyst layer. Porous carbon with mesopores around 20 nm has positive effect on mass transport in cell performance. However, there is still a lacking in works evaluating the optimum conditions of mesoporous support in a fuel cell, where the effects of the preparation of the catalytic layer are crucial. This different result would indicate that the MC is promising for catalyst support in fuel cells, a forthcoming work analyzing in detail the optimum conditions for the formulation of catalytic ink in a catalyst layer will be presented.

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