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Electrochemical and fuel cell evaluation of PtAu/C electrocatalysts for ethanol electro-oxidation in alkaline media

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ARTICLE INFO

Article history:
Received 13 February 2014
Received in revised form
14 April 2014
Accepted 22 April 2014
Available online 23 May 2014

Keywords:
PtAu/C electrocatalysts
Alkaline fuel cell
Ethanol oxidation reaction

ABSTRACT

PtAu/C electrocatalysts in different atomic ratios and supported on Vulcan XC 72 carbon were tested for ethanol electro-oxidation in alkaline media. The electrocatalysts were prepared using borohydride as reducing agent. PtAu/C X-ray diffraction (XRD) patterns showed peaks characteristic of Pt face-centered cubic (fcc) structure and carbon. PtAu/C (70:30) and (50:50) XRD patterns showed a shift to lower values of 2θ when compared to Pt/C, this way suggesting the formation of PtAu alloy. Transmission electron micrographs showed the nanoparticles with particle size between 4 and 6.5 nm for all PtAu/C electrocatalysts. Electrochemical characterization of the PtAu/C materials suggested the PtAu/C (50:50) as the most promising material for ethanol electro-oxidation while experiments in single fuel cell suggested PtAu/C (70:30). The discrepancy in the results obtained can be explained by the electrode construction since PtAu/C (50:50) yields a much thicker electrode than PtAu/C (70:30), due to the Pt load is the same. The best results obtained with PtAu/C electrocatalysts could be explained by the presence of Pt and Au in close contact (alloy) associated to the extend in the platinum lattice parameters since these properties could contribute to the C–C breaking bond.

Introduction

Alkaline direct ethanol fuel cells have recently attracted worldwide attention because they are low-cost and show promise as clean and efficient power sources [1,2]. Direct alcohol fuel cells can use liquid fuels without a reforming step. They also have a compact design. These advantages make this technology an attractive possibility for satisfying the rapidly growing demand for compact power sources [2,3].

Among the alcohols used as fuels, ethanol has been recognized as the most promising since it is a carbon-neutral, less toxic and it has many unique physicochemical properties including high energy density. Thus, it is easy to transport and storage, as well as handle and it has been proved by

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http://dx.doi.org/10.1016/j.ijhydene.2014.04.169

researches to have a lower crossover rate and affects cathode performance less severely than methanol [1,2,4].

However, the complete ethanol oxidation reaction is hard to achieve due to the difficulty of breaking C–C bonding to form CO_2 and H_2O and also to the formation of intermediates that poison Pt/C anodes, reducing the fuel cell performance [5,6]. In order to overcome these difficulties, some parameters could be changed such as the synthesis method, the particle structure, the particle size and the catalyst component [7,8].

Nevertheless, electrocatalysts that are more active and selective than Pt/C resulted in combining Pt with other elements [5]. Moreover, bimetalic catalysts with alloy structures are an attractive topic of catalytic research applied in ethanol oxidation due to their enhanced activity and stability [6].

Considering binary catalysts, although gold has been demonstrated to be chemically inactive among noble metals due to its electronic configuration, Au is known to be reactive for CO oxidation. Moreover, supported Au catalysts are also promising catalysts in various practical areas such as water photosplitting, hydrogen production, CO removal, CO gas sensors and air-purification devices for removing trace quantities of CO from ambient. Also, gold metal finally dispersed on metal oxides has been demonstrated to be a promising catalyst in a number of catalytic reaction including complete oxidation of hydrocarbons, hydrogenation and NO reduction [8–13].

Aiming the improvement of direct ethanol fuel cells in alkaline media, this work contemplates a deep study considering electrochemical and also fuel cells experiments using PtAu/C in different atomic ratios as work electrodes/anodes of a single fuel cell. The PtAu/C materials were prepared by the borohydride reduction process and supported on Vulcan XC 72 carbon.

Experimental

PtAu/C electrocatalysts (20 wt.% of metals loading) in different atomic ratios (PtAu/C (50:50), PtAu/C (70:30) and PtAu/C (90:10)), Au/C and Pt/C were prepared by the borohydride reduction process [14,15] using $H_2PtCl_6{\cdot}6H_2O$ (Aldrich) and $HAuCl_4{\cdot}3H_2O$ (Aldrich), as metal sources and used as work electrodes/anodes of a direct ethanol fuel cell. In the borohydride process, the Vulcan XC72 was firstly dispersed in an isopropyl alcohol/water solution (50/50, v/v) and the mixture homogenized under stirring, being the metals sources added and put on an ultrasonic bath for 5 min. After that, a solution of $NaBH_4$in 0.1 mol L^{-1} KOH was added, in one portion, under stirring at room temperature and the resulting solution was maintained under stirring for more 15 min. After this procedure, the final mixture was filtered and the solids washed with distillated water and dried at 70 °C for 2 h.

All the prepared materials were characterized by XRD using a Rigaku diffractometer model Miniflex II, using Cu Kα radiation source (0.15406 nm). The X-ray diffraction patterns were recorded in the range of $2\theta = 20°–90°$ with a step size of 0.05° and a scan time of 2 s per step. Transmission electron microscopy (TEM) images were also carried out using a JEOL transmission electron microscope model JEM-2100 operated at 200 kV.

This work contemplates two studies, electrochemical and fuel cell conditions. The electrochemical experiments were taken by using a three-electrode conventional cell, using as reference and work electrodes the AgAgCl(KCl 3 mol L^{-1}) and Pt wire, respectively. The work electrode was prepared using the thin porous coating technique as proposed by De Souza et al. and Silva et al. [16,17]. The electrochemical characterizations were made by cyclic voltammetry conducted at a scan rate of 10 mV s^{-1} in KOH 1 mol L^{-1} in presence and absence of ethanol 1 mol L^{-1} and also by amperometric curves, recorded in the same electrolyte containing ethanol at −0.35 V for 1800 s. All measurements were conducted at room temperature.

Considering the real conditions, the experiments were conducted using a single cell with 5 cm^2 of area. The temperature was set to 75 °C for the fuel cell and 85 °C for the oxygen humidifier. All electrodes were constructed with 1 mg of Pt per cm^2 in the anode or in the cathode excepted for Au/C which contained 1 mg of Au per cm^2. In all experiments a commercial Pt/C (BASF) was used as cathode and the electrocatalysts were painted over a carbon cloth in the form of a homogeneous dispersion prepared using Nafion® solution (5 wt.%, Aldrich).

After the preparation, the electrodes were hot pressed on both sides of a Nafion® 117 membrane at 125 °C for 3 min

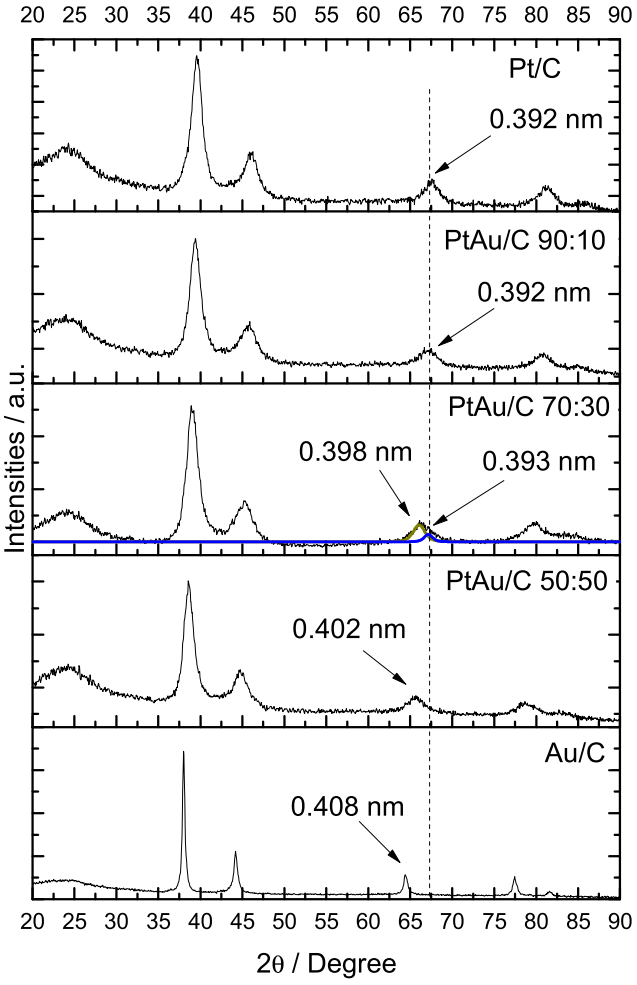

Fig. 1 – X-ray diffraction patterns of the PtAu/C, Pt/C and Au/C electrocatalysts.

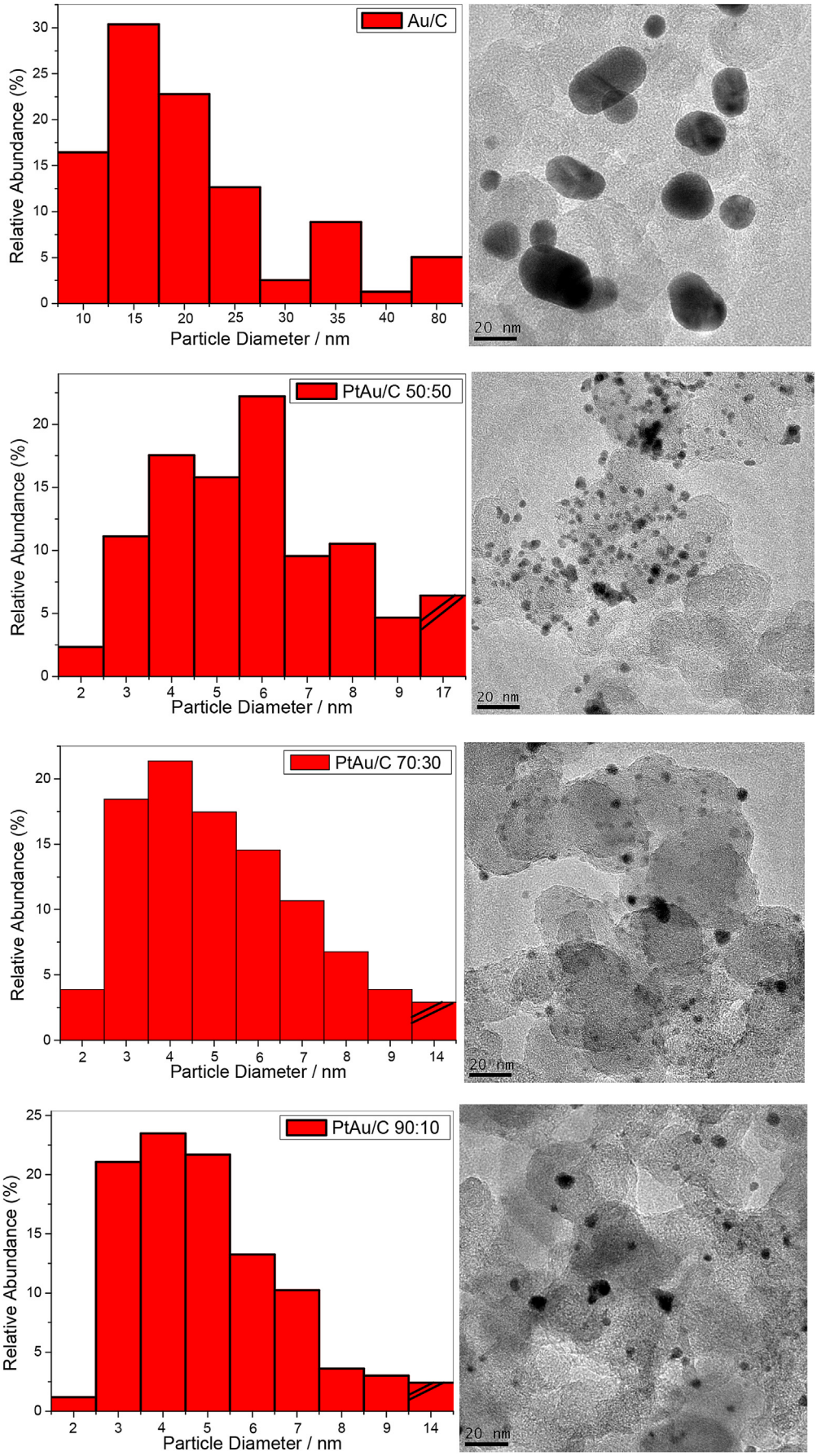

Fig. 2 – TEM micrographs and histograms of the particle size distribution of Au/C, PtAu/C (50:50), PtAu/C (70:30) and PtAu/C (90:10).

under a pressure of 247 kgf cm^{-2}. Prior to use, the membranes were exposed to KOH 6 mol L^{-1} for 24 h as it has already been proposed by Hou et al. [18]. The fuel, 2.0 mol L^{-1} C_2H_5OH and 2.0 mol L^{-1} KOH were delivered at 1.0 mL $min^{-1}$and the oxygen flow was regulated at 150 mL min^{-1}. Polarization curves were obtained by using a potentiostat/galvanostat PGSTAT 302NAutolab.

It has been demonstrated that even using existing ion conductors and catalysts, adding an alkaline (NaOH or KOH) to ethanol would improve the performance of the fuel cell which is attributed to the increase in the ionic conductivity of the membrane and also to the kinetics of the ethanol oxidation reaction to be further sped up [4].

Results and discussion

XRD patterns of the PtAu/C, Pt/C and Au/C electrocatalysts were shown in Fig. 1. All PtAu/C diffractograms showed four peaks in $2\theta = 39°$, 46°, 67° and 81°, which are associated with the (111), (200), (220) and (311) planes, characteristic of Pt face-centered cubic (fcc) structure [16,17] and a broad peak at about 25° associated with the hexagonal structure of the Vulcan XC72 support [19,20]. Peaks associated to Au were not evidenced in the PtAu/C electrocatalysts, suggesting the formation of amorphous phases that can not be observed by XRD [21] or the formation of alloyed phases.

For PtAu/C (70:30) and PtAu/C (50:50) XRD patterns there was a shift to lower values of 2θ when compared to Pt/C, suggesting the formation of PtAu alloy. Moreover, the lattice parameters were also calculated for all the PtAu materials and the values obtained are inserted on Fig. 1. The lattice parameter of PtAu/C (70:30) and PtAu/C (50:50)were higher than the Pt, what indicates the formation of alloy phases between Pt and Au or their inter-diffusion of Pt and Au atoms in the fcc lattice with elongated Pt–Pt inter-atomic distance [22].

Zhou et al. [22] studied the activity of PtAu electrocatalysts and also observed a downshift of the XRD patterns and associated this shift to the presence of PtAu in alloyed phases. Furthermore, they attributed the downshift to the lattice parameter expansion of the PtAu electrocatalysts as observed in the present work. They also affirm that the stronger Pt–Au interaction and the expanded Pt lattice parameter in alloyed PtAu/C contributed to the higher specific activity towards ethanol oxidation.

Su et al. [6] studied the activity of PdAu electrocatalysts and observed that the diffraction peaks of the Pd_3Au and Pd_3AuNi catalysts were located at lower 2θ values with respect to those Pd. This result was associated with the formation of binary Pd_3Au and ternary Pd_3AuNi alloy and also to the expansion of the Pd lattice. Moreover, Su et al. [6] suggested that the expansion of the Pd lattice could be favorable for ethanol absorption.

TEM micrographs and histograms of particle mean diameter distribution for the PtAu/C and Au/C electrocatalysts are shown in Fig. 2. All prepared materials showed the particles well dispersed on the support, even though some small particle agglomerations can be observed. The nanoparticles mean diameter were determined by counting about 100 randomly chosen particles from the relevant TEM images [23,24]and the particles are with average diameter of 18.5 nm for Au/C, 6.4 nm for PtAu/C (50:50), 5.8 nm for PtAu/C (70:30) and 4.0 for PtAu/C (90:10).

Bai et al. [25] synthetized PtAu nanoparticles using sodium borohydride reducing reagent and obtained nanoparticles with an average of 3.5 nm and also a narrow size distribution. Moreover, Yi et al. [26] used the same methodology to prepare Pt/C and PtSn/C electrocatalysts and obtained also a narrow distribution with the average particle size of 3.5 nm and 3 nm for Pt/C and PtSn/C, respectively.

Fig. 3 shows the cyclic voltammograms of PtAu/C, Au/C and Pt/C electrocatalysts in 1.0 mol L^{-1} KOH. PtAu/C voltammograms display a well-defined hydrogen oxidation region in comparison to pure platinum. Furthermore, the double layer and oxides formation/reduction regions were also observed [27]. Among all PtAu electrocatalysts, PtAu (50:50) presented the largest charge–discharge current of the double layer. This behavior is characteristic of binary electrocatalysts containing transition metals and it can be explained by the better material dispersion on carbon and/or the formation of ultrafine particles [28].

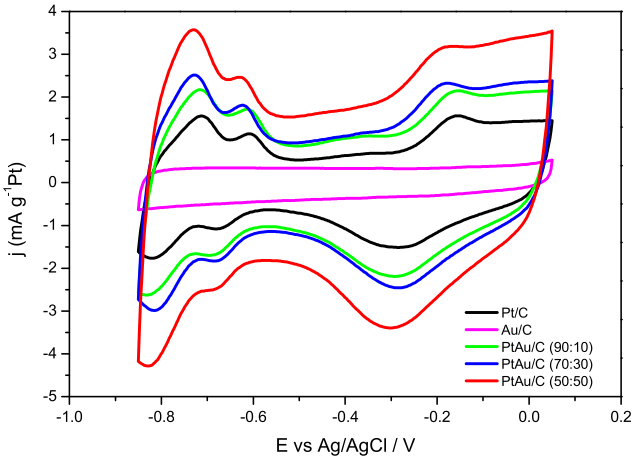

Fig. 3 – Cyclic voltammograms of PtAu/C electrocatalysts, Pt/C and Au/C in 1.0 mol L^{-1} KOH and scan rate of 10 mV s^{-1}.

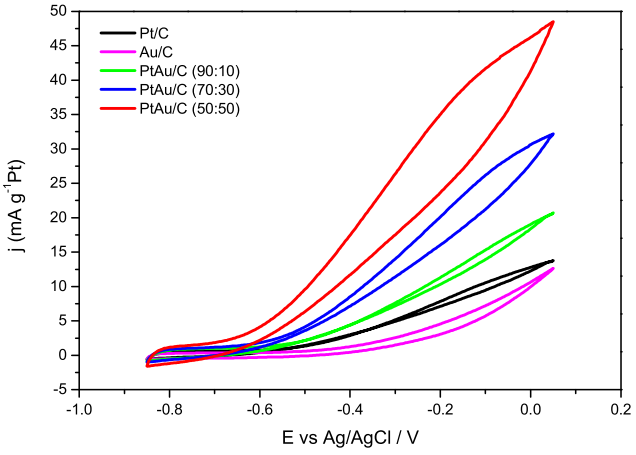

Fig. 4 – Cyclic voltammograms of PtAu/C, Pt/C and Au/C in presence of 1.0 mol L^{-1} KOH and 1.0 mol L^{-1} of ethanol. Scan rate of 10 mV s^{-1}.

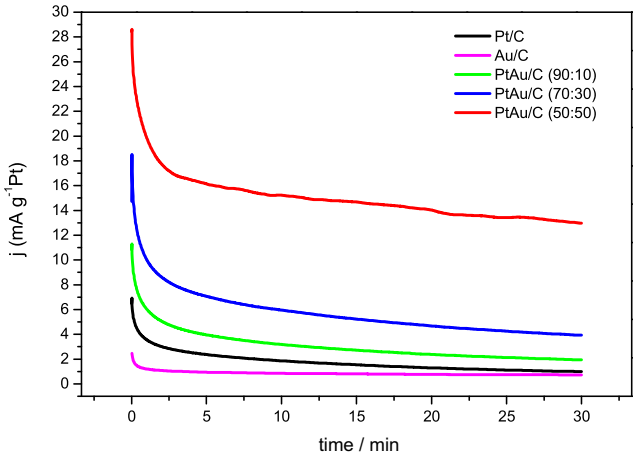

Fig. 5 – Current-time curves at −0.35 V in 1 mol L^{-1} ethanol and 1.0 mol L^{-1} KOH solution for PtAu/C electrocatalysts, Pt/C and Au/C.

Fig. 4 shows the CV of Pt/C, Au/C and PtAu/C in different atomic ratios in presence of 1.0 mol L^{-1} ethanol and 1.0 mol L^{-1} KOH. The CVs were normalized by the amount of Pt in the elctrocatalysts. By Fig. 4 it is possible to observe that PtAu/C (50:50) showed the best electrocatalytic activity toward ethanol electro-oxidation. After PtAu/C (50:50), the PtAu/C (70:30) and the PtAu/C (90:10) showed better activities toward ethanol electro-oxidation when compared to Pt/C and Au/C. Moreover, the ethanol oxidation starts at a potential of about -0.50 V for Pt/C.

It is also important to stress that just Au/C showed a final current density similar to Pt/C. This observation shows the good activity of Au/C in alkaline media as already mentioned by other authors [7,13,29].

From the chronoamperometry results, on Fig. 5, the PtAu (50:50) also showed the best electrocatalytic activity. The current density measured for PtAu/C (50:50) is about thirteen times higher than the one obtained for Pt/C. These results could be attributed to an optimal composition between Pt and Au.

Han et al. [13], working with Au/Pt nanodendrites modified electrodes, observed that when the molar ratio of Au to Pt is equal to 3 the nanodentrites electrocatalysts exhibited the highest peak current density and with the further increase in Pt content the peak current decrease. This observation can also be made in this paper since the PtAu/C (50:50), with more Au content, showed the highest current density while with the increase in the Pt content PtAu/C (70:30) and PtAu/C (90:10) there was a decrease in the current density.

According to Zhou et al. [22] Au works indirectly by decreasing the adsorption of poisoning intermediates on Pt sites and increasing the capability of free Pt sites to supply $-OH_{ads}$ species. Also, gold can extend the platinum lattice parameter by alloying. These properties are beneficial to the effects of catalyst structure change on ethanol C–C bond cleavage. Additionally, it has been reported that the addition of Au to Pt could increase the dehydrogenation and also the C–C bond cracking activity [22,30,31].

Additionally, according to Li et al. [32] Au can upshift the d-band energy of the Pt layer and a more a higher-lying d-band energy characterizes a more reactive surface that tends to bind adsorbates more strongly and enhances the kinetics of the dissociation reaction producing these adsorbates.

Then, the best results obtained on chronoamperometry measurements using PtAu/C compared to Pt/C, might be associated to the PtAu alloyed phases associated to the extend in the platinum lattice parameters since these properties could contribute to the C–C breaking bond.

Fig. 6 shows the performances of a single DEFC operating at 75 °C with PtAu/C (50:50), PtAu/C (70:30), PtAu/C (90:10), Pt/C and Au/C as anode electrocatalysts and Pt/C BASF as cathode in all experiments. Table 1 summarizes the results obtained by these experiments. From Fig. 6 and Table 1 it is possible to observe that the open circuit voltage of the fuel cell follows the order:PtAu/C (70:30) > PtAu/C (50:50) > PtAu/C (90:10), ≥Pt/C > Au/C. The values of maximum power density also follow the sequence of the open circuited potential. The experiments at 75 °C on single DEFC showed that the power density for the PtAu/C (70:30) was three times higher than the one obtained using Pt/C.

The results on single DEFC show the PtAu/C 70:30 as the best material toward ethanol electro-oxidation. This result does not mean to the one obtained in electrochemical experiments which indicate the PtAu/C (50:50) as the best material. This might be associated to the anode construction, since the Pt load is constant (1 mg_{Pt} cm^{-1}) and then the anode composed of PtAu/C (50:50) is thicker than the PtAu/C (70:30). Consequently, the PtAu/C (50:50) is more resistive than the PtAu/C (70:30) electrode what also difficult the fuel diffusion

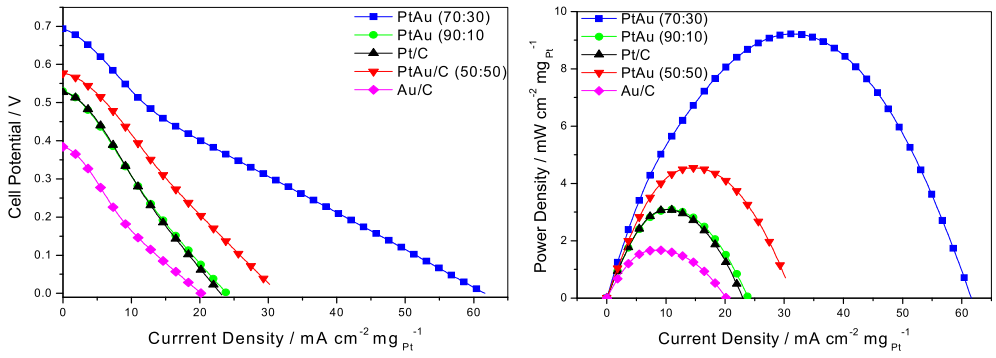

Fig. 6 – Direct ethanol fuel cell performance of the PtAu/C electrocatalysts, Pt/C and Au/C.

Table 1 – Main results obtained using a direct ethanol fuel cell.

Catalyst	Open circuit potential /V	Maximum power density /mWcm^{-2}
Au/C	0.38	1.6
Pt/C	0.53	3.0
PtAu/C 50:50	0.58	4.5
PtAu/C 70:30	0.69	9.0
PtAu/C 90:10	0.53	3.0

through the catalytic layer. Then the best result obtained with PtAu/C might be associated with the effect generated between Pt and Au in close contact (alloy) that contributes to the C–C cleavage.

Conclusions

Borohydride process showed to be an efficient method to prepare PtAu/C electrocatalysts for ethanol electro-oxidation in alkaline media. This method yields electrocatalysts with mean particle size between 4 and 6.5 nm for all electrocatalysts. Electrochemical characterization of the synthetized materials suggested PtAu/C (50:50) as the most promising material for ethanol electro-oxidation in alkaline media while single cell experiments suggested PtAu/C (70:30) as the best material for the same process. The fuel cell discrepancy toward ethanol electro-oxidation could be explained by the electrode construction since PtAu/C (50:50) yields a much thicker electrode than PtAu (70:30) because of the Pt load. The best results obtained with PtAu/C electrocatalysts could be explained by the presence of PtAu alloyed phases associated to the extend in the platinum lattice parameters since these properties could contributed to the C–C breaking bond.

Acknowledgments

The authors wish to thank Laboratório de Microscopia do Centro de Ciências e Tecnologia de Materiais (CCTM) by TEM measurements, FAPESP (2013/01577-0, 2011/18246-0, 2012/22731-4, 2012/03516-5) and CNPq (150639/2013-9) for the financial support.

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