

Synthesis of Pt/Carbon Xerogel Electrocatalysts for PEM Fuel Cells by the Multiple SEA Method

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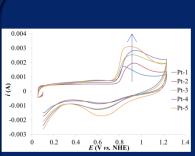


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The catalytic layers used in Proton exchange membrane fuel cell (PEMFC) are classically based on Pt particles supported on carbon black (Pt/C). The structure of these catalytic layer is however not optimal, resulting in incomplete utilization of the mass of Pt loaded onto the carbon support [1]. In order to reduce the cost of the catalytic layer without decreasing its performance, two parameters can be improved: the support and the synthesis of the Pt particles. With respect to the support, commonly used carbon blacks do not display optimal properties for electrocatalysis: they may contain high amount of chemical impurities, and the final structure of the electrodes is hardly tuneable [2]. A possible solution to these drawbacks is to use synthetic nanostructured materials with a controllable, reproducible texture and with a pure, known and constant chemical composition; carbon xerogels exhibit such properties [3]. With respect to the synthesis of the Pt particles, highly dispersed Pt/carbon xerogel catalysts can be prepared by the Strong Electrostatic Adsorption (SEA) method [4, 5]. This technique consists in impregnating the support, e.g. carbon xerogel, with a solution of metal precursor, e.g. hexachloroplatinic acid (H.PtCls), under pH control (2.4 - 2.5 for the impregnation of carbon xerogels by hexachloroplatinic acid [6]). After impregnation by the precursor, the impregnated support is dried and submitted to reduction treatment under H₂ flow. The impregnation-reduction step was repeated up to five times to increase the Pt loading. The five resulting catalysts were then characterized by physicochemical and electrochemical methods.

Multiple SEA Method Modified SEA method: • High concentration of H₂PtCl₆ • Re-use of the solution Optimal pH Addition of carbon xerogel I h stirring Drying Filtration Reduction Next impregnation

Physicochemical Characterization 0,0870 2.3 0.7 2.7 Pt-2 14.7 0.0895 22 0.7 27 3.0 23 19.0 0.0581 2.1 0,0475 22.2 2.4 Pt-4 0.0512 2.1 0.7 2.8 Pt-5 TEM micrograph Results obtained by ICP-AES, image analysis Fraction of particles (%) 30 25 20 15 10 5 of TEM micrographs and XRD Pt-2 Pt-3 Pt-4 Pt-5 Pt-3 Particle size (nm) Particle size distribution



CO stripping on the five catalysts measured at 20 mV/s in liquid electrolyte (H₂SO₄ 1 mol/L)

	S (m²/g _{Pt}) ±10%	d _{co} (nm) ±10%	SA ₈₅ (mA/cm² _{Pt}) ±10%	SA ₉₀ (mA/cm² _{Pt}) ±10%	<i>b</i> (mV/dec) ±10%
Pt-1	109	2.5	0.052	0.0114	-69
Pt-2	93	3.0	0.044	0.0093	-72
Pt-3	94	2.9	0.049	0.0102	-79
Pt-4	96	2.9	0.047	0.0093	-78
Pt-5	96	2.9	0.060	0.0105	-72

Electrochemical Characterization

XRD patterns of the five catalysts

Results obtained by CO stripping and ORR

Same particle size (~2.5 nm)

Tafel curves of the five catalysts

High dispersion



Increase of the Pt electroactive area
The specific activity for ORR remains constant
The Tafel slope (= reaction mechanism) remains constant

The multiple SEA method allows obtaining well dispersed Pt/carbon xerogel catalysts with high weight percentage up to 25 wt.%, and particle size close to ca. 2.5 nm according to TEM, XRD and CO stripping measurements. Moreover, the multiple SEA technique leads to a nearly threefold decrease in the consumption of Pt compared to the SEA method from which it is inspired. Although the highest Pt weight percentage obtained in this study is 25 wt.%, studies are in progress to determine the maximum loading that can be achieved without alteration of the metal dispersion. Further work is in progress to test the performance of these catalysts in real PEMFC conditions, and compare it to that of commercial catalysts. Other analyses will be performed so as to determine the optimal Pt weight percentage, and the optimal thickness of the catalytic layer by modifying these two variables in a series of membrane-electrodes assemblies.

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