The multiple SEA method: a method to synthesize Pt/carbon xerogel catalysts for Proton Exchange Membrane Fuel Cells (PEMFC)

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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 (H2PtCl6), 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
- 1 h stirring
- Filtration
- Drying
- Reduction
- Next impregnation
- Re-use of the solution

### Physicochemical characterization

<table>
<thead>
<tr>
<th>Pt</th>
<th>Pd</th>
<th>Au</th>
<th>Cu</th>
<th>Ni</th>
<th>Co</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>d50 (nm)</td>
<td>2.5</td>
<td>0.052</td>
<td>0.014</td>
<td>0.009</td>
<td>0.009</td>
<td>0.009</td>
</tr>
<tr>
<td>b (mV/dec)</td>
<td>-69</td>
<td>-72</td>
<td>-79</td>
<td>-78</td>
<td>-72</td>
<td></td>
</tr>
</tbody>
</table>

### Electrochemical characterization

<table>
<thead>
<tr>
<th>Pt</th>
<th>Pb</th>
<th>Sn</th>
<th>Zn</th>
</tr>
</thead>
<tbody>
<tr>
<td>E (V vs. NHE)</td>
<td>-0.004</td>
<td>0.004</td>
<td>-0.032</td>
</tr>
</tbody>
</table>
| Pt loading | Increase of the Pt electroactive area
The specific activity for ORR remains constant
The Tafel slope (= reaction mechanism) remains constant

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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. 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-electrode assemblies.