



## Methodology for the design of accelerated stress tests for non-precious metal catalysts in fuel cell cathodes



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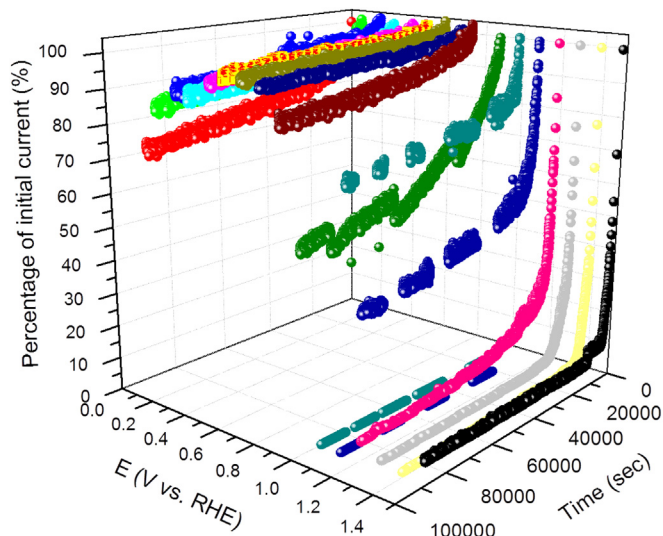
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### HIGHLIGHTS

- Electrocatalysis of ORR in Alkaline Fuel Cells.
- Design of accelerated stress test for NPMCs for fuel cell cathodes.
- Durability of NPMC cathodes in alkaline fuel cells.

### GRAPHICAL ABSTRACT



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### ABSTRACT

In this work we propose systematic methods for testing non-precious group metal catalysts and support degradation alkaline fuel cell cathodes. In this case study, we used a cathode composed of a pyrolyzed non-precious metal catalyst (NPMC) on activated carbon. The vulnerabilities of the cathode components were studied in order to develop the methodology and design an accelerated stress test (AST) for NPMC-based cathode in alkaline environment. Cyclic voltammetry (CV), chronoamperometry (CA) and impedance spectroscopy (EIS) were used to characterize the electrochemical behavior of the cathode and to follow the changes that occur as a result of exposing the cathodes to extreme operating conditions. Rotating ring disk electrode (RRDE) was used to study the cathodes kinetics; Raman spectroscopy and X-ray fluorescence (XRF) were used to study the structural changes in the electrode surface as well as depletion of the catalysts' active sites from the electrode. The changes in the composition of the electrode and catalyst were detected using X-ray diffraction (XRD). For the first time, we show that NPMC degrade

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rapidly at low operating potentials whereas the support degrades at high operating potentials and developed a tailor-made AST to take these into account.

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## 1. Introduction

The need for alternative energy is undisputable. One of the most promising alternative energy technologies today are fuel cells. This is due to the great advancement made in recent years, which includes: better engineering, heat and mass transfer optimization and cheaper catalysts based on non-precious group metals (NPMC) [1–3]. Due to the high costs associated with catalysts for polymer-electrolyte membrane fuel cells, alkaline fuel cells (AFC) technology has re-emerged in recent years with hopes for lower costs. One of the major advantages of using AFCs is their ability to work efficiently without precious metals both at the anode and the cathode [4,5]. The oxygen reduction reaction (ORR), in the cathode, is known to be the major limiting factor in the energy-conversion efficiency of fuel cells due to its sluggish kinetics [6] and it is more favorable in alkaline environment compared to acidic environment [7]. AFCs are easy to handle and they have an excellent suitability for alternating loads. Although they appear large and complicated, they can be built in small compact modules which can then be combined into large generators [8]. AFCs were found to produce less negative environmental impact such as human hazard and pollution than an equivalent solid oxide or phosphoric acid fuel cells [9]. Moreover, even their intolerance to carbon dioxide coming from air does not carry weight these days since effective, low cost techniques for removal of unwanted reaction products have improved significantly [10]. Overall, AFC technology, has great advantages [11] and in order to improve its lifetime and durability, a better understanding of failure mechanisms is required.

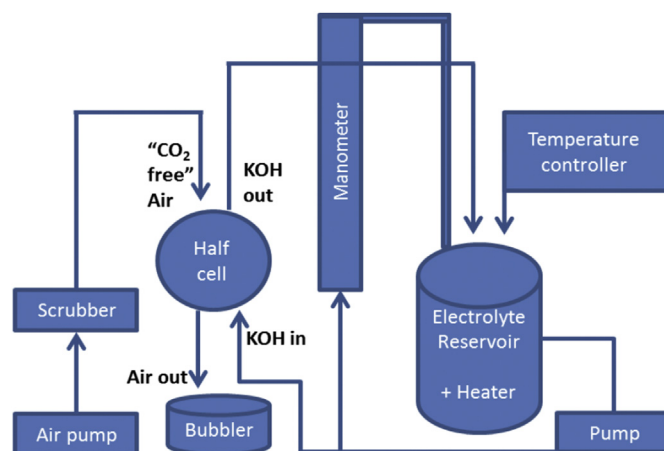
As the interest in AFCs rises, the development of new materials to increase their activity and lower their cost, the question of their durability has become critical. The durability testing protocols suggested by agencies such as the US DOE is limited to the study of platinum on carbon in acid. These protocols have become the generic testing protocols to all fuel cell catalysts but they possess one significant flaw: they do not necessarily take into account the vulnerabilities of other types of catalysts. When studying the durability of fuel cells, one can conduct a constant lifetime test; however, this method is time-consuming and expensive. Instead of long-term measurements, the fuel cells industry along with government agencies such as the US-DOE and researchers develop protocols and perform accelerated stress tests (ASTs). ASTs are methods for determining the durability of the whole cell or a particular component in the fuel cell system in short periods of time by exposing the cell to extreme, yet realistic, operating conditions that may cause some of its components to degrade rapidly (e.g. support, catalyst and membrane). Using such methods, the weaknesses of the fuel cell can be determined and then improved in order to increase its lifetime. The performance, degradation rate and component damage level under specific working conditions are examined during and after the ASTs. The aim of the AST is to predict the fuel cell's lifetime and explain the probable failure and degradation mechanisms. So far, most of the AST protocols were designed for commercial polymer electrolyte membrane fuel cells (PEMFCs) which consist on Pt-based catalysts, carbon supports and Nafion membranes. In the case of other catalysts, supports and membranes, specifically in AFCs, these ASTs become irrelevant due to the different chemical and electrochemical properties of the

different components. For example, in some of the US-DOE ASTs, the electroactive surface area (ECSA), an indication of the catalysts activity, of Pt-based catalysts is measured from the hydrogen adsorption/desorption peaks: this cannot be done with non-precious group metal catalysts since most of them do not adsorb hydrogen. Hence, ASTs need to be considered as case sensitive, or at least divided into categories of materials, since not all fuel cells are composed of the same materials and/or operate under the same conditions. Therefore, other methods and measurable parameters need to be found in order to assess the catalyst degradation in such cases. Here, we developed the methodology which will allow groups who are studying non-conventional materials for fuel cells to study the stability and durability of their catalysts and supports, develop case-sensitive ASTs for their fuel cells in general and for AFCs in particular.

In this paper, we present the methodology used to develop an AST in half-cell for a cathode based on carbon support (activated carbon) and non-precious metal catalyst for ORR (pyrolyzed porphyrin) in an alkaline fuel cell. We do this using a variety of electrochemical techniques such as chronoamperometry, cyclic voltammetry and impedance spectroscopy, as well as XRF, XRD and Raman spectroscopy. The methodology and data presented in this work are based on a real life test of a commercial alkaline fuel cell, measurements of transition potentials and their duration.

## 2. Experimental

The electrodes studied in this work are gas diffusion electrodes (GDE) consisting of pyrolyzed cobalt porphyrin (the catalyst, support and cathodes were manufactured and supplied by GenCell Ltd) incorporated in a carbon support on top of a nickel mesh (the current collector). The cobalt loading on the carbon support after the pyrolysis is 0.5wt. These electrodes were tested for durability in a half-cell test bench (HCTB) shown in Scheme 1, which controls the cell temperature, electrolyte flow and gas (oxygen or argon) flow, connected to an Autolab System (Metrohm Autolab, Netherlands).



Scheme 1. Layout of the cathodic Half Cell Test Bench.

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