Fuel Cell Catalyst Layer Evaluation using a Gas Diffusion Electrode Half-Cell: Oxygen Reduction Reaction on Fe-N-C in Alkaline Media

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Abstract

Anion exchange membrane fuel cells (AEMFC) are a promising technology to allow the application of non-precious metal catalysts. While many of such catalysts have been identified in numerous recent fundamental research studies, reports evaluating these catalysts in realistic AEMFC catalyst layers together with stability assessments are rare. In the present work we show that fast and reliable evaluation and optimization of Fe-N-C-based oxygen reduction reaction (ORR) catalyst layers can be achieved using a gas diffusion electrode (GDE) half-cell approach. To set a benchmark in such measurements, a commercial Pajarito Powder Fe-N-C catalyst and commercial Aemion™ ionomer are used. It is demonstrated that the ORR performance can be increased significantly by fine-tuning of the ionomer activation time. Furthermore, the optimized Fe-N-C-based catalyst layer shows very high stability with no observable performance deterioration after 5000 cycles in the 0.6 – 1.0 V vs. RHE potential window.

Keywords:

Fe-N-C, Oxygen Reduction Reaction, Gas Diffusion Electrode, Alkaline Fuel Cell, Non-noble metal catalyst, Alkaline Ionomer
1. Introduction

After decades of optimization, proton exchange membrane fuel cells (PEMFC) with Pt-group metal (PGM) catalysts have reached commercialization level. [1] However, the price for those devices is still high. The membrane electrode assembly (MEA) with PGM-based catalyst layers accounts for approximately one third of the overall device costs. [2] Therefore, the search for efficient, durable and cheap electrocatalysts and their implementation in real devices are two of the main topics in fuel cell research. [3] Recently, catalysts synthesized from earth-abundant, inexpensive and easily exploited materials as iron, nitrogen, and carbon (Fe-N-C) exhibit promising oxygen reduction reaction (ORR) activities, which opens new opportunities to significantly reduce the overall fuel cell technology costs. [4] Still, for this kind of catalyst the acidic environment of conventional PEMFCs imposes stability challenges, mainly due to Fe dissolution and carbon corrosion. [5-7] When operated in an alkaline environment instead, the durability of non-PGM materials can be improved significantly. [8] Additionally, the intrinsic activity of those materials can be slightly increased at high pH values. [9] Therefore, anion exchange membrane fuel cells (AEMFC) could facilitate the widespread application of non-PGM catalysts and hence significantly reduce overall fuel cell device costs. Recently tremendous progress in AEMFC development has led to performances exceeding power densities of PEMFCs using non-PGM catalyst materials. [10] This progress mainly arises from optimized mass transport of water, OH\(^-\) and the reactant gases, which is influenced by (i) membrane and ionomer materials, (ii) catalyst layer design and (iii) operating conditions. For more details, the readers are referred to excellent reviews in the field. [10-13] As those mass transport phenomena have a huge impact on the fuel cell performance, appropriate methods should be established to examine and optimize this influencing parameters both properly and rapidly.

Hitherto Fe-N-C research and development in PEMFC and AEMFC has mainly been conducted with either thin-film rotating disk electrode (TF-RDE) or MEA setups. Both suffer from
limitations evaluating catalyst layer effects on the single cell performance. Thin catalytic layers < 100 μg cm\(^{-2}\) are suggested for a valid application of TF-RDE, but often very high amounts of catalyst are used for those measurements leading to uncontrolled hydrodynamics and potentially misleading intrinsic results. [14] Additionally, the Fe-N-C catalyst most active in RDE, does not automatically lead to the best performance in MEA [15, 16], which is not really surprising taking into account the different mass transport effects in a very thin catalyst layer on a solid glassy carbon disk in RDE experiments and a realistic catalyst layer on a gas diffusion layer used in MEA experiments. Especially because experiments with Fe-N-C catalysts in both PEMFC and AEMFC have already revealed the impact of catalyst layer parameters (e.g. type of ionomer, ionomer content and distribution and manufacturing condition) on the morphology and transport behaviour in the electrode and therefore the single cell performance. [17-19] Generally it has to be pointed out, that a fuel cell electrode is a complex structure including catalyst, ionomer and void space, where each one of these plays a crucial role for electrode function. [12] This can only be investigated insufficiently with TF-RDE experiments. Hence MEA experiments are widely used to study catalyst layer effects on the fuel cell performance. But MEA experiments also introduce major drawbacks as they are (i) time and material intensive, (ii) do not allow direct insights into one single electrode (usually lack of a reference electrode) and (iii) are often difficult to compare due to deficiency of standardized test protocols and conditions for the evaluation of non-PGM catalysts. To bridge the gap between RDE and MEA experiments and to combine their advantages, recently different groups have conducted experiments with electrochemical half-cells using gas diffusion electrodes (GDE) as working electrodes. [20-22] Those GDE experiments allow fast and comparable testing at standardized operating conditions and provide dedicated insights into one single electrode with realistic catalyst layer parameters at relevant potential and current ranges. Therefore, this method can be an optimal supplement to drastically shorten the time from successful catalyst synthesis to an operating electrode for fuel cell applications.
In this communication, we present the first implementation of this novel GDE method for non-PGM catalysts in alkaline environment. As the ORR is still the limiting half-cell reaction [12], we focus on cathode catalyst layers and will address anode catalyst layer optimization in further work.

2. Material and Methods

2.1 Catalyst ink and electrode preparation

The PGM-free high pH GDE was fabricated from an ink comprised of a total of 20 wt% solids in 1-propanol. The solid fraction was made up by commercial 70 wt% Fe-N-C (PMF-011904, Pajarito Powder) and commercial 30 wt% ionomer (Aemion™ HNN5-00-X, Ionomr) with ion exchange capacity (IEC) \( \sim 1.4 - 1.7 \text{ meq g}^{-1} \). The ionomer was dissolved in the solvent and then added to the catalyst powder. The resulting ink was stirred for one hour, placed in an ultrasonication bath for one hour, stirred overnight, sonicated again for another hour on the next day and after this, stirred until usage. The ink was applied onto a Freudenberg H23C8 gas diffusion media (4 x 4 cm) with an automated film applicator (ZAA 2300, Zehntner). The wet film thickness was determined by adjusting the gap-height on the doctor blade. It was set to 170 µm, which resulted in an average loading of \( \sim 1.3 - 1.5 \text{ mg cm}^{-2} \). Subsequently the samples were dried at 40 °C for 1 h and at 40 °C under reduced pressure for an additional hour. The Fe-N-C loading of the GDEs was measured by weighing (Sartorius Cubis®, 0.001 mg) the samples before and after the catalyst ink deposition and solvent evaporation.

2.2 GDE half-cell measurements

For the electrochemical measurements the GDE setup, electrochemical components and protocols presented in a previous work [22] were used. For more detailed information, see also Supporting Information. All experiments were carried out in minimum two batches of experiments in 1.0 M KOH (EMSURE®, Merck). Potentials were converted to reversible
hydrogen electrode (RHE) reference. Reference electrode potential against RHE was measured before every experiment with the same batch of electrolyte ($E_{Ag/AgCl} = 1.015 \pm 1.026 \ V_{RHE}$).

3. Results and Discussion

As described above, GDE half-cell experiments can be used to investigate and optimize single catalyst layer parameters in a fast and dedicated way. One of those parameters is the preconditioning or activation of the ionomer used in the catalyst layer. As the ionomer is present in the iodide form, an exchange of those iodide ions with hydroxide ions is required to obtain a hydroxide conductive polymer. [23] In the literature this activation is mostly done by immersing the alkaline electrode into 1 M KOH solution. But different activation protocols have been applied, also varying for different ionomers. Some research groups dip the electrode into the solution for $40 \pm 60 \ min$ [24, 25], $5 \pm 6 \ h$ [26], $24 \ h$ [27] or $48 \ h$ [28, 29]. However, the impact of immersion time for activation of alkaline electrodes has never been compared. Therefore, the samples were differentially preconditioned by immersing them into 1 M KOH for several intervals (2 x 20 min, 24 h, 48 h and 72 h) and subsequently washed thoroughly with ultrapure water to remove the impurities. In Figure 1 the polarization curves in $O_2$ and cyclic voltammograms (CV) in Ar of alkaline Fe-N-C electrodes are compared, which have been activated in 1 M KOH for various timeframes.

From the comparison it can be clearly seen that the activation procedure effects the electrode`s performance. While immersing the electrode into 1 M KOH for 2 x 20 min or 24 h does not lead to a significant improvement of the ORR performance, a pre-treatment of the alkaline GDEs in 1 M KOH for 48 h leads to best activities and mass transport properties. Therefore, it can be concluded that using Aemion™ ionomers 48 h of pre-conditioning in 1 M KOH are required to replace all the iodide ions with hydroxide ions and obtain the optimal hydroxide
conductivity. Surprisingly a drastic decrease in mass transport properties (but not intrinsic activity, see low-current region in Figure 1B) can be observed after 72 h of pre-treatment in 1 M KOH. This is probably due to catalyst or ionomer degradation in aqueous alkaline environment.

It has to be noted that this activation data is very specific to the alkaline ionomer which is used for GDE manufacturing. But the example shows how GDE experiments can be used to drastically accelerate electrode evaluation and activation. Whereas one single cell experiment almost takes one day of experiments, due to sophisticated MEA fabrication in alkaline, assembling, heating up, cooling down and disassembling of the cell, all the experiments for this activation study with repetitions could be conducted in less than four working days. Additionally, in half-cell experiments, factors such as anodic reaction limitations and carbonation of the alkaline ionomers [25, 30] can be excluded and therefore, the effect of one single parameter can be investigated exclusively.

After the impact of ionomer activation was investigated, the durability of the GDEs in alkaline environment as well as a possible impact of carbonation should be assessed. The carbonation of ionomers can lead to significant problems in AEMFC, mainly due to changes in conductivity behaviour and therefore mass transport limitations, but also electrocatalytic effects. [25, 30] Although carbonation of the ionomer mainly occurs when operating AEMFC with ambient air, it can already take place during the manufacturing process or sample storage as the exchange from OH\textsuperscript{−} to HCO\textsubscript{3}/CO\textsubscript{3}\textsuperscript{2−} occurs very fast. Nevertheless, it has been found out, that at high current densities all HCO\textsubscript{3}/CO\textsubscript{3}\textsuperscript{2−} are exchanged by OH\textsuperscript{−} again (“self-purging”). [30, 31] Therefore, the samples have first been tested towards their ORR activity up to 2 A cm\textsuperscript{−2} three times consecutively to check for any effect of ionomer carbonation on ORR activity determination. Subsequently they have been exposed to a load cycling protocol comprising 5000 triangular cycles between 0.6 and 1.0 V vs. RHE performed at a scan rate of 100 mV s\textsuperscript{−1}
in Ar-saturated electrolyte as used in previous degradation studies for non-PGM catalysts. [8] In Figure 2, the cathode’s activity towards ORR during this protocol is compared on the example of one 48 h activated sample. For reasons of clarity only one sample is displayed, but the trends shown in Figure 2 have been confirmed in all other experiments.

From Figure 2 two conclusions can be drawn: (i) There is no significant performance loss over 5000 degradation cycles. A slightly worse performance in comparison to the fresh samples can be attested, but the difference is minor and lays very close to the error bars of GDE testing at those high current densities. Therefore, the results from experiments in aqueous electrolyte can be confirmed [8] where Fe-N-C catalysts proved to be suitable for AEMFC applications in terms of durability. (ii) Carbonation does not play a significant role in GDE testing in alkaline environment, as the performance over the first consecutive polarization curves remains stable. This can be attributed to the constant purging of the adjacent aqueous electrolyte with Ar for about 20 min prior to the ORR experiments, when CVs in Ar are recorded. This shows, that GDE experiments are a suitable tool to evaluate the degradation of only one single catalyst layer. The impact of carbonation could be studied further in detail by purging different concentrations of CO₂ into the aqueous electrolyte and thereby facilitate the carbonation of the ionomer.

A catalyst system should always be compared to recent state-of-the-art developments to provide a comprehensive evaluation to the readers. Unfortunately, this is not always done appropriately, which can lead to difficulties in comparing results between different research groups. In Figure 3 we provide a comparison of the present results to the state-of-the-art performances measured in AEMFC experiments with: (i) the same catalyst, (ii) Fe-N-C catalysts in general and (iii) PGM catalysts.
In previous AEMFC experiments with the commercial Fe-N-C catalyst from Pajarito Powder (empty squares), Hossen et al. [26] have been able to achieve current densities up to approximately 700 mA cm$^{-2}$. In comparison with our present results with the same catalyst it can be seen that the onset of the polarization curve in the activation region is in a comparable range, even slightly higher at Hossen’s single cell experiments, probably due to different operating conditions in AEMFC testing (60 °C, 1.4 bar) and higher catalyst loading (3.5 vs. 1.6 mg$_{\text{Cat}}$ cm$^{-2}$). However, when exceeding low current densities severe reaction limitations can be observed in the high current region. There can be multiple explanations for that: (i) catalytic limitations at the Pt/C anode, (ii) mass transport-limitations in either the cathode’s or the anode’s catalyst layer, (iii) ohmic resistance in the membrane. And probably each of these factors plays a role in limiting the overall reaction. But in the conducted single cell experiment it is difficult to reveal which effect is the most severe, stressing again the advantage of the presented GDE half-cell method. The benchmark for Fe-N-C catalysts in alkaline environment was just recently set by Firouzjaie et al. [10] using a catalyst from Jaouen’s group [8]. The better performance of their catalyst system in comparison with the one presented in the present work can be explained by: (i) their use of a catalyst tuned for alkaline environment, instead of a commercial catalyst developed especially for acidic environment; (ii) improved mass-transport through catalyst layer engineering. Especially Mustain’s group has developed methods to drastically improve AEMFC performance through dedicated catalyst layer design. [10, 12] This is not only crucial working with non-PGM catalysts, but also in AEMFCs using PGM catalysts, confirmed by the fact that also the benchmark PGM system in AEMFC has been developed by the same group. [32] This again highlights the importance of catalyst layer parameters on AEMFCs performance. However, in single cell experiments it is often difficult to reveal the impact of those parameters and compare with studies from other groups as (i) both half-cell reactions and mass-transport phenomena in the membrane can play a crucial role on the cell’s performance and (ii) no standard operating procedure has been established.
yet, which leads to various different testing protocols and testing parameters complicating a valid comparison between different literature results. Therefore, standardized half-cell experiments using GDE setup could play an important role accelerating the understanding and optimization of catalyst layer parameters to further improve AEMFC performance.

4. Conclusions and Outlook

AEMFCs are a promising technology to allow the application of non-PGM catalysts to fuel cells due to their improved stability in alkaline media. Nevertheless, a lot of effort still has to be made in understanding the impact of catalyst layer parameters and subsequently optimizing them. For PEMFC this optimization took decades. Therefore, faster methods should be developed to help accelerating this research. GDE half-cell experiments are proposed as one of such methods to bridge the gap between TF-RDE and MEA testing. Both, RDE and MEA will still play an important role for catalyst synthesis evaluation (RDE) and evaluating operating parameters and full cell phenomena such as water management (MEA). Nevertheless, GDE experiments allow faster and dedicated evaluation of the effect of catalyst layer parameters on AEMFC performance. In this study this has been demonstrated on the examples of (i) ionomer activation, where immersing the GDE with commercial Aemion™ ionomer in 1 M KOH for 48 h leads to the best electrode performance, and (ii) Fe-N-C catalyst stability, where the commercial Pajarito Powder catalyst showed high stability during 5000 load cycles between 0.6 – 1.0 V vs. RHE. Due to the simplicity of the setup this experiments could be conducted in a fraction of the time necessary for MEA testing. The obtained results have been compared to benchmarks in literature. This comparison revealed the importance of understanding and optimizing catalyst layer parameters, such as catalyst loading, type of ionomer, ionomer content and distribution, and the morphology of the catalyst layer. Further work will be dedicated to understanding the impact of those parameters on both anode and cathode performance. Also
the effect of carbonation of the ionomer will be addressed by using different CO$_2$ saturation in
the aqueous electrolyte.

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Captions

Figure 1: Comparison of polarization curves of alkaline Fe-N-C cathodes (Pajarito Powder, 1.5 – 1.7 mg\text{cat} cm\textsuperscript{-2}) with Aemion\textsuperscript{TM} ionomer and different pre-treatment procedures applied. Tested in 1.0 M KOH. A: Cyclic voltammograms (100 mV s\textsuperscript{-1}) with Ar. B&C: Tafel plot and polarization curve for the oxygen reduction reaction with pure O\textsubscript{2}.

Figure 2: Comparison of consecutive polarization curves of alkaline Fe-N-C cathodes (Pajarito Powder, 1.6 mg\text{cat} cm\textsuperscript{-2}) with Aemion\textsuperscript{TM} ionomer (48 h activated in 1 M KOH) before (blue) and after (red) 5000 degradation cycles (100 mV s\textsuperscript{-1}, 0.6 – 1.0 V vs. RHE, Ar). Tested in 1.0 M KOH with O\textsubscript{2}. Inset: Cyclic voltammograms of same samples in Ar.

Figure 3: Comparison of the present work (blue squares, 1.6 mg\text{Fe-N-C} cm\textsuperscript{-2}) with benchmark results of AEMFC experiments with (a) the same commercial catalyst from Pajarito Powder (empty squares, Fe-N-C cathode: 3.5 mg\text{Fe-N-C} cm\textsuperscript{-2}, Pt/C anode: 0.2 mg\text{Pt} cm\textsuperscript{-2}) [26], (b) Fe-N-C catalysts (light blue triangles, Fe-N-C cathode: 0.9 mg\text{Fe-N-C} cm\textsuperscript{-2}, PtRu/C anode: 0.6 mg\text{PtRu} cm\textsuperscript{-2}) [10] and (c) Pt/C catalyst (yellow, Pt/C cathode: 0.56 mg\text{Pt} cm\textsuperscript{-2}, PtRu/C anode: 0.986 mg\text{PtRu} cm\textsuperscript{-2}) [32].
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