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A standard electrolyzer test cell design for evaluating catalysts and cell components for anion exchange membrane water electrolysis



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ABSTRACT

Anion exchange membrane water electrolysis technology is an environmentally friendly method for hydrogen production without carbon dioxide emissions. The development of this technology is hindered by the sluggish rates of the hydrogen and oxygen evolution reactions, demanding the rational design of electrocatalysts for these processes. Although numerous hydrogen and oxygen evolution electrocatalysts are reported in the literature, there is currently no standard set of conditions or cell design under which they can be tested; variance in testing conditions and water electrolyzer components from one study to another therefore hampers a full and coherent comparison of novel electrocatalysts for ration exchange membrane water electrolysis. Herein, a standard water electrolyzer test cell was designed and optimized, such that this test cell can be adopted as a universal framework for evaluating novel electrocatalysts (and indeed other cell components) for anion exchange membrane water electrolysis. The performance of this water electrolyzer was evaluated using a commercial Pt/C hydrogen evolution reaction electrocatalyst, with an optimized cell found to deliver current densities of 1.40 A cm⁻² and 2.74 A cm⁻² at cell voltages of 2.0 V and at 60 °C using Fumasep FAA-3-50 and Sustainion® X37-50 membranes, respectively. Guidance for assembling and operating the water electrolyzer is provided, along with computer-aided design drawings of the electrolyzer and its components to allow others to construct their own identical cells for benchmarking catalysts and other cell components for anion exchange membrane water electrolyzer is provided, along with computer-

1. Introduction

Hydrogen is an attractive replacement for fossil fuels, which can be used in fuel cells or internal combustion engines, delivering high energy density (\approx 143 MJ kg⁻¹) and releasing water as the only by-product [1]. The vast majority of the world's hydrogen is currently produced by steam-methane reforming and the water gas-shift processes [2]. These H₂ production processes are carbon-intensive, fossil-fuel-based methods, operating at around 800–1000 °C; therefore, other environmentally friendly and energy-efficient hydrogen production methods must be adopted for producing hydrogen sustainably.

Electrochemical water splitting is a sustainable and environmentally friendly alternative to the steam-methane reforming and water gas-shift methods for hydrogen production, by which hydrogen is ultimately derived from water with essentially zero carbon footprint [3]. Water electrolyzers that operate at temperatures below 100 °C and at ambient pressure can be categorized into acidic and alkaline systems based on the type of electrolyte employed and can be further divided into

zero-gap and finite-gap water electrolyzers [4,5]. The terms finite-gap and zero-gap relate to the distance between the membrane/diaphragm separating the cathode and anode electrodes. Zero-gap water electrolysis systems (where the anode and cathode catalysts are in direct contact with the membrane separator, with no interstitial liquid electrolyte between the catalysts) outperform finite-gap water electrolysis systems in terms of performance. For example, at 60-80 °C, finite-gap alkaline water electrolyzers generally achieve maximal current densities on the order of 0.2-0.4 A cm⁻² compared to zero-gap anion exchange membrane water electrolyzers (0.2-1.0 A cm⁻²) and zero-gap proton exchange membrane water electrolyzers $(0.6-2 \text{ A cm}^{-2})$ [6]. The superior performance of zero-gap designs is due in large part to the much lower internal resistance inherent in such cells, which in turn is due to the considerably shorter anode-cathode distances than those found in finite-gap designs. Zero-gap electrolyzer designs are therefore seen as a most promising approach for mass production of hydrogen [4,5].

The deployment of anion exchange membrane water electrolyzers is seen as especially attractive since nonprecious materials, such as Ni and

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Fig. 1. A schematic illustration of the anion exchange membrane water electrolyzer developed in this study. "AEM" = anion exchange membrane.

Fe, can be used due to the less corrosive alkaline environment compared to that found in proton exchange membrane water electrolyzers (the latter generally requiring expensive precious metal catalysts) [7]. Nevertheless, the rational design of stable electrocatalysts with high activity towards hydrogen evolution reaction (HER) and oxygen evolution reaction (OER) is still critical due to the sluggish electrode reactions (particularly the OER), which in turn results in a high overpotential requirement (η) . Overpotential here means the additional potential required to drive the water splitting process over and above that predicted on the basis of thermodynamics [8]. Numerous HER and OER electrocatalysts have been investigated in anion exchange membrane water electrolyzers; however, studies to date have operated diverse water electrolyzers, with diverse components and with different operating conditions [6]. Consequently, the systematic comparison of different electrocatalysts or other cell components such as membranes and gas diffusion layers is hampered, as there is no universally-accepted benchmark anion exchange membrane electrolyzer device to act as a starting point for innovation, with each study instead suggesting its own baseline (where the other cell components beyond those specifically under test may or may not be comparable to those in other studies) [9, 10]. Thus, constructing and characterizing a benchmark water electrolyzer to be adopted in anion exchange membrane water electrolysis research for investigating novel electrocatalysts and other cell components is crucial.

In this study, an anion exchange membrane water electrolyzer system was built (Fig. 1) using different configurations consisting of stainless steel fiber paper as an anode gas diffusion layer/anode catalyst and two cathode gas diffusion layers, and Fumasep FAA-3-50 or Sustainion® X37-50 Grade RT as the anion exchange membranes. These different configurations were investigated by electrochemical techniques using 1 M KOH, and the performance of the electrolyzer was evaluated using a commercial HER (Pt/C) electrocatalyst, and using FAA-3-50 and Sustainion X37-50 membranes. Through these efforts, a system consisting of affordable, available, and durable materials was developed as a benchmark anion exchange membrane water electrolyzer, to be used as a universal basis for testing electrocatalysts and other cell components, allowing a coherent comparison and evaluation of new anion exchange membrane water electrolyzers. Moreover, the designs of the cell components developed in this study are available as open-access computer-aided design (CAD) files, which can be downloaded and modified by the reader from the University of Glasgow's open access data repository (see also the Supporting Information).



Fig. 2. A diagram of the fully-assembled flow cell developed in this work.

2. Experimental

2.1. Water electrolyzer components, setup, and assembly

A flow cell (Fig. 2) with an active area of 13 cm² (3.6 cm \times 3.6 cm, although the gas diffusion layers were cut to a size of 3.7 cm \times 3.7 cm, to prevent any possible leakage) was assembled as follows with compression force of 5.65 Nm on each bolt (see Supporting Information and Fig. S1a for a full description and diagram of the cell assembly process). A pair of titanium plates (thickness: 3 mm) were used as current collectors, each with a serpentine flow design containing six channels through which the electrolytes were supplied to the water electrolyzer (and through which the gases evolved were removed). Deionized water (resistivity = 15 M Ω -cm) was used across all experiments for making 1 M KOH. This 1 M KOH was supplied to both the anode and the cathode sides simultaneously using Norprene tubing (Merck Life Science UK Limited, 4.78 mm internal diameter) and using two Fisherbrand GP1100 General Purpose peristaltic pumps, both using a flow rate of 40 mL min⁻¹. Two commercial membranes, Fumasep FAA-3-50 (supplied by FuelCellStore) and Sustainion® X37-50 Grade RT (supplied by Dioxide Materials) were used as anionic exchange membranes. Stainless steel fiber paper was used as an anode gas diffusion layer. At the cathode, 60% Pt on Vulcan carbon (the Pt loading mass on carbon was 0.5 mg cm⁻²) with Nafion dispersion - PTFE-treated (5 wt%) deposited on carbon cloth (W1S1011) or 0.5 mg cm⁻² 60% Pt on Vulcan carbon (the Pt loading mass on carbon was 0.5 mg cm^{-2}) with Nafion dispersion - PTFE-treated (5 wt%) deposited on Sigracet 22 BB carbon paper were used as cathode gas diffusion layers. All the gas diffusion layers were supplied by FuelCellStore, except the stainless steel fiber paper, which was supplied by Dioxide Materials. Polytetrafluoroethylene (PTFE) gaskets (FuelCellStore) with different thicknesses (0.127 mm, 0.254 mm, and 0.508 mm) were used to prevent electrolyte leakage and any possible direct contact of the bipolar plates. The temperature inside the flow cell was controlled by heating the water reservoirs using hot plates, and internal cell temperature was monitored using K-type thermocouples inserted into the outlet and inlet of the cathodic and anodic sides. Temperature measurements were monitored by a Pico TC-08 data logger and PicoLog software. Guidance for assembling and

Table 1

The experimental parameters used in the EIS tests.

DC Potential (V)	2.0
AC Potential (mV rms)	100
Initial Frequency (kHz)	10
Final Frequency (mHz)	500
Points/Decade	6.0



Fig. 3. The equivalent circuit model used in the EIS analysis. Circuit components are defined in the main text.

operating the flow cell, and a table (Table S1) of the technical specifications of the gas diffusion layers and membranes can be found in the Supplementary information (Fig. S1a–m). Furthermore, computeraided design (CAD) drawing files of the water electrolyzer components were created to allow other researchers to manufacture an identical water electrolyzer. These are available for open access download at https://doi.org/10.5525/gla.researchdata.1356.

2.2. Electrolyte characterization

Any leached metal residuals were detected in the electrolyte reservoirs using inductively coupled plasma optical emission spectroscopy (ICP-OES) after each stability test; after neutralizing the 1 M KOH electrolytes with HNO₃ and diluting them 5-fold with 5% HNO₃. The dimensions of the gas diffusion layers (3.7 cm \times 3.7 cm) before electrolysis were confirmed using a digital caliper and a ruler.

2.3. Anion exchange membrane preparation

The Fumasep FAA-3-50 and Sustainion X37-50 membranes were delivered in a dry form and were pre-treated before use, following the manufacturers' instructions. The membranes were submerged in 1 M KOH at room temperature (around 20 °C) for at least 24 h to convert them into the OH-form. They were stored under 1 M KOH in closed containers to prevent dryness and CO_2 contamination, in which CO_3^{-1} formation might influence the ionic conductivity.

2.4. Electrochemical characterization

All the electrochemical tests were carried out using the abovementioned flow cell at 60 °C (unless otherwise mentioned), with a flow rate of 40 mL min⁻¹, using a BioLogic SP-150 potentiostat with an 80 A booster. The data were acquired and analyzed using EC-Lab[@] software (version 11.43). In addition, the assembled flow cell and components were subject to a pre-treatment prior to collecting data on each membrane electrode assembly configuration by applying a fixed potential of 2.0 V for 1 h at 40 °C.

The polarization curves were measured over a potential range of 0 V to 2.0 V at a scan rate of 10 mV s⁻¹, and the chronopotentiometric tests were performed at 1.0 A cm⁻² at 60 °C.

The electrochemical impedance spectroscopy (EIS) analysis was performed under the experimental parameters shown in Table 1, unless otherwise mentioned. The Nyquist plots generated by the EIS measurements were fitted using an equivalent circuit model (Fig. 3), which was applied according to the methods developed in the literature [11–14]. In the equivalent circuit, L1 (inductor) refers to the connectors and cables used while taking measurements, R1 represents the ohmic resistance of the flow cell components, R2 and R3 depict the cathodic and anodic charge transfer resistances, respectively, and the Q2 and Q3 represent the constant phase elements of the cathodic and anodic processes, respectively.



Fig. 4. (a) Polarization curves and (b) Nyquist plots (at 60 °C only) of the water electrolyzer using stainless steel fiber paper at the anode and 0.5 mg cm⁻² Pt/C deposited on two different cathode gas diffusion layers: carbon cloth and Sigracet carbon paper.



Fig. 5. The chronopotentiometric evaluation of the water electrolyzer at 1 A cm⁻² using stainless steel fiber paper as the anode catalyst and 0.5 mg cm⁻² Pt/C deposited on two different gas diffusion layers at the cathode at 60 °C.

3. Results and discussion

3.1. Characterization of the generic alkaline water electrolyzer

The performance of the water electrolyzer was tested using stainless steel fiber paper at the anode and 0.5 mg cm^{-2} Pt/C (HER electrocatalyst), with 5 wt% PTFE, 35% Vulcan carbon, and Nafion dispersion as an ionomer binder (the use of Nafion as an ionomer/binder in anion exchange membrane water electrolysis has considerable precedent [15-18]), deposited on two different cathode gas diffusion layers: carbon cloth and Sigracet carbon paper. A Fumasep FAA-3-50 membrane was employed. As shown in Fig. 4a, excellent overall high performance was achieved using both cathode gas diffusion layers. The Pt deposited on carbon cloth configuration (1.40 A cm⁻² at 2 V cell potential and at 60 °C) outperformed the Pt deposited on Sigracet carbon paper configuration (1.24 A cm⁻² at 2 V cell potential and at 60 °C). The difference in performance between the two configurations was attributed to the physical structure of the two gas diffusion layers. At high current densities, the carbon paper-based gas diffusion layers show inferior performance compared to the carbon cloth-based gas diffusion layers due to the more torturous structure of carbon papers when compared with carbon cloth [19]. The high tortuosity causes more mass transport limitations due to issues with removing water and evolved gases, leading to bubble formation blocking the catalyst active sites [20].

The EIS analysis of both configurations was carried out in the potentiostatic mode at 2.0 V using a varied frequency from 10 kHz to 500 mHz (6 points per decade) with a sinusoidal excitation signal of 100 mV rms. As shown in the Nyquist plot (Fig. 4b), the EIS analysis agreed with the linear sweep voltammetry results. The configuration where carbon cloth was used as the cathode gas diffusion layer achieved slightly lower ohmic resistance ($\approx 211 \text{ m}\Omega \text{ cm}^2$) compared to the



Fig. 6. (a) Polarization curves and (b) Nyquist plots (at 60 °C only) of the water electrolyzer using a Sustainion membrane, stainless steel fiber paper at the anode and 0.5 mg cm⁻² Pt/C deposited on two different cathode gas diffusion layers: carbon cloth and Sigracet carbon paper.



Fig. 7. The chronopotentiometric evaluation of the water electrolyzer at 1 A cm⁻² using a Sustainion membrane, stainless steel fiber paper as the anode catalyst, and 0.5 mg cm⁻² Pt/C deposited on (a) Sigracet carbon paper and (b) carbon cloth at 60 °C.

configuration using Sigracet carbon paper ($\approx 227 \text{ m}\Omega \text{ cm}^2$). The cathodic and anodic charge transfer resistances achieved by the configuration using carbon cloth were lower than using Sigracet carbon paper as well; 1.07 m Ω cm² (carbon cloth) and 1.16 m Ω cm² (Sigracet carbon paper), and 30.7 m Ω cm² (carbon cloth) and 55.3 m Ω cm² (Sigracet carbon paper), respectively.

The stability of the water electrolyzer was evaluated by a galvanostatic electrolysis at 1.0 A cm⁻² for 25 h at 60 °C (Fig. 5). An increase in the required cell potential from 1.97 V to 2.14 V (Sigracet carbon paper) and from 1.92 V to 2.07 V (carbon cloth) was observed. The increase in the required cell potential most likely occurred due to a passivation layer formed on the stainless steel gas diffusion layer surface over time, decreasing the electrical conductivity of the interface between the stainless steel fiber paper and the membrane (i.e. the electrolyte) [6]. Effects on the cell's performance by more extensive corrosion of the stainless steel was excluded since consistently low Fe concentrations (\approx 0.3 ppm) were detected by ICP-OES in the anolyte streams of all the various configurations, with no detectable Ni content in these anolytes [21]. No Pt content was detected in the carbon cloth and Sigracet carbon paper.

3.2. The effect of the membrane on the water electrolyzer

The performance of the water electrolyzer with a different membrane was evaluated employing the same aforementioned two configurations, but using the Sustainion X37-50 membrane instead of Fumasep FAA-3-50 as the anion exchange membrane. As shown in the polarization curves (Fig. 6a), the performance of the configuration using Pt deposited on carbon cloth (2.74 A cm⁻² at 2 V cell potential and at 60 °C) gave superior performance compared that obtained using the Pt deposited on Sigracet carbon paper (2.13 A cm⁻² at 2 V cell potential and at 60 °C).

As shown in the Nyquist plot (Fig. 6b), lower ohmic resistance was obtained by using the carbon cloth configuration ($\approx 124 \text{ m}\Omega \text{ cm}^2$) than the Sigracet carbon paper configuration ($\approx 132 \text{ m}\Omega \text{ cm}^2$). The cathodic



Fig. 8. Nyquist plots of the water electrolyzer before and after electrolysis for 205 h at 1 A cm⁻² and at 60 °C using a Sustainion membrane, stainless steel fiber paper at the anode, and 0.5 mg cm⁻² Pt/C deposited on carbon cloth.

and anodic charge transfer resistances achieved were 0.84 m Ω cm² (carbon cloth) and 0.938 m Ω cm² (Sigracet carbon paper) and 17.1 m Ω cm² (carbon cloth) and 27.5 m Ω cm² (Sigracet carbon paper), respectively; agreeing with the linear sweep voltammetry results.

The effect of using a Sustainion membrane on the water electrolyzer's stability was investigated by a galvanostatic electrolysis at 1.0 A cm⁻² for 25 h at 60 °C using the Sigracet carbon paper configuration (Fig. 7a) and a longer test for 205 h and at 60 °C using the carbon cloth configuration (Fig. 7b), since this configuration achieved the best performance. In the stability test of the Sigracet carbon paper configuration, the potential required cell potential to deliver 1 A cm⁻² increased from an initial 1.79 to 2.01 V at 25 h. Meanwhile, for the carbon cloth configuration, the required cell potential to deliver 1 A cm⁻² increased from 1.78 V and stood at a peak of 2.10 V after 205 h. As confirmed by ICP-OES analysis of the electrolytes employed in both configurations after each stability test, the Fe concentration was around 0.3 ppm and no Pt was detected in the catholyte after the stability test of both configurations. These concentrations detected by the ICP-OES were similar to the concentration detected in the cases where the FAA-3-50 membrane was used, indicating a consistent effect on the water electrolyzer performance by the anode gas diffusion layer (i.e. the stainless steel fiber paper). Again, the increase in cell voltage required to maintain a current density of 1 A cm⁻² can be attributed to the decrease in the electrical conductivity at the anode side caused by passivation of the stainless steel gas diffusion layer. The drop in electrical conductivity was confirmed by performing EIS analysis after the stability test for 205 h (Fig. 8), and comparing this to the ohmic resistance of the system before (124 m Ω cm^2) and after (232 m Ω cm²) electrolysis. Over longer electrolysis durations, however, stainless steel substrates in alkaline media show excellent electrocatalytic activity towards OER, since such substrates develop a rich metal oxide layer composed of mixed oxides of Ni, Fe and Cr, acting as OER active sites, at high anodic potential under OER operation, as reported in the literature [22]. Consequently, the effects of passivation on the surface of stainless steel fiber paper seem to be offset somewhat over longer-term stability tests.

According to the combined linear sweep voltammetry, EIS, and stability tests results, using the Sustainion membrane allowed higher current densities to be achieved, with lower resistances, and longer stability. The Sustainion X37-50 membrane achieved the required combination of high current densities and stable performance. This higher performance can be attributed to the higher OH⁻ conductivity of the Sustainion membrane of 115 mS cm⁻¹ at 60 °C compared to the OH⁻ conductivity of FAA-3-50 of 55 mS cm⁻¹ at 100 °C [23,24]. Although higher performance was obtained using the Sustainion membrane, it must be noted that the Fumasep FAA-3-50 membrane is a cost-effective alternative for short-term tests, since the price of the FAA-3-50 membrane (\$63.00 for 20 cm \times 30 cm) is almost four times lower than the price of Sustainion® X37-50 membrane (\$255.00 for 22 cm \times 28 cm) at time of writing. However, for long-term stability tests (>24 h) it is recommended to use the Sustainion membrane due to its superior

Table 2

Comparison of the performance of various anion exchange membrane water electrolyzers at 60 $^\circ\text{C}.$

Anion exchange membrane	Anode (catalyst loading: mg cm ⁻²)	Cathode (catalyst loading: mg cm ⁻²)	Current density (A cm ⁻²)	Ref.
Sustainion X37- 50	Stainless steel	Pt/C (0.5)	2.74 at 2.0 V	This work
FAA-3-50	Stainless steel	Pt/C (0.5)	1.40 at 2.0 V	This work
FAA-3-50	IrO ₂ (4)	Pt/C (0.4)	1.5 at 1.9 V *	[26]
FAA-3-50	IrO ₂ (2)	Pt/C (2)	1.0 at 1.8 V	[27]
Sustainion Grade T	IrO ₂ (2)	Pt/C (0.5)	1.8 at 2.0 V	[28]
Sustainion X37- 50	NiFe ₂ O ₄ (2)	NiFeCo (2)	1.0 at 1.9 V	[18]
Sustainion X37- 50	NiFe ₂ O ₄ (1.8)	Raney Ni (2.7)	0.744 at 1.8 V	[29]
Sustainion Grade T	NiFe ₂ O ₄ (1.8)	Raney Ni (14.5)	0.84 at 1.8 V	[29]
Sustainion X37- 50	NiFe ₂ O ₄ (2)	NiFeCo (3)	1.0 at 1.9 V	[30]

*At 70 °C.

durability [18,25]. A list of the performance of different anion exchange water electrolyzers using different components from the literature is shown in Table 2.

4. Conclusions and future directions

Herein, an anion exchange membrane water electrolyzer was constructed and optimized as a universal benchmark for testing new electrocatalysts and other cell components in a standardized fashion. When using stainless steel fiber paper as a combined anode gas diffusion layer and OER electrocatalyst and Pt/C as an HER electrocatalyst, the benchmark water electrolyzer showed excellent performance of 2.74 A $\rm cm^{-2}$ and 1.40 A $\rm cm^{-2}$ at 2.0 V and at 60 $^{\circ}\rm C$ using carbon cloth and Fumasep FAA-3-50 and Sustainion® X37-50 membranes, respectively. The water electrolyzer showed a moderate decline in performance over extended duration testing, which was primarily attributed to the formation of a mixed-oxides passivation layer on the stainless steel fiber paper surface over extended time under high anodic polarization, decreasing the electrical conductivity of the stainless steel fiber paper. Further work to aid in the standardization of procedures for anion exchange membrane water electrolyzers that should be addressed include optimization of electrocatalyst deposition on gas diffusion layers or membranes. Together with the present study, such standardization will enable innovation in, and proper comparison of, anion exchange membrane water electrolyzers for the mass production of hydrogen in a sustainable manner.

Appendix A. Supplementary data

Supplementary data associated with this article (component designs, assembly procedure and Table S1) can be found at https://doi.org/10.5525/gla.researchdata.1356

CRediT authorship contribution statement

Abdulhai H. Faqeeh: Conceptualization, Methodology, Data curation, Investigation. Mark D. Symes: Conceptualization, Supervision, Writing – review & editing.

Declaration of Competing Interest

The authors declare no conflicts of interest.

Data availability

Data will be made available on request.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.electacta.2023.142030.

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