

Supporting Information

Beyond Catalysis and Membranes: Visualizing and Solving the Challenge of Electrode Water Accumulation in AEMFCs

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Experimental

Ethylene Tetrafluoroethylene (ETFE-BTMA) Membrane Synthesis

The ETFE-BTMA membrane was prepared from pre-formed ETFE films (25 μm thickness, Nowofol Kunststoffprodukte GmbH (Germany)) using the peroxidation (pre-irradiation in air) method previously reported.¹ The ETFE films were subjected to electron-beam irradiation in air to 30 kGy total absorbed dose (using a 4.5 MeV Dynamatron Continuous Electron Beam Unit at Synergy Health, South Marston, UK). As the irradiation step is performed in air, immediate reaction of the radicals that are formed with O_2 molecules leads to the creation of peroxide and hydroperoxide groups on the polymers. The peroxidated ETFE films then act as a solid-state free-radical initiator for the subsequent graft polymerization step. After irradiation, the films were transported back to the laboratory in dry ice before they were stored in a freezer at -40°C (the peroxide groups are stable for around 6 months at this temperature²).

For the grafting step, the electron-beamed films (ca. 15 cm $\times$ 15 cm) were immersed in an aqueous dispersion of VBC (vinylbenzyl chloride, 5%vol. mixture of 3- and 4-isomers; 500 – 100 ppm *tert*-4-butylcatechol and 700 – 1100 ppm nitromethane inhibitors, Sigma-Aldrich and used without the removal of inhibitors) in sealed vessels along with addition of dispersant (1%vol. 1-octyl-2-pyrrolidone, Sigma-Aldrich). The solutions were purged with N_2 for 2 h before the vessel was sealed and heated at 70°C . After the reaction period, the films were removed from the grafting mixture and washed in toluene (reagent grade, Fisher Scientific); this process is employed to remove excess unreacted VBC and any poly(VBC) homopolymer (not bound to the ETFE base material) that may be present. The resulting intermediate ETFE-*g*-poly(VBC) films were subsequently dried at 70°C for 5 h in a vacuum oven to remove all traces of solvent.

To quaternize, the intermediate films were then submerged in the aqueous TMA solution (aqueous trimethylamine solution, 45 wt%, Sigma-Aldrich) at ambient temperature for 24 h, then washed in DI water (ultra-pure deionized water with a resistivity of 18.2 $\text{M}\Omega\text{ cm}$), and finally heated in fresh DI water; this procedure was adopted to remove any excess TMA from the resulting quaternized ETFE-*g*-poly(vinylbenzyltrimethylammonium) anion exchange membranes (AEMs). Final conversion to the chloride-anion-form ETFE-BTMA AEMs was conducted as follows: the as-synthesized AEMs were submerged in aqueous NaCl (1 M) for 15 h with one change of NaCl solution during this period to ensure complete ion-exchange. The resulting AEMs were then soaked in water to remove any excess NaCl-derived co- and counter-ions. The final desired radiation-grafted AEM(Cl^-) films were stored in water until required and were not allowed to dry out at any point before subsequent measurements/experiments were conducted.

Perfluorinated Anion-exchange Membrane (PFAEM) synthesis

Synthesis of the perfluorinated anion exchange membrane (PFAEM Gen 2) has been previously described in detail³ and is summarized herein. A perfluorosulfonic acid precursor in the sulfonamide form was acquired from 3M with equivalent weight (in sulfonic acid form) of 825 g/mol. A linking molecule, iodohexyltrimethyl ammonium iodide, was synthesized by adding an excess of trimethylamine to 1,6-dibromohexane in tetrahydrofuran at 0°C and stirring overnight at 23°C. The precipitated product was filtered, washed repeatedly with hexane, and dried under vacuum. This intermediate bromide salt was then converted into iodide form by ion exchange with an excess of potassium iodide in acetonitrile under reflux for 12 h. The resulting iodide salts were precipitated with chloroform, filtered, and dried under vacuum.

PFAEM Gen 2 was prepared by adding an excess (~1.3x) of the iodohexyltrimethyl ammonium iodide linker to a 0.05 M solution of sulfonamide precursor polymer in dimethylformamide and an excess of potassium carbonate. The reaction mixture was stirred at 120°C for 48 h. 4 equivalents of iodomethane were then added to the intermediate (zwitterionic) polymer at room temperature. The reaction mixture was then stirred for 12 h at 100°C in a pressurized glass flask. The reaction mixture (after cooling to 23°C) was poured into a 5-fold volume excess of 70% methanol solution. Following 5 h of continuous stirring at 23°C, the precipitated ionomer was filtered, washed 3 times with 70% methanol solution, and dried at 50°C under vacuum.

Membranes (iodide counterion form) were prepared by dissolving PFAEM at 120°C for 2 h in a 5% w/w solution in dimethylacetamide, then hand spreading over a 6" x 6" square of fiberglass-reinforced Teflon and evaporating overnight at 60°C. Resultant membranes were annealed in a hot press at 160°C and 800 psi for 10 minutes, then converted to the hydroxide ion form by immersion in 1 M NaOH for 1 hour at 23°C (exchanged 2x).

Anion-exchange ionomer (AEI) powder synthesis

The synthesis of the AEI powder was reported previously⁴ and is summarized here. ETFE powder (Z8820X, AGC Chemicals Europe) with a particle size of 20 – 30 µm was peroxidated in air using an electron-beam with a total absorbed dose of 70 kGy. The resulting “activated” powder was then submerged in a solution containing VBC, 2-propanol (reagent grade, Sigma-Aldrich), and Surfadone LP-100 (ISPCorp) with a volume ratio of 1.00:3.95:0.05. The mixture was purged with N₂ for 2 h, and then sealed and heated for 72 h at 60°C. The powder was recovered by filtration, washed with toluene (reagent grade, Fisher Scientific), and dried at 50°C under vacuum, resulting in ETFE-*g*-poly(VBC) grafted powders.

The powder was quaternized by submersion in an aqueous TMA solution (TMA, 50%wt. in water, Acros Organics) for 5 h at ambient temperature. The resulting powder was washed 5 times with DI water, and then heated in DI water for 18 h at 50°C. After a further water wash step (5 × DI water) the powder was dried for 5 d at 40°C under vacuum. The final resulting anion-conducting AEI powder (in the most chemically stable Cl⁻ form), that was used in all of the electrodes in this manuscript, possessed an IEC of 1.24 ± 0.06 mmol g⁻¹ (*n* = 3).

Materials and gas diffusion electrode (GDE) preparation

First, the AEI powder was ground with a mortar and pestle for 10 min to reduce the amount of aggregated particles. Next, 100 – 150 mg of one of two carbon supported catalysts was added to the AEI in various ratios (approx. 20%wt., detailed in Table 1 and Table S1) along with 1 mL DI water: 40% Pt on Vulcan carbon (Alfa Aesar HiSPEC 4000, Pt nominally 40%wt., supported on Vulcan XC-72R carbon), or 40%wt. Pt + 20%wt. Ru on vulcan carbon (Alfa Aesar HiSPEC 10000, Pt nominally 40%wt., and Ru, nominally 20%wt., supported on Vulcan XC-72R carbon). For some

anode electrodes various amounts (loadings detailed in Table 1 and Table S2) of catalyst free vulcan carbon (XC-72R, Cabot) was added to the mixture in order to increase the macro porosity and water capacity of the catalyst layers. This carbon addition was performed with catalyst free carbon to maintain the accurate electrode comparisons with identical catalysts. The catalyst-AEI mixture was then ground with a mortar and pestle for 10 min, a length of time that was selected because it invariably produced a visually and texturally homogenous slurry, suggesting that no AEI agglomerates remained. Then, 2 mL of 2-propanol (Fisher Chemical Optima) was added to the mortar and ground for a further 5 min, after which the catalyst slurry was completely transferred to a LDPE vial, and combined with an additional 7 mL of 2-propanol to produce a low viscosity ink. Each ink was homogenized in an ice-chilled ultrasonic bath (Fisher Scientific FS30H) for 60 min, maintained between 5 °C and 10 °C.

The resulting catalyst inks were used to fabricate gas diffusion electrodes (GDEs) by hand spraying onto a larger area gas diffusion layer (GDL, Toray TGP-H-060 with 5% PTFE wetproofing (0% PTFE wetproofing was used in previous work given in Fig. S2 and Table S1)) with an Iwata Eclipse HP-CS using 15 psig N₂ (Airgas Ultra High Purity). 5 cm² GDEs were then cut from the larger sprayed electrode for use in the cell hardware. Generally, the target total platinum group metal loading of the resulting GDEs was $0.6 \pm 0.1 \text{ mg cm}^{-2}$, and the actual catalyst loading for each data set is given in the tables and/or Fig. captions.

MEA assembly and Anion Exchange Membrane Fuel Cell (AEMFC) testing

Prior to MEA and fuel cell assembly, the GDEs and AEMs were separately immersed in aqueous KOH (1 M, Fisher Chemical, pellets/certified ACS) for 60 min, replacing the solution every 20 min to ensure complete ion-exchange. During this hydration and ion-exchange process, no substantial electrocatalyst or AEI particles were observed to wash off the GDE surface, showing adequate adhesion of the catalyst layers onto the GDLs. Excess aqueous KOH and water were removed from the electrodes and membrane with a laboratory cloth prior to assembly. Each set of GDEs and AEM were pressed together in-cell to form the membrane electrode assembly (MEA) with no prior hot pressing. The MEAs were secured in 5 cm² Fuel Cell Technologies hardware between two single pass serpentine flow graphite plates using 6 mil (150 μm) PTFE gaskets with 20% pinch (5.1 N·m torque). An 850E Scribner Fuel Cell Test Station was used for all testing. H₂ and O₂ gas feeds were supplied to the anode and cathode, respectively, at various flow rates and dew points without back-pressurization (*ca.* 1 atm absolute). Throughout this manuscript, the dew points of the supplied gases will be identified in an *Anode/Cathode* format with the dew points reported in °C, *e.g.* 57/55 would correspond to an anode dew point of 57°C and a cathode dew point of 55°C. The cell temperature was held constant at 60°C $\pm$ 0.5°C for all experiments. The temperature of the heated gas follow lines between the fuel cell test stand and the cell were maintained at 5°C above the respective gas dew points.

All of the polarization curves shown were collected under potentiometric control at a scan rate of 10 mV·s⁻¹. Linear sweeps – in lieu of point-by-point collection was used in order to better tease out flooding issues under water starved and flooded conditions. When the water management issues were well controlled, there is no significant difference between point by point and linear sweep polarization curves, which is shown in previous work for multiple high performing cells.⁵ Additionally, tests were repeated after multiple hours and varying the testing conditions to ensure stability, recoverability, and repeatability.

Recent AEMFC Progression

Interest AEMFCs has erupted in recent years, which can be seen both from the both in publications (Fig. S1) and citations.

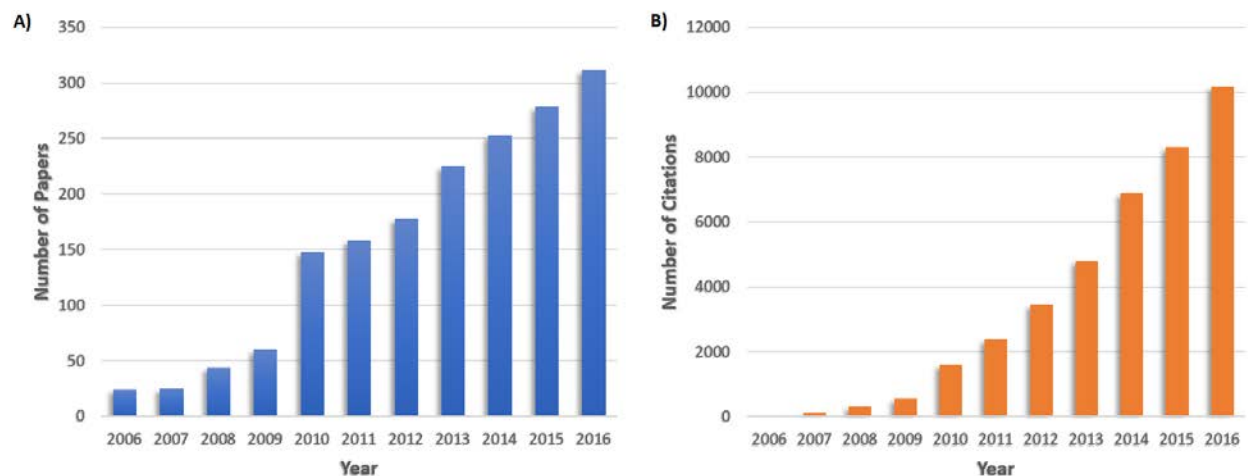


Fig. S1. Report of the number of publications (A) and citations (B) for the “Anion Exchange Membrane Fuel Cell” from 2006-2016 showing the explosive growth in interest in AEMFCs since the turn of the century, and, more specifically, since 2006.⁶

There has also been a significant increase in cell performance (Table S1 and Fig. S2) in recent years. Several key factors have led to step changes in achievable power density and current density. One of the most significant and surprising findings has been that not only is it not necessary to run the feed gas streams at full humidity, but it is extremely detrimental to performance – especially at higher currents. This finding runs counter to what was widely believed, as both the cathode and the membrane need significantly more water than in proton exchange membrane fuel cells (PEMFCs). The power and current density gains resulting from operating at optimized dew points (OD) vs full humidity (FH) can be observed in Fig. S2 A & B. In the Pt (without wetproofing) anode both the maximum power density and current density are tripled (185 mW cm^{-2} to 670 mW cm^{-2} and 700 mA cm^{-2} to 2100 mA cm^{-2}). When the anode catalyst is replaced with PtRu, optimizing the dew points increases the maximum power density by 40% (1000 mW cm^{-2} to 1400 mW cm^{-2}) and the achievable current density by 25% (3050 mA cm^{-2} to 3800 mA cm^{-2}). The addition of 5% PTFE wetproofing to the gas diffusion layer improves the water management in the electrodes, increasing the power density by 60% (670 mW cm^{-2} to 1060 mW cm^{-2}) and increasing the current density by 70% (2100 mA cm^{-2} to 3600 mA cm^{-2}). Another large step change in AEMFCs was the addition of ruthenium to the anode catalyst⁷ (Fig. S2 A & B), with the improvements in the kinetic overpotential resulting in a peak power density of 33% (1060 mW cm^{-2} to 1400 mW cm^{-2}). All of the cells described here and performance shown in Fig. S2 were tested in our lab with the ETFE-BTMA membrane and ionomer, and assembled as described in the preceding experimental section. These three significant findings were crucial to the prior state-of-the-art performance reported by our research team.⁵

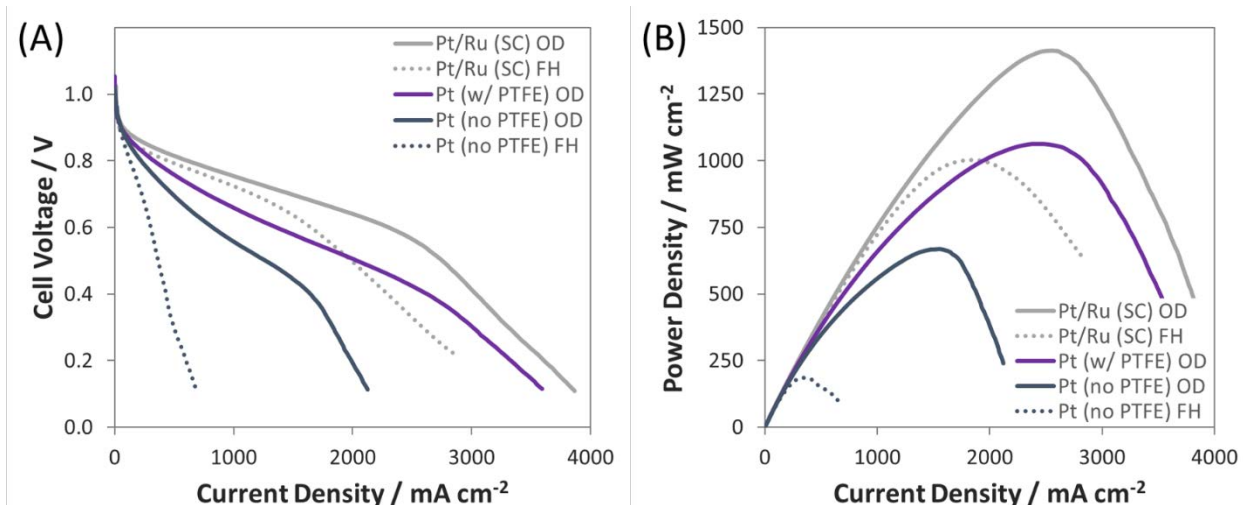


Fig. S2. A) i-V and B) i-Power curves ($10 \text{ mV}\cdot\text{s}^{-1}$ forward scans) showing the progression of recent discoveries to reach the current literature state-of-the-art.⁵ In all cases, a radiation grafted ETFE membrane with a BTMA headgroup was used. The cell temperature was 60°C with a flow rate of $1.0 \text{ L}\cdot\text{min}^{-1}$ for H_2 and O_2 at the anode and cathode, respectively. The gas feed dew points were either 60°C (FH, dotted lines), or optimized (OD, solid lines). Further cell and electrode details in Table S1.

Table S1 Electrode composition of progression.⁵

Anode/Electrode Type:	Pt (no PTFE)	Pt (w/ PTFE)	PtRu (SC)	PtRu (IC)
GDL Wetproofing	No wetproofing	5% PTFE	5% PTFE	5% PTFE
Anode Catalyst	Pt	Pt	PtRu	PtRu
Anode (Pt or PtRu) Loading, mg cm^{-2}	1.25	0.84	0.67	0.67
Cathode Pt Loading, mg cm^{-2}	1.25	0.84	0.53	0.53
Optimized Dew Points Anode/Cathode, $^\circ\text{C}$	57/55	53/51	54/57	54/57
Carbon weight %	48 %	48 %	32.0 %	41.4 %
AEI weight %	20.0 %	20.0 %	20.0 %	17.2 %
AEI:C ratio	0.417	0.417	0.625	0.417
AEI:C:Pt ratio	0.625 : 1.5 : 1	0.625 : 1.5 : 1	0.625 : 1.0 : 1	0.625 : 1.5 : 1

Table S2 Published AEMFCs with 1.0 W cm^{-2} or greater peak power density.

AEM Chemistry	Peak Power Density W cm^{-2}	Cell Temp $^\circ\text{C}$	A/C Dew Points $^\circ\text{C}$	Back Pressure kPa_g	A/C Pt Loading mg cm^{-2}
ETFE-BTMA (this work)	1.9	60	45/46	0	0.5/0.5
LDPE-BTMA ⁸	1.5	80	80/80	0	0.4/0.4
PF AEM Gen 2 ³	1.4	60	50/53	15	0.5/0.5
aQAPS ⁷	1.0	60	60/60	100	0.3/0.4

Neutron Imaging Cell and Operation

To investigate the water imbalance in the AEMFC and identify flooding and mass transport limitations, the cell was rebuilt in with a IC anode in special operando cell and analyzed using neutron imaging, a quantitative, non-destructive measurement of localized liquid water. The hardware has been previous described in detail⁹ and is summarized herein. Gold plated combination current collector and flow fields with a single serpentine flow pattern and active area of 1.2 cm^2 were assembled with a Pt cathode and a PtRu(IC) anode. Both the ETFE-BTMA and PFAEM membranes were utilized with identical electrodes and assembled with 6-mil gaskets to achieve a 20% pinch. The cells were then humidified and broken in under the same protocol used for the 5 cm^2 cells. It should be noted that the NIST cells are primarily optimized for the imaging techniques, and due to their smaller size, assembly requirements, and flow field pattern (designed for optimal imaging of the electrodes and membrane), they do not achieve the same maximum performance as the Fuel Cell Technologies 5 cm^2 hardware.

The cell was first optimized in the neutron beam facility, first humidified, broken in, and then the dew points were optimized at a constant current density of 1.5 A cm^{-2} . The current was held constant during all images in order to control the water production and consumption rate at the anode and cathode. After optimization the images were taken in 1-hour cycles. The first 20 minutes were to allow equilibration for any changed conditions, after which 20 images were taken, each image collected over two minutes. The high-resolution neutron images were collected on the BT-2 beam line at the NIST Center for Neutron Research (NCNR), and captured with a high-resolution CCD box with an MCP detector and special resolution of about 6.5 microns. For each data point the 20 images were averaged, combined, deconvoluted, and analyzed using NCNR software & protocols (for the crashed condition in the ETFE-BTMA cell the 20 images were split into two groups and analyzed separately, giving the before and after crash images/data). The conditions that were varied during these experiments were the feed stream dew points, which were raised and lowered from the optimal conditions, and the current density, which directly affect the water production and consumption rate at the anode and cathode respectively. The effect of these factors on water in the catalyst layers, GDLs, and membrane were examined through this operando visualization technique, which provides simultaneous information on the membrane hydration, water movement through the membrane, flooding behavior over the entire cell, and how these factors are coupled with the current and voltage.

The voltage response over portions of these tests are given in Fig. S3 for the ETFE-BTMA test, and Fig. S4 for the PFAEM test. With the ETFE-BTMA membrane, it can be observed that as the dew points are increased, the operating voltage is driven down, until it reaches a point where the flooding overtakes the electrodes, and the current plummets to zero. The visualization and quantification of this event is shown in Fig. 2. With the PFAEM, the current drops under similarly observed flooding events as the ETFE-BTMA, however the current does not fall to 0. More interestingly, when the dew points are relaxed, the current is able to return to the set value of 1.5 mA cm^{-2} , but the cell is just barely stable since the cell voltage remains ca. 0 V. When the set current is lowered to 1.0 mA cm^{-2} (and to 0.5 mA cm^{-2}), the flooding is immediately relieved, and the voltage recovers (Fig. S4). It is also observable in the in-plane neutron radiographic image in Fig. 2C, where the last panel shows the flooding of the electrodes has been completely alleviated. These observations show that the flooding behavior of the cells is reversible and the preferred method of relaxation is a brief decrease in cell current. This suggest that while the feed gas dew points have a large effect on electrode flooding, focus should be directed at the catalyst layer and GDL to tune the water management within the electrode and membrane to optimize cell performance and further AEMFC capabilities.

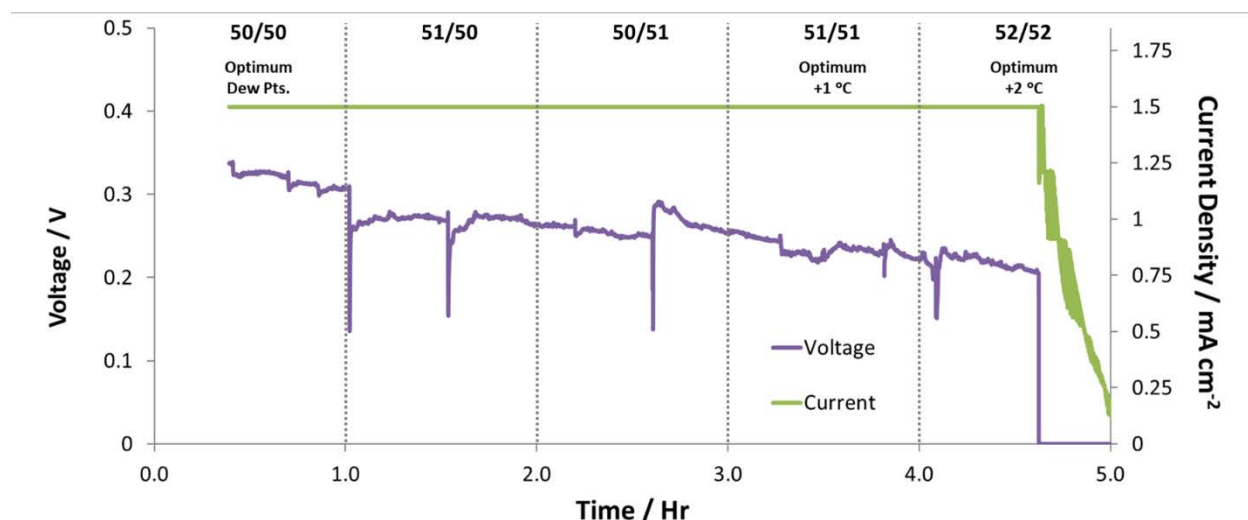


Fig. S3. Voltage and current density over time with the ETFE-BTMA cell for varying feed gas dew points of the NIST 1.2 cm² radiography cell, demonstrating the effect of flooding, including the decreasing voltage and significant flooding event resulting in the cell crash at approximately 30 minutes after the Optimum +2 °C condition is started.

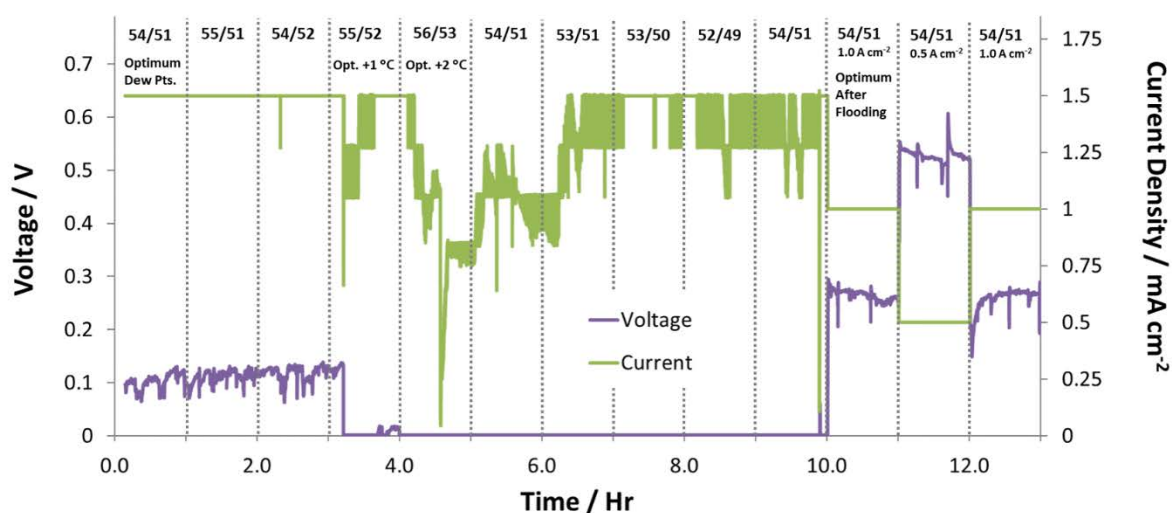


Fig. S4. Voltage and current density over time with the PFAEM cell for varying feed gas dew points current densities of the NIST 1.2 cm² radiography cell, demonstrating the effect of flooding and recovery through lowered set current density.

ECSA Measurements by CO-Stripping

Determining the electrochemically active surface area (ECSA) of platinum-based fuel cell electrode layers via cyclic voltammetry (CV) is an important diagnostic test for PEMFCs to determine electrode layer catalytic activity. However, the well-defined platinum features that allow easy quantification for PEMFC do not always show up in a corresponding test for alkaline exchange membrane fuel cells (AEMFC). Instead, carbon monoxide (CO) is first electrochemically adsorbed to the catalyst active sites, and then stripped away via CV. The resulting current allows facile quantification of the ECSA for AEMFC that is otherwise not available.

The ECSA was determined by passing pure CO gas at 0.2 L min⁻¹ over the anode electrode, where the cell was held at a potential of 0.2 V vs hydrogen at the cathode electrode for 10 minutes to ensure complete adsorption of CO at all active sites. Following the CO adsorption, nitrogen was

purged for 30 minutes to remove excess CO, then a CV was run between 0.07 and 1.2V for three cycles, where CO was stripped from all electrochemically active platinum sites during the first cycle. The areal difference between the first and second cycle (in Watts) is then used to calculate the ECSA of the tested electrode (Fig. 3E).

Polarization Curve Deconvolution

The reduction in operating cell voltage in a fuel cell is caused by the sum of three overpotentials based on the kinetic, ohmic, and mass transfer limitations of the operating cell, and can be represented by:

$$E_{cell} = E_{rev} - \eta_{\Omega} - \eta_{kinetic} - \eta_{MT} \quad [1]$$

The first step to breaking out the overpotentials of the polarization curves is to isolate the ohmic overpotential or iR free cell voltage using the current and ohmic resistances at each point as shown:

$$E_{iR-free} = E_{cell} + iR_{\Omega} \quad [2]$$

where iR_{Ω} is also the ohmic overpotential. The iR values are calculated at each point along the polarization curve using the high frequency intercept and current interrupt technique on the built-in frequency response analyzer in the test stands. It is expected that the ohmic loss plotted vs current density in a PEMFC is typically a straight line, however, this is not the case in the AEMFC, as increased membrane hydration can decrease the hydroxide transfer resistance within the membrane, causing a slight decrease in the slope of the overpotential as the current increases (Fig. 3B). The resulting calculated iR-free voltage only has the kinetic and mass transfer overpotentials, the sum of which can be calculated by subtraction of the iR-free voltage from the reversible cell potential, which is calculated from the thermodynamic components of the reactions. At cell voltages of greater than 0.85 V and operating currents of less than 100 mA cm⁻² it can be assumed that mass transfer limitations are not a factor,¹⁰ and utilizing this assumption Tafel analysis is performed on the “kinetic region” calculated overpotential:

$$\eta_{kinetic} \propto m \log i + b \quad [3]$$

where the previous assumptions are validated by the fit, as mass transfer limitation would cause a change in the slope of the Tafel analysis. The resulting fit is then used to extrapolate the kinetic overpotentials through the entire curve. The final component of the deconvolution, the mass transfer overpotential, is then calculated by subtracting the kinetic and ohmic overpotentials from the reversible cell potential.

Addition of Excess Carbon to Anode

Too much carbon can certainly be added to the electrode layer, where the kinetic losses and inaccessibility of the catalyst overtake the mass transport gains from the added carbon. This was observed when even more carbon was added to the anode (denoted EC for excess carbon), with a AEI:C:Pt ratio of 1.25:3.5:1. This anode, with a resulting power density of only 1.1 W cm⁻², demonstrates that not surprisingly you can add too much carbon and dilute the catalyst to detriment. It should also be noted that the lower open circuit potential of 0.9 V also suggests kinetic limitations as the primary limiting factor in the higher carbon. In this cell the total ionomer percentage was limited to 20 wt%, despite the effect this has on the AEI:C ratio, in order to prevent an even larger change to the C:Pt ratio, and completely drown out the platinum in the catalyst layer. A comparison of all full loading anodes is given in Table S2 and the polarization of the EC anode is given in Fig. S5.

Table S3 Catalyst, carbon, and ionomer loadings and ratios of all full loading PtRu anode electrodes.

Anode Type:	SC	IC	BC	EC
PtRu Loading, mg cm ⁻²	0.67	0.71	0.71	0.75
Carbon Loading, mg cm ⁻²	0.45	0.71	1.07	1.76
Carbon weight %	32.0 %	41.4 %	*48.0 %	56.0 %
AEI weight %	20.0 %	17.2 %	*20.0 %	*20.0 %
AEI:C ratio	0.625	*0.417	*0.417	0.357
AEI:C:Pt ratio	0.625 : 1.0 : 1	0.625 : 1.5 : 1	0.94 : 2.5 : 1	1.25 : 3.5 : 1

* optimized value

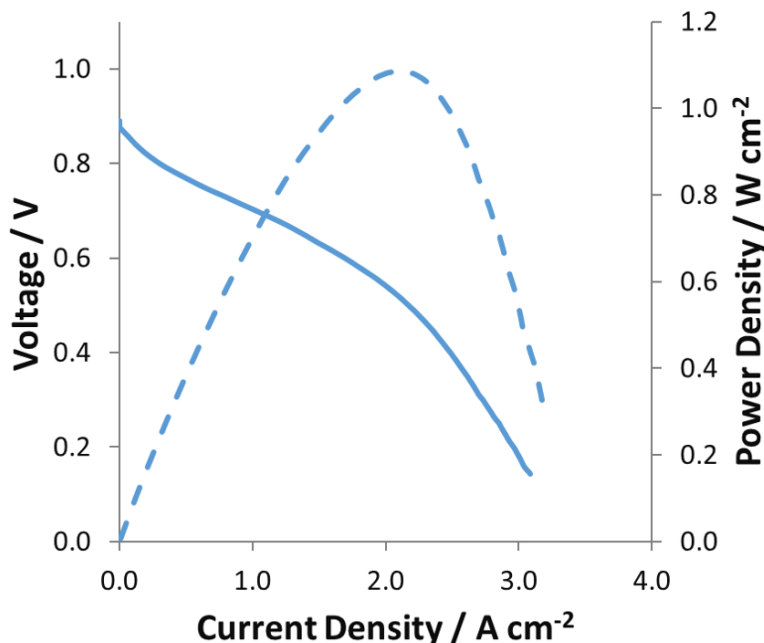


Fig. S5. i-V and i-Power curves (10 mV·s⁻¹ forward scans) for the EC anode. A radiation grafted ETFE membrane with a BTMA headgroup was used with a cell temperature of 60° and a flow rate of 1.0 L·min⁻¹ for H₂ and O₂ at the anode and cathode, respectively. The gas feed dew points were optimized at 52 °C /52 °C for the anode/cathode. Further cell and electrode details given in Table S2.

Stability Test

A cell with an IC anode with a PtRu loading of 0.75 mg cm⁻² and cathode with a platinum loading of 0.53 mg cm⁻² was broken in and tested with polarization curves, voltage holds and current holds for 8 hours, at which point dew points were optimized at 52 °C/52 °C (anode/cathode) and stable performance was achieved. A constant current of 600 mA cm⁻² was drawn from the cell for 200 hours, after which the current was changed to 550 mA cm⁻² for 10 hours and back to 600 mA cm⁻² for 10 hours to assess stability and recoverability under changing current conditions. After the current change the load, temperature, fuel, and oxidant gases were removed to simulate a hard shut down and cold start. Nitrogen replaced the H₂ and O₂ and the cell was allowed to naturally cool for 8 hours. Following the shut down the cell was brought up to the previous optimized temperature, H₂ and O₂ were applied, and the stability test was immediately resumed. It can be observed that after the brief relaxation in current the cell resumed the previous state of operation at 0.6 V₀. However, after the cold shutdown the cell returned to operation near 0.95 V₀ and followed the same gradual decline to stable operation near 0.6 V₀. V₀ in this cell is 0.625 V.

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