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Recent progress in understanding the catalyst layer in anion exchange membrane electrolyzers – durability, utilization, and integration

Emily K. Volk, ^a Melissa E. Kreider, ^b Stephanie Kwon ^{*c} and Shaun M. Alia^{*b}

Anion exchange membrane water electrolyzers (AEMWEs) are poised to play a key role in reducing capital cost and materials criticality concerns associated with traditional low-temperature electrolysis technologies. To accelerate the development and deployment of this technology, an in-depth understanding of cell materials integration is essential. Notably, the complex chemistries and interactions within the catalyst layer (consisting of the anode/cathode catalyst, anion exchange ionomer, and their interfaces with the transport layers and membrane) collectively influence overall cell performances, lifetimes, and costs. This review outlines recent advances in understanding the catalyst layer in AEMWEs. Specifically, electrode development strategies (including catalyst deposition techniques and configurations as well as transport layer design strategies) and our current understanding of catalyst-ionomer interactions are discussed. Effects of cell assembly and operational variables (including compression, temperature, pressure, and electrolyte conditions) on cell performance are also discussed. Lastly, we consider cutting-edge *in situ* and *ex situ* diagnostic techniques to study the complex chemistries within the catalyst layer as well as discuss degradation mechanisms that arise due to the integration of cell components. Simultaneously, comparisons are made to proton exchange membrane water electrolyzers (PEMWEs) and liquid alkaline water electrolyzers (LAWEs) throughout the review to provide context to researchers transitioning into the AEMWE space. We also include recommendations for standard operating procedures, configurations, and metrics for comparing activity and stability.

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Broader context

Hydrogen (H₂) production from water electrolysis allows for the efficient conversion of low-cost renewable electrons to stored chemical energy, thereby limiting the curtailment of variable renewable energies (e.g., wind and solar) and creating a promising long-term energy storage medium. This ultimately provides green pathways for carbon-intensive and hard-to-decarbonize sectors including transportation; steel, cement, and ammonia production; and chemical synthesis, and facilitates the energy transition from fossil fuels to variable renewable energy sources. Current barriers to water electrolysis penetration in the market originate from the costly and rare materials required by current state-of-the-art PEMWEs and the low efficiency and incompatibility with intermittent operation of commercial LAWES. AEMWEs have the potential to offset these concerns by providing green H₂ production at high operating current densities and with the potential for intermittent operation, while utilizing earth-abundant, non-platinum group metal (PGM) materials. Understanding the materials, interfaces, and interactions within the catalyst layer of these devices is a critical next step to facilitate the development and deployment of AEMWE technology.

1. Introduction

Green H₂ produced *via* electrochemical water splitting has emerged as a key energy carrier to facilitate the integration

and storage of low-cost, variable renewable electrons and, ultimately, decarbonization across sectors.^{1–3} Current industrial methods, however, face cost and scalability challenges.^{2,4} Liquid alkaline water electrolyzers (LAWEs) traditionally suffer from low operating current densities (on the order of 100 s of mA cm⁻²),⁵ highly caustic electrolyte (> 7 M KOH) with increased maintenance and operational costs, and incompatibility with H₂ backpressure, thus requiring costly H₂ gas separation and compression upstream for industrial implementation.² State-of-the-art proton exchange membrane water electrolyzers (PEMWEs) allow for both high operating

^a Advanced Energy Systems Graduate Program, Colorado School of Mines, Golden, Colorado 80401, USA

^b Chemistry and Nanoscience Center, National Renewable Energy Laboratory, Golden, Colorado 80401, USA. E-mail: salia@nrel.gov

^c Department of Chemical and Biological Engineering, Colorado School of Mines, Golden, Colorado 80401, USA. E-mail: kwon@mines.edu



current densities ($2\text{--}5\text{ A cm}^{-2}$ at $1.8\text{--}2.0\text{ V}$)^{6–8} and significantly decreased H_2 separation costs with the ability to apply H_2 backpressure.^{2,4} The harsh, acidic near-surface environment in PEMWE systems, however, requires the use of expensive and rare platinum group metal (PGM) materials for catalysts and component coatings (transport layers and separators),² imposing capital cost⁴ and materials criticality^{9,10} concerns. Further, there is interest to move away from the fluoropolymer materials often used for the membrane and ionomer in PEMWE systems due to high costs and growing concerns over the lasting effects of fluoropolymers on human health and the environment.¹¹

Anion exchange membrane water electrolyzers (AEMWE) are an emerging technology that encompass the benefits of both LAWE and PEMWE technologies. Like PEMWEs, AEMWE systems utilize polymer-based electrolytes and a zero-gap approach to achieve high operating current densities ($>1\text{ A cm}^{-2}$ at 1.8 V).^{1,12–14} AEMWE systems can also allow for the application of H_2 backpressure to minimize H_2 separation costs.^{2,15,16} More importantly, these systems operate with a near-surface alkaline environment, which allows for the use of non-PGM materials for catalysts and other components,^{2,15,17} thus significantly reducing capital cost and materials criticality



Emily K. Volk

Emily Volk is currently a PhD candidate at the Colorado School of Mines in the Advanced Energy Systems Graduate Program. She is co-advised by Dr Stephanie Kwon and Dr Shaun Alia. Her thesis work centers on non-platinum group metal oxide catalysts for the oxygen evolution reaction in alkaline media and their integration into catalyst layers for electrochemical devices. Specifically, she is interested in

the electrochemical and physical characterization of activation/deactivation mechanisms relevant to the catalyst layer. Before coming to Mines, Emily worked as a Scientist for Pall Corporation and obtained her BS in Chemical Engineering from the University of Rochester.



Melissa E. Kreider

Melissa Kreider is a postdoctoral researcher in the Electrochemical Engineering and Materials Chemistry Group at NREL. Her current work focuses on materials integration and catalyst layer engineering to improve the performance and durability of anion exchange membrane water electrolyzers. She is interested in using electrochemical and materials characterization to understand electrocatalyst activation and

degradation processes. Prior to joining NREL, she completed her PhD in Chemical Engineering at Stanford University with Prof. Thomas Jaramillo, where she developed electrocatalysts for fuel cell applications and utilized in situ X-ray spectroscopic, diffraction, and mass spectrometric techniques to characterize catalyst evolution during reaction.



Stephanie Kwon

Stephanie Kwon is an assistant professor in the Chemical and Biological Engineering department at Colorado School of Mines. Her research interest centers on heterogeneous catalysis and sustainability. She brings together cutting-edge research tools, including in situ spectroscopy, microkinetic modeling, and density functional theory calculations, to understand surface reactions at a molecular-level. In doing so,

she aims to tackle our current energy and environmental problems by providing novel catalytic systems with improved energy and atomic efficiency. Her current research projects include CO_2 conversion, low temperature H_2O electrolysis for H_2 production, biomass conversion (via aldol condensation catalysis), benzene alkylation, and alkene oxidation catalysis.



Shaun M. Alia

Shaun Alia is a senior scientist in the Electrochemical Engineering and Materials Chemistry Group at NREL. His research interests involve understanding electrochemical and degradation processes, materials integration, and component development. Within the HydroGEN Energy Materials Network, Dr Alia is a Technology Lead in Low Temperature Electrolysis and a capability owner in materials development and testing. He is

further active within in situ durability, diagnostics, and accelerated stress test development as a Durability Liaison for the H2NEW consortium. Previously, he worked in several areas related to electrochemical energy conversion and storage, including hydrogen and direct methanol fuel cells, capacitors, and batteries.



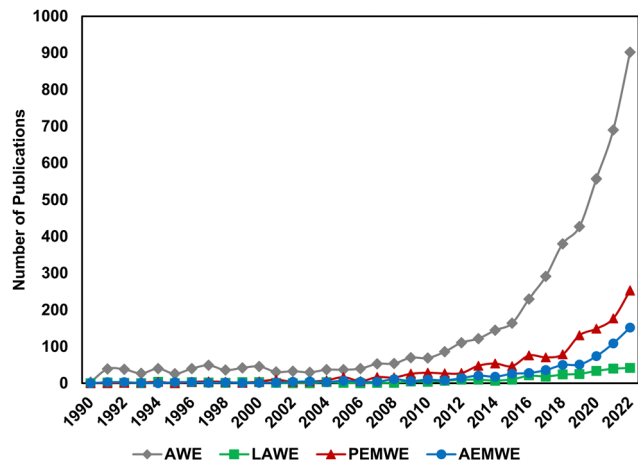


Fig. 1 Number of publications mentioning “alkaline electrolysis (grey, AWE)”, “liquid alkaline electrolysis” (green, LAWE), “proton exchange membrane electrolysis” (red, PEMWE), and “anion exchange membrane electrolysis” (blue, AEMWE) over the last 30 years. Data obtained from Web of Science on 08/01/2023.

concerns. Furthermore, AEMWE systems typically operate with dilute supporting electrolytes (*i.e.*, 0.1–1 M KOH or K_2CO_3), or, possibly, with pure water feeds,¹⁸ reducing operating cost and materials durability concerns related to the high-concentration electrolytes typically used in LAWE systems.

Over the last decade, interest in low temperature electrolysis technologies has risen rapidly. Fig. 1 shows Web of Science search results for the keywords “alkaline electrolysis” (grey diamonds), “liquid alkaline electrolysis” (green squares), “proton exchange membrane electrolysis” (red triangles) and “anion exchange membrane electrolysis” (blue circles) by year. Alkaline electrolysis is by far the largest field, with renewed interest developing in the late 20th century. AEMWE and PEMWE have comparatively skyrocketed in relevance in the last 5 years; search results for “anion exchange membrane electrolysis” yielded 761 publications, 485 of which were published in the last 5 years (*i.e.*, 2019–present). Because “alkaline electrolysis” may have inadvertently included results from AEMWE, we also looked at the trends for “liquid alkaline electrolysis”; interestingly, this term emerges in 1991 and doesn’t increase above 5 publications per year until 2015, at the same time as AEMWE interest begins to rapidly increase, suggesting its emergence was to help distinguish research in traditional LAWE *vs.* novel AEMWE. As the AEMWE system is an emerging technology within the field, there is a need to translate the lessons learned from PEMWE and LAWE to AEMWE and to elucidate the differences among these technologies.

Several reviews have covered recent component-level advances in AEMWE technology,^{19–23} including for membranes/ionomers^{24–29} and catalysts.^{30–34} A significant improvement has been made over the course of the last 5–10 years in enhancing the ionic conductivity of anion-exchange polymers ($> 100 \text{ mS cm}^{-1}$)³⁵ and prolonging the stability of AEMWE cells (stability tests of up to 10 000 h have been reported).^{24–27,29,35–42}

More recently, several studies have reported novel strategies for transport layer design by modifying the porosity and morphology of materials to enhance AEMWE performance.^{43,44} These improvements, alongside advances in catalyst development, have enabled AEMWE cell performances exceeding 3 A cm^{-2} at reasonable voltages (1.8–2.2 V).^{13,14}

The reported performances of AEMWE, however, still fall short of PEMWE counterparts. Both the oxygen (OER) and hydrogen evolution (HER) reactions typically require higher overpotentials in AEMWE relative to their PEMWE analogues, with the OER as the more-limiting reaction in both conditions.⁸ HER kinetics drop by several orders of magnitude when moving from acid to base,^{45,46} and although intrinsic OER kinetics have been shown to be similar or slightly improved in alkaline *vs.* acidic electrolytes, OER kinetics are more limited in AEMWE than PEMWE at the device level. This discrepancy is likely due to differences in catalyst–ionomer interactions and less-than-optimal understanding of materials integration, ink rheology, and electrode properties that can limit OER kinetics at the device level. Furthermore, transport layer development in AEMWE is significantly lacking compared to the PEMWE space, and advances are needed to improve catalyst–transport layer interfaces and the transport of liquid reactants and gaseous products. Many questions remain regarding key device-level variables, including ideal operating conditions and the role of the ionomer within the catalyst layer. Few reviews have discussed such questions and research related to the integration of the catalyst layer with other device components.

This review aims to cover recent advances in understanding the catalyst layer in AEMWEs, with a focus on the chemistry and physics behind the integration of materials at the device level. To provide context for this review, the current state-of-the-art in AEMWE cell components, including catalyst, membrane, and transport layer design, will be briefly summarized in Section 2. Next, recent advances in electrode development, including catalyst deposition techniques and configurations, catalyst–transport layer design strategies, and catalyst–ionomer integration will be discussed in Section 3. The effects of cell assembly and operational variables (Section 4) as well as *in situ* and *ex situ* diagnostic techniques that can be used to understand processes within the catalyst layer will be reviewed (Section 5). Lastly, single-cell stability, with a focus on degradation mechanisms that arise due to the integration of components, will be discussed in Section 6. Throughout this review, key comparisons between AEMWE and LAWE and PEMWE will be made to facilitate a knowledge transfer for researchers transitioning into the emerging AEMWE space.

2. Overview of AEMWE cell components and recent component-level advances

AEMWEs commonly use a cell hardware configuration analogous to that widely used in PEMWE and PEM fuel cell (PEMFC) technologies (Fig. 2a). However, there is yet no consensus on a



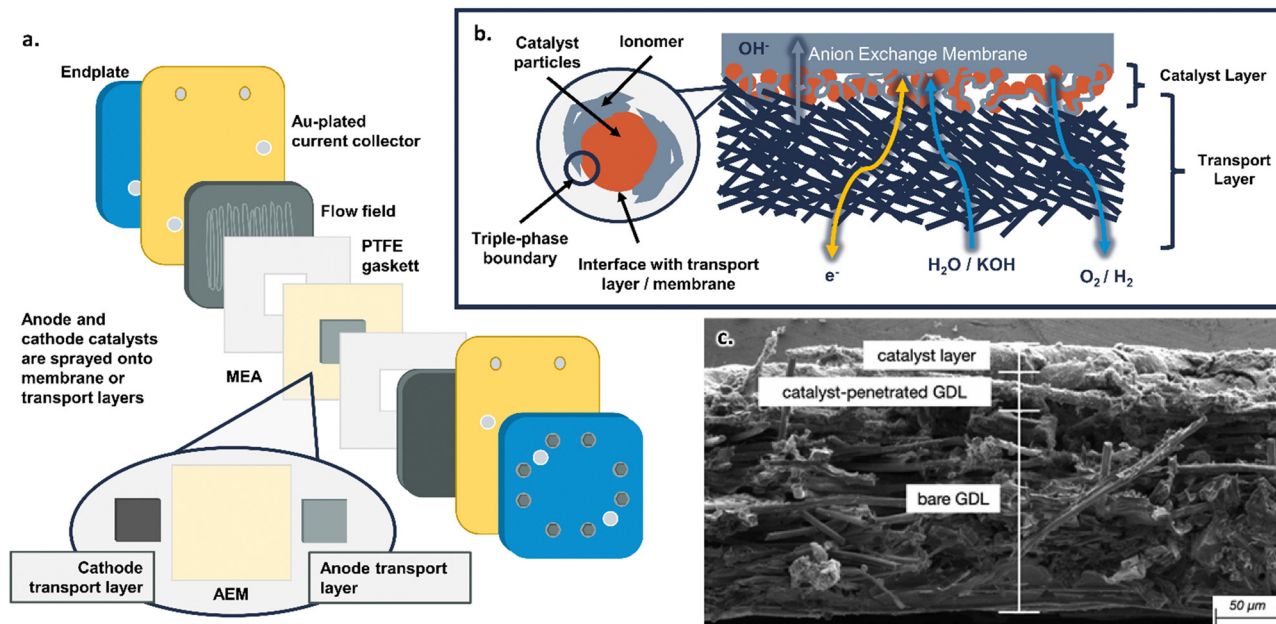


Fig. 2 (a) Schematic of the components in an AEMWE. (b) Schematic of the catalyst layer in an AEMWE. (c) SEM image of an anode catalyst layer in an AEM fuel cell showing the transport layer fibers, catalyst layer, and interfacial region. Reprinted from J. Marie Mora *et al.*, Analytical-based simulation approach for an anion exchange membrane fuel cell, *Energy Conversion and Management*, **273**, 116382, Copyright (2022), with permission from Elsevier.⁶⁵

standard configuration for AEMWE. This is in sharp contrast to the PEMWE components and assembly protocols that have been standardized in several benchmarking efforts across the US, EU, and internationally,^{47–49} including in the Future Generation Membrane Electrode Assembly (FuGeMEA) configuration outlined by the U.S. Department of Energy (DOE) H₂NEW Consortium.^{48,50} This current lack of effective benchmarking and baselining within AEMWE reflects the current status of the field and demonstrates the potential for developing and optimizing components across AEMWEs. Common cell components used in AEMWEs, as well as areas for future research, are outlined below. Note, the cell components discussed here specifically relate to research and development efforts at the single-cell level; commercial stack components and configurations likely have different design considerations, which will be addressed but are not the focus of this review.

At either end of an AEMWE are the cell end plates (shown in blue, Fig. 2a). These end plates have threading for bolts that provide compression to the cell. The end plates are often made of aluminium (Al) or stainless steel, which are chosen to apply necessary compression and to withstand the mild temperatures of AEMWE (60–80 °C). Beyond performing the desired function, these materials choices should not have a significant impact on electrolyzer performance or other materials integration. It is worth noting that stainless steel exhibits a higher alkaline stability than Al,¹⁷ which may be beneficial in case of a cell leakage. In PEMWE systems, the end plates are often made of anodized Al.⁵¹

The next components in the cell are the current collectors (shown in gold, Fig. 2a). The current (or voltage) is supplied

through the current collectors by leads connected to a power supply or a potentiostat. They thus must be made of materials with high electronic conductivity to minimize their resistance contributions to the overall cell configuration. Most commonly, Au-plated Cu is used for the current collectors for both AEMWE and PEMWE.⁴⁷

The current collectors are then in contact with the flow fields or bipolar plates (shown in grey, Fig. 2a) through which the liquid reactant (*i.e.*, water or supporting electrolyte) flows into the cell. Flow fields for AEMWEs are most often made of stainless steel or Ni due to their alkaline compatibility, analogous to the materials choices in LAWE, without the need for the PGM coatings used in PEMWEs. Yet, one must be cautious in using stainless steel because it is prone to dissolution and can introduce Fe contaminants into the electrolyte, which have been shown to significantly impact the performance of Ni and Co materials; specifically, Fe contaminants have been shown to boost the OER activity of these catalytic systems.^{52,53} Fe contamination in the electrolyte has also been shown to negatively impact long-term durability in pure-water feeds due to interactions with the ionomer (details in Section 6).⁵⁴ The use of Ni for flow field materials also has some concerns. Although Ni is stable against dissolution in alkaline and oxidizing environments, it is likely to passivate (*i.e.*, form a less-conductive oxide layer) and deform over time, decreasing cell performance. It is therefore necessary to routinely polish Ni flow fields to avoid complications such as increased contact and high frequency resistances.

In comparison, PEMWE systems utilize flow fields made of Ti due to its acidic stability. Pt or Au protective coatings are



often used to help with longevity and minimize contact resistances,⁵⁵ although the use of PGM or noble-metal-based materials can contribute to cost concerns. Ti oxidation (*i.e.*, to TiO₂) can lead to increased contact resistances, which can result in a decrease in overall cell performance.⁵⁶ Learning from these experiences in the PEMWE field, the use of such conductive coatings may be a good strategy to pursue for AEMWE flow fields to prevent increased contact resistances caused by Ni oxidation and thus to suppress cell degradation.

At the center of the cell is the membrane electrode assembly (MEA). The MEA is composed of the anion exchange membrane, anode/cathode catalyst layers, and transport layers, all framed by polytetrafluoroethylene (PTFE) gasketing (for research cells with no H₂ backpressure) with a typical total thickness of <1000 μm. Each individual component within the MEA will be discussed in the following paragraphs.

The anion exchange membrane functions as the electrolyte material in AEMWEs, analogous to the proton-conducting membranes used in PEMWEs. The membrane is typically made of a conductive polymer containing positively charged functional groups (often N-based) to allow for the transport of OH⁻, although there is yet no standardization for the membrane choice. This contrasts with PEMWEs, where Nafion is often used as the standard proton-conducting material. Several reviews have highlighted recent advances in polymer chemistry for anion exchange membranes and ionomers,^{24–29} including those that are commercially available.²⁶ Recent developments in cation functional groups, side chain engineering, cross-linking strategies, and backbone development have resulted in anion exchange polymers that have high ionic conductivity and alkaline stability. Such developments allow for extended durability testing for AEMWE cells and bring the technology closer to commercialization.

On either side of the anion exchange membrane are the transport layers, which are also called gas diffusion layers (GDLs) or porous transport layers (PTLs). The transport layer facilitates the transport of liquid reactants (*i.e.*, water or supporting electrolyte) and gaseous products (*i.e.*, H₂ at the cathode, O₂ at the anode; Fig. 2b) and must be electronically conductive to allow electrons to move between the catalyst particles and the current collectors. It is also important to note that, on the anode side, the transport layer must be able to withstand high potentials (*i.e.*, often up to and exceeding 2 V) and an aqueous, alkaline environment (*i.e.*, pH of 12–14). Common materials for transport layers in AEMWEs include C paper and Ni, Ni alloy, or stainless steel meshes and foams,^{21,57,58} although C is not a viable choice for the anode transport layer because it oxidizes and degrades at high potentials and pH.^{59,60}

Comparatively, transport layers in PEMWEs often consist of C paper at the cathode and Pt-coated Ti at the anode.⁶¹ Ti is chosen due to its acid compatibility (*i.e.*, it passivates but does not corrode) and manufacturability, while Pt provides necessary electronic conductivity.⁶¹ Transport layer properties are critical for facilitating interfacial contact with the catalyst layer and can heavily affect site accessibility. For PEMWEs, research

has suggested that the use of a microporous layer (MPL) at the catalyst-layer interface can help facilitate interfacial properties, including improved ionic and electronic contacts and increased active-site accessibility.^{62,63} The MPL is usually a few microns thick and has a smaller mesh size than the bulk of the transport layer to facilitate these improved interfacial contacts. Such use of an MPL is currently less studied in the AEMWE community, although a recent study by Razmjooei *et al.* showed an improvement in performance by utilizing a transport layer topped with a Ni MPL in an AEMWE.⁴³ This work suggests that AEMWE performance could be improved by engineering the interface between the catalyst and transport layer material; this will be discussed further in Section 3.2. Lastly, transport layer design also impacts membrane durability; transport layers can provide structural support to the membrane and prevent excessive creep (caused by swelling) into the catalyst/transport layers. Such behaviors can occur when surface porosity is too high, especially under H₂ backpressure operation, as has been shown in PEMWEs.⁶⁴

PTFE gaskets frame the transport layers and facilitate desired compression to the cell. Note that gasketing can be used for ambient pressure cells, but other methods are likely required for high-pressure operation; pressurized operation and effects of cell compression are discussed in Section 4.1. The thickness of the gaskets can be adjusted individually for the cathode and anode, allowing for different compressions as well as the use of different transport layer thickness on either side.

In between the transport layers and anion exchange membrane are the anode and cathode catalyst layers. In this review, the catalyst layer specifically refers to (1) the catalyst particles themselves, (2) the anion-conducting polymer (referred to as the ionomer) dispersed with the catalyst particles, (3) their interface with the porous transport layer, and (4) their interface with the anion exchange membrane (shown schematically in Fig. 2b and as a cross-sectional SEM image from JM Mora *et al.*⁶⁵ in Fig. 2c). Anode and cathode catalysts can be deposited either on the membrane (catalyst coated membrane, or CCM, approach) or on the transport layer (catalyst coated substrate, or CCS, approach); the pros and cons of these approaches are discussed in Section 3.2. The most common deposition methods for anode and cathode catalyst layers involve spraying from an ink (which can ultimately translate to manufacturing techniques, such as a roll-to-roll coating), though other deposition methods will be discussed in detail in Section 3.1. The catalysts are often deposited as a mixture with the ionomer (Fig. 2b); the role of ionomers and details of catalyst-ionomer integrations are discussed in Section 3.3.

Both anode and cathode catalyst layer designs involve many of the same variables and considerations, including catalyst loading, ionomer content, and electrolyte conditions, among others. Anode catalyst layers, however, unlike cathode catalyst layers, must be able to withstand high voltages and thus highly oxidative conditions. Further, cathode catalyst layers have different hydration considerations, especially when AEMWEs



are operated cathode dry (discussed further in Section 4.2). Similarly, the electrolyte flow configuration (*i.e.*, DIW *vs.* supporting electrolyte, cathode wet *vs.* dry) will affect necessary ionomer contents and these needs may vary between anode and cathode catalyst layers. These topics are discussed further in Section 3.3.

The most commonly used catalysts in PEMWE are Pt/C and IrO₂ for HER and OER, respectively.² The acidic near-surface environment of PEMWEs necessitates the use of these expensive and rare PGM materials, as non-PGMs oxidize into soluble forms at such conditions.¹⁷ In fact, even Ir-based materials become thermodynamically unstable at the harsh OER reaction conditions in both acid and base;^{17,66,67} Ir has been shown to dissolve *via* multiple routes at high anodic potentials, including *via* the formation of IrO₃ (g) and, subsequently, to soluble IrO₄²⁻,⁶⁷ raising concerns over the long-term durability of Ir in electrolyzers.

Like in PEMWE, Pt/C and IrO₂ have historically been common choices for AEMWE catalysts. However, the alkaline environment of AEMWEs also allows for the use of non-PGM materials. First-row transition metals (*e.g.*, Ni, Fe, Co, and Mn) have been studied in many forms, including metallic nanoparticles, polycrystalline metals, sulfides, nitrides, and oxides,^{68–71} which are shown to be active for OER in alkaline media. Metallic species often exhibit higher OER reactivities than the respective oxides, but oxidative conditions (*i.e.*, high potential and pH) at the anode likely mean that any starting material eventually converts to an oxidized (*i.e.*, oxide or (oxy)hydroxide) form *in situ*.⁷² Recent works have explored the effects of varying composition (*e.g.*, mixed metals and/or heteroatom X-ides, including nitrides, sulfides, phosphides, *etc.*),^{73–75} of the use of different oxide forms,^{76–78} and the use of supports^{79–81} to enhance catalytic activity, active surface area, and electronic conductivity; such advances in catalyst development have been reviewed previously.^{30–34}

For the HER in AEMWE systems, many studies still use PGM-based catalysts, primarily Pt or PtRu on high surface area C, analogous to those used in PEMWE.^{82–84} More recent works have focused on developing alternative non-PGM materials, including CoNiO₂,⁸⁵ NiCu oxide,⁸⁶ and Ni/CeO₂-La₂O₃/C.⁸⁷ Performance enhancements have been observed for transition metal phosphides and sulfides, such as VCo-P⁸⁸ and Fe_{0.2}Ni_{0.8}-P_{0.5}S_{0.5},⁸⁹ and there has been significant interest in NiMo-based alloys.^{12,90–92} It is instructive to note that the optimal loadings for non-PGM cathode catalysts in an MEA are typically several times greater than those for PGM catalysts; for example Tricker *et al.* found optimized cathode loadings of 0.3 mg cm⁻² and 1.6 mg cm⁻² for Pt/C and CoNiO₂/C, respectively.⁸⁵ The necessity of higher loading may be due to lower accessible surface areas and/or lower per site activity compared to PGM catalysts supported on high surface area carbons. These materials are often unstable and induce performance drops on the order of 100–200 mV at 10 mA cm⁻² in three-electrode, rotating disc electrode (RDE) studies *vs.* PGM cathode catalysts;^{93–95} more work is therefore required to develop non-PGM catalyst options for HER. Promising catalysts must also be tested in AEMWE

devices, since RDE activity is not necessarily predictive of performance in a device due to significant differences in the catalyst layer environment and transport processes, as has been demonstrated for PEMFCs.⁹⁶

For both anode (OER) and cathode (HER) catalysts, considerations must also be made to material cost, availability, and criticality. As discussed, PGMs, such as Ir, have been identified as having high material criticality,^{9,10} motivating the development of non-PGM materials for OER and HER catalysts as well as, more largely, AEMWE cell components. However, not all non-PGMs have equal material costs or criticalities. For example, Co has appeared alongside PGMs on critical materials lists globally;^{9,10,97} considerations to the long-term viability and costs of sourcing different catalyst materials must be weighed alongside measures of activity and stability.

The following sections will dive deeper into recent advances in understanding how these individual components integrate in AEMWEs, with specific focus on the catalyst layer and how key interfaces impact the overall cell performance (*e.g.*, overpotentials, accessibility of active sites, stability, *etc.*).

3. Recent advances in electrode development and configurations

Electrode manufacturing, including catalyst deposition methods, configurations, and composition, have been shown to impact the overall performance of PEMFCs, PEMWEs, and, more recently, AEMWEs. The following sections will explore recent advances in electrode development, including catalyst deposition techniques (Section 3.1), strategies for integrating catalysts with membranes and transport layers (Section 3.2), and catalyst-ionomer interactions and integration strategies (Section 3.3).

3.1 Catalyst deposition techniques

There are numerous strategies for depositing catalytic materials onto the membrane or transport layer. These deposition strategies can affect the type and nature of active sites (*i.e.*, chemical structures of the catalytic materials), the degree to which active sites are exposed, and the network of electronic and ionic conductivities between catalysts particles as well as between the catalyst and the transport layer or membrane.

The most commonly used technique is spraying a catalyst ink, composed of catalyst particles, ionomer, water, and alcohol (*e.g.*, *n*-propyl or isopropyl alcohol, ethanol, *etc.*), onto a substrate (*e.g.*, membrane or transport layer).^{13,98–100} This method has also been widely implemented in PEMWE research.¹⁰¹ Typically, spraying can be performed by hand using an airbrush (Fig. 3a) or by using an automated ultrasonic spray system (Fig. 3b), both of which provide varying degrees of control regarding deposition homogeneity. Typical, the tuning variables for these spraying techniques include ink composition (including solvent choice and concentration as well as catalyst/ionomer ratios) and preparation (including sonication and icing procedures), deposition temperature, and deposition



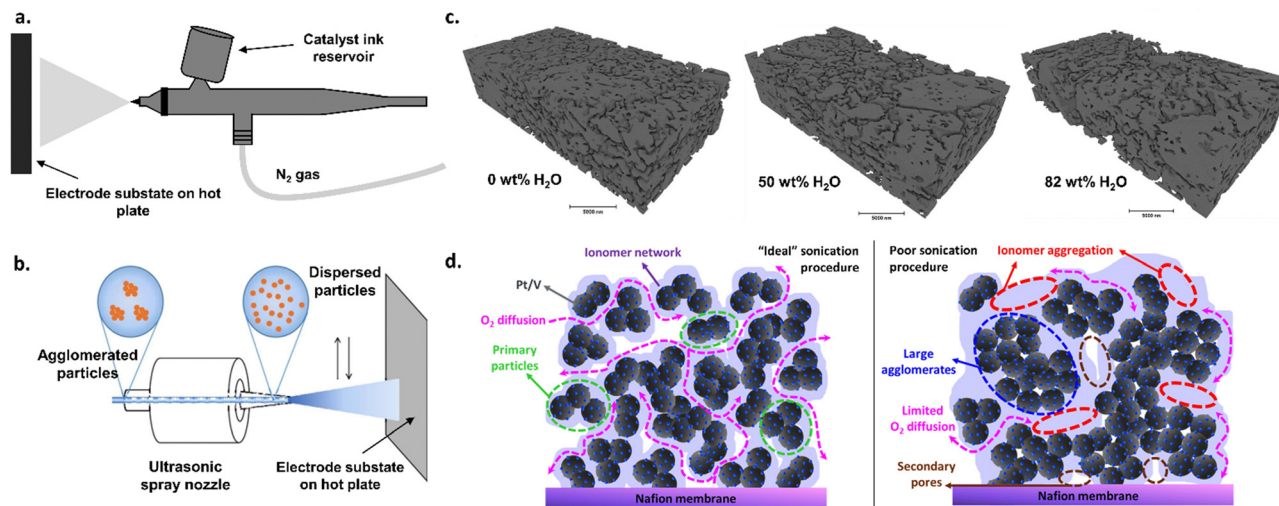


Fig. 3 (a) Schematic of the airbrush spraying method for catalyst deposition. (b) Schematic of an ultrasonic spray system. Adapted from Yarlagadda *et al.*, *J. Electrochem. Soc.*, 2017 with permission.²⁰⁴ (c) Nano-CT segmented phase contrast images for catalyst layers prepared with different water contents in the ink. Reprinted from L. Osmieri *et al.*, Utilizing ink composition to tune bulk-electrode gas transport, performance, and operational robustness for a Fe–N–C catalyst in polymer electrolyte fuel cell, *Nano Energy*, **75**, 104943, Copyright (2020), with permission from Elsevier.¹⁰² (d) proposed catalyst layer structure for a PEMFC cathode with an “ideal” sonication procedure vs. a “poor” sonication procedure. Reprinted with permission from Wang *et al.*, *ACS Appl. Energy Mater.* Copyright (2019) American Chemical Society.¹⁰⁴

time. Ink compositions need to be optimized for each catalyst-ionomer pair, which can otherwise lead to significant discrepancy between research groups and research publications, as shown previously in the PEMWE and PEMFC spaces.^{102–107} For example, the water and alcohol content in the ink solution affects the surface tension of the ink, which, in turn, impacts the dispersion of the catalyst in the ink and the ink drying rates, all of which can significantly impact catalyst layer microstructure (Fig. 3c), as shown previously for PEMFCs.^{105,107} The ionomer content can similarly impact ink rheology as well as catalyst dispersion, which has been shown to have significant impacts on catalyst layer microstructure in PEMFCs^{103,105} and PEMWEs.¹⁰⁶ Catalyst agglomeration in inks can lead to less-than-ideal surface areas of nanoparticle catalysts, impacting activity. Different sonication procedures can help mitigate catalyst and ionomer agglomeration, as has been shown in PEMFCs (Fig. 3d),¹⁰² and such procedures must be developed on a case-by-case basis depending on the catalyst-ionomer pair used. The results depicted in Fig. 3c and d show that ink design choices can have substantial effects on catalyst layer structure; such lessons from these results are importantly transferable to AEMWEs. Although optimizing each variable can be challenging and time consuming, the abundance of tuning variables can provide a high degree of control in terms of catalyst layer thickness and microstructure. These spraying techniques also have the advantage of facile scalability, adaptation to existing commercial manufacturing methods (*i.e.*, roll-to-roll coating),¹⁰⁸ and the ability to further optimize for cost and processing needs.

Ink-spraying methods generally require the use of an ionomer or a polymeric binder to adhere the catalyst particles to the substrate (*i.e.*, membrane or transport layer). The ionomer may

also serve as an ion conductor that helps to shuttle OH[−] ions through the catalyst layer to/from catalyst active sites. This is especially critical in pure water operation, where the ionomer phase is the sole ion conductor. Yet, such ionomers or polymers can inhibit electronic conductivity pathways between catalyst particles and between the catalyst and transport layers, as well as block catalyst active sites, leading to decreased cell performance. The catalyst can also facilitate oxidation and loss of the ionomer, leading to instability within the catalyst layer and to long-term durability concerns,^{54,109,110} which will be discussed in more detail in Sections 3.3 and 6.

In supporting electrolyte, anions in the bulk-liquid phase provide additional OH[−] conductivity and an ionomer may not be required as an ion conductor. As a result, catalyst deposition techniques that do not require the use of ionomer or polymeric binder can be used, reducing concerns of site-blocking by the ionomer and of inhibited electronic conductivity. If desired, an ionomer can be integrated after electrode coating to facilitate operation in pure water feeds. Such “direct” deposition methods that bypass the use of ionomers include electrodeposition, chemical vapor deposition, physical vapor deposition (*e.g.*, sputtering),¹¹¹ and plasma deposition,^{91,112,113} which differ in the ways to deposit active catalysts on to the cell components.

In the electrodeposition method, the substrate material (often the transport layer) is submerged in an electrolyte containing metal ions of the desired catalyst type, and potential is applied to deposit the metals on the substrate.^{114,115} These materials can be grown as oxides or oxidized *ex situ* to the desired form. Unlike airbrush spraying, electrodeposition methods avoid ink rheology concerns and discrepancies; instead, catalyst layer thickness and structure can be precisely controlled *via* changes to the concentration and identity of the



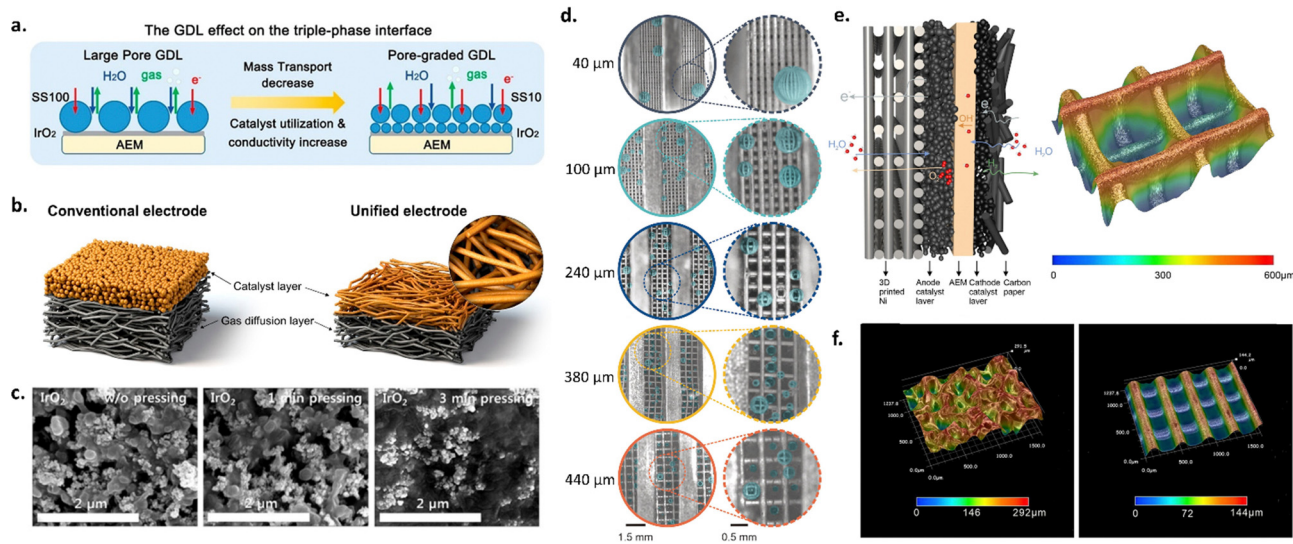


Fig. 4 (a) Schematic of different transport layer strategies and the impact on the catalyst layer–AEM interface. Reprinted with permission from Xu *et al.*, *ACS Energy Letters*. Copyright (2021) American Chemical Society.¹²⁶ (b) Unified electrode strategy. Reprinted with permission from Park *et al.*, *ACS Catal.* Copyright (2022) American Chemical Society.¹²³ (c) SEM images of catalyst layer surfaces with different hot-pressing times. Reprinted from A. Lim *et al.*, A study on electrode fabrication and operation variables affecting the performance of anion exchange membrane water electrolysis, *Journal of Industrial and Engineering Chemistry*, **76**, 410–418, Copyright (2019), with permission from Elsevier.¹³³ (d) Photos of O₂ gas bubbles evolved at 1 A cm⁻² in AEM electrolyzers with different 3D-printed Ni transport layer pore sizes (40 μm, 100 μm, 240 μm, 380 μm, 440 μm). (e) Schematic of the anode and cathode catalyst layers in an AEM electrolyzer (left) and optical microscope image of the 3D-printed Ni transport layer with a pore size of 240 μm (right). (f) Optical microscope images of a commercial Ni foam transport layer vs. a 3D-printed Ni transport layer with a pore size of 240 μm. (d)–(f) Were reprinted with permission from Huang *et al.*, *Angewandte Chemie International Edition*, Copyright (2023) John Wiley and Sons.⁴⁴

for PEMWEs.^{129–131} Hot pressing utilizes a heated hydraulic pump to compress the sprayed catalyst layer at different pressures and temperatures. Hot pressing has several advantages, including decreasing ohmic resistances. Several studies have utilized this technique for AEMWE systems. For example, Cho *et al.* studied the effects of changing the temperature of hot pressing (*i.e.*, room temperature, 50, and 80 °C) for AEMWEs consisting of an IrO₂ anode and Pt/C cathode both with PTFE as the binder material. The authors found that increasing the hot-pressing temperature decreased polarization resistances and led to increased activity; for a cell hot-pressed at 80 °C, the activity was 1.9× higher than for a sample with no pressing.¹³² Later, Lim *et al.* investigated differences in pressing time and found that cell performance decreased with increased pressing time (*i.e.*, from 161 to 118 mA cm⁻² at 1.6 V as the pressing time increased from 0 to 3 min), which they attributed to structural deformation of the catalyst layer and increased blocking of catalyst active sites by the PTFE binder; this deformation with increased hot-pressing time is shown in Fig. 4c.¹³³ The results of these two studies suggest that hot pressing can be advantageous, but that considerations must be made to treatment times to avoid deformation and performance losses.

Focusing on the challenges of transporting electrons, gases, and water through the anode transport layer, Huang *et al.* fabricated 3D Ni transport layers with straight-through pores and variable grid sizes, which were then coated with a NiFe catalyst. They found that there was a trade-off with grid (pore) size between mass transport and catalyst utilization, and thus the best performance was achieved with a gradient in grid size.

Fig. 4d shows the results of their study and the differences in bubble formation for transport layers with different pore sizes. Compared with a transport layer with a pore size of 240 μm throughout, using a top layer with a pore size of 240 μm over a bulk pore size of 550 μm led to a decrease in overpotential of 38 and 100 mV at 1 and 2 A cm⁻², respectively.⁴⁴ The surface morphology and porosities of these 3D Ni transport layers led to improved mass transport vs. a commercial Ni foam transport layer; the differences in these transport layers are shown in optical images from the authors in Fig. 4e and f. Other works have also used transport layer materials directly as electrodes, similar to the approach used in liquid alkaline electrolysis; such works include the study of Ni foam,^{134–137} Ni felt,^{57,138} and SS felt⁵⁷ directly as catalysts in AEMWEs.

3.3 Ionomer content and catalyst/ionomer interactions

The ionomer is often used in the catalyst layer for multiple purposes: (a) to adhere catalyst materials to the membrane or transport layer, (b) to provide ionic conductivity for OH⁻ species (product in HER and reactant in OER) through the catalyst layers, and (c) to facilitate ideal rheological characteristics in catalyst inks for spraying deposition methods. In most studies, the same polymer is used for the ionomer and membrane. Many ionomers/membranes have been developed by research groups^{139–141} and commercially; typical commercial ionomers include Aemion, Sustainion, Fumasep FAA-3, and PiperION.²⁶ Important properties for ionomers include their ion exchange capacity (IEC), water uptake, and swelling, which can deviate from the properties of the bulk membrane



(or another polymeric binder) is viable as an alternative binding agent to ionomer.^{132,146}

Careful consideration of catalyst–ionomer pairs and investigation into any possible reactions between such materials are critical to gain deeper understanding of possible AEMWE degradation mechanisms. Such knowledge has direct implications for catalyst layer design, where electronic and ionic conductivity pathways must be created and maintained. Ultimately, understanding ionomer degradation mechanisms and implementing this knowledge into catalyst layer design can help facilitate the development of AEMWEs with long operational lifetimes and assist their wide-scale deployment.

4. Effects of cell configuration and operating conditions

While many cell assembly variables and cell operating conditions have been standardized for PEMWE systems,^{48,50} such variables remain undecided in the AEMWE space. As discussed in Section 2, discrepancies remain over material choices for cell hardware, including end plates, flow fields, and transport layers. Such material choices alongside changes to assembly and operational variables can directly affect reaction kinetics and mass transport rates, and thereby lead to differences in cell overpotentials and stabilities. This emphasizes the need for understanding and optimizing for these choices, as well as for standardization across laboratories to ensure comparable results. Changes to assembly and operational variables, including cell compression, temperature, and pressure will be discussed in Section 4.1, electrolyte variables and pre-treatment procedures will be discussed in Section 4.2, and the need for and recommendations for establishing baseline operation and standardized protocols will be discussed in Section 4.3.

4.1 Effects of cell assembly and operational variables

Cell assembly variables related to material choices and electrode configurations have been discussed in Sections 2 and 3, respectively. Cell compression is another variable that has been studied for AEMWEs and PEMWEs, which can be changed by altering transport layer or PTFE gasket thicknesses. Gasket stiffness can also play an important role in cell compression; more-stiff gaskets will prevent additional compression occurring when the cell is closed, whereas less-stiff gaskets can lead to additional compression. Higher cell compression can lead to better contact between the catalyst layer and the transport layers/membrane, which, in turn, can decrease Ohmic and kinetic losses and improve cell performance. However, overly high cell compressions can cause membrane tearing or deformation, decreasing cell longevity. Therefore, understanding the implications of cell compression on performance and durability as well as determining preferred compression ranges are important for advancing AEMWE design.

In the PEMWE space, increasing cell compression has been shown to reduce interfacial contact resistances and decrease cell overpotentials, although it may have detrimental effects on

the mass transport of gaseous products by constricting pores in the transport layer and increasing hydrogen crossover through the membrane.¹⁵² For AEMWE systems, recent works provide insights into the effects of varying cell compression on overall performance. For example, Xu *et al.* systematically evaluated several AEMWE single cells with different levels of cell compression. They found that increasing cell compression led to higher cell activities, which they hypothesized was related to increased interfacial contact.¹²⁶

Temperature is an important operational variable, as changes to temperature directly affect kinetic rate constants, thermodynamic equilibrium potentials, and ionic conductivities. The temperature range for low temperature electrolysis technologies (including PEMWE and AEMWE) is generally limited to below 100 °C (*i.e.*, the boiling point for water), with operation commonly between 50 and 90 °C.² Lim *et al.* evaluated the effects of changing cell operating temperatures in the range of 50 to 90 °C and found that at a constant overpotential of 591 mV, there was a +50 mA cm⁻² increase in current density for every 10 °C increase in temperature. They attributed this higher activity to increased ionic conductivity in the membrane, ionomer, and electrolyte and to increased electrochemical kinetics.¹³³ Ionic conductivity is sensitive to temperature in the range of 0 to 100 °C, which is known to increase with temperature. For example, OH⁻ transport *via* the Grotthuss ion hopping mechanism is sensitive to temperature in this range.¹⁵³ The authors further saw a 22.6% decrease in Ohmic resistance and a 21.9% decrease in kinetic resistances measured by *in situ* electrochemical impedance spectroscopy (EIS) as the temperature increased from 50 to 90 °C, demonstrating that higher temperatures are desirable for high cell activities.

Similarly, in a recent study by Capri *et al.*, the authors studied an AEMWE containing Fumasep FAA3-50 membrane and ionomer, NiFe₂O₄ anode catalyst, and Pt/C cathode catalyst at different temperatures (30–60 °C) and saw a similar increase in performance with increasing temperature. At 2.2 V, they reported an increase of +500 mA cm⁻² with every 10 °C increase in temperature; their cell produced 1.35 A cm⁻² of current at 30 °C and 3 A cm⁻² at 60 °C, one of the highest reported current densities for AEMWE. The authors similarly attribute this performance gain to an increase in both ionic conductivity and electrochemical kinetics, supported by decreasing Ohmic and kinetic resistances observed in *in situ* EIS.¹³

These works have clearly shown that operating at higher temperatures has positive effects on cell activities. Increased temperatures, however, can also accelerate component degradation rates, leading to longevity concerns. Furthering understanding of how temperature impacts component-level and cell durability will be critical, including membrane/ionomer degradation pathways, catalyst–ionomer interactions, and transport layer and flow field oxidation.

Application of H₂ backpressure is another cell operational variable of interest. H₂ backpressure applied at the cathode can reduce H₂ separation and compression costs by mitigating downstream processing costs.^{2,4} In fact, commercial PEMWEs often operate with 15–30 bar of backpressure at the



electrolyte and how it can affect catalyst layer structure and overall cell activity and stability are still debated.

Near surface pH changes have been suggested to play a role in catalytic activities and stabilities. Lei *et al.* compared AEMWE performance in pure water vs. two different supporting electrolytes, 10 mM KOH and a phosphate buffer. These two supporting electrolytes had a similar pH of 12 but were expected to experience different near-surface pH swings during water splitting reactions. The authors showed that near-surface pH changes led to catalyst morphological changes for both pure water and 10 mM KOH operation; such changes are evident in SEM images of the anode catalysts before and after the reactions, as shown in Fig. 5a–d. They further demonstrated that using a phosphate buffer precluded this reconstruction, as shown in Fig. 5e and f. In the pure water and KOH-fed cases, the authors hypothesized that there was a loss in ionic contact related to this reconstruction (Fig. 5g and h). Such reconstruction led to cell deactivation in pure water operation, although this effect was less prominent in KOH-fed cells because the additional ionic conductivity provided by the supporting electrolyte was sufficient to overcome losses in the ionomer network.⁸²

Zhang *et al.* similarly noted surface reconstruction effects for Ni, Fe, and Co-containing oxide catalysts at different pH conditions (*i.e.*, pH = 7, 12, and 14). Specifically, the authors showed a decreased crystallinity for all samples in X-ray diffraction (XRD) measurements after testing (*i.e.*, 6 h at 0.5 mA cm⁻²) in pure water conditions. They also suggested that high pH (*i.e.*, 14) was necessary to enhance stability of these non-PGM catalysts, especially those containing Fe.¹⁶⁵ Such results indicate the importance of understanding both near-surface pH effects and the role of the ionomer in AEMWEs with supporting electrolyte.

The identity of cations has also been reported to impact cell performance. For example, some works have suggested that

cations can interact with surface-adsorbed *OH and affect OER activities.^{161,166} Kiessling *et al.* demonstrated that cations with larger charge densities have more significant stabilization of adsorbed *OH, resulting in cell activities in the order of K⁺ > Na⁺ > Li⁺, where K⁺ yielded the highest cell performance.¹⁶¹ Possible cation effects on HER have also been proposed. For example, Chen *et al.* proposed that, with increasing pH, positively charged cations co-adsorb with OH, shifting the H adsorption peak potential. They showed that this shift was also impacted by cation strength, with further delays in the H adsorption potential in the order of Li⁺ < K⁺ < Na⁺ < Cs⁺, where Cs⁺ resulted in the largest delay.¹⁶⁷ These results further provide insights into the disputed non-Nernstian pH dependency of HER. Additional discussion on the role of cations in HER can be found in the review by Jia *et al.*¹⁶⁸

Changes to electrolyte composition during testing have been shown to impact cell performance. For example, during operation in pure-water, the activity and durability can be affected if there is residual KOH (or other supporting electrolyte) in the reactor lines.^{146,160} Hassan *et al.* demonstrated this in their study of an AEMWE with an IrO₂ anode catalyst, PtNi cathode catalyst, and XION membrane (from Xeryg Inc.). They showed that when there was residual KOH in the lines, cell activity was initially higher but degraded faster.¹⁶⁰ Similarly, Kiessling *et al.* reported significant activity loss after switching from 1 M KOH to pure water and back to 1 M KOH (Fig. 5j).¹⁶¹ Relatedly, Lei *et al.* showed that extended AEMWE operation using a phosphate buffer as the supporting electrolyte led to phosphorylation of the membrane/ionomer and subsequent performance losses. They demonstrated that these losses could be reversed with brief exposure to 0.01 M KOH feeds (Fig. 5i).⁸² These results suggest that electrolyte identity and flow protocols can significantly change performance results.

Electrolyte flow configuration can also play a role in overall cell performance. Cathode dry operation is desirable due to the

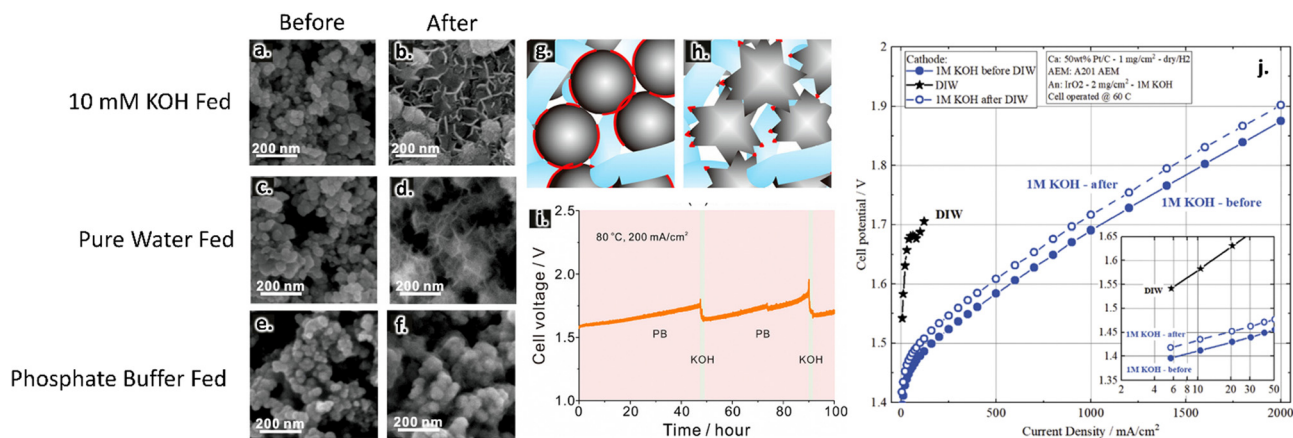


Fig. 5 Anode catalyst layers before testing (a), (c) and (e) and after testing (b), (d) and (f) in 1 mM KOH (a) and (b), pure water (c) and (d), and phosphate buffer (e) and (f) at 200 mA cm⁻². Images collected using SEM imaging. Proposed mechanism of catalyst deactivation in pure water feed; blue phase represents the ionomer, grey phase represents the catalyst, and the red phase indicates regions of catalyst-ionomer contact (g) and (h). Prolonged stability test in phosphate buffer where the solution was periodically refreshed with KOH (i). Reprinted with permission from Lei *et al.*, *ACS Sustainable Chemistry & Engineering*, Copyright (2022) American Chemical Society.⁸² Effect of pre-feeding DIW on AEM activity (j). Reproduced from Kiessling *et al.*, *Journal of the Electrochemical Society*, Copyright (2021).¹⁶¹



well-known differences in achievable performance based on operating temperature, PGM *vs.* non-PGM catalysts, and pure water *vs.* supporting electrolyte operation, but little consensus has been reached in the field about what conditions should be considered standard. Consequently, there is a lack of baseline protocols and metrics for cell testing that would allow for direct comparison across research groups.

As a start, the AEMWE field can borrow some best practices from PEMWE. There have been efforts within the EU⁴⁹ and the U.S. DOE's H2NEW consortium^{48,50} to develop standard testing protocols with set MEA materials, loadings, and processing steps to serve as a benchmark for comparison. There have also been efforts to eliminate intra-group performance variation due to differences in test stations and protocols through round robin testing of identical MEAs.⁴⁷ Importantly, these benchmarks do not need to have the highest performance or most advanced materials; instead, the emphasis is on using commercial materials that are widely available to all research groups. Initial efforts to standardize protocols for AEMWE have been conceptualized and started through the U.S. DOE's HydroGEN consortium¹⁷⁴ and within the IEA,¹⁷⁵ but, as of yet, decisions regarding appropriate baselining and benchmarking remain undecided.

Because there are several concurrent lines of research in AEMWE, there could be several benchmarks, for example covering PGM and non-PGM catalysts, pure water and supporting electrolyte operation, and different classes of membrane chemistries. Along with having defined materials, the benchmarks would also need a standard testing protocol and performance and/or durability metrics for comparison. Methods for taking polarization curves, impedance spectroscopy, and durability tests should be standardized. Standard metrics for comparing performance (*e.g.*, voltage at 1 A cm⁻², current density at 1.8 V or at a voltage efficiency of 68%¹⁷⁶) and durability (*e.g.*, degradation rate in mV h⁻¹ at 1 A cm⁻²) are also necessary.

Based on the current state of the field, a reasonable benchmark cell, particularly to serve as a comparison for novel membranes and anode catalysts (*i.e.*, PGM cathode, supporting electrolyte), would be a commercial NiFeO_x anode catalyst and Pt/C cathode catalyst deposited onto Ni and C transport layers, respectively. While many forms of NiFeO_x have been studied, one of the most common, commercially available forms is NiFe₂O₄. This material has been discussed in the literature as a possible non-PGM benchmark catalyst for alkaline OER.⁷⁰ Although NiFe₂O₄ lacks desirable materials properties (its composition contains more Fe than has been reported preferable,^{52,177} it has relatively poor electronic conductivity, and available particle sizes are relatively large (~30 nm)), it is commercially available in relatively large quantities. Furthermore, for catalysts with similar particle sizes, differences between three different commercial suppliers of NiFe₂O₄ have been shown to be low in RDE testing;⁷⁰ NiFe₂O₄ may therefore be a reasonable candidate for a benchmark OER catalyst. The best choices for the anion exchange membrane and ionomer are yet to be known, which has implications for the choice of

benchmarking temperature and supporting electrolyte. As a baseline to maximize performance, we recommend 1 M KOH and 80 °C, although these operating conditions may not be accessible to all membrane chemistries and can hasten degradation. While activity metrics at either a particular current density or voltage are relevant for demonstrating progress towards performance targets, such as the 3 A cm⁻² at 1.8 V (2026 target for PEMWE from the U.S. DOE),⁸ a current density-based metric is preferred for comparison due to the widely varying high frequency resistances (HFR) in AEMWEs that significantly affect measured cell voltages. Specifically, we recommend reporting the HFR-free voltage (to account for differences in cell hardware and membrane resistances) at 1 A cm⁻², an industrially relevant current density. For durability assessments, the U.S. DOE PEMWE and LAWE targets are stated in terms of a mV h⁻¹ increase at a constant current density;⁸ we therefore recommend that durability measurements be conducted at a moderate, constant current density of 1 A cm⁻².

In addition to establishing common performance metrics, it will be important to standardize best practices for materials characterization before and after testing. Component-level characterization of catalysts is common, but additional characterization of fully assembled cells is necessary to understand changes within the catalyst layer. The following section will discuss recent advances in *in situ* and *ex situ* techniques to understand such changes.

5. *Ex situ* and *in situ* diagnostic techniques to understand changes in the catalyst layer

Characterization of the used electrode and membrane materials is necessary to understand processes within the catalyst layer for AEMWEs. Changes to the chemical states of the catalysts, to the distribution of catalyst and ionomer in the catalyst layer, and to the morphology and porosity of the catalyst layer are all of interest to study. Relevant characterization techniques include microscopy, tomography, spectroscopy, X-ray techniques, inductively coupled plasma-mass spectrometry (ICP-MS), and gas chromatography-mass spectrometry (GC-MS) analysis, among others.

Ex situ characterization, which often involves comparing materials before and after testing, provides valuable insights into changes that occurred during testing. It is also typically easier to implement than *in situ/operando* measurements since no specialized cell hardware is required. In RDE studies, it has become the standard to provide pre- and post-test characterizations of catalysts to show what changes have or have not occurred, such as the conversion of “X-ide” (*i.e.*, sulfides, phosphides, *etc.*) OER catalysts to the (oxy)hydroxide form.⁷² There are many differences between the operating environments of RDE and MEAs, however, meaning that there is a need to also implement this type of *ex situ*, post-test



to study bulk and surface chemistries, as well as catalyst layer morphology. This is vital to understanding catalyst activity and degradation mechanisms involving catalysts, ionomers, and membranes.

In half-cell, three-electrode measurements, *in situ* X-ray absorption (XAS); XPS, Raman, and UV-visible spectroscopies; and XRD have been used to characterize compositional, phase, and structural changes of HER and OER catalysts.^{186–190} On-line ICP-MS has further been used to probe the pH- and potential-dependent dissolution of metals from catalysts. While significant insights have been gained from this work that have helped to shape catalyst development efforts, it is important to recognize that materials changes and rates of degradation can be very different in an MEA environment *vs.* in a liquid electrolyte. For example, an on-line ICP-MS study of a PEMFC gas diffusion electrode half-cell found an order of magnitude lower rate of Pt dissolution compared to an analogous study in a three-electrode flow cell.¹⁹¹ They attributed this change to differences in mass transport that shifted the equilibrium potential for dissolution and favored redeposition of dissolved species.

To specifically understand the interactions between the different components in the catalyst layer, *in situ* MEA characterization is needed. Peng *et al.* used *operando* X-ray computed tomography to understand catalyst layer utilization and mass transport in an ultra-low Ir loaded PEMWE using various Ti transport layers with uniform porosity.¹⁹² They found a significant inverse relationship between porosity and contact area, with an intermediate contact area percentage found to be optimal for catalyst utilization. Although mass transport resistance is minimized for a transport layer with the highest porosity, kinetic and ohmic overpotentials are minimized at intermediate porosities and contact areas. Ampurdanés *et al.* utilized a similar PEMWE cell with X-ray transparent Kapton windows in both the end plates and holes in the current collectors to conduct *in situ* Co K-edge and Ir L₃-edge XAS measurements in transmission mode (Fig. 7d).¹⁹³ Further development of these *in situ* cells, particularly for AEMWE devices, is needed.

Other characterization methods provide insights into system-level operation and the origin of performance losses. On-line GC-MS is often used for Faradaic efficiency and H₂ crossover measurements. These measurements can be used to assess the effects of operating conditions on H₂ crossover. *In situ* EIS is often used to study changes in ohmic, kinetic, catalyst layer, and mass transport resistances to better understand the origin of overpotentials within the MEA. The development of methods to quantify the electrochemical surface area (ECSA) of non-PGM oxide catalysts is also necessary. Currently, ECSA determination is largely limited to estimations of the double layer capacitance, either from cyclic voltammetry^{68,194} or EIS^{195,196} measurements. These methods have inherent errors and are reported to provide surface areas accurate to within an order of magnitude.^{68,194}

Recently, there have been efforts to develop reference electrodes that can be integrated into the single cell to separate the

contributions of the anode and the cathode.^{85,147} Xu *et al.* demonstrated the use of a Ag/AgCl reference electrode, connected to the cell *via* a hydrated strip of membrane, to measure polarization curves and impedance spectroscopy. They found that changes to the morphology of the anode transport layer led to decreased anode overpotential and charge transfer resistance with no impact on cathode performance, whereas an increase in cathode transport layer thickness affected both the anode and cathode.¹²⁶ While there are challenges to successful implementation of reference electrodes, such as maintaining hydration and good connection with the MEA, reference electrodes will be a powerful tool for understanding the impacts of materials integration and operational choices on individual components of the MEA.

To understand and improve AEMWE performance and durability, it is necessary to understand the origin of losses, the condition of materials within the device, and degradation mechanisms. *Ex situ* characterization of cell components and the catalyst layer, such as through microscopy and spectroscopy, allows for post-mortem analysis of material changes and effects on interfaces and integration. *In situ* techniques have been employed extensively in half-cell, three-electrode measurements, but it should not be assumed that these insights will correlate directly with material behavior in single cells due to differences in the reaction environment and the increased importance of interactions between components in the single cell. Further efforts to design cells and techniques for *in situ* characterization in AEMWEs are needed, and *ex situ* materials characterization should be employed more frequently for improved understanding of the catalyst layer.

6. Catalyst layer stability and stability testing protocols

Currently, AEMWE cells have demonstrated lifetimes on the order of 10 000 h, but this likely needs to increase by at least an order of magnitude to be competitive with other technologies.⁴

Individual, component-level stability of catalysts^{21,31,197} and membranes^{21,40,172,198} has been reviewed previously. For catalysts, it is generally understood that oxidation and dissolution are the biggest contributors to degradation. For membranes, swelling and oxidation of the backbone are key concerns. These effects can be compounded and made more complicated at the device level, where the integration of components and cell operating variables can have dramatic effects on stabilities. This section will discuss durability considerations for AEMWE operation, with a focus on degradation mechanisms that arise due to the integration of components and cell operational variables.

Membrane hydration is a concern for long-term AEMWE operation, especially in cathode dry operation (discussed in Section 4.2 based on the results of Koch *et al.*¹⁷¹). Wang *et al.* further assessed water transport within AEMWEs by evaluating the effect of changing catalyst layer properties on the mass-transport resistance of water in AEMWEs. In their first study,



they modified catalyst distribution and thickness on a series of different transport layers, finding that denser catalyst layers promoted more efficient water transport.¹⁶⁹ In a later study, these authors investigated the effects of membrane thickness and catalyst layer porosity to further elucidate the origins of concentration (*i.e.*, transport) overpotentials and to understand water transport in AEMWEs. They concluded that water transport through the membrane is limiting. They further monitored the relative humidity of evolved H₂, finding that when this value decreased, cell performance decreased concurrent with an increase in overall cell resistances.¹⁷⁰ Long periods of rest (*i.e.*, 12+ h) have also been shown to lead to membrane dehydration,¹⁹⁹ which is important to consider especially for dynamic operation of AEMWEs.

Membranes, ionomers, and supporting electrolytes are also subjected to carbonation when the cell or electrolyte reservoirs are exposed to the atmosphere.^{198,200–202} Such carbonation can decrease the ionic conductivity of the ionomers, leading to decreased performance. Carbonation can also change the near-surface pH, affecting activities and stabilities. Parrondo *et al.* initially observed this degradation route for a water-fed AEMWE single cell with a polysulfone-based membrane; they found that short-term degradation was attributable to CO₂ intrusion and subsequent carbonation.¹⁹⁸ This degradation route has also been noted in anion exchange membrane fuel cells (AEMFCs)^{200,201} and CO₂ electrolyzers,²⁰² both of which often employ the same types of anion exchange membranes utilized in AEMWEs. For example, Zheng *et al.* concluded that CO₂ intrusion in an AEMFC resulted in a Nernstian thermodynamic shift in anode potential caused by a pH change associated with increased carbonation as well as decreased kinetics resulting from a lack of OH[−] reactants.²⁰⁰ Such phenomena may translate to AEMWEs.

Other pH effects are critical for AEMWE stability. For example, Mayerhöfer *et al.* studied a CuCoO_x catalyst with an Aemion membrane and ionomer from Ionomr in a single cell AEMWE in both pure water and 0.1 M KOH. They concluded that without supporting electrolyte, the near-surface pH was not basic enough to keep CuCoO_x in a thermodynamically stable form and led to the dissolution of both Cu and Co; these materials are predisposed to dissolve at low pH conditions.¹⁷ They conclude that supporting electrolyte is therefore required to maintain the thermodynamic stability of non-PGM catalysts and that anion exchange polymers are insufficient to provide such a basic environment.¹⁴⁸ Relatedly, Zhang *et al.* evaluated the pH-stability relationship for Ni, Fe, and Co-containing oxides and found that all materials tested had improved stability at high pH (*i.e.*, pH = 14) vs. weakly alkaline (*i.e.*, pH = 12) or pure-water (*i.e.*, pH = 7) conditions. The stability of Fe-containing materials suffered the most with decreasing alkalinity (*i.e.*, at pH 7 and 12 vs. 14) due to increased Fe dissolution measured with ICP-OES.¹⁶⁵

Krivina *et al.* also observed Fe dissolution in pure-water operation. They found that, among a series of 5 non-PGM catalysts (Co₃O₄, NiO, NiCoO₂, Ni_{0.5}Co_{0.5}Fe₂O₄, and NiFe₂O₄), a Ni-rich surface was formed after durability testing in pure

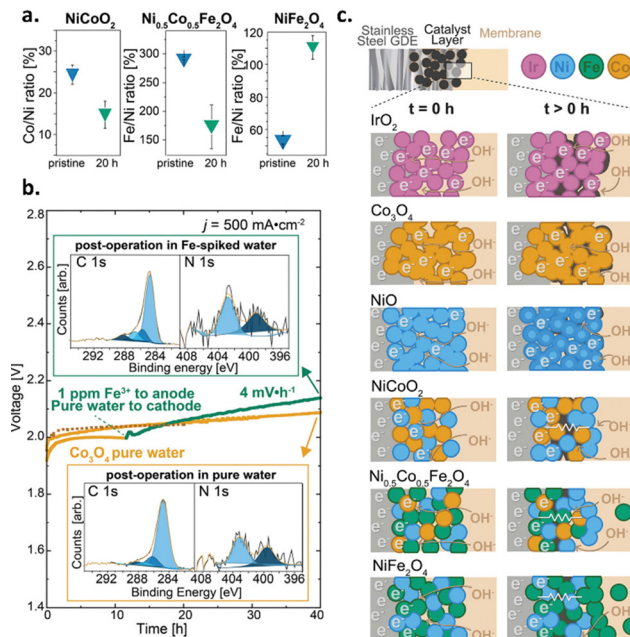


Fig. 8 (a) Metal ratios before and after testing in pure water for bimetallic oxide catalysts measured with XPS. (b) The role of solvated iron species in single-cell degradation, where Fe-spiked water showed increased oxidation of the ionomer vs. pure water evidenced with XPS (inserts). (c) Proposed mechanism of single-cell degradation for six oxide catalysts. Reprinted with permission from Krivina *et al.*, *Advanced Materials*, Copyright (2022) John Wiley and Sons.⁵⁴

water (20 h, 500 mA cm^{−2}) for Co-containing catalysts, whereas for NiFe₂O₄ an Fe-rich surface was formed, as evidenced by XPS results (Fig. 8a). The authors attributed this change to preferential leaching of Fe and redeposition onto the catalyst surface. The authors then intentionally introduced Fe³⁺ species to the electrolyte, resulting in an increased degradation rate that they attributed to a disruption in the catalyst–ionomer network by the dissolved Fe species (Fig. 8b).⁵⁴ Future works that investigate whether this effect persists in supporting electrolyte operation, as well as those focused on the development of methods to stabilize Fe in OER catalysts, are critical.

Additionally, membrane and ionomer swelling at AEMWE operating conditions, especially in pure water feeds, can lead to cell degradation^{98,142,143} and catalyst detachment and delamination^{1,148} Chen *et al.* studied the effects of polymer swelling in the membrane vs. the ionomer in the catalyst layer separately, to probe if there was a difference in how performance was affected. This study used Ir as the anode catalyst, Pt/C as the cathode catalyst, and HMT-PMBI as the membrane and ionomer. In doing so, they demonstrated that their crosslinking strategy resulted in a 4× decrease in volumetric ionomer swelling. With different degrees of crosslinking in either the membrane or ionomer in the catalyst layer, the authors were able to selectively change the water uptake capacity and probe ionomer swelling for each location individually. They concluded that membrane swelling has a minimal impact on overall performance in pure-water feeds, but found that



preventing ionomer swelling in the catalyst layer lead to a 4-fold increase in the lifetime of the cell.⁹⁸ These results suggest that limiting ionomer swelling in the catalyst layer is essential to achieving high cell stabilities.

To isolate the electrochemical stability of ionomers (*i.e.*, without interference from the catalyst) in different supporting electrolytes and oxidizing conditions, Krivina *et al.* used a quartz crystal microbalance to measure mass loss as a function of applied potential for films of ionomer. They found that the ionomers gained mass at open circuit voltage due to hydration, particularly in pH 10 and 14 electrolyte. Aemion fully delaminated at 700 mV overpotential in pH 10, whereas Sustainion and PiperION had 5–10% mass loss in pH 14 at the 700 mV overpotential.²⁰³ These results show that ionomer can be oxidized and degraded due to exposure to oxidizing potentials and alkaline electrolyte, even without the presence of a catalyst.

Catalyst–ionomer interactions can also lead to degradation of the ionomer and cell deactivation. This is especially critical in pure water feeds, where the ionomer is essential to provide ionic conductivity through the catalyst layer. Krivina *et al.* recently conducted a comprehensive assessment of degradation mechanisms for AEMWEs operated in pure water with different PGM (IrO₂) and non-PGM (NiO, Co₃O₄, NiCoO₂, Ni_{0.5}Co_{0.5}Fe₂O₄, and NiFe₂O₄) anode catalysts and with PiperION membranes and Versogen ionomers.⁵⁴ Using post-test XPS (C, N, and F 1s) spectra, they found that IrO₂ facilitated significant ionomer degradation, which they attributed to high electronic conductivity and more active sites for ionomer oxidation reactions. They found that non-PGM catalysts also facilitated ionomer degradation, but to a lesser extent due to the decreased electronic conductivity of these systems compared to IrO₂. Fig. 8c shows the proposed effects of these losses on cell performance; IrO₂ and Co₃O₄ are expected to have sufficient electronic conductivity to maintain accessibility to active sites that are co-located with remaining ionomer at the membrane interface. Conversely, NiCoO₂, Ni_{0.5}Co_{0.5}Fe₂O₄, and NiFe₂O₄ are predicted to have insufficient electronic conductivity through the catalyst layer, creating a gap between regions accessible to electrons and to ions. Understanding and controlling these catalyst–ionomer interactions will be essential for improving durability in pure water operation.

AEMWE currently lacks standard operating and reporting protocols, including for durability testing. Many studies evaluate cells using potentiostatic or galvanostatic holds at suggested operating levels (*e.g.*, around 2.0 V or 1 A cm⁻²).^{38,178,204} While these tests provide insights into degradation mechanisms that arise due to the harsh, oxidative operating conditions, the practical operation of AEMWE electrolyzers coupled with variable renewable energy sources will likely operate at on/off operation. To assess the effects of such operation on durability, other studies have investigated the use of cycling tests, where the voltage or current is cycled from high to low,^{40,179} and periodic voltage bias.²⁰⁵ Campagna-Zignani *et al.* evaluated cells with a 1 A cm⁻² hold and with 0.2 to 1 A cm⁻² cycles. They found that for the current hold case, the performance leveled off after initial deactivation. For

the cycling test, they observed similar behavior, but the steady-state activity was less than that for the hold test. They attributed this to semi-reversible losses observed during the cycling tests.³⁸ Further investigations into the effects of cycling on the chemistry of the catalysts (*e.g.*, change to phase, crystal structure), the chemistry of the ionomer (*e.g.*, if there are accelerated degradation/oxidation pathways) and to the catalyst layer structure are necessary to understand how these cells will behave in practice.

The timescales for comparing stability are also critical. Many cells show dramatic changes in the first 10s of hours of operation, and then level off to a steady state degradation rate. For example, Campagna-Zignani *et al.* tested a cell composed of NiFe anode, NiMo/KB cathode, and Fumatech FAA-3-50 membrane at 1 A cm⁻² and observed initial rapid degradation for 100, which leveled off at 80% of the initial efficiency over the remaining 2000 h operation.³⁸ Appropriate timescales for reporting and comparing stability must therefore be established. Further, the development and validation of accelerated stress tests that can be completed on shorter time scales, and which are employable by many researchers, are necessary.

7. Conclusions and future outlooks

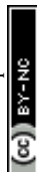
Interest in AEMWE is rising rapidly as the need for lower-cost, green H₂ production technologies compatible with intermittent variable renewable energy sources rise. Recently, component-level advances have dramatically enhanced AEMWE activities and stabilities, and cells with high operating current densities (*e.g.*, >3 A cm⁻²) and up to 10 000 h operation have been reported. While significant, these achievements fall short of commercialization goals, and a critical next step in AEMWE research will be to expand on these component-level advances and move towards a more-rigorous understanding of device integration, especially on understanding, designing, and developing the catalyst layer.

While AEMWE can adopt some catalyst-layer design strategies from the PEMWE and PEMFC spaces, fundamental differences in the relevant chemistries for water splitting reactions, ionomer and catalyst materials, and degradation mechanisms necessitate AEMWE-specific research efforts. In terms of materials, questions surrounding the relevance of the electronic conductivity of non-PGM oxide catalysts, which have much lower conductivities than the PGMs used in PEM systems, remain. Furthermore, while many PEMWE materials choices have been standardized, there is no such consensus in the AEMWE community regarding the preferred choices for catalysts, membranes and ionomers, transport layers, and/or electrolyte composition.

For AEMWE electrode design, decisions must be made about preferred deposition methods and configurations. Spray techniques lend themselves most readily to developed manufacturing techniques, such as roll-to-roll coating, used in the PEMFC space. Modification and optimization of ink formulations, rheological parameters, and spray temperature and times



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