



Energy Materials Network
U.S. Department of Energy



HydroGEN
Advanced Water Splitting Materials

HydroGEN: Low Temperature Electrolysis

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National Renewable Energy Laboratory

Project ID # P148A

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DOE Hydrogen Program 2021 Annual Merit Review and Peer Evaluation Meeting

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Overview - LTE Technology Relevance / Impact

Proton exchange membrane (PEM)

- Gas Crossover
- Membranes
- Catalyst Materials
- Catalyst Loading
- PTL Materials

Anion exchange membrane (AEM)

- Membranes
- Catalyst
- Ionomer
- Electrolyte feed
- BOP Materials

Common Barriers

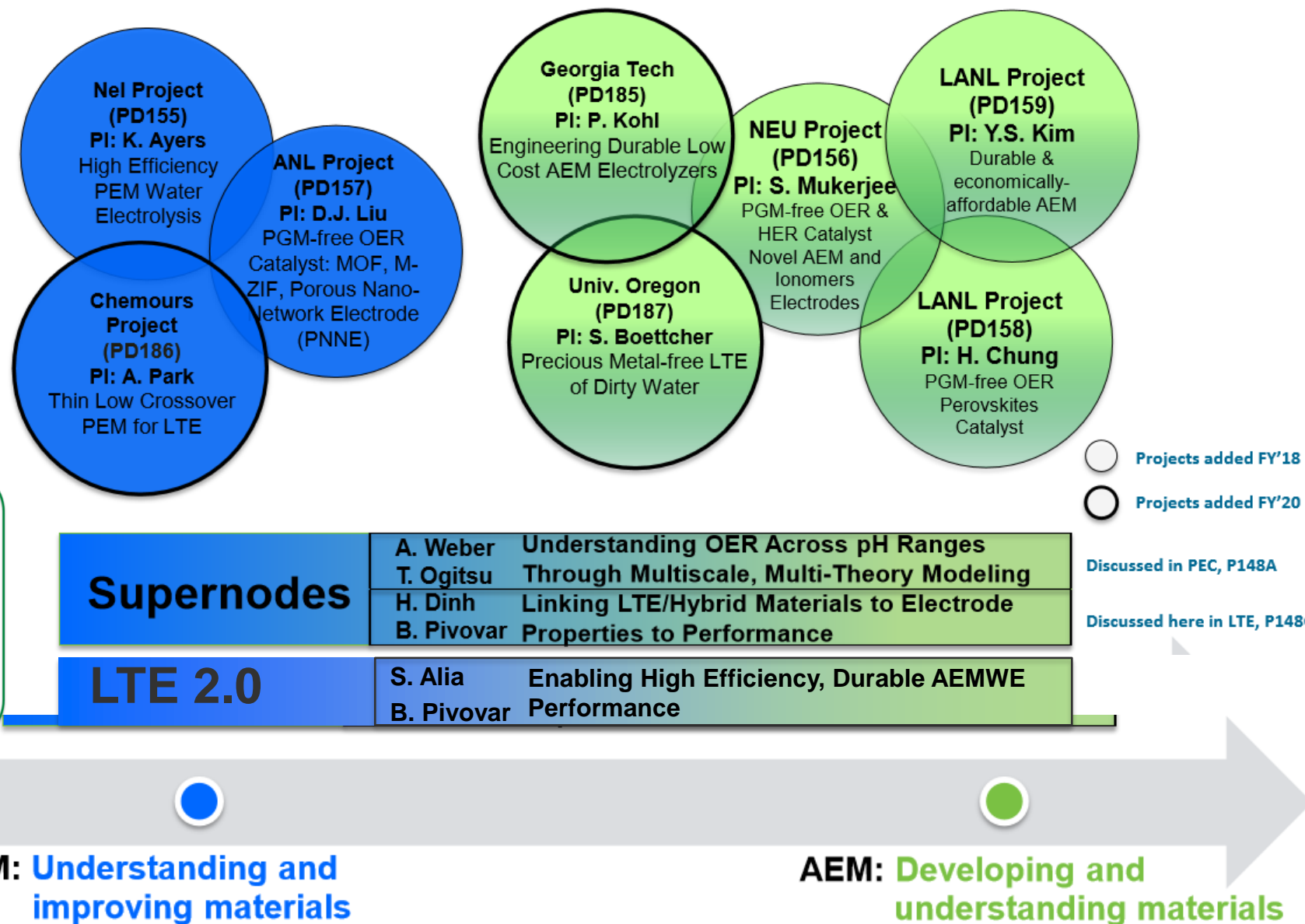
- Material Integration
- Material Cost
- Understanding Interfaces and Interactions



Approach: HydroGEN 2.0 Project Added to LTE Activities

HydroGEN LTE Projects

- 8 FOA projects with 41 nodes
 - 3 currently supported (in Accomplishments)
 - 5 with closeout contributions (in Technical Backup)
- 2 Supernodes with 14 nodes
- LTE 2.0 with 4 nodes



Support through:
Personnel
Equipment
Expertise
Capability
Materials
Data

LTE Node Labs

ONREL
NATIONAL RENEWABLE ENERGY LABORATORY



Sandia
National
Laboratories

Lawrence Livermore
National Laboratory

LTE FOA Projects

nel • Argonne
NATIONAL LABORATORY

Northeastern University
Center for Renewable Energy Technology

Los Alamos (2)
NATIONAL LABORATORY

Georgia Institute
of Technology

Chemours • UNIVERSITY OF OREGON



Collaboration and Coordination - HydroGEN LTE Node Utilization

Lab	Node	2.0	Super	Chemours	GT	Oregon	NEL	ANL	NEU	LANL1	LANL2	Total
LLNL	Computational Materials Diagnostics and Optimization of PEC Devices							✓				1
LBNL	DFT and Ab Initio Calculations							✓		✓		2
LBNL	Multiscale Modeling of Water-Splitting Devices	✓	✓	✓	✓		✓		✓		✓	7
SNL	LAMMPS								✓			1
NREL	Electronic-Structure Modeling for Atomistic Understanding of Catalytic Materials	✓										1
NREL	Novel Membrane Fabrication		✓			✓	✓		✓			4
SNL	Separators for Hydrogen Production					✓				✓	✓	3
NREL	Multi-Comp. Ink Development, High-Throughput Fabrication, & Scaling		✓		✓		✓	✓	✓			5
SNL	Advanced Electron Microscopy							✓				1
NREL	Catalyst Synthesis, Ex situ Characterization & Standardization		✓				✓	✓				3
LBNL	Ionomer Characterization and Understanding	✓	✓	✓		✓	✓		✓		✓	7
NREL	In Situ Testing Capabilities	✓	✓	✓	✓	✓	✓		✓	✓	✓	9
LBNL	Understanding Inks and Ionomer Disp.		✓			✓						2
SNL	Near Ambient Pressure E-XPS									✓		1
NREL	Surface Analysis Cluster Tool							✓		✓		2
LBNL	Probing and Mitigating Corrosion						✓					1
LBNL	PEC In Situ Testing using X-Rays									✓		1
LBNL	Water Splitting Device Testing										✓	1
SRNL	Fabrication and Characterization of Components for H ₂ Production		✓									1

Nodes

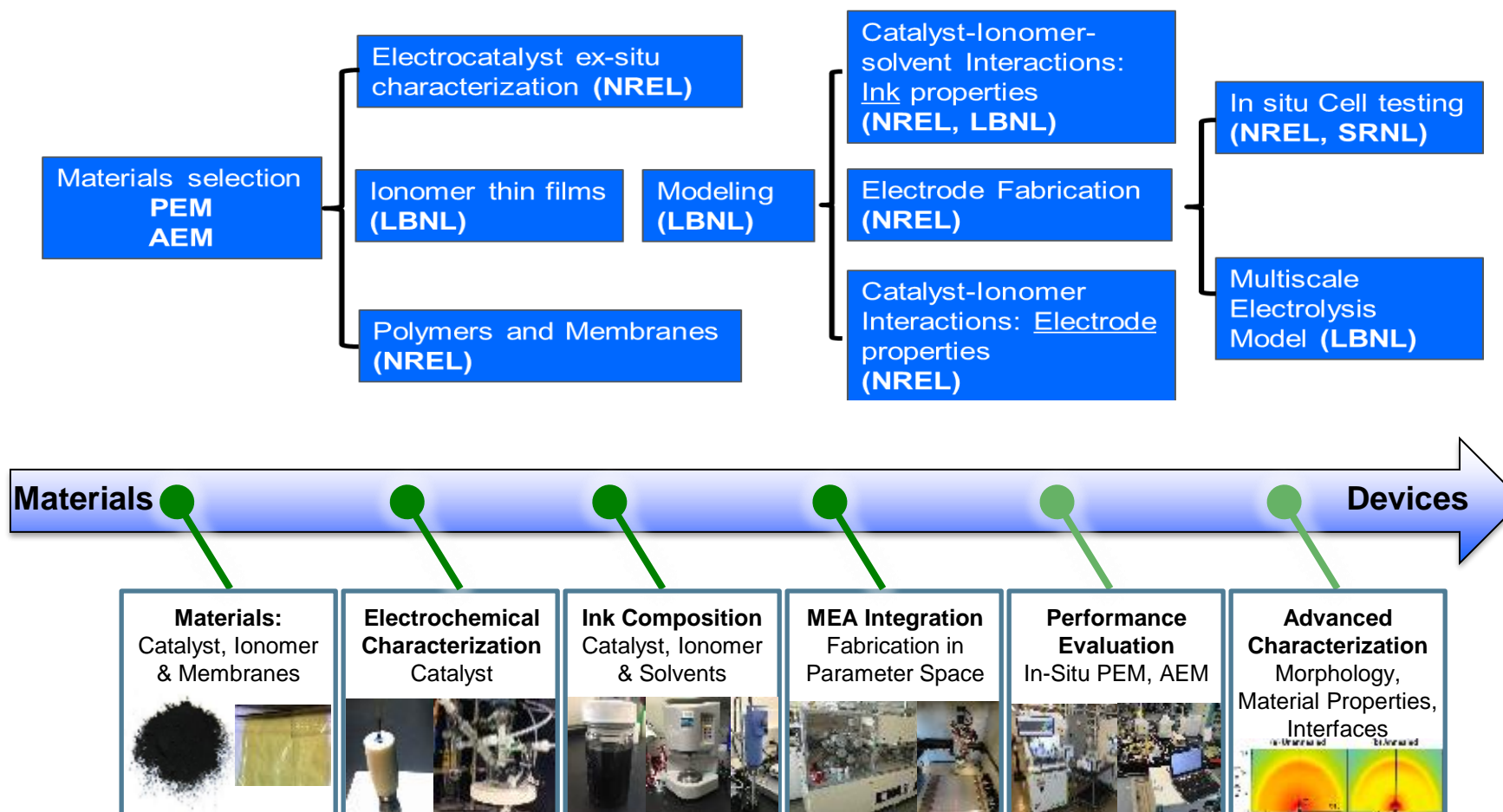
- Computation
- Materials Synthesis
- Processing and Scale Up
- Characterization



LTE Supernode Approach:

Linking LTE/Hybrid Materials to Electrode Properties to Performance

Goals: Create true understanding between *ex-situ* and *in-situ* performance. Identify how material properties are linked to electrode properties and how these are linked to electrolyzer performance.





LTE Supernode Accomplishments:

Roll-to-roll Performance (R2R), Ionomer Impact on Coating Quality



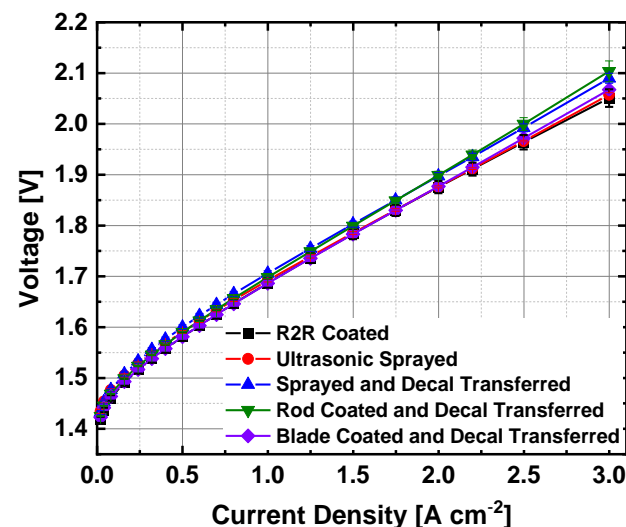
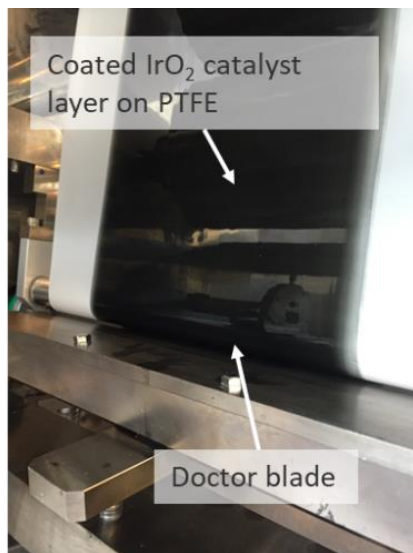
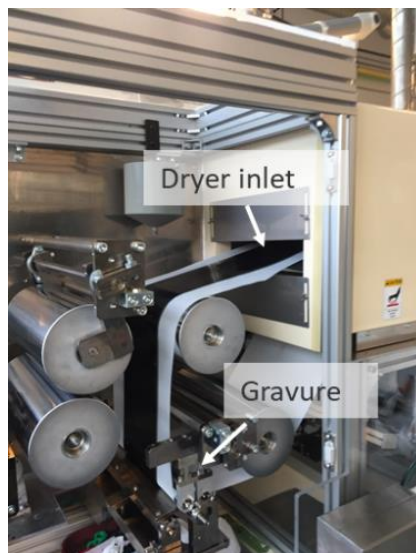
NREL
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8
Nodes



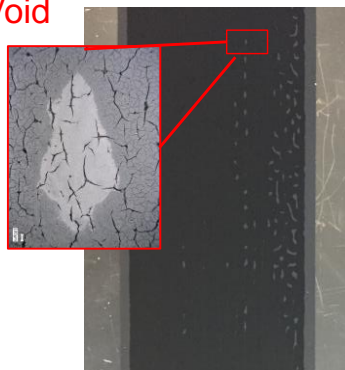
- Demonstrated scalable electrode fabrication methods with similar performance to lab-scale methods
- R2R-coated anodes have high loading uniformity and are defect free

I/C = 0.3

I/C = 0.6

I/C = 0.9

Void



8 cm

Coating Method: Slot die
Pt loading: 0.3 mg/cm²
Substrate: SGL 29BC

I/C	Notes
0.3	Numerous voids
0.6	Sporadic voids
0.9	Highly uniform

Showed that increasing ionomer to carbon ratio (I/C) leads to improved coating quality, likely due to better dispersion of Pt/C



LTE Supernode Accomplishments:

Hybrid Cycle, Ionomer Impact on Performance



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**8
Nodes**



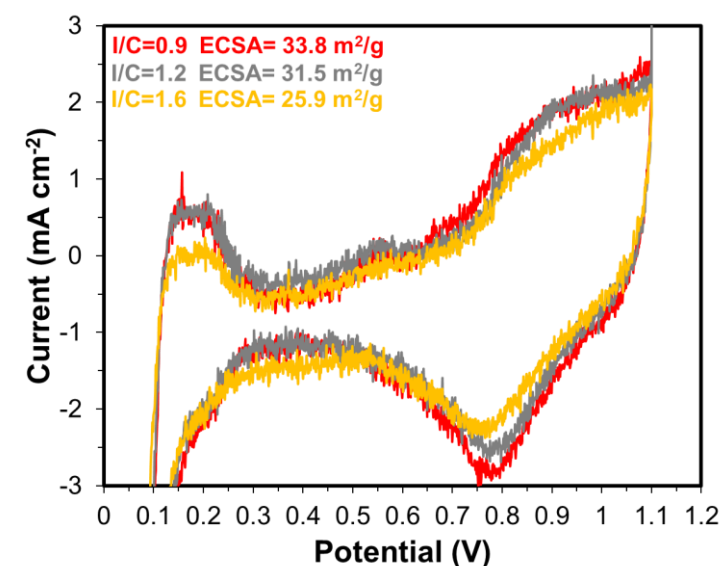
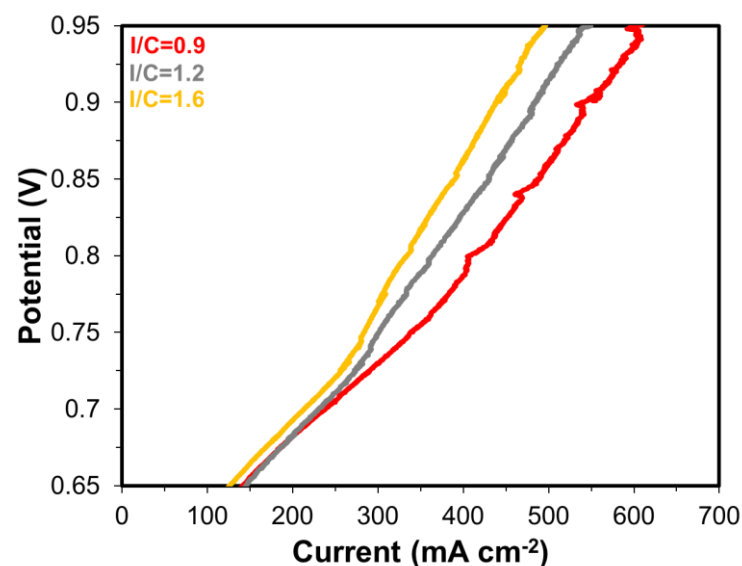
Thermochemical: 800-900 °C



Electrochemical: 80-140 °C
-0.172 V vs SHE (30 wt% & 20 °C)



Net Reaction



- Optimization of ionomer content allows for improved electrode design
- Increasing the I:C ratio significantly impact the high current density region
- Decrease in performance can be partially attributed to lower available electrochemical surface area



LTE 2.0 Approach:

Enabling High Efficiency, Durable AEM Electrolysis Performance

Goals: Determine the role of the supporting electrolyte and the limiting factors behind water operation in AEM electrolysis.

- Evaluate AEM's ability to approach PEM performance/durability in water feeds, leveraging component development efforts in seedling projects
- Examine ionomer-electrolyte effects, how the ionomer's interaction with charged species enhances or diminishes catalytic activity
- Elucidate the role of conformational disorder and molecular motions to changes in catalyst activity via *ab initio* molecular dynamics simulations of the ionomer in contact with the catalyst surface.

State of the Art (Point A)	Overvoltage for AEMWE operation with DI water is in excess of 100 mV of Nafion 112 at 2 A/cm ² .
End of Project Milestone (Point B)	Demonstrate overvoltage for AEMWE operation with deionized water that is within 50 mV of Nafion 112 at 2 A/cm ² . Demonstrate an overvoltage increase for AEMWE operation with deionized water that is within 0.15 mV/h during 500 h of operation at 1 A/cm ² . (NREL, LBNL)

Point A: at the beginning of the project (October 2020)

Point B: at end of project (3 years)



LTE 2.0 Approach:

Linking Modeling, Characterization, and Performance

Materials Selection

Electronic Structure
Modeling (NREL)

Ionomer Thin Films
(LBNL)

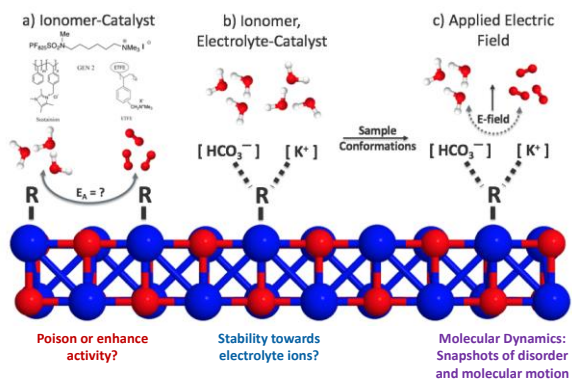
In situ Cell Testing
(NREL)

Multiscale Model
(LBNL)

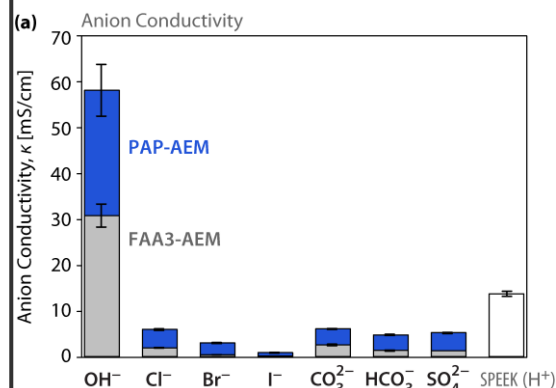
Materials

Devices

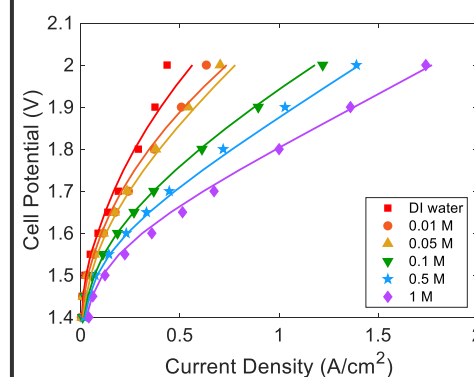
Materials Properties: Catalyst



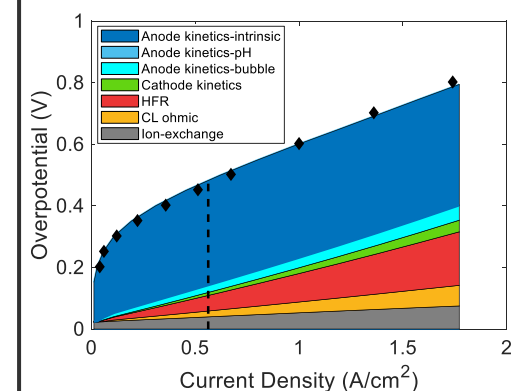
Materials Properties: Membrane



Device: Performance



Device: Analysis



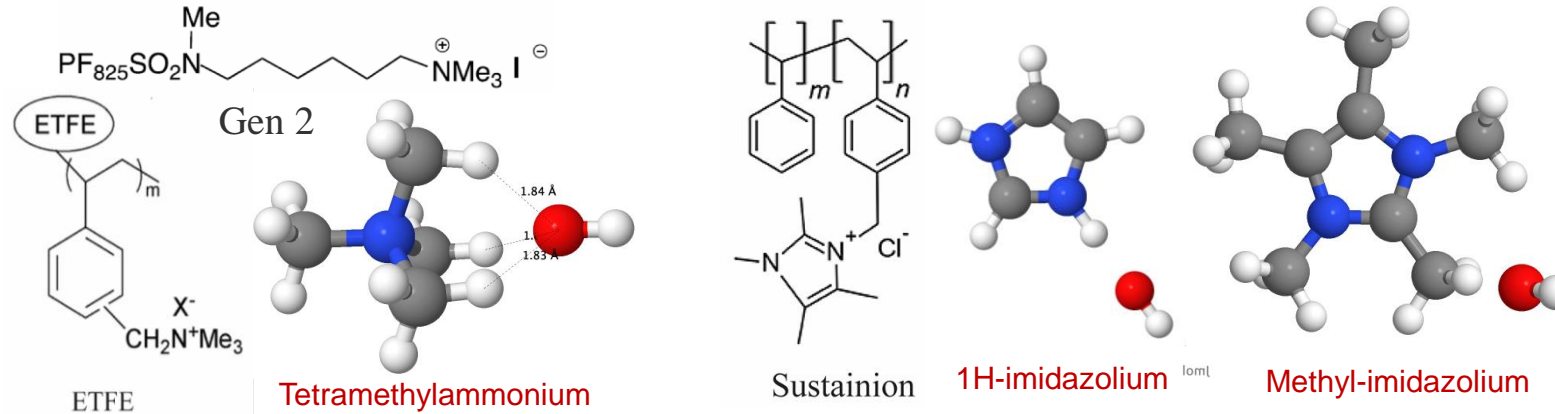


LTE 2.0 Accomplishments:

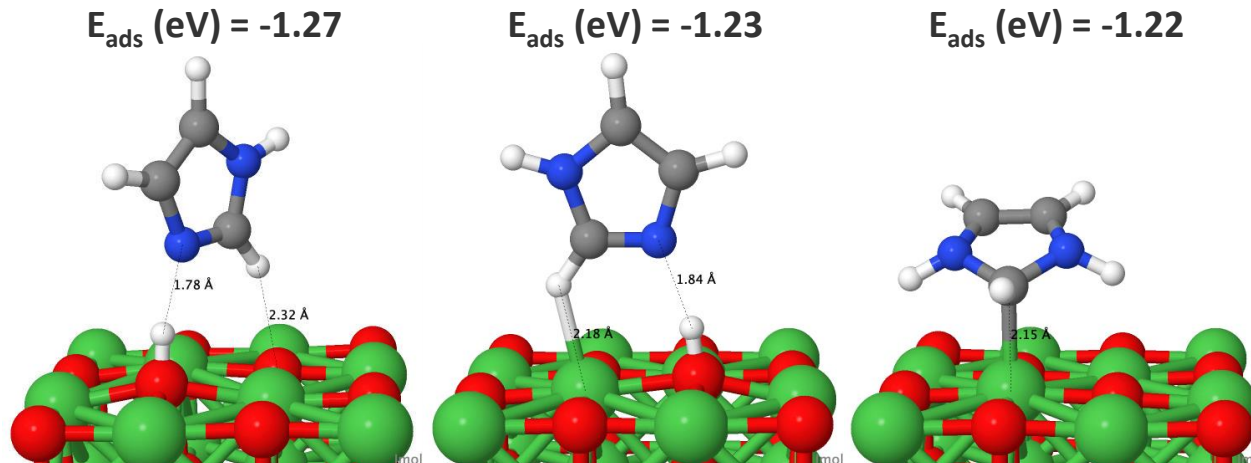
Understanding Ionomer-Electrolyte Effects on Alkaline Oxygen Evolution

Electronic-Structure Modeling

Approximate Ionomer with Smaller Organic Fragments: Gain insights into how N^+R group may transport OH^- to catalyst



Ionomer-Catalyst Interactions: N^+R group can poison activity by blocking sites or influence catalysis by aiding OH^- to catalyst



Preliminary calculations on (2x2) NiO (100) indicate:

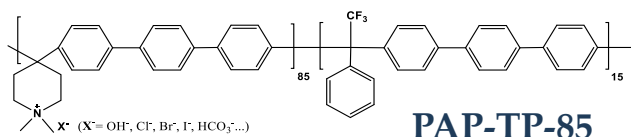
- Imidazolium *more weakly bound* than OH^* ($E_{ads} = -2.12$ eV)
- N^+R group can *react with surface* via deprotonation, supply protons



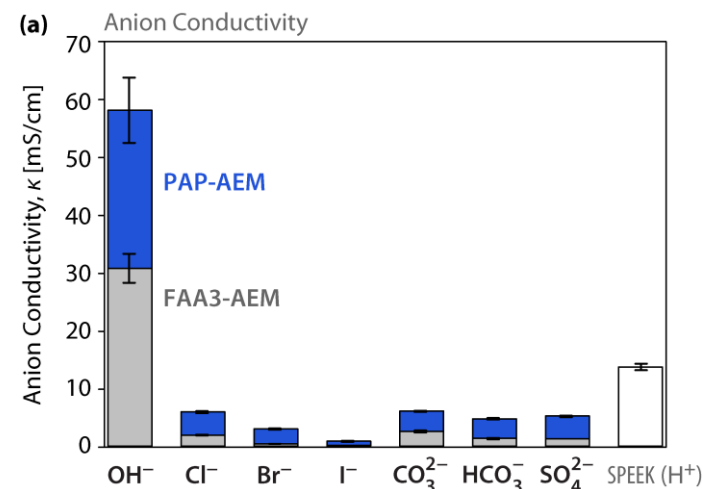
LTE 2.0 Accomplishments:

Membrane Hydration, Structure, and Conductivity

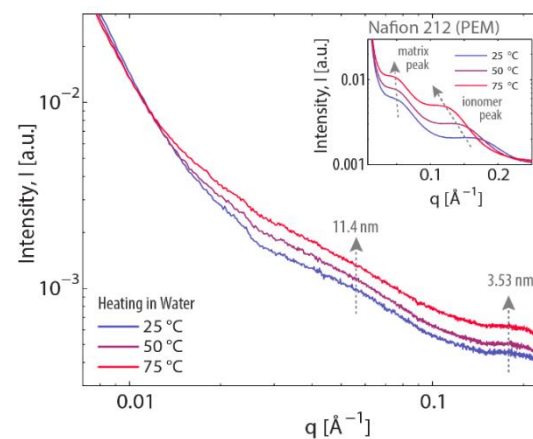
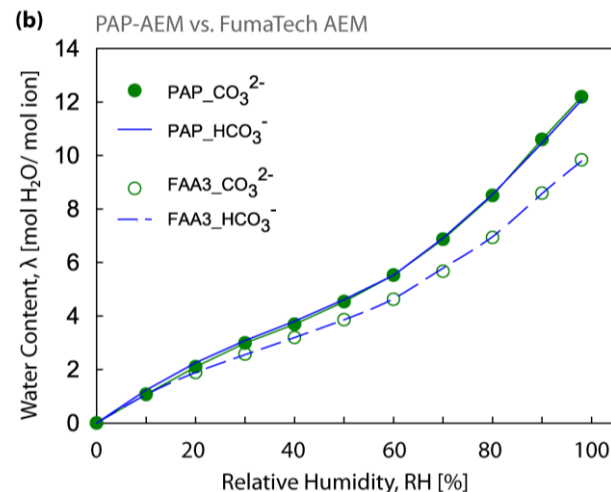
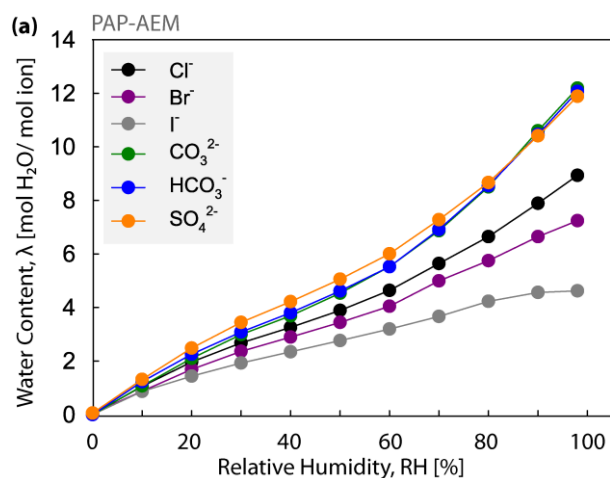
- Anion form impacts hydration and conductivity



- PAP-AEM has higher conductivity than FAA3
- Conductivity is strongly impacted by anion size
- The nano-morphology does NOT change with hydration, counter-ion, and temperature



Conductivity in water in various anion forms: Higher conductivity of PAP-AEM (in blue) compared to FAA3



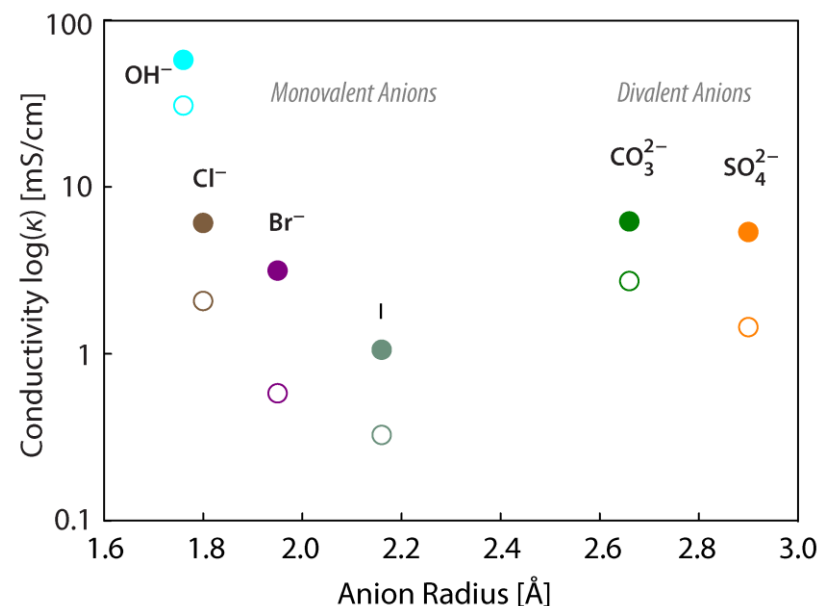
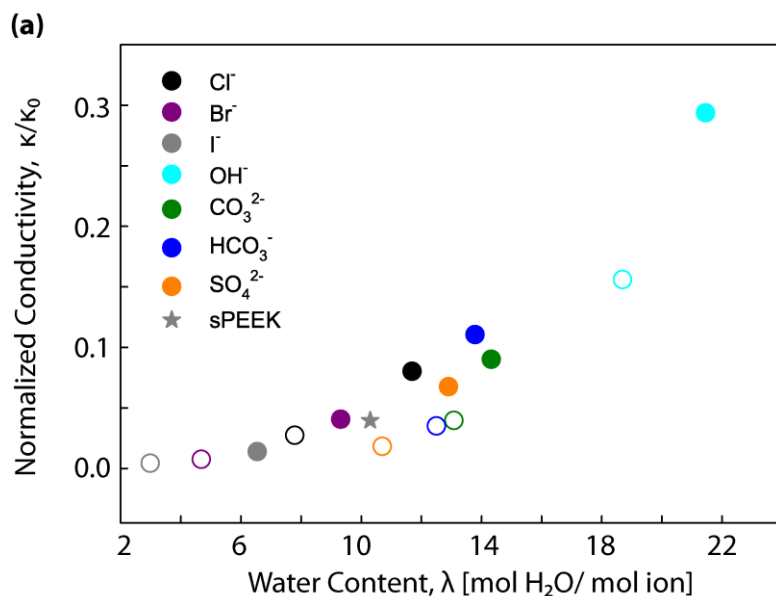
X. Luo, S. Rojas-Carbonell, Y. Yan, A. Kusoglu, *Structure-transport relationships of poly(aryl piperidinium) anion-exchange membranes: Effect of anions and hydration*, **J. Membrane Science**, 598 (2020) 117680.



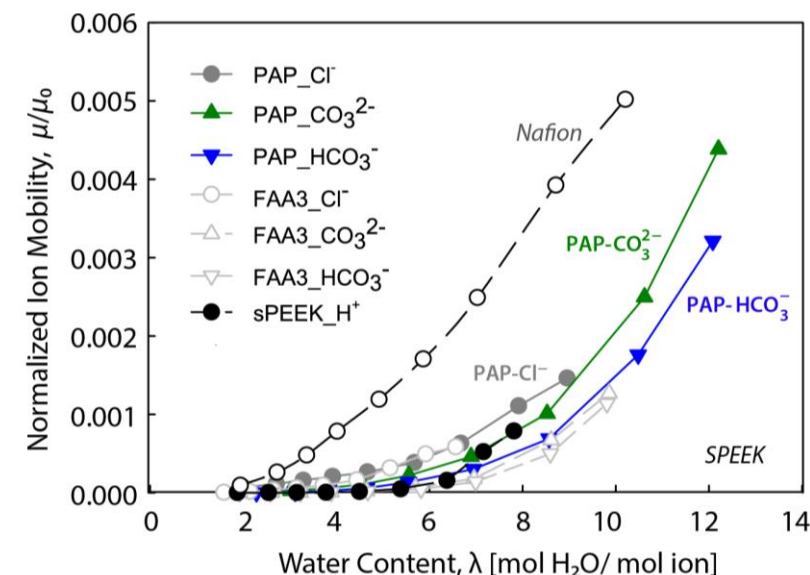
LTE 2.0 Accomplishments:

Membrane Hydration Governs Conductivity

Conductivity increases with hydration for all anion forms (PAP: closed symbols)



Comparison of ion mobility for various AEMs and PEMs, effect of hydration



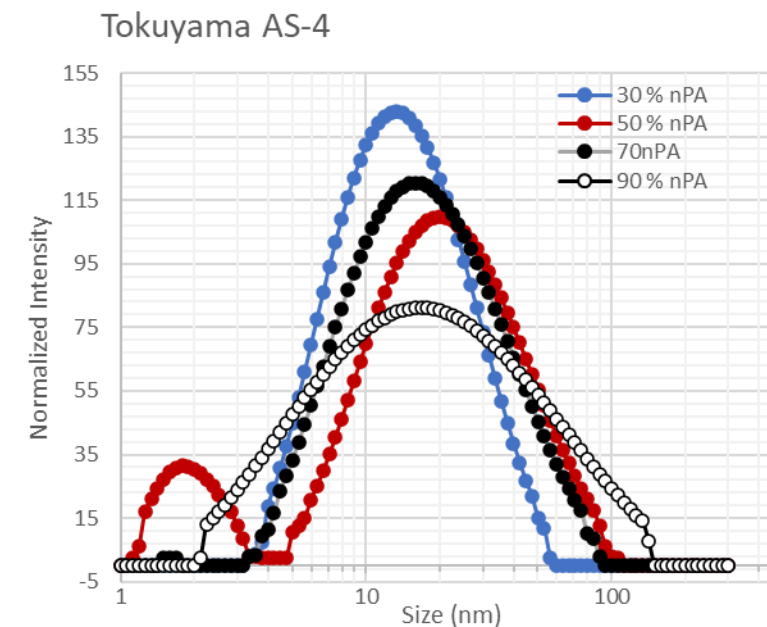
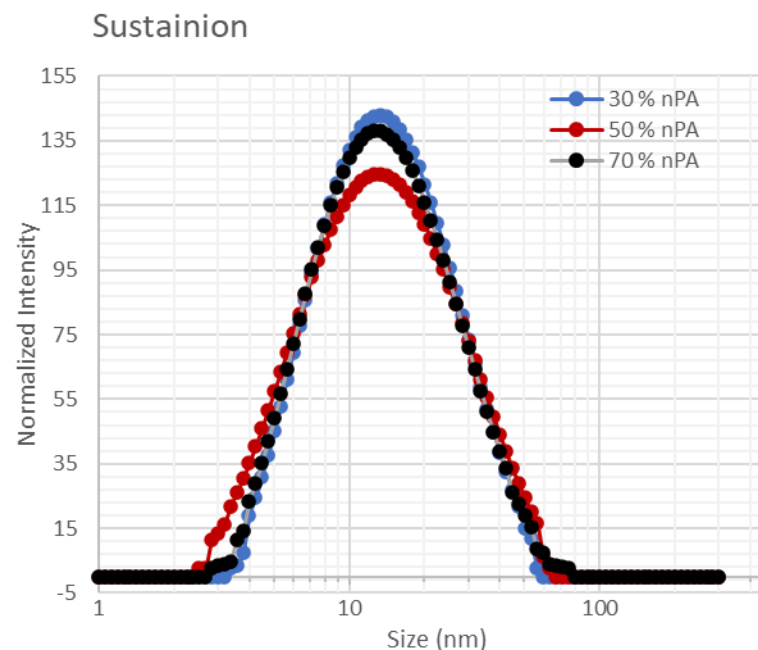
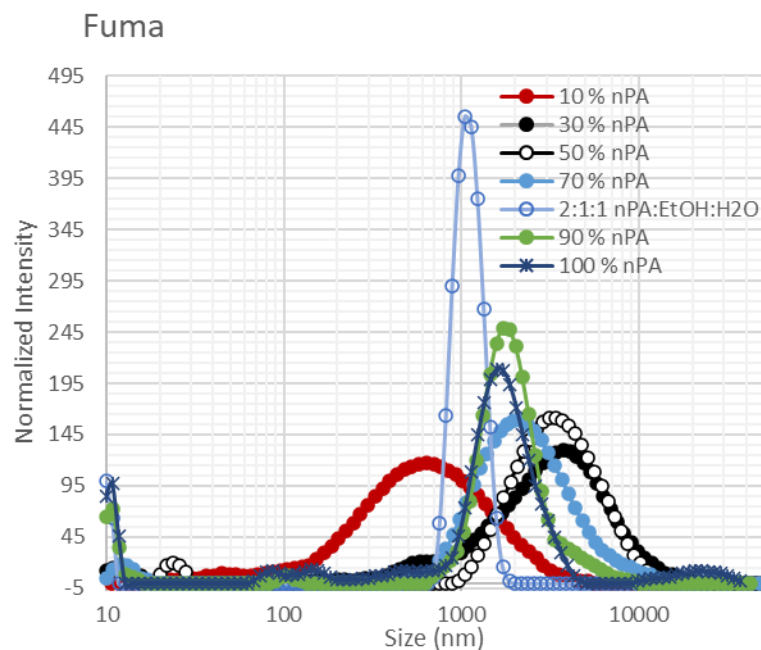
- Ion mobility plays a more important role than anion concentration and strongly dependent on hydration level (water content)
- Membrane conductivity: CO₃²⁻ > HCO₃⁻

X. Luo, S. Rojas-Carbonell, Y. Yan, A. Kusoglu, *Structure-transport relationships of poly(aryl piperidinium) anion-exchange membranes: Effect of anions and hydration*, **J. Membrane Science**, 598 (2020) 117680.



LTE 2.0 Accomplishments:

Impact of Ionomer Ink Composition



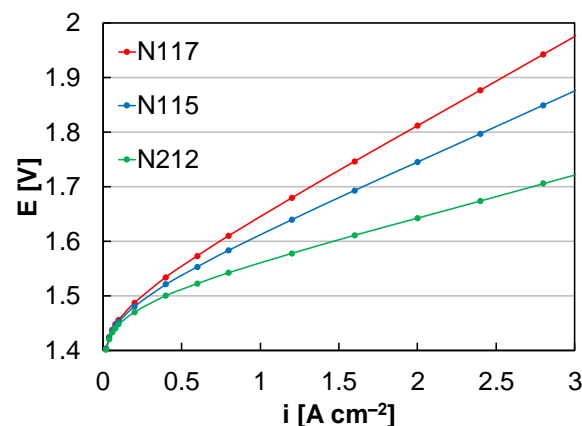
- Fumatech
 - FAA-3 shows much larger aggregates than Sustainion (XB-7) and Tokuyama (AS-4)
 - Shows broadening of distribution and increase in size for intermediate water content
 - Adding of 25 % ethanol narrows distribution
- Sustainion shows hardly any change with water content
- Tokuyama AS-4 shows broadening of distribution with decreasing water content



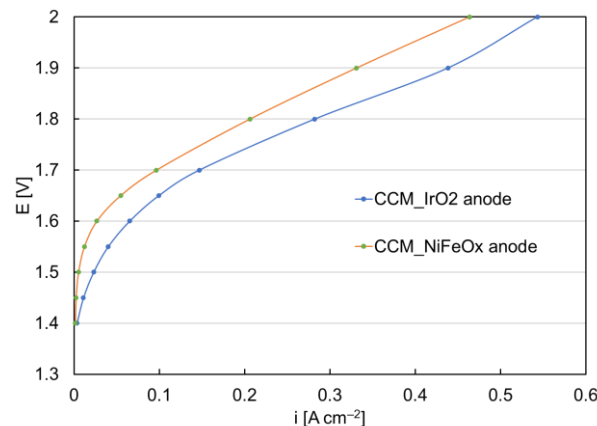
LTE 2.0 Accomplishments:

In-situ Electrolysis Performance

PEM Comparisons

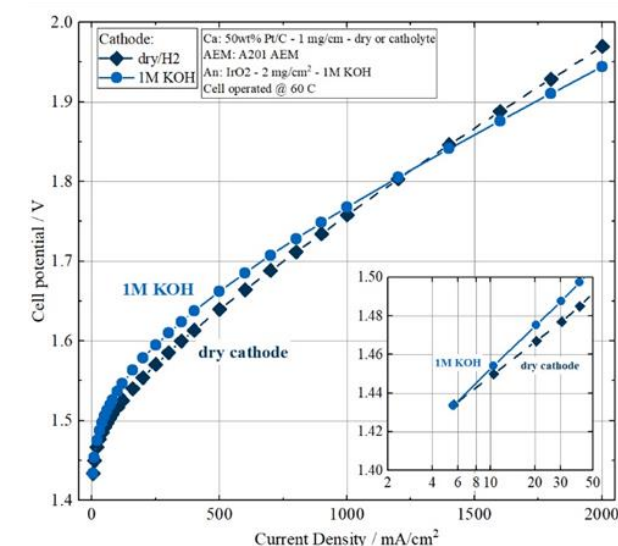
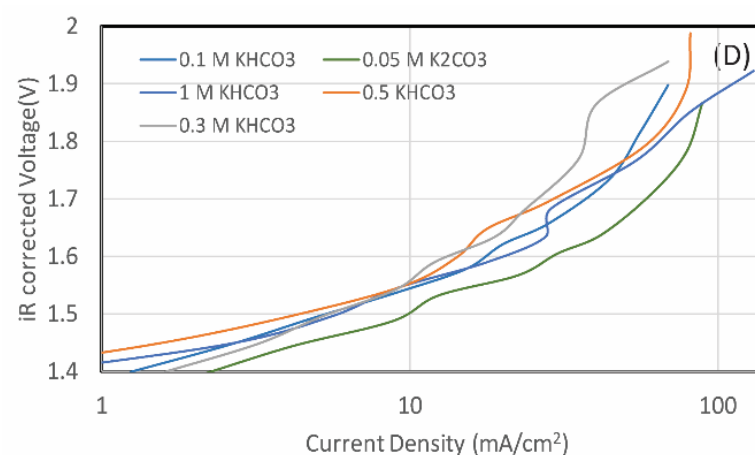
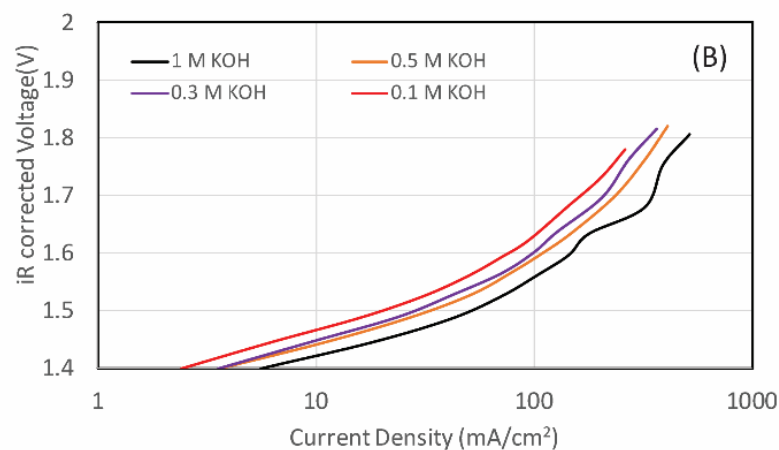


Current AEM Status



- Set AEM performance milestones based on PEM comparisons (membrane thickness normalized)
- AEM performance generally lower without supporting electrolyte, alkalinity and conductivity improve performance (alkalinity critical)
- Dry cathode generally results in lower performance (high current density), durability

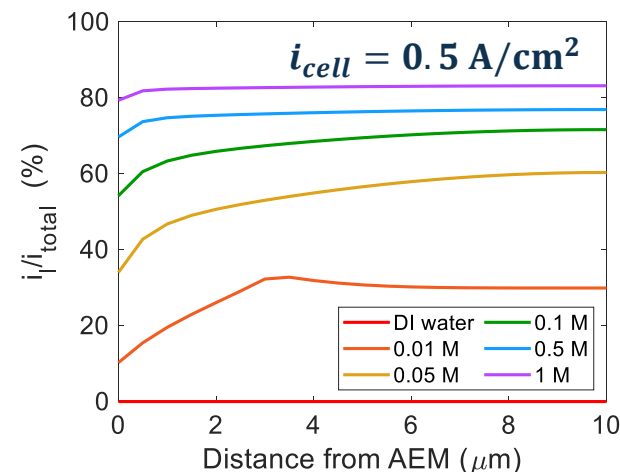
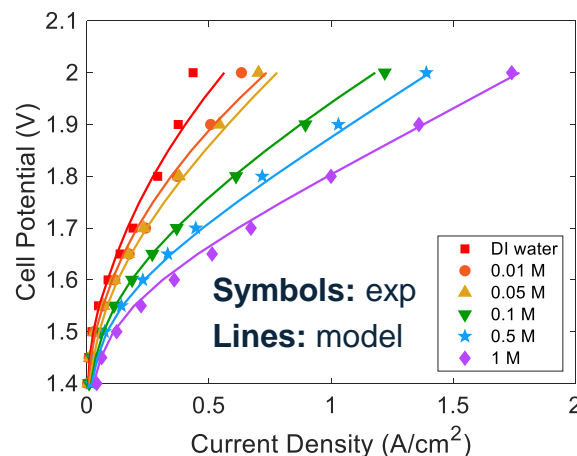
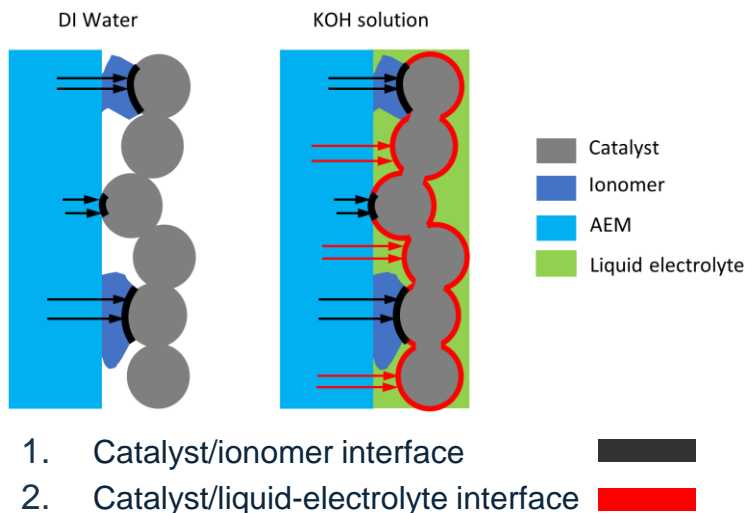
Role of Supporting Electrolyte



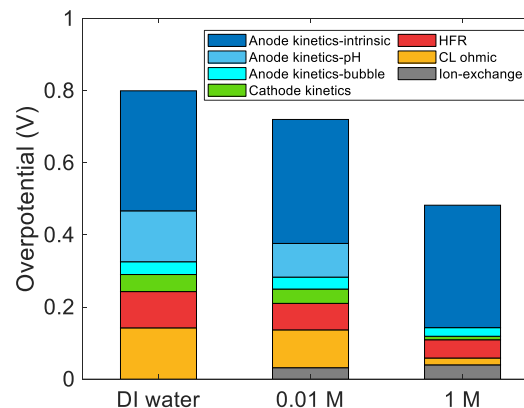
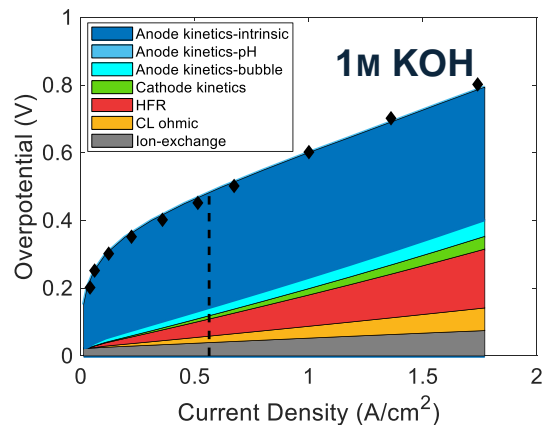
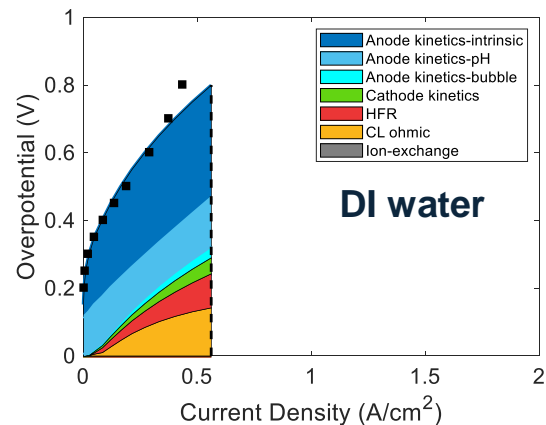


LTE 2.0 Accomplishments:

Impact of External Electrolyte on Performance



- Surface area increases with c_{OH^-} by additional ion-transport pathways
- More uniform current distributions from catalyst/liquid-electrolyte interface at high c_{OH^-}



Compositions at $0.56 \text{ A}/\text{cm}^2$

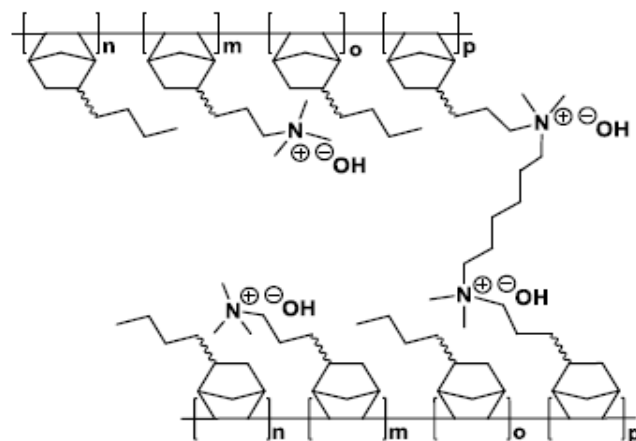
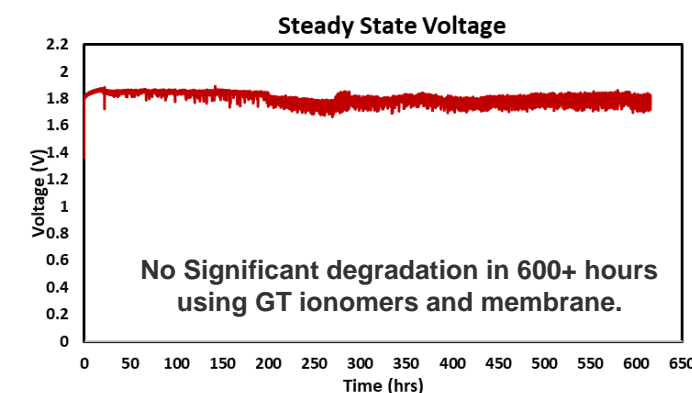
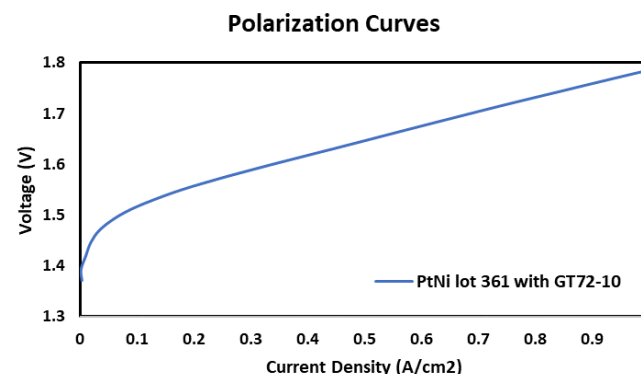
- Anode kinetics a significant factor in nonideal performance
- In unsupported electrolyte, increased overpotential largely due to a pH effect on anode kinetics and catalyst layer ohmic losses



P185 (P. Kohl): High-Performance AEM LTE with Advanced Membranes, Ionomers and PGM-Free Electrodes



Goals: To enhance and combine state-of-the-art alkaline polymer electrolyzer components into one optimized membrane electrode assembly (MEA) system to achieve the DOE targets for low temperature electrolysis (LTE)



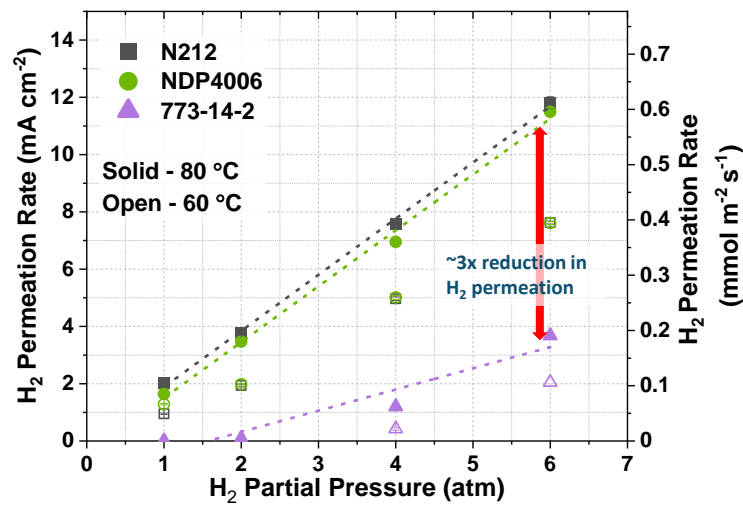
- Produced anion conducting polymer with conductivity >180 mS/cm (80°C) at the kg scale
- Developed OER and HER catalysts with high surface area (>30 m²/g unsupported, 400 m²/g supported)
- HER and OER ionomers for LTE MEAs stable at 500 mA/cm² and 1.75 V for >100 hour
- Achieved >600 h of durability with no losses using project-developed membranes, catalysts, and ionomers



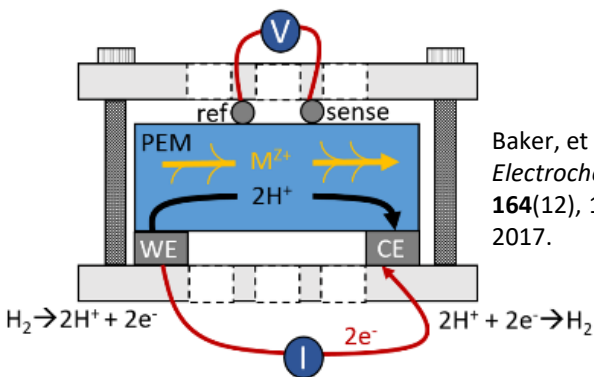
P186 (A. Park): Performance and Durability Investigation of Thin, Low Crossover Proton Exchange Membranes for Water Electrolyzers



Goals: Develop thin, reinforced membrane with performance and durability additives that enables high current density and long lifetime in PEMWE systems.

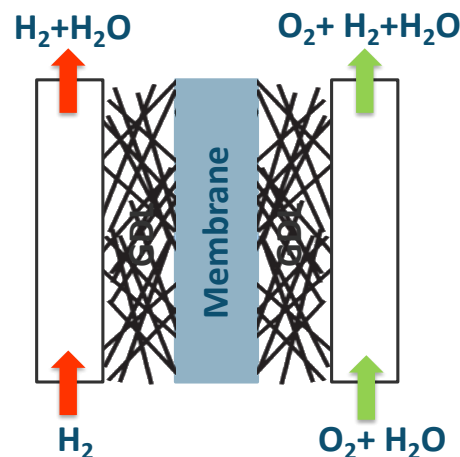


Active area: 50 cm², Cell temperature: 80 °C, O₂ partial pressure: 1 atm, titanium single serpentine flow field

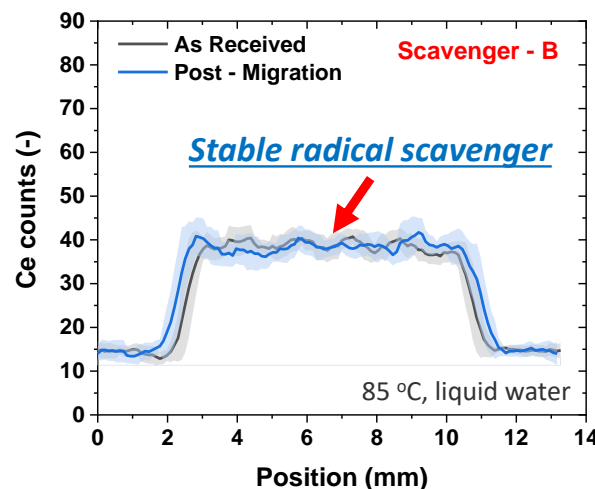


Baker, et al., *J. Electrochem. Soc.*, **164**(12), 1272-1278, 2017.

Ex-situ H₂ Permeation Measurement



- H₂ crossover measurement in H₂/O₂ environment simulated with ex-situ cell
- Demonstrated 3x reduction in effective H₂ crossover with thin membranes (~50 μm with GRC, 1-6 atm H₂)



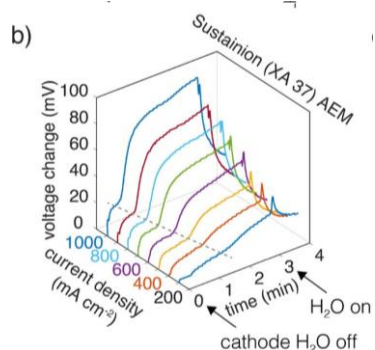
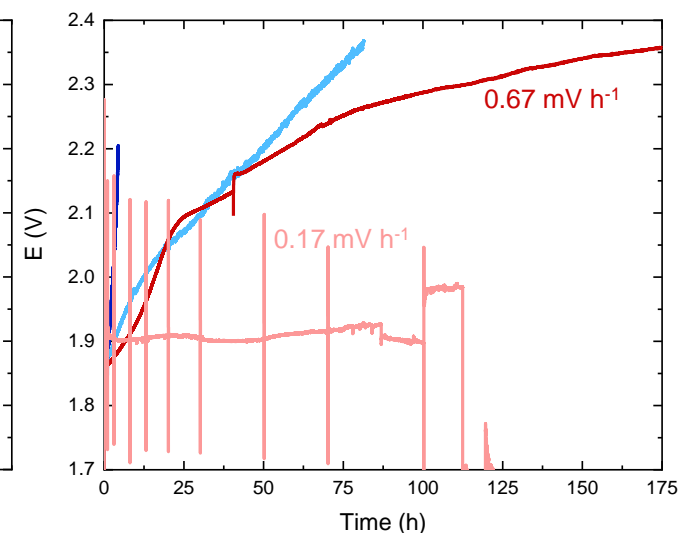
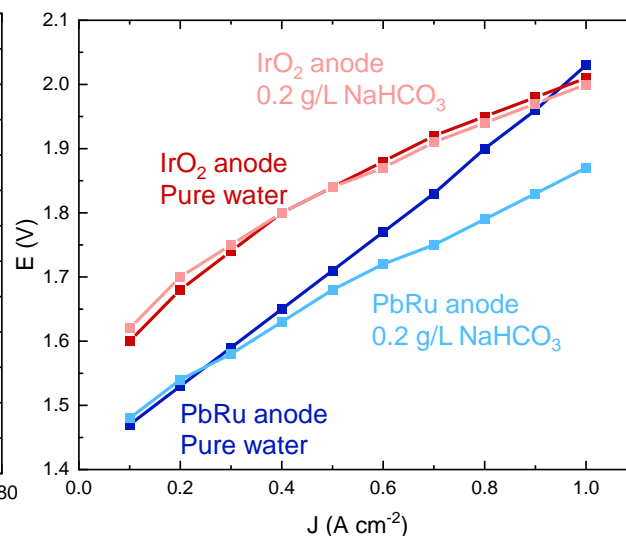
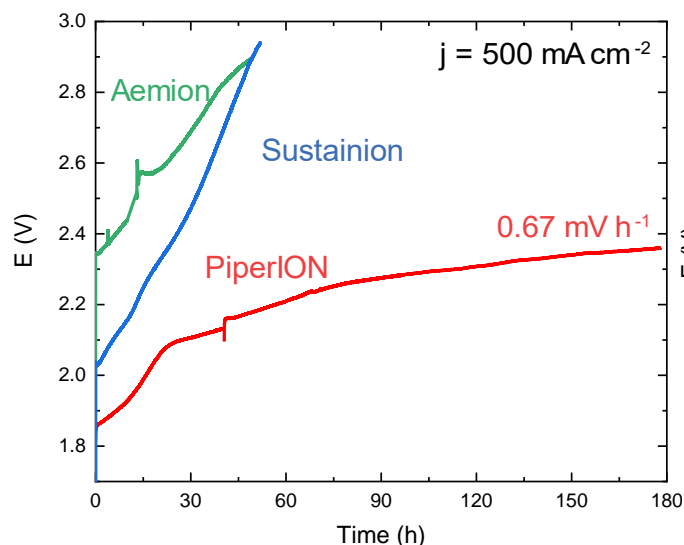
- Three candidate scavengers surveyed for retention in high temperature, water saturated PEMWE environment
- Scavenger morphology/composition has significant impact on mobility within PEMWE working environment



P187 (S. Boettcher): Pure Hydrogen Production through Precious-Metal-Free Membrane Electrolysis of Dirty Water



Goals: Develop a technical understanding of performance degradation of alkaline and bipolar membrane electrolyzers in pure and dirty water and engineer impurity tolerant systems.



- Demonstrated significant improvements to AEM performance and durability
- Degradation rate below 5 mV h⁻¹ for over 100 h, below 1 mV h⁻¹ for the final 20 h
- PbRuO_x catalyst has shown selectivity for OER over Cl reactivity. Stability of IrO_x and PbRuO_x catalysts were compared in water and carbonate solutions at relevant seawater concentration
- Reversible and irreversible degradation is observed in the absence of water flow to water-consuming electrode



Summary of Accomplishments

- HydroGEN LTE supported 8 FOAs (41 nodes), 2 Supernodes (14 nodes), LTE 2.0 (4 nodes)
- **Separated the impacts of supporting electrolytes and built understanding for performance differences based on catalyst-ionomer interactions (LTE 2.0).** While supporting electrolytes improve performance by improving anode kinetics (alkalinity), materials have been developed to start bridging the gap between AEM/PEM performance.
- **Demonstrated that iridium oxide anodes fabricated by scalable coating methods (roll to roll) produce comparable PEM electrolysis performance to those fabricated by lab-scale methods (LTE Supernode).** This effort identified the potential to fabricate PEM electrolysis electrodes at much higher rates than current lab methods, contributing to per unit cost reductions.
- **Achieved AEM electrolysis durability of no performance loss for greater than 600 h (P185, P. Kohl).** Demonstrated at 1 A/cm² using project-developed catalysts (Pajarito Powder), membranes, and ionomers (Georgia Tech).
- **Demonstrated a three-fold reduction in the effective hydrogen crossover rate with thin membranes in PEM electrolysis (P186, A. Park).** Achievement enables high current density performance while improving upon lifetime.
- **Durability losses as low as 0.17 mV h⁻¹ achieved in electrolytes at seawater-relevant concentrations (P187, S. Boettcher).** Improved understanding of performance degradation enables the engineering of impurity tolerant systems.



Collaboration, Effectiveness

- 3rd Annual Advanced Water Splitting Technology Pathways Benchmarking & Protocols workshop, March 1-3, 2021
- Interfacing between HydroGEN and IEA Annex 30 in benchmarking
- Contributions to the Meta Data development for the HydroGEN Data Center

Seedling Leads

Kathy Ayers
Shannon Boettcher
Chris Capuano
Hoon Chung
Yu Seung Kim
Paul Kohl
Di-Jia Liu
Sanjeev Mukerjee
Andrew Park

Seedling Teams



Supernode and 2.0 Teams



Shaun Alia
Guido Bender
Huyen Dinh
Mai-Anh Ha
Saad Intikhab
Allen Kang
Ross Larsen
Scott Mauger
Janghoon Park
Jason Pfeilsticker
Bryan Pivovar
Michael Ulsh
James Young



Nemanja Danilovic
Julie Fornaciari
Ahmet Kusoglu
Jessica Luo
Adam Weber
Guosong Zeng
Jeremy Zhou



Elise Fox
Héctor Colón-Mercado



Future Work

- LTE 2.0
 - Improve cell performance and durability through materials integration approaches, leveraging component development in the seedling projects
 - Improve our understanding of ionomer-electrolyte effects, through systematic studies, diagnostics, and simulations
- Leverage HydroGEN nodes to enable successful completion and continuation of the seedling projects, depending on which budget period they are in
- Any proposed future work is subject to change based on funding levels



Technical Backup Slides





Tech Transfer and Publications

- LTE HydroGEN supports numerous companies through FOA Projects
- Continual development of IP

Publications

- J.L. Young, Z. Kang, F. Ganci, S. Madachy, G. Bender, “PEM electrolyzer characterization with carbon-based hardware and material sets”, *Electrochem. Commun.*, 124 (2021) 106941.
- Z. Kang, M. Pak, G. Bender, “Introducing a novel technique for measuring hydrogen crossover in membrane-based electrochemical cells”, *Int. J. Hydrogen Energy*, 46 (2021) 15161-15167.
- J. Park, Z. Kang, G. Bender, M. Ulsh, S.A. Mauger, “ Roll-to-roll production of catalyst coated membranes for low-temperature electrolyzers”, *J. Power Sources*, 479 (2020) 228819.
- M.-A. Ha, R.E. Larsen, “ Multiple Reaction Pathways for the Oxygen Evolution Reaction May Contribute to IrO₂ (110)’s High Activity”, *J. Electrochem. Soc.*, 168 (2021) 024506.
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Tech Transfer and Publications

Publications (Continued)

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Contribution to Achievement of DOE Targets or Milestones

- These projects will contribute to the achievement of the following DOE milestones from the Hydrogen Production section of the Hydrogen and Fuel Cell Technologies Office Multi-Year Research, Development, and Demonstration Plan:
 - Hydrogen Levelized Cost (Production): \$2/kg
 - Energy Efficiency: 43 kWh/kg



Awards

- Bryan Pivovar was awarded the 2021 Energy Technology Division Research Award of the Electrochemical Society and the 2021 US Department of Energy Secretary's Honor Award
- DOE Postdoc award: Eun Joo Park (LANL) seedling project



K. Ayers: High Efficiency PEM Water Electrolysis

nel



UCI IRVINE



OAK RIDGE
National Laboratory

NREL
Transforming ENERGY

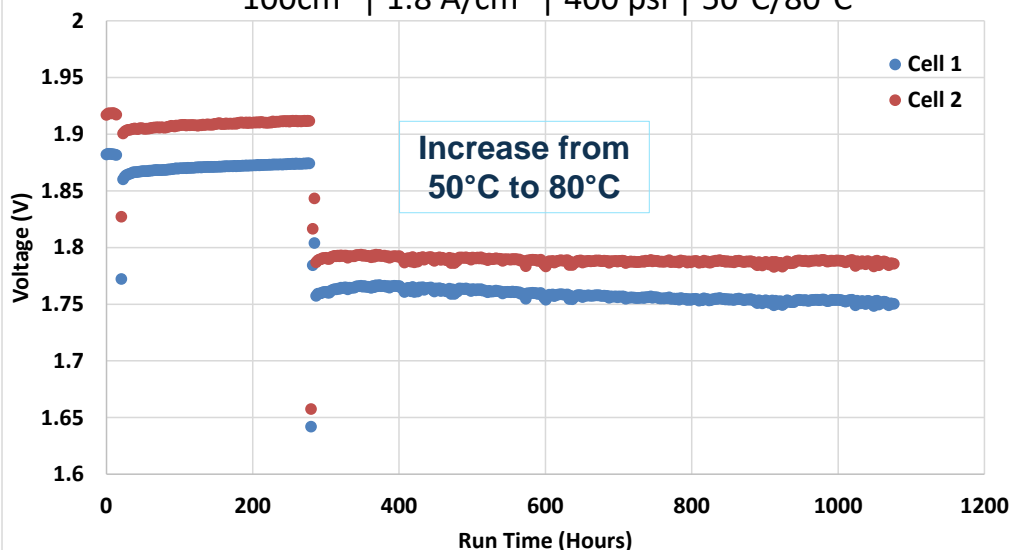


7
Nodes

Goals: To incorporate all elements of the advanced membrane, catalyst, electrode fabrication techniques, and cell modeling to realize a reliable MEA configuration with efficiency meeting the 43 kWh/kg targets

RND2035601 Steady State

100cm² | 1.8 A/cm² | 400 psi | 50°C/80°C

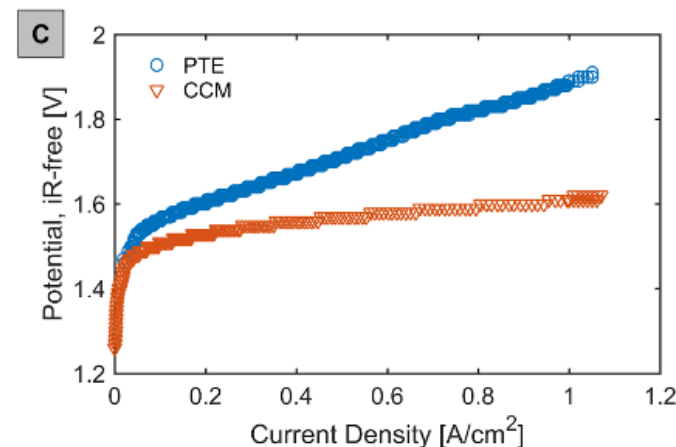
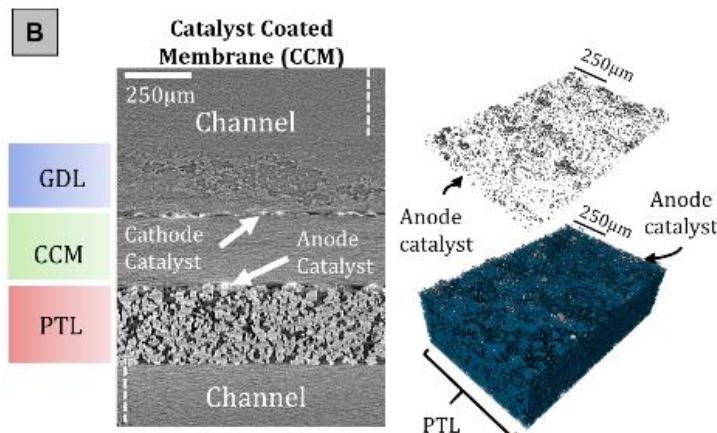


Anode Loading

0.6 mg/cm² IrO_x

Cathode Loading

0.1 mg/cm² Pt/C



- Catalyst layer morphology is resolved, inhomogeneous distribution
- Better polarization for CCM (at 1 A/cm² 250 mV difference)
- Durability testing conducted over 1000 hours in 100 cm² stack
- Stable voltages achieved with high surface area IrO_x showing a degradation rate of 0 μV/hr



D.-J. Liu: PGM-free OER Catalysts for PEM Electrolyzer



Lawrence Livermore
National Laboratory



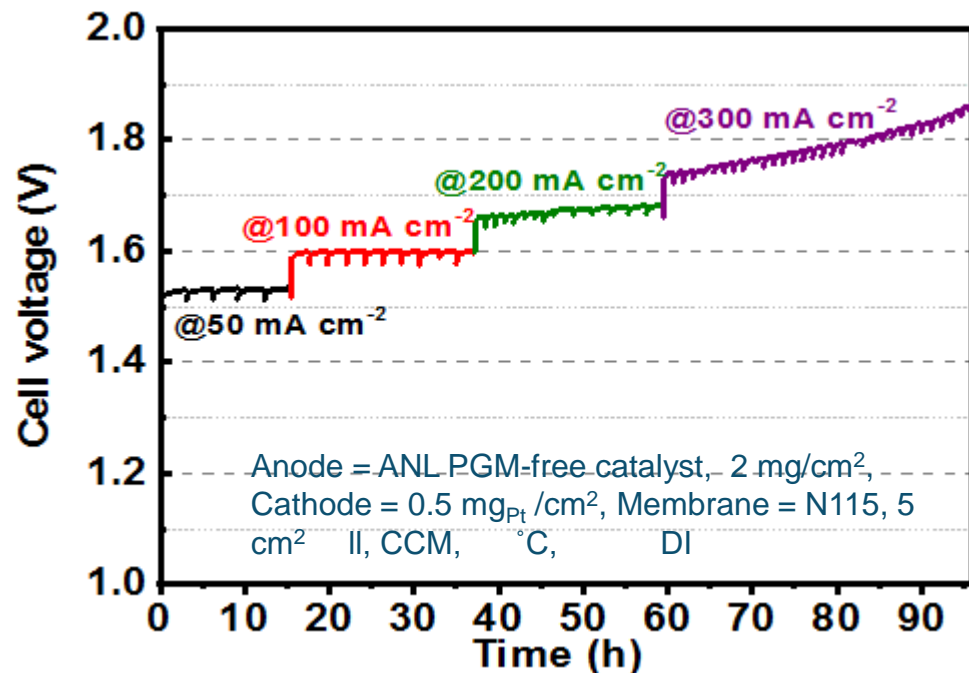
Sandia
National
Laboratories



5
Nodes

Goals: To develop platinum group metal-free oxygen evolution electrocatalysts as a viable replacement for iridium in proton exchange membrane water electrolyzers.

PEMWE cell voltage decay rates at 50, 100, and 200 mA/cm² meet the target of < 2mV/hr. At 300 mA/cm², the voltage increase exceeded the target value



@ Current density (mA/cm ²)	Duration (hr)	Ave. Cell Voltage (V)	Decay rate (mV/hr)
50	14.7	1.528	0.55
100	21.5	1.600	0.35
200	22.1	1.672	0.93
300	36.7	1.799	3.17

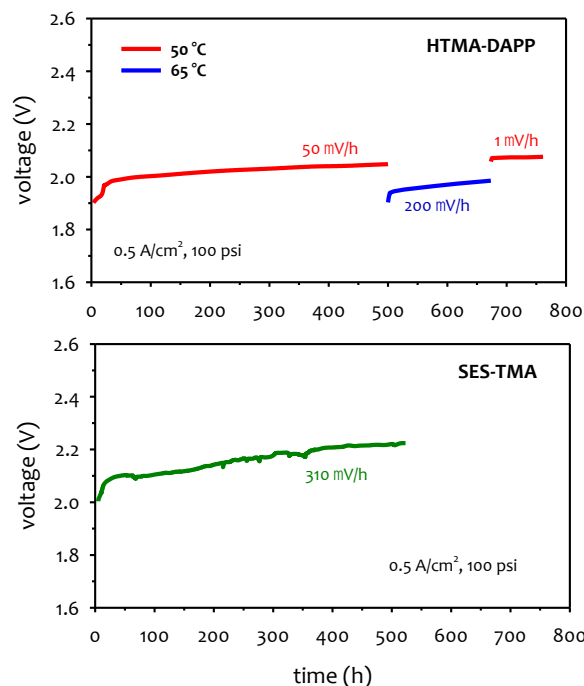
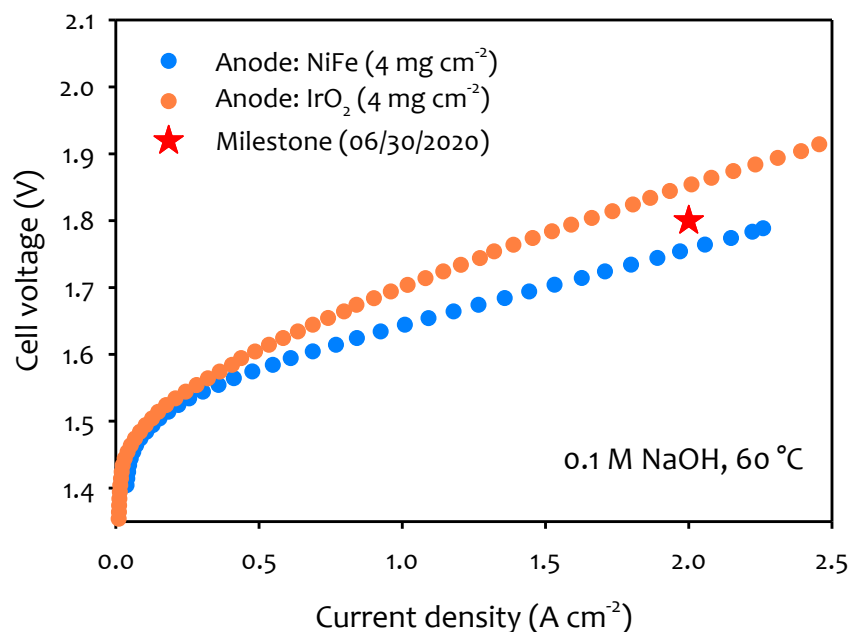
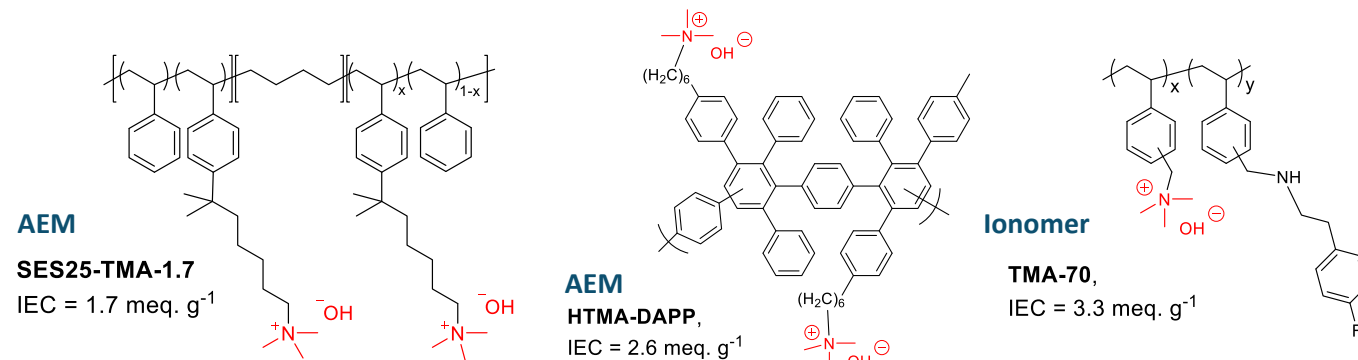
Progress demonstrates that PGM-free OER catalyst offers a promise as the replacement of Ir in PEMWE. Further development could lead to a major cost reduction in low-cost hydrogen production through low-temperature PEMWE electrolyzer



Y. Kim: PGM-free OER Catalysts for PEM Electrolyzer



Goals: Prepare durable and economically-affordable alkaline hydroxide conducting SES materials and demonstrate high performance and durability in AEM-based water electrolysis.



- Met the performance target using PGM-free anode
- AEM lifetime > 500 hours
- HTMA-DAPP AEM showed lower degradation rate (50 mV/h for HTMA-DAPP vs. 310 mV/h for SES-TMA)
- On track for AEM electrolyzer performance target, < 0.1 mV/h over 300 hours at 1 A/cm² using PGM-free anode



S. Mukerjee: Novel PGM-free Catalysts for Alkaline HER and OER



Northeastern
University



ADVENT

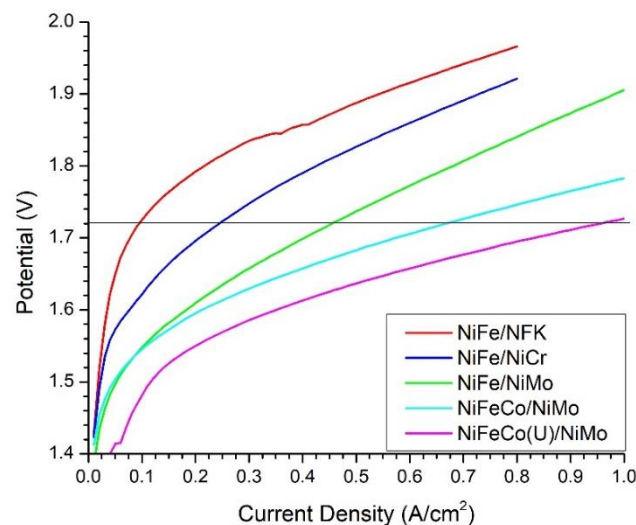


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3
Nodes

Goals: Decrease the cost of hydrogen production via water electrolysis using high-performing PGM-free catalysts and a novel, temperature-stable anion exchange membrane.



- A novel electrode architecture with Pt-free catalysts led to a performance of 1 A/cm² at 1.726 V.
- Performance decay of 0.02 mV/hr at 1 A/cm² for 30 hours.

End of Project Targets

Description	Milestone	Status
HER catalyst	Overpotential below 150 mV at 500 mA/cm ² in H ₂ pump cell	200 mV
OER catalyst	Overpotential below 100 mV @ 500 mA/cm ²	93 mV
AEM membrane and ionomer	Area specific resistance of 0.08 Ω·cm ²	0.08 Ω·cm ²
	Less than 10% IEC after 1000 hr treatment in 90°C 1M KOH	3.74% loss of IEC
Cell Performance	1.72 V at 1 A/cm ²	1.726 V @ 1 A/cm ²
	Less than 1 mV performance decay after 48hrs of sustained operation at 1 A/cm ²	0.02 mV/hr decay after 30 hours