Advanced Water-Splitting Technology Pathways Benchmarking & Protocols Workshop

Breakout Session Summaries Low Temperature Electrolysis (LTE)

September 21-22, 2023

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LTE Breakout Sessions

Session ID	Торіс	Lead	Note Taker
L1-A	Priority Research Opportunities - Membrane	Andrew Motz (Nel Hydrogen)	Chris Topping (Tetramer)
L1-B	Priority Research Opportunities - Catalyst	Barr Zulevi (Electric Hydrogen) & Shaun Alia (NREL)	Noor UI Hassan (NREL)
L2-A	PTL Research Opportunities and Test Protocols	Chris Capuano (Nel Hydrogen)	Emily Volk (NREL)
L2-B	Liquid Alkaline Test Protocols to be Written	Meital Shviro (NREL)	Sergio Bakovic (Nel Hydrogen)
L3-A	Priority Research Opportunities - Stack & Cell Testing Gates for Qualification	Bryan Pivovar (NREL) & Chris Capuano (Nel Hydrogen)	Ai-Lin Chan (NREL)
L3-B	Liquid Alkaline Priority Research Opportunities	Grace Lindquist (Hgen)	Sergio Bakovic (Nel Hydrogen)
L5-A	Accelerated Stress Tests- Catalyst	Shaun Alia (NREL)	Melissa Kreider (NREL)
L5-B	Accelerated Stress Tests- Membrane	Andrew Park (Chemours)	Kathy Ayers (Nel)

Session Summary – Part 1

Summary of Discussion	Consensus and Dissenting Opinions
 Discussion of research needs and opportunities related to: 1. PEM-WE 2. AEM-WE 3. Liquid Alkaline Electrolyzers 	 Consensus: Membrane needs include: Increased current density (and related conductivity); Thinner – but maintaining low H2 crossover; Understanding of hydrocarbon (HC) failure mechanisms (including timescales and mass transfer rates); Improved / meaningful AST protocols combined with long term testing. Water management essential for HC Improved understanding of degradation mechanisms will become even more important as we move to thinner membranes / HCs Dissenting Opinions: "New materials essential" vs "PFSAs are good enough" (and can survive regulatory concerns)
 Key Take-Aways / Discussion Points Durability is a key issue for both PFSA and HC How well can durability (/swell etc) be addressed with reinforcements ? How to address need for increased current density Consequences of moving to thinner membranes Mechanical challenges of lower resistance membranes Creep into PTL + other interface / integration issues Need for infrastructure for testing degradation (AST protocols, increased long term testing facilities, effective ex situ tests) How relevant are cycling AST methods to 'always on' electrolyzers ? Development of effective modelling Dynamics vs capacity – modelling pressure fluctuations For PFSA replacement – need for alternative membranes and ionomers? Options to demonstrate quality / reliability to end users – certification ? Enhanced break in understanding to allow equilibration of membrane behavior (especially for novel HC / non-PFSA membranes)	<section-header><section-header><list-item><list-item><list-item><list-item><list-item><list-item><list-item></list-item></list-item></list-item></list-item></list-item></list-item></list-item></section-header></section-header>

Summary of Discussion	Consensus and Dissenting Opinions
Discussion of research needs and opportunities related to:	Consensus: Need to define target current density (current DOE = 2 A/cm ² @ 1.8 V) Need for meaningful AST methods
 PEM-WE AEM-WE Liquid Alkaline Electrolyzers 	Dissenting Opinions: Define standard electrolyte concentration vs Define two or more benchmark concentrations (high + low) for different membranes vs Define 'acceptable window' of concentration

Key Take-Aways / Discussion Points

- Trend observed towards increasing electrolyte concentration (1M ٠ KOH)
 - Need to define the best conditions for durability
 - Compatibility of all component materials with electrolytes
- Evaluation of electrolyte/system cost vs operating expense benefits
- How much does electrolyte mask degradation ?
- Membrane and ionomer challenges are different may need different chemistries
- Need for ionomer/catalyst co-development

- How relevant is Fenton's test to AEM? Is there an equivalent?
- May need 3 stage screening eg (i) hot KOH soak, (ii) O2 purged KOH soak. (iii) electrochemical backbone attack
- Degradation testing is often visual (vs specific BOL/EOL data)

Action Items

- Develop high temperature base stability protocols
- Develop meaningful AST methods
- Define a baseline material

Session Summary – Part 3

Summary of Discussion

Discussion of research needs and opportunities related to:

- 1. PEM-WE
- 2. AEM-WE
- 3. Liquid Alkaline Electrolyzers

Consensus and Dissenting Opinions

Consensus:

Lots of work to be done in this area Need to increase current density High current density is dominated by ohmic losses Current separators cannot operate with pressure differential

Dissenting Opinions:

Do we need a new separator material or are existing materials good enough?

Key Take-Aways / Discussion Points

Design may be simpler compared with some other devices but membranes are not readily available

Key issues to address include:

- Porosity vs mechanical stability of separators;
- Doping;
- Need for materials able to cope with high base concentrations;
- Trade-off between use of thinner separators vs stability to higher/differential pressures and maintaining low crossover;
- Possible use of composite structures

Action Items

Lots of work to be done in this area

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Chris Topping (Scribe)	Tetramer Technologies, LLC
Bryan Pivovar	NREL
Sarah Park	NREL
Chris Capuano	Nel Hydrogen
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Adam Weber	LBNL
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Session ID: L1B Title: Catalyst Research Needs

 Summary What are research needs for electrocatalysts? Test protocols ASTs, Benchmarking, use FC as template New materials needed for new approaches: low-PGM for PEM, stable for AEM 	 Consensus and/or dissenting opinions Need unique physical characterization methods AEM: DI vs electrolyte drive different needs
 Key Take Aways Meaningful EChem testing protocols are needed across WE Rely on consortia for protocols Maturity of platforms drive different needs low-PGM for PEM, stable for AEM "Standard" physical characterization may be sufficient 	 Action Items Who is final decider on protocols? Should AEM protocols be updated since HydroGen is on "Pause"? What is correct path to new matls and directions?

Session ID: L1B Title: Catalyst Research Needs

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Grace Lindquist	Hgen	
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George Roberts	Nel Hydrogen	
Girish Srinivas	TDA Research	
Meghan Vander Woude	Chemours	
Sergio I Perez Bakovic	Nel Hydrogen	
Mohamed Abdelrahman	Moleaer Inc.	
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method that others can implement?

 Summary of discussion MPLs have been shown to improve performance and allow for lower loadings – however, this may be an issue at high current densities (water starvation concerns). Trade needs to be understood Suggested PTL/MPL testing and validation protocols: Corrosion % (including of coatings), bulk density, through plane resistance, in plane resistance, porosity, tensile strength, flexural strength Assemble cell w/out membrane to help assess what part of HFR comes from PTL vs membrane Can use other diagnostics, e.g. non-Faradaic impedance, to get more information We need a consensus across the community for PTLs, especially in terms of MPL, coatings, testing 	 Consensus and/or dissenting opinions How to assess resistances of PTLs w/ and w/out MPLs, in-situ vs. ex-situ, with or without compression Need for inspection methods – voids, abnormal height regions, etc – currently visual, need for something more automated (thermal, IR, etc) AEM transport layer development is further behind, degradation can be more accelerated. Larger spacing between PTL fibers, higher fluidity of membranes/polymers, etc. How, if at all, do test protocols/metrics differ? → some say not at all, some say metrics for success may vary PTL thickness are trending thinner to make overall cell thinner (cost concerns, etc) → can lead to lost mechanical strength Need for a corrosion metric or not? For PEM, Ti will likely passivate vs. dissolve. Need to understand if this is a concern w/ Ni or SS in AEM. Also, coating dissolution may be a concern Au vs Pt coating? → consensus we do not want Au, it is not stable
 Key Take-Aways Standard cell for community to use to test new components - Work w/ other consortiums What do non uniformities in PTL mean for the device in terms of performance and long term durability Writing and validating protocols – more than just one lab/company How are materials handled prior to cell assembly needs to be standardized (cleaning/surface prep) MPL included in PTL discussions, should or should not coating process be included in discussion? Whose job should it be to coat PTLs? Companies may not want to share IP, but there is a need for consistency in coating. Turn to national labs? Share a standardized 	 Action Items Development and validation of protocols for PTL testing Development of a roughness metric. How do we quantify surface roughness, what is the effect in a cell How to quantify and assess PTL defects Procedure for cleaning, preparing PTLs Need for shareable and implementable coating methodology

Session ID: L2-A Title: PTL Research Opportunities and Protocols Workshop

Name	Affiliation
Emily Volk	NREL
Melissa Kreider	NREL
Shaun Alia	NREL
Ai-Lin Chan	NREL
Chris Topping	Tetramer Technologies
Meghan Vander Woude	Chemours
Andrew Park	Chemours
Andrew Motz	Nel Hydrogen
George Roberts	Nel Hydrogen
Carina Schramm (online)	TUM
Johan Buurma (online)	TNO
Matthias Kornherr (online)	TUM
Bilal Iskandarani	University of California, Irvine
Mohamed Abdelrahman	Moleaer Inc.
Nadia Tolouei	University of California, Irvine
Balasubramanian Lakshmanan	Versogen
James Vickers	DOE

 Summary of discussion First session on liquid alkaline Initial protocol assessing performance of baseline components was discussed. Suggestion on how to update the existing protocol and standardization. 	 Consensus and/or dissenting opinions Developing activation protocol to obtain reproducible data is required The development of the activation procedure will allow understanding of the components during the electrochemical process. Fe level should be monitored and understood. Implementation of EIS measurements
 Key Take-Aways Incorporation of KOH purification is needed. Incorporation of shut down in operation protocol is needed. Updating the protocol might be needed to reach the full activation of the components and assessment of the performance. Incorporation of additional testing partners 	 Action Items Obtain protocol for KOH purification Update the exciting protocol with additional measurements

Session ID: L2-B Title: Liquid Alkaline Test Protocols to be Written

Name	Affiliation
Luigi Osmieri	LANL
Ahmed Farghaly	ANL
Rito Yanagi	Yale University
Sarah Park	LANL
Kathy Ayers	NEL Hydrogen
Yu Seung Kim	LANL
Hassan UI Noor	NREL
Grace Lindquist	Hgen
Bryan Pivovar	NREL
Sergio Perez Bakovic	NEL Hydrogen

Session Action Item Assignments

Session ID: L2-B Title: Liquid Alkaline Test Protocols to be Written

Name	Affiliation	Action Item	Target Due Date
Ahmed Farghaly	ANL	Draft protocol for KOH purification	12/30/2023
Meital Shviro	NREL	Update protocol with additional measurements	06/30/2024

Session ID: : L3-A Title: Priority Research Opportunities - Stack & Cell Testing Gates for Qualification

Summary of discussion

- Validation/Verification NREL is working on increasing and enhancing test station capability. Open test beds in 25-100 kW range with flexibility to test different supplier stacks (connections, voltage range, current range etc).
- Baseline hardware and testing protocols for PEM stacks needs further development for community.
- The components, PTLs and membranes can be different from AEM electrolysis, but the testing methodology of stacks can be leveraged from PEM.
- Round robin activities are ongoing in LA. Verification of PTL protocols can follow this model for final validation

Consensus and/or dissenting opinions

- PEM: Break in protocols need to be discussed, to know you are measuring changes in the test and not just typical stabilization that occurs in the first few hundred hours of operation.
- Water quality is a key factor in degradation. Temperature control and where control sensor is located in test stand should be developed for consistency.
- AEM: there is less baseline testing protocol, because it varies from different cell configurations. Pressurized operation can lead to shorting in the cell (membrane breaks). The quality of electrolyte is critical.
- LA: LA is not tolerant for differential pressure. Electrolyte quality impacts on LA performance.

Key Take-Aways

- Material screening can be done with single cells. After stack built by OEMs, stacks are tested in national labs as the validating group.
- Short stack testing: leverage testing reproducibility, understanding degradation mechanisms, fabrication processes and material screening.
- Increasing test station capability is on the way.
- Pressurized operation is available for PEM (30 bar). It might be challenging for AEM and LA. NREL has a 25cm2 stack design that will be become available open source.
- Need to identify people to finish writing protocols
- Identify how protocols will be verified and validated (industry, academia, or labs)

Action Items

- PEM: elevated pressure capable cell stack needed, as well as test stations for validation. Hardware, protocols, and test stands need to be standardized for consistency across test locations.
- AEM: the configuration of cell design is needed. Material screening (catalysts, polymers and PTLs) is on the process.
- LA: conditioning protocols need to be studied and developed since there is funding directed at this technology again though the DOE.

Name	Affiliation
Yu Seung Kim	LANL
Chris Capuano	Nel
Kathy Ayers	Nel
George Roberts	Nel
Chris Topping	Tetramer Tech, LLC
Andrew Park	Chemours
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Balsu Lakshmanan	Versogen
Andrew Motz	Nel
Nadia Tolouei	University of California, Irvine
Mohamed Abdelrahman	Moleaer Inc.
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Ai-Lin Chan	NREL

Session Action Item Assignments

Session ID: : L3-A Title: Priority Research Opportunities - Stack & Cell Testing Gates for Qualification

Name	Affiliation	Action Item	Target Due Date
Guido Bender	NREL	Develop and release an open source 25cm2 single cell stack capable of 30bar differential pressure	12/31/2023
Chris Capuano	NEL	Develop draft test stand P&ID, showing location, control, and measurement points for data collection	3/31/2024
?	?	Identify and develop cell stack break-in procedures for all technologies	TBD
?	Labs?	Verify test stand, stack, and break-in procedure and compare against multiple locations	TBD

Session ID: L3-B Title: Liquid Alkaline Priority Research Opportunities

<u>Summary</u>

Discussed priority research opportunities for:

- Increased current density operation
- Improved durability
- Renewables integration

Consensus and/or dissenting opinions

Need to develop new catalyst materials?

- Anode no, focus on electrode/catalyst integration and advanced structures/increased ECSA
- Cathode yes, particularly for non PGMs

Key Take-Aways

- Addressing shutdown degradation mechanisms is high priority for improved efficiency and durability
- Wide parameter space to explore for advanced separator development but extent of efficiency improvements is unclear

Action Items

 No assigned action items but there is a request for further DOE guidance defining technical targets for pressurized operation and durability/lifetime targets for renewable power

Session ID: L3-B Title: Liquid Alkaline Priority Research Opportunities

Name	Affiliation
Su Min Ahn	LANL
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Sur •	 split discussion into AEM/liquid alkaline and PEM. Discussed major degradation mechanisms, particularly related to the catalyst and ionomer, and current state of ASTs for each technology. Liquid alkaline has need to understand full-electrode and system level degradation mechanisms. Discussed how to choose baseline materials and design protocols that are relevant for different material choices and can isolate particular degradation modes (ex. catalyst vs PTL vs membrane). Discussed need for protocols to reflect real operating conditions and/or correlate to realistic lifetimes. 	 Consensus: The field needs baselines and ASTs. The variety of materials used for AEMWE is a challenge for baselining and developing a universal AST protocol. In situ diagnostics (ex. reference cell, EIS) and ex situ characterization are needed to complement the AST protocols. Undetermined: What metrics should be used to compare degradation? How to probe or identify individual component degradation mechanisms?
<u>Ke</u> √ ●	 <u>All 3 LTE technologies would benefit from a methodology to assess metal oxide surface area.</u> H2NEW is developing protocols for PEMWE and liquid alkaline ASTs, which will need to be validated by external partners. HydroGEN is developing baselines and performance and durability for AEMWE. Need feedback from OEMs on how to make ASTs relevant to actual operating conditions and to help with extending these to stack-level. 	 Action Items Improve communication in the community outside of these annual meetings. Protocol development by H2NEW and HydroGEN consortia. Industry/academic/national lab partners needed for protocol validation that comes out of H2NEW and HydroGEN projects: UCI, Moleaer, Hgen, Nel, LANL expressed interest in being involved in this process.

Session ID: L5A Title: ASTs – Catalyst

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Shaun Alia	NREL]
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Emily Volk	NREL	1
Luigi Osmieri	LANL	1
Ahmed Farghaly	ANL	1
Meital Shviro	NREL]
Grace Lindquist	Hgen]
George Roberts	Nel Hydrogen]
Yulia Kukula	Arizona State University]
Sergio I Perez Bakovic	Nel Hydrogen]
Meghan Vander Woude	Chemours]
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Session ID: 154 Session Action Item Assignments

Jession ID. LJA	
Title: ASTs – Cataly	/st

Name	Affiliation	Action Item	Target Due Date
H2NEW		Protocol development	
HydroGEN		Protocol development	
?	?	Communication	
Nadia E.Tolouei Bilal Iskandarani	UCI	Validation	
Mohamed Abdelrahman	Moleaer	Validation	
Grace Lindquist	Hgen	Validation	
Sergio I Perez Bakovic	Nel	Validation	
Luigi Osmieri	LANL	Validation	

 Summary Must understand what is to be measured to quantify failure Possible failure modes include chemical, mechanical, and electrochemical degradation. Must consider scale at which to operate (stack vs single cell) 	 Consensus need individual tests to probe failure modes before combining. Tests must be done fully hydrated. Debate: specific protocols vary between PEM/AEM/HC PEM. Incorporate liquid Alkaline?
 Key Take-Aways Need an enhanced understanding of relevant failure modes, especially with new PTLs and membranes Likely need different protocols/stressors for different membrane types 	 Action Items Work with H2NEW/OEMs to gather real life failure data to guide testing protocols Develop protocols that match the real life failure data.

Session ID: L5-B Title: Accelerated Stress Tests-Membrane

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Kathy Ayers	Nel
Chris Capuano	Nel
Andrew Motz	Nel
Bryan Pivovar	NREL
Yu Seung Kim	LANL
Eun Joo (Sarah) Park	LANL
Andrew Park	Chemours
Meital Shviro	NREL
Chris Topping	Tetramer
Jeff Martin	Shell
Ai-Lin Chan	NREL
Noor Ui Hassan	NREL

Session Action Item Assignments

Session ID: L5-B Title: Accelerated Stress Tests-Membrane

Name	Affiliation	Action Item	Target Due Date
While specific owners were not assigned, a reasonable expectation would be for Topic 2a awardee and H2New to tackle many of these issues in concert. H2New and/or HydroGEN 2.0 to define protocols for low TRL technologies and liquid alkaline		Intentional PTL defects to penetrate membrane	
		Force edge stresses (e.g., through membrane shims)	
		Define mechanical protocols for both tensile and compressive failures	
		Define fluoride inventory and threshold for failure (for PFSA)	
		Define appropriateness of gas sensors for durability monitoring (pinhole/thinning of membrane)	
		Define how to incorporate liquid alkaline separators	