



BETO 2021 Peer Review: Electrocatalytic CO₂ Utilization

Jack Ferrell – NREL

CO₂ Upgrading

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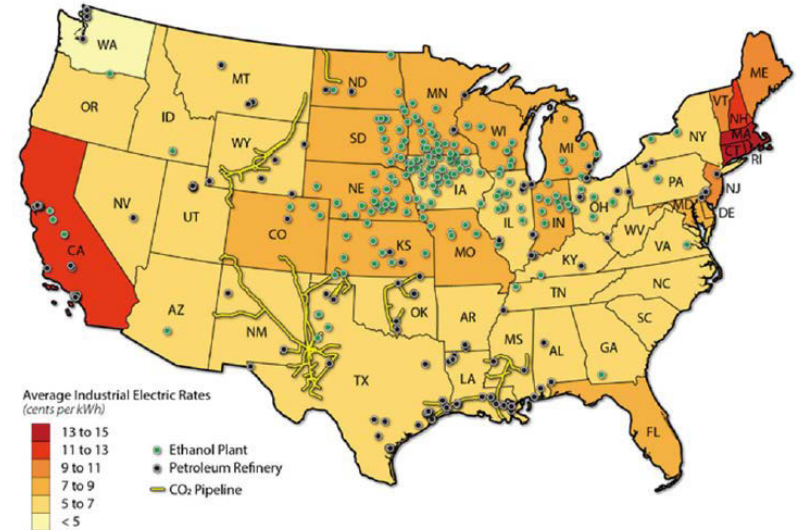
BIOENERGY TECHNOLOGIES OFFICE



Project Overview: Motivation





CO₂ – a ubiquitous feedstock:

- CO₂ is available from a range of point sources, as well as in the atmosphere. The only feedstock measured in gigatons
- Domestic biorefineries emit a very pure CO₂ waste stream, and represent an *early opportunity for deployment* of CO₂ utilization technologies
- Multiple forces combine to make capture + utilization of CO₂ attractive
 - Climate change (some incentives today for CO₂ capture/use)
 - Increase C utilization of existing processes
 - Inexpensive, intermittent electricity available & increasing
- ***Additional R&D needed to enable the economical electrochemical conversion of CO₂ to value-added products, either through C₁ intermediates to be further upgraded (e.g., CO; TRL 3-4), or directly to products (e.g., C₂H₄, C₂H₅OH; TRL 1-3)***








Market Trends




Product

-  Anticipated decrease in gasoline/ethanol demand; diesel demand steady
-  Increasing demand for aviation and marine fuel
-  Demand for higher-performance products
-  Increasing demand for renewable/recyclable materials




Feedstock

-  Sustained low oil prices
-  Decreasing cost of renewable electricity
-  Sustainable waste management
-  Expanding availability of green H₂
-  Closing the carbon cycle

Capital

-  Risk of greenfield investments
-  Challenges and costs of biorefinery start-up
-  Availability of depreciated and underutilized capital equipment

Social Responsibility

-  Carbon intensity reduction
-  Access to clean air and water
-  Environmental equity

NREL's Bioenergy Program Is Enabling a Sustainable Energy Future by Responding to Key Market Needs

Value Proposition

- Enable long-term and stable operation of low-temperature CO₂ electrolyzers, by developing diagnostics, standard procedures for testing, and focusing on degradation pathways

Key Differentiators

- Focus on diagnostics and standard testing procedures
- Leverage NREL expertise on both low-temperature electrochemical technologies and enabling capabilities within the ChemCatBio Consortium

Quad Chart Overview

Timeline

- 10/1/2019
- 9/30/2022

	FY20	Active Project
DOE Funding	\$300k	\$900k

Project Partners

- *Feasibility Study of Utilizing Electricity to Produce Intermediates from CO₂ and Biomass (2.1.0.304)*
- *Advanced Catalyst Synthesis and Characterization (ACSC) (2.5.4.304)*
- *Consortium for Computational Physics and Chemistry (CCPC) (2.5.1.307)*

Barriers addressed

- Ct-E: Efficient Low-Temperature Deconstruction
- Ct-H: Efficient Catalytic Upgrading of Gaseous Intermediates to Fuels and Chemicals

Project Goal

Enable stable and long-term operation of low-temperature CO₂ electrolyzers, by investigating scalable MEA architectures, studying catalyst and support degradation, and creating standard methods for testing and product quantification

End of Project Milestone

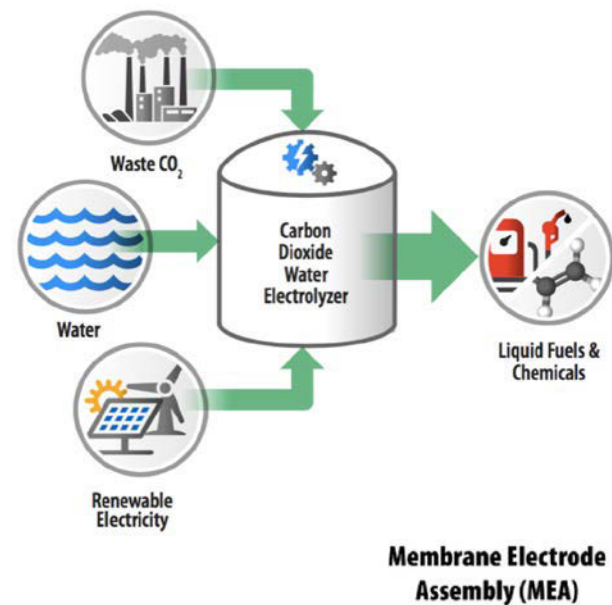
Demonstrate stable (<10% change in cell voltage) and long-term (>100 hours) electrocatalytic CO₂ conversion in a membrane-electrode-assembly (MEA) at relevant current density (>200 mA/cm²)

Funding Mechanism

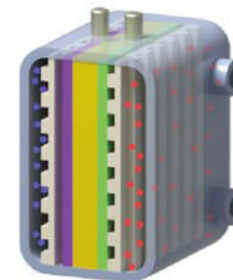
AOP Project (continuing)

1 – Management: Project Overview

- Project started in FY18
 - Collaboration with Feasibility Study (2.1.0.304) to determine technical feasibility and research needs¹
 - Focused on both electrochemical and thermochemical CO₂ upgrading
- Moved into ChemCatBio in FY20
 - Electrochemical pathway only
- Project Structure / Communication / Collaborators
 - 1 task, applied R&D
 - Regular contact with BETO TM
 - Frequent contact with other BETO CO₂ utilization projects
 - Collaborate with NREL researchers with expertise in low-temperature electrochemical systems (EERE - Hydrogen and Fuel Cell Technologies Office)
 - Leverage ChemCatBio capabilities in catalyst synthesis and characterization (ACSC project) and modeling (CCPC)



Membrane Electrode Assembly (MEA)



¹Energy Env. Sci., 2020, 13, 472-494

1. Management: ChemCatBio Foundation – FY21

Integrated and collaborative portfolio of catalytic technologies and enabling capabilities

Catalytic Technologies

Catalytic Upgrading of Biochemical Intermediates

(NREL, PNNL, ORNL, LANL)

Upgrading of C1 Building Blocks

(NREL)

Upgrading of C2 Intermediates

(PNNL, ORNL)

Catalytic Fast Pyrolysis

(NREL, PNNL)

Electrocatalytic CO₂ Utilization

(NREL)

Enabling Capabilities

Advanced Catalyst Synthesis and Characterization (ACSC)

(NREL, ANL, ORNL)

Consortium for Computational Physics and Chemistry (CCPC)

(ORNL, NREL, PNNL, ANL, NETL)

Catalyst Deactivation Mitigation for Biomass Conversion

(PNNL)

Industry Partnerships

(Phase II Directed Funding)

Opus12 (NREL)

Visolis (PNNL)

Sironix (LANL)

Cross-Cutting Support

ChemCatBio Lead Team Support (NREL)

ChemCatBio DataHUB (NREL)

2 – Approach: Alkaline Exchange Membrane MEAs

Advantages:

- High Faradaic efficiency to products
- Stable operation
- Scalable MEA architecture

Technical Challenges:

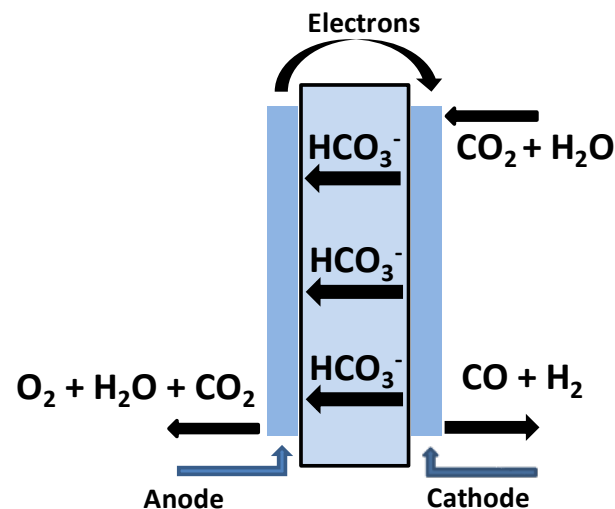
- CO₂ crossover (50%)
- Long-term membrane durability
- Catalyst & support degradation

Approach:

- Probe membrane durability in MEA environment
- Test new AEM membrane materials
- *Quantify catalyst and carbon support degradation*
- *Standard methods for testing & product analysis*

Go/No Go: Demonstrate Stable & Long-term Operation (March 2021)

Alkaline Exchange Membrane (AEM)



100s mA/cm²
@ ~3V

2 – Approach: Proton Exchange Membrane MEAs

Advantages:

- Nafion durable & well-understood membrane material
- No CO₂ crossover issues

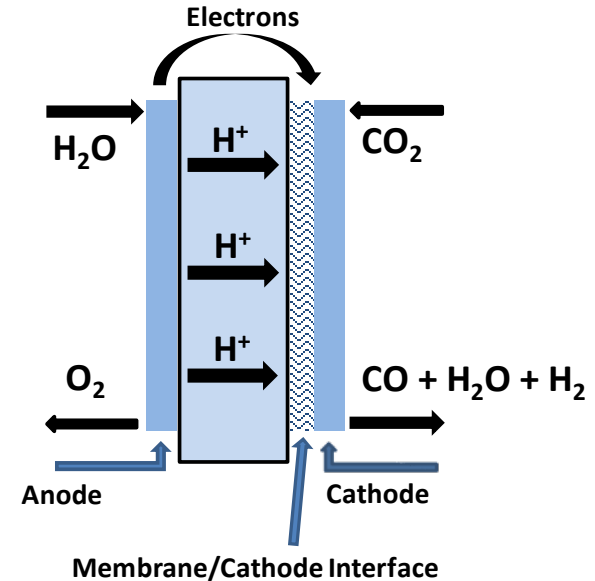
Technical Challenges:

- Acidic environment – significant hydrogen evolution
 - Buffer layer required at membrane/cathode interface
 - Stability & scalability of buffer layer architectures
- Catalyst & support degradation

Approach:

- Quantify hydrogen evolution kinetics
- *Use kinetics to develop integrated MEA model*
- *Quantify catalyst and carbon support degradation*
- *Standard methods for testing & product analysis*

Proton Exchange Membrane (PEM)

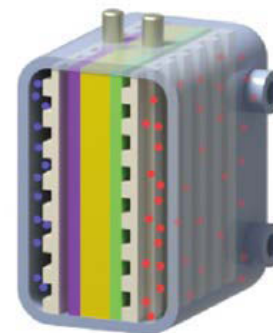


100s mA/cm²
@ ~3V

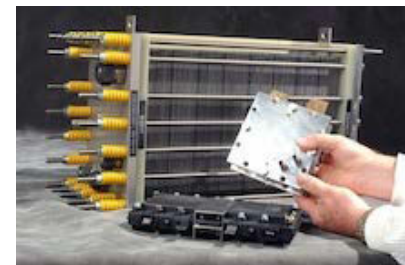
3 – Impact: Addressing Technical Barriers

- Improving biorefinery carbon utilization
 - Ethanol biorefineries large source of pure industrial CO₂ waste stream
 - Valorization of this waste stream, enabled by inexpensive renewable electricity, will improve biorefinery economics
- Published review article in *Energy & Environmental Science*¹
 - Focused on key technical challenges CO₂ utilization
 - Identified research needs
- Addressing top technical barriers¹ for low-temperature CO₂ electrolysis
 - Increasing long-term stability
 - Scalable MEA architectures
 - Optimizing reaction conditions (electrolyte, pH, mass transport)
 - Increasing single-pass CO₂ conversion

Membrane Electrode Assembly (MEA)



Single MEA



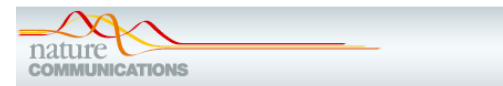
Electrolyzer Stack

¹*Energy Env. Sci.*, 2020, 13, 472-494

3 – Impact: Addressing Community Needs

Recent publications identify common needs for enabling CO₂ conversion

- Standardized testing protocols
 - MEA fabrication
 - Electrochemical testing
 - Product analysis: *Standard NMR method developed (FY21 Q2)*
- Accelerated durability test needed
 - Cells and systems need to get to 1000s of hours – long-term durability testing not practical
 - Degradation routes (catalysts, membranes, catalyst supports) must first be understood
 - *Catalyst and catalyst support degradation*



COMMENT

<https://doi.org/10.1038/s41467-020-19185-8> OPEN

The future of low-temperature carbon dioxide electrolysis depends on solving one basic problem

Joshua A. Rabinowitz¹ & Matthew W. Kanan¹

Carbonate formation is the primary source of energy and carbon losses in low-temperature carbon dioxide electrolysis. Realigning research priorities to address the carbonate problem is essential if this technology is to become a viable option for renewable chemical and fuel production.

Journal of Materials Chemistry A



PERSPECTIVE

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Cite this: *J. Mater. Chem. A* 2020, 8, 2257

Towards accelerated durability testing protocols for CO₂ electrolysis†

U. O. Nwabara,¹ M. P. de Heer,² E. R. Cofell,³ S. Verma,⁴ E. Negro⁵ and Paul J. A. Kenis^{1*}

In recent years, the electrochemical reduction of CO₂ (ECO₂RR) to value-added chemicals, fuels, and intermediates has been proposed as a promising option for utilizing excess CO₂ emissions. ECO₂RR could be integrated into existing CO₂-emitting industrial processes to mitigate emissions. To get to that stage, however, ECO₂RR cells and systems need to exhibit lifetimes of thousands of hours, similar to other commercially viable electrochemical systems. Accelerated durability testing (ADT) has been employed to rapidly screen the stability of these other electrochemical systems. Currently, most ECO₂RR studies only report durability for tens of hours. Yet, once the ECO₂RR field reaches longer system lifetimes as a whole, ADT studies will become necessary. In this perspective, we evaluate accelerated durability studies employed for fuel cells, water electrolyzers, and chlor-alkali systems and apply the knowledge to suggest an appropriate ECO₂RR ADT protocol, which is currently lacking.

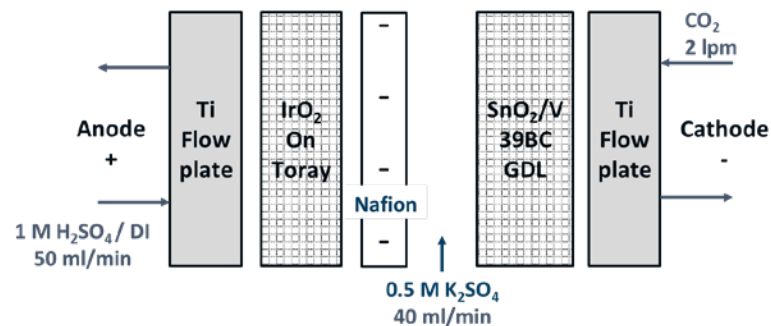
Received 3rd September 2020
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DOI: 10.1039/D0JA00695a

rsc.li/materials-a

4 – Progress: Baseline Existing MEA Architectures

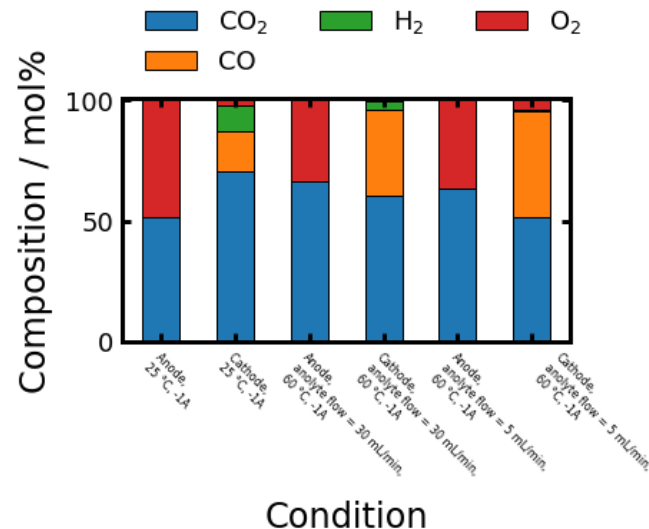
PEM MEAs

- Nafion membrane
- 150 mA/cm² @ ~3.5V (25 cm² cell)
- Flowing liquid buffer layer (K₂SO₄)
- SnO₂ catalysts selectively produce formate
- Significant hydrogen evolution occurred (~50% selectivity to H₂)



AEM MEAs

- Sustainion membrane (Dioxide Materials)
- 155 mA/cm² @ ~3.3V (5 cm² cell)
- High CO selectivity (98%) on Ag catalyst
- Significant CO₂ crossover (from cathode to anode) observed (50%)



4 – Progress: Membrane Integrity Diagnostic

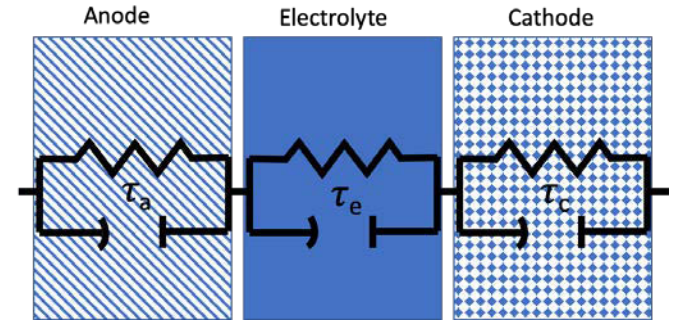
In situ Membrane Integrity

- CO₂ utilization MEAs employ very thin membranes (50 μm) – pinholes can develop during MEA fabrication
- Pinholes lead to parasitic losses and safety concerns

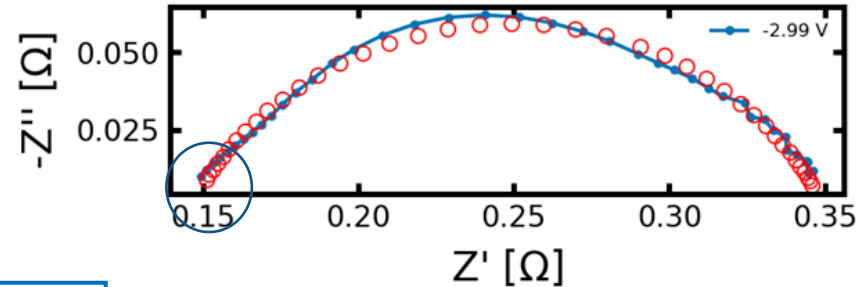
Electrochemical Impedance Spectroscopy (EIS)

- EIS is an easily-deployed, fast technique that can probe operating MEAs
- Test multiple MEAs with and without defects
- Membrane resistance defined as high-frequency (>38 kHz) limit of real impedance (Z') - circled
- Method yields area specific resistance (ASR) for membrane of operating MEAs
 - 1.52 Ω-cm² (<10% RSD) – below this membrane is compromised

EIS – Equivalent Circuit Model



EIS – Nyquist Plot



Diagnostic quickly detects if membrane has defects

4 – Progress: New Alkaline Exchange Membranes (AEMs)

Durability concern for existing AEM materials

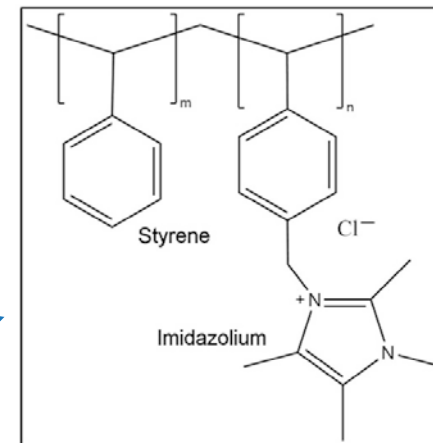
- Nafion (PEM membrane) is well-developed and durable, due to perfluorinated membrane backbone
- Significant recent AEM development for alkaline fuel cells – with a goal of increased durability
- NREL developed a new perfluorinated AEM membrane¹
 - Showed enhanced durability in AEM fuel cell testing (>500 hours @ 600 mA/cm²) and record performance with powdered ionomer in electrode
- Leading AEM for CO₂U is Sustainion – hydrocarbon backbone

Testing Perfluorinated AEM for CO₂U

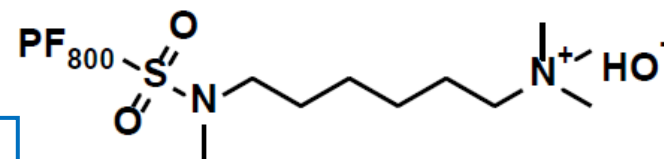
- Promising results – 200 mA/cm² @ -3.5V
- Ag cathode catalyst – CO main product
- No degradation seen in cell voltage over 1-hour test

New more durable membrane materials needed – initial results promising

Sustainion Membrane (Dioxide Materials):



NREL Perfluorinated Membrane:²



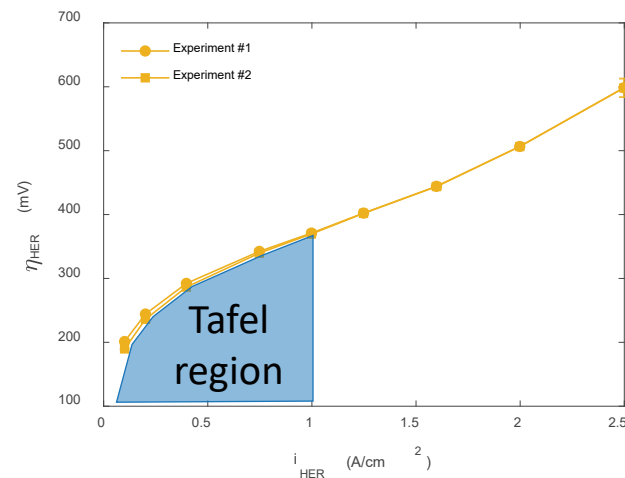
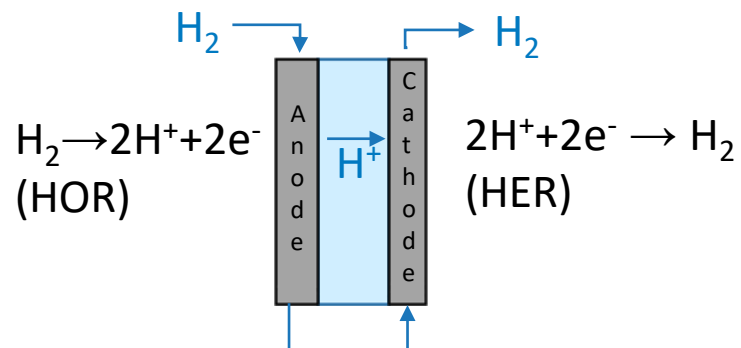
¹Advanced Ionomers & MEAs for Alkaline Membrane Fuel Cells, DOE Hydrogen and Fuel Cells Program, PI Bryan Pivovar;

²ECS Transactions, 80 (8) 957-966 (2017)

4 – Progress: Hydrogen Evolution Reaction (HER) Kinetics

Kinetic info lacking for CO₂U MEAs

- Setup experiment based on asymmetric Pt/Nafion/Pt MEA¹
 - High Pt loading (0.4 mg/cm²) at anode – HOR kinetics fast
 - Low Pt loading (0.03 mg/cm²) at cathode – HER kinetics dominate
 - Reproduced previous results on Pt
- Fabricated CO₂U MEAs: Pt/Nafion/Ag
 - Ag catalyst used to selectively form CO from CO₂
 - 0.4 and 0.8 mg/cm² Ag catalyst loading at cathode
- Evaluated HER kinetics on CO₂U MEAs
 - 40, 50, 60, 70 & 80 °C
 - Constant gas flow (1-2 slpm H₂), RH (100%), and P (150 kPa)
 - Extract Tafel slopes (semi-log plot)
 - Simplification of Butler-Volmer equation
 - Extrapolate to $\eta = 0$ to get exchange current density (i_0)
 - HER 10³ – 10⁴ slower on Ag than Pt
- *Moving forward: Integrated MEA modeling efforts in collaboration with CCPC*



¹Journal of The Electrochemical Society, 154 7 B631-B635 2007

Summary

- Management
 - Moved into ChemCatBio Consortium – leverage capabilities in catalyst synthesis & characterization and modeling
 - Close connection with Feasibility Study – technical feasibility and analysis
- Approach
 - Applied R&D on scalable single-membrane MEAs
 - Focus on enabling long-term & stable operation
- Impact
 - Identified technical challenges, targeted R&D to address challenges
 - Addressing specific needs of CO₂ utilization community (standard protocols, degradation)
- Progress
 - Baselined PEM and AEM MEAs
 - Developed membrane integrity diagnostic
 - New alkaline membrane material tested
 - Hydrogen evolution kinetics will enable integrated modeling efforts

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2019 Peer Review Comments

- This is a comprehensive project, and advancements were made in catalyst development. System design results can help demonstrate the variability of the approach. The connections between electrochemical and thermochemical pathways can be articulated to identify the synergies, and the linkage between the identified BETO barriers and deliverables can help shape future priorities.
 - Response: We thank the reviewer for the feedback and agree that there are clear synergies between the CO₂ conversion routes such as the synthesis of catalyst materials that are being leveraged across both pathways and characterization efforts that focus on key intermediates that are similar in both cases. Future deliverables will have metrics that directly address BETO MYP barriers through the incorporation of TEA in upcoming years
- This is a great team leveraging ChemCatBio efforts. Active collaborations with other expert groups. Thus, the team has a great potential to significantly advance the thermo and electrochemical catalysts for CO₂ conversion to C₂+. The consideration of using a wide range of CO₂ sources (e.g., ambient to industrial sources beyond ethanol plants) and the investigation of challenges associated with different CO₂ waste streams would further improve the potential impact of this study.
 - Response: We thank the reviewer for their positive comments and agree that the investigation of realistic CO₂ sources will bring additional value to the project. In the initial 2-years of the project we focused on developing a synthetic platform of catalyst materials and an understanding of the corresponding reactivity. Additionally, we have developed capabilities for the testing of different membrane-electrode-assemblies which are required for performing relevant research (including screening of different CO₂ sources).
- This project aims to benchmark and then further develop both electrochemical and thermochemical catalysts for the conversion of CO₂.

2019 Peer Review Comments

- This project aims to benchmark and then further develop both electrochemical and thermochemical catalysts for the conversion of CO₂.
- While the thematic goals are highly relevant to the BETO mission, the project has presented insufficient evidence to show that specific efforts in this project have a good chance of advancing the state of the art.
 - Response: On the thermochemical side, we sought to develop a synthetic strategy for a tunable catalyst system to develop a fundamental understanding of the impact of catalyst features on the resulting product slate. We acknowledge that the metal carbide system is only one type of catalyst system used for CO₂ reduction, but we believe that it affords the ability to rationally tune the catalyst structure and advance the state of the art over industrial materials. On the electrochemical side, our catalyst development efforts have yet to find a material that can outperform the state-of-the-art copper catalyst. However, electrocatalyst development with a tunable materials platform (transition metal phosphides) have been useful for studying electrochemical CO₂ reduction, and we are actively exploring new materials systems. Additionally, much work has been spent developing capabilities for catalyst testing in the relevant environment (in a membrane-electrode-assembly), and these capabilities are required to advance the current state-of-the-art.
- The performers have proposed to improve both electrochemical and thermochemical conversion of CO₂ to C₂+ products. The performers will be collaborating with the Feasibility study from NREL. The performers are suggested to focus on one of the two approaches (likely electrochemical) to maximize chance of success. The performers should also more clearly list out what the metrics of success (MoS) and the rationale for choosing those targets.
 - Response: In the future we will more closely align metrics of success for the project with BETO MYP barriers which will be facilitated with ongoing TEA efforts to compare electrochemical and thermochemical approaches.

Publications / Presentations

- “Transforming the carbon economy: challenges and opportunities in the convergence of low-cost electricity and reductive CO₂ utilization.” R. Gary Grim, Zhe Huang, Michael T. Guarnieri, Jack R. Ferrell III, Ling Tao* and Joshua A. Schaidle*, *Energy & Environmental Science*, 2020, 13, 472-494. <https://doi.org/10.1039/C9EE02410G>
- Downes, C.A.; LiBretto, N; Harman-Ware, A.E.; Happs, R.E.; Ruddy, D.; Baddour, F.; Ferrell, J.R. III; Habas, S.E.*; Schaidle, J.A.* “Electrocatalytic CO₂ Reduction over Cu₃P Nanoparticles Generated via a Single-Source Molecular Precursor Route” *ACS Appl. Energy Mater.* 2020, 3 (11), 10435-10446. <https://doi.org/10.1021/acsaem.0c01360>
- “Using waste CO₂ to increase ethanol production from corn ethanol biorefineries: Techno-economic analysis.” Zhe Huang, Gary Grim, Joshua Schaidle, Ling Tao*, *Applied Energy*, 2020, 280, 115964. <https://doi.org/10.1016/j.apenergy.2020.115964>
- Jack Ferrell, Courtney A. Downes, Ahn T. To, Susan E. Habas, Frederick E. Baddour, Kurt M. Van Allsburg, Erick A. White, “CO₂ Utilization: Electrochemical and Thermochemical Pathways” Invited Oral Presentation, Chemical Catalysis for Bioenergy Consortium: Addressing Deactivation during Biomass Conversion. *257th ACS National Meeting & Exposition*, Orlando, FL. April 3rd, 2019.
- Anh To, Courtney Downes, Frederick Baddour, Susan Habas, Erick White, Jack Ferrell, “CO₂ Utilization: Recent Results with Metal Carbides and Phosphides” Poster presentation. *Carbon Capture, Utilization and Storage: Gordon Research Conference*. May 5-10, 2019. Les Diablerets, Switzerland.

FY20 Milestones

Milestone Name/Description	Criteria	End Date	Type
Develop <i>in situ</i> MEA Diagnostics	Develop <i>in situ</i> electrochemical diagnostics tailored for CO ₂ utilization MEAs. Electrochemical impedance spectroscopy (EIS) and supporting measurements will be used to quantify <i>in situ</i> electrocatalyst activity, CO ₂ crossover (from cathode to anode), ohmic resistances of different parts of the MEA, and identify processes that lead to performance degradation (e.g., pH drift, membrane degradation, catalyst degradation, leakage current). Electrochemical process models will be developed, and model parameters will be used for benchmarking and MEA development in subsequent tasks. Target <10% variability (defined as % RSD) for the whole cell resistance from the EIS measurement.	12/31/2019	Quarterly
Alkaline Exchange Membrane (AEM) Membrane Development	While AEM materials operate at a pH favorable for CO ₂ utilization, persistent problems remain including loss of CO ₂ reactant (via bicarbonate crossover from cathode to anode) and membrane degradation. We will adapt new fluorinated AEM membrane materials to improve durability while maintaining high current density. Demonstrate degradation rates <10 mV/hr at current density >100 mA/cm ² .	3/31/2020	Quarterly
Proton Exchange Membrane (PEM) MEA Development	While PEM materials (e.g., Nafion) are very durable and have been proven for other applications, they operate at a pH that is too low to facilitate electrochemical CO ₂ utilization. Perform PEM MEA development, including efforts to reduce the pH at the membrane/cathode interface. Potential strategies to reduce the pH include the addition of solid or liquid-containing buffer layers, as well as the incorporation of AEM ionomer materials into the cathode catalyst layer. Demonstrate stable performance, defined here at maintaining >50% Faradaic efficiency to non-hydrogen products for at least 4 hours.	6/30/2020	Quarterly
Determine most promising type of membrane-electrode-assembly (MEA) for integration and <i>in situ</i> testing of state-of-the-art electrocatalysts.	At least 2 different types of MEAs will be tested: proton exchange membrane (PEM) and anion exchange membrane (AEM) MEAs. By integration of the same state-of-the-art electrocatalyst (e.g., Ag catalyst for selective formation of CO), performance and any stability issues will be compared for PEM and AEM MEAs. The most promising MEA will be the focus of future MEA development in this project, and will be used for <i>in situ</i> testing and baselining of electrocatalysts in the electrolyzer environment. Target maintaining high Faradaic efficiency (>50% to non-hydrogen products) for at least 8 hours, at a relevant current density (>100 mA/cm ²).	9/30/2020	Annual SMART

FY21 Milestones

Milestone Name/Description	Criteria	End Date	Type
Evaluate Hydrogen Evolution Reaction Kinetics in the MEA Environment.	Measure kinetic parameters including Tafel Slopes and Exchange Current Densities for the Hydrogen Evolution Reaction (HER) in a proton exchange membrane (PEM) MEA. HER kinetics remain unexplored for CO ₂ Utilization MEAs, and these parameters will inform modeling and scaleup efforts, as well as strategies to limit hydrogen evolution in PEM MEAs. Hydrogen evolution is a persistent problem that significantly reduces the Faradaic efficiency of CO ₂ conversion in PEM MEAs. Kinetic data produced in this milestone will inform future meso-scale modeling efforts in collaboration with the CCPC.	12/31/2020	Quarterly
Develop Standard Method for Product Analysis from CO ₂ Utilization MEAs.	Agreed-upon Standards remain a need in the CO ₂ Utilization community. As electrochemical CO ₂ conversion can create various products, standard analytical methods are needed to quantify these products. Currently, there is no consensus on product quantification, that is, many different techniques and analytical methods are used. We will develop at least one standard analytical method for product quantification and will deliver a Laboratory Analytical Procedure (LAP) with this milestone. The LAP can later be published on an NREL website, where it will be free and publicly available.	3/31/2021	Quarterly
Risk Identification	Based on subject matter expert interviews, critical literature review, and research to-date in the Electrocatalytic CO ₂ Utilization Project (2.3.1.316) develop a risk register for the CO ₂ reduction pathway evaluated in the FY21 Q4 Annual SMART milestone in the Feasibility Study Project (2.1.0.304), characterize the risks based on probability and impact, and identify responsible parties. Technical risks will be evaluated on both a unit operations level and a systems integration level. This is a joint milestone with the Feasibility Study of Utilizing Electricity to Produce Intermediates from CO ₂ and Biomass (2.1.0.304).	6/30/2021	Quarterly
Integration of Electrocatalysts into CO ₂ Utilization MEAs.	Integrate at least two (2) electrocatalysts into CO ₂ utilization MEA cathodes. Develop methods for reliable application of electrocatalysts and identify any differences between electrocatalyst activity in the <i>ex situ</i> catalyst screening test (in aqueous solution) and <i>in situ</i> activity in the MEA cathode. This milestone is in collaboration with the Advanced Catalyst Synthesis and Characterization (ACSC) project within ChemCatBio.	9/30/2021	Annual SMART

Go/No Go

Name	Description	Criteria	Date
Long-term operation at relevant current density.	Stable and long-term electrocatalytic CO ₂ conversion at significant current density remains a challenge. Develop MEA architectures that can enable stable operation at sufficient current density.	Demonstrate stable (degradation rates < 10 mV/hr) and long-term (>24 hours) electrocatalytic CO ₂ conversion in a membrane-electrode-assembly (MEA) at relevant current density (>100 mA/cm ²). This degradation rate represents an upper-bound for what will be needed for scale-up.	3/31/2021

***On track to complete the
Go/No Go on time***