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13. SUPPLEMENTARY NOTES The views, opinions and/or findings contained in this report are those of the author(s) and should not be construed as an official Department of the Army position, policy or decision, unless so designated by other documentation.
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14. ABSTRACT
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15. SUBJECT TERMS
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a. REPORT UU	b. ABSTRACT UU	c. THIS PAGE UU	19b. TELEPHONE NUMBER 425-445-8358

# RPPR Final Report

## as of 13-Jan-2022

Agency Code: 21XD

Proposal Number: 77269CHSB1

Agreement Number: W911NF-20-P-0072

### INVESTIGATOR(S):

**Name:** Todd Brix  
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**Principal:** Y

Organization: **OCO Inc.**

Address: P.O. Box 190, Blue River, OR 974130190

Country: USA

DUNS Number: 081218648

EIN:

**Report Date:** 19-Feb-2022

Date Received: 11-Jan-2022

**Final Report** for Period Beginning 20-Aug-2020 and Ending 19-Feb-2022

**Title:** On-site Electro-synthesis of Potassium Formate from Recycled CO<sub>2</sub>

**Begin Performance Period:** 20-Aug-2020

**End Performance Period:** 19-Feb-2022

**Report Term:** 0-Other

Submitted By: Todd Brix

Email: toddbrix@ocochem.com

Phone: (425) 445-8358

**Distribution Statement:** 1-Approved for public release; distribution is unlimited.

### STEM Degrees:

### STEM Participants:

**Major Goals:** The primary goal of the Phase I option was to modify the gas diffusion electrodes (GDEs) yield improved performance in terms of Faradic Efficiency (FE) stability over time in preparation of the Phase II work.

A variety of experiments were designed and executed to do this including methods to reduce GDE wetting, increase GDE hydrophobicity via hydrophobic additives to the catalyst ink, adding a Gas Diffusion Layer (GDL), and changing the reactor hydrodynamic design

**Accomplishments:** The work done in this Phase I options period helped in making significant optimizations to the GDE (including the modification of the gas diffusion layer) to enhance its performance, and these optimizations have been built upon to further the performance of the GDE as reported in the Phase II monthly reports thus far. Apart from that, the learnings from the unsuccessful methods in enhancing the GDE and reactor performance has led to a better understanding of the process which will assist in better planning and approach to meet the targets of Phase II.

Specific Accomplishments Include:

- 1) Successfully fabricating, applying and testing a gas diffusion layer consisting of a PTFE mixture.
- 2) Applying a porous polymer layer on top of the GDE to reduce wetting of the GDE
- 3) Applying a porous structure of the CO<sub>2</sub> gas side of the GDE to help gas distribution and increase GDE electrical contact:
- 4) Testing several different GDEs were prepared with modified binder content and gas diffusion layer (GDL) with varying PTFE wt% coatings

The work done in this Phase I options period helped in making significant optimizations to the GDE (including the modification of the gas diffusion layer) to enhance its performance, and these optimizations have been built upon to further the performance of the GDE as reported in the Phase II monthly reports thus far. Apart from that, the learnings from the unsuccessful methods in enhancing the GDE and reactor performance has led to a better understanding of the process which will assist in better planning and approach to meet the targets of Phase II.

**Training Opportunities:** Nothing to Report

**Results Dissemination:** Nothing to Report

# RPPR Final Report

as of 13-Jan-2022

**Honors and Awards:** Nothing to Report

**Protocol Activity Status:**

**Technology Transfer:** OCO's work in improving and scaling the electrochemical synthesis of potassium formate, supported by the US Army, led to several discussions with several companies and national laboratories including Pacific Northwest National Laboratory (using formate as a hydrogen carrier in a flow reactor-based energy storage application, a biochemical organism design company (using formate as a microbial feedstock) an oil and gas services company (using formate as a drilling/completion fluid).

This interest is indicative of the need for more sustainable and modular ways to make potassium formate for a wide variety of commercial applications in addition to its major use today as a road de-icing chemical.

**PARTICIPANTS:**

**Participant Type:** PD/PI

**Participant:** Arun S. Agarwal

**Person Months Worked:** 1.00

Project Contribution:

National Academy Member: N

**Funding Support:**

**Participant Type:** Other (specify)

**Participant:** Jared Hague

**Person Months Worked:** 2.00

Project Contribution:

National Academy Member: N

**Funding Support:**

**Partners**

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I certify that the information in the report is complete and accurate:

Signature: Todd Brix

Signature Date: 1/11/22 3:48PM



## Monthly Report

Proposal Title:	<b>On-site Electro-synthesis of Potassium Formate from Recycled CO<sub>2</sub></b>		
Purchase Order #	W911NF20P0072	Time Period	November 11 – December 10, 2021
Proposal #	A201-028-0212	Monthly Report #	4/4
CIN:	GFEB500116777870000	ITEM NO	000304
Contractor Name	OCO, Inc.	UNIT QUANTITY	1
Contact	Todd Brix	UNITS	1
Address	16642 NE 162 <sup>nd</sup> CT, Woodinville, WA 98072	UNIT PRICE	\$13,471.00
		AMOUNT	\$13,471.00
Planned (Former) Work Location	49356 McKenzie Highway Vida, OR 97488-9711	Contracting Officer	Jessica Harrington (919) 549-4336 Jessica.m.harrington14.civ@mail.mil
New ( as of 11/2) Work Location	350 Hills Street Richland, WA 99354		
UIC:	W6QK43	Direct Labor Hours	20
Federal Service Code	AZ11	Direct Labor Dollars Paid:	\$2,767.50
Contractor Type:	Prime	Command of the Requiring Activity	W911NF

### Key Work and Updates

As reported in the month 3 monthly report of the current Phase I options period, electrochemical tests conducted on modified gas diffusion electrodes (GDEs) yielded improved performance in terms of Faradic Efficiency (FE) stability over time. In this period, we evaluated methods to reduce wetting of the GDE to help maintain high CO<sub>2</sub> mass transport within the GDE and thus maintain the electrochemical performance as a function of time. Unfortunately, these attempts did not yield desired results. However, they helped eliminate these methods as potential solutions to improving the hydrophobic character of the GDE and help with better uniform CO<sub>2</sub> distribution.

#### Applying a porous polymer layer on top of the GDE to reduce wetting of the GDE:

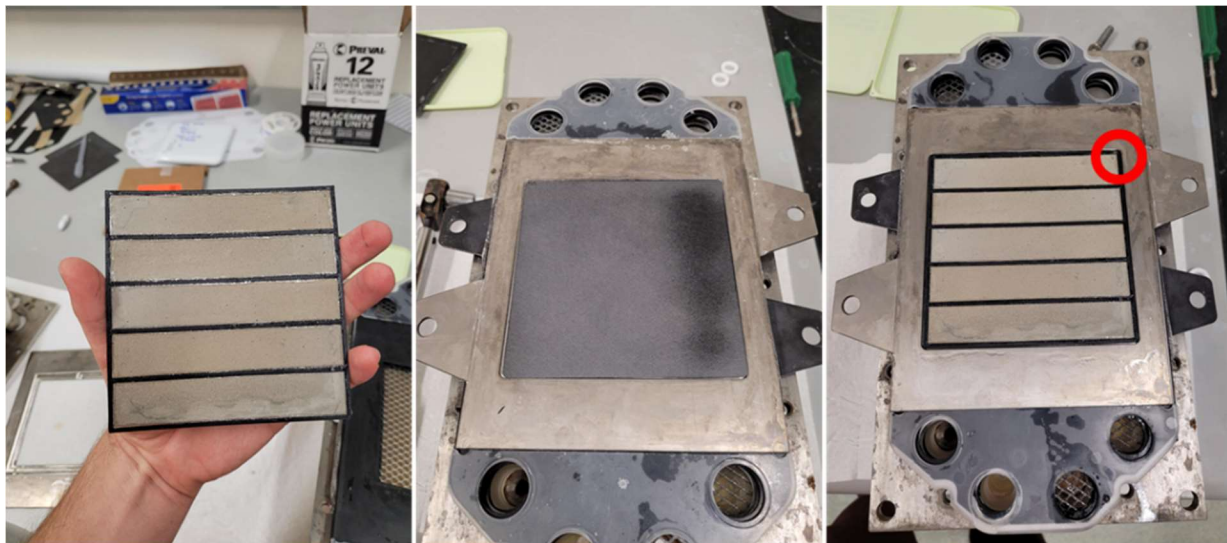
Some recent literature suggested that application of a porous polymer layer on top of the catalyst layer (via spraying of a separate layer of dilute polymer solution on top of the catalyst

layer of the previously prepared GDE) would potentially create a new, hydrophobic barrier that better assists in keeping the electrolyte from wetting the catalyst layer. This method was attempted to make PTFE porous layers on nano Sn on C coated toray paper GDEs by spraying dilute solutions (1% PTFE) of polymer. 3 different electrodes were prepared, where the quantity of spray polymer volumes were varied to impact perceived thickness of polymer layer (or improve hydrophobicity). However, contrary to expectation, there was no impact on the %FE performance as compared to the electrode without the polymer top coating. Possibly, the polymeric spray, which was made in isopropanol, dissolved the catalyst layer and mixed it with the polymer, such that, instead of a secondary top polymer layer, a new composite catalyst and polymer layer was potentially created, which would be quite similar to the original catalyst layer.

### **Applying a porous structure of the CO<sub>2</sub> gas side of the GDE to help gas distribution and increase GDE electrical contact:**

A tin based porous structure attached to a 3-D printed backing was obtained as indicated in Figure 1 and placed behind the GDE, on the CO<sub>2</sub> gas side. The hypothesis here is that the presence of the fine porous metal structure in contact with the GDE will help with the CO<sub>2</sub> gas distribution and reduce/eliminate wetting of the carbon GDE by maintaining a uniform flux of CO<sub>2</sub> across the height of the electrode, along with providing better electrical contact since the GDE is in direct contact with the metal porous structure.

After initial setup *trial and error*, the structure was successfully integrated with OCO's 100 cm<sup>2</sup> cell. Sub-optimal electrochemical full cell performance indicated in Figure 2 point to aspects of porous structure that need to be significantly modified & optimized to be of value.



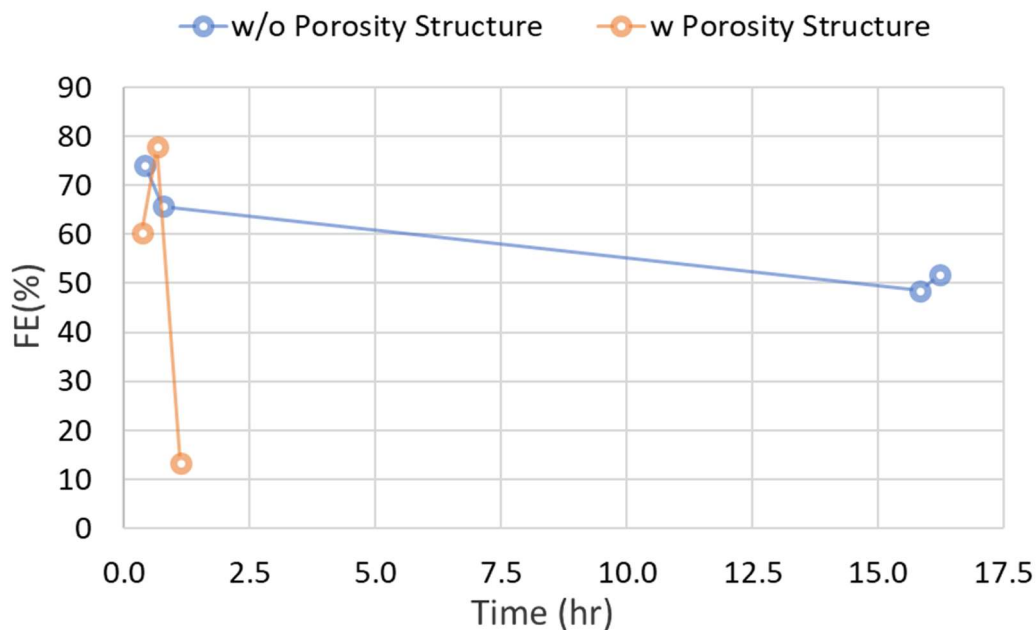
**Figure 1. Porosity structure integrated on the gas side of the GDE in the 100cm<sup>2</sup> cell.**

Upon integration and running the electrochemical cell with the porosity structure (Figure 1, above shows the setup, and Figure 2, below, indicates the results and comparison with and without porosity structure) the following observations were made:

- ~15% lower current density at same  $V_{cell}$  → metal porous structure unable to provide conductive path potentially due to gap between GDE & porous structure



- Same initial %FE as without porosity structure (<1 hr)
- Steep fall is FE to low value (<15%) due to electrolyte seeping between porosity structure & GDE within an hour, causing the CO<sub>2</sub> distribution to worsen and hence, low %FE



**Figure 2. Comparison for FE performance with and without the porosity structure in 100cm<sup>2</sup> cell.**

**Upcoming Month Tasks.**

This is the 4<sup>th</sup> and final report of the options period, which marks the end of the Phase I options work, while the work on the Phase II continues. The work done in this Phase I options period helped in making significant optimizations to the GDE (including the modification of the gas diffusion layer) to enhance its performance, and these optimizations have been built upon to further the performance of the GDE as reported in the Phase II monthly reports thus far. Apart from that, the learnings from the unsuccessful methods in enhancing the GDE and reactor performance has led to a better understanding of the process which will assist in better planning and approach to meet the targets of Phase II.