

Carbon Dioxide Collection and Pressurization Technology

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The Martian atmosphere is composed of about 95% carbon dioxide which can be processed to produce useful consumables such as oxygen and, with a hydrogen source, methane. Leading technologies for processing CO₂ are solid oxide electrolysis (SOE), reverse water gas shift (RWGS) and the Sabatier reaction. Typically, these processes benefit from a high purity carbon dioxide source. A significant feed rate can be accomplished if that source is pressurized. Reactive Innovations has developed an electrochemically-driven technology platform which can be customized to fulfill the many individual separation requirements of ISRU technology processing streams. In recent NASA-funded programs, our technology was shown to be highly adaptable in performing selective separation of species from gas and liquid streams. Recent work has utilized this general approach to not only separate but pressurize carbon dioxide. In addition we applied this reactive chemistry to our tubular reactor platform for further advantages in reducing weight and volume for space-based applications. We previously reported results (ICES-2016-134) which demonstrated that selection of individual components, including redox carrier, ionic liquid, and membrane material play a significant role in advancing the mechanism of carbon dioxide separation and pressurization. This paper provides an update on our most recent work which has shown further improvements to the technology.

Nomenclature

ISRU	= In Situ Resource Utilization; using resources at the site of exploration
SOE	= solid oxide electrolysis
RWGS	= reverse water gas shift
SILM	= supported ionic liquid membrane
MEA	= membrane electrode assembly
IL-PVDF	= ionic liquid polyvinylfluride composite membrane
mil	= unit of length equal to 0.001 inch
psia	= pressure measurement in pounds per square inch, absolute; includes atmospheric pressure

I. Introduction

Mars is the ultimate destination of NASA's human exploration program. In Situ Resource Utilization (ISRU) is a key technology required to enable such missions. The goals of using resources that are available at the site of exploration and pursuing the philosophy of "living off the land" instead of bringing it all the way from Earth are to achieve a reduction in launch and delivered mass for exploration missions, a reduction in mission risk and cost, enable new missions not possible without ISRU, and to expand the human presence in space. Past studies have shown making propellants and other mission critical consumables (life support and power) in situ can significantly reduce mission mass and cost,¹ and also enable new mission concepts.

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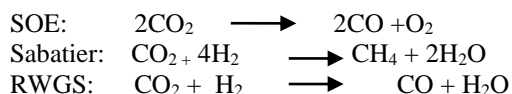
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Table 1. Mars atmosphere composition²

Viking Atmospheric Measurements		
Composition	carbon dioxide	95.32%
	nitrogen	2.7 %
	argon	1.6%
	oxygen	0.13%
	carbon monoxide	0.07%
	water vapor	0.03%
	neon, krypton, xenon	trace
	ozone, methane	
Surface Pressure	100-900 Pa, depending on altitude average 700 Pa	

While NASA's understanding of Mars' resources continues to evolve--significantly with the recent indication of the presence of water, a well-known resource is in the Martian atmosphere. The Martian atmosphere (Table 1) is composed of about 95% carbon dioxide which can be processed to produce useful consumables such as oxygen and methane. Leading technologies for processing CO₂ are solid oxide electrolysis (SOE), reverse water gas shift (RWGS) and the Sabatier reaction. The SOE process dissociates Martian CO₂ into O₂ and CO via a high temperature (~1000°C) electrochemical process through a thin zirconia membrane. The Sabatier and RWGS processes use a catalyst to react CO₂ with H₂ to produce

CH₄, and H₂O, and CO and H₂O respectively. The chemical processes are shown below:



Typically, these processes benefit from a high purity carbon dioxide source. A significant feed rate can be accomplished if that source is pressurized. An example feed rate goal is to supply a Sabatier reactor with 88g CO₂/hr at 50 psia.³ To meet this goal, there have been a variety of methods studied to capture and pressurize carbon dioxide from the Martian atmosphere including: freezing at cryogenic temperature, mechanical compression, liquefaction at high pressures, adsorption pumping, separation using membranes, sorption by ionic liquids in both liquid form and supported membranes, acid-base reaction with amines, preparative chromatography, and molecular sieves. A recent study conducted by NASA³ concluded that freezing was the most advantageous of these when considering power usage, collection rate, and CO₂ purity. However, the freezing technology requires ancillary storage tanks adding to the mass and volume of the CO₂ collection system.⁴

Another important application for CO₂ separation technologies involves air revitalization where carbon dioxide must be removed from the cabin of spacecraft and spacesuits. Some separation technologies being considered for this application include physical, centrifugal processes, adsorption (pressure swing) techniques,⁵ and chemical absorption processes.

Our approach relies on a self-regenerating supported ionic liquid membrane, where a redox carrier selectively binds and releases carbon dioxide into a pressurized gas stream which can be fed directly into the ISRU reactive processes. Alternatively, our technology could be used to continuously remove CO₂ from space habitats.

II. Technical Approach

Reactive Innovations has developed an electrochemically-driven technology platform which can be customized to fulfill the many individual separation requirements of ISRU technology processing streams. The enabling component of our technology capitalizes on the unique customizable properties of ionic liquids in performing room temperature efficient separations. In recent NASA-funded programs, our technology was shown to be highly adaptable in performing selective separation of species from gas and liquid streams. In one program, we demonstrated carbon dioxide separation from a CO₂/N₂ stream at varying concentrations using a planar electrochemically modulated cell. Our recent work utilizes this general approach to not only separate but pressurize carbon dioxide. In addition, we are applying this reactive chemistry to our tubular reactor platform for further advantages in reducing weight and volume for space-based applications.

Our technology relies on a facilitated transport mechanism. The concept proceeds through the redox binding affinity of a carrier molecule. In the reduced state this molecule binds CO₂. By changing the potential, the carrier is oxidized and CO₂ is released. The redox carrier is carefully selected to maximize the "swing" in the redox potential to increase the effective binding and release of CO₂.

In our concept, an ionic liquid is used to dissolve the redox carrier molecule. The ionic liquid medium is an enabling component for demonstrating facilitated transport for this space application because of its many advantageous properties, which include near zero vapor pressure, wide liquidus range, thermal stability, non-explosive, and wide electrochemical window. In our approach, the ionic liquid/redox carrier is imbibed in a membrane, such as Nafion, forming a supported ionic liquid membrane (SILM). Electrodes are pressed against the solid membrane and supply a potential to reduce and oxidize the redox carrier at the cathode and anode respectively forming an electrochemically modulated device as shown schematically in Figure 1. Gas mixtures containing carbon dioxide are introduced at the cathode. As a potential is applied, carbon dioxide is selectively bound to the reduced form of the redox carrier and is carried across the membrane to the anode where it can be released.

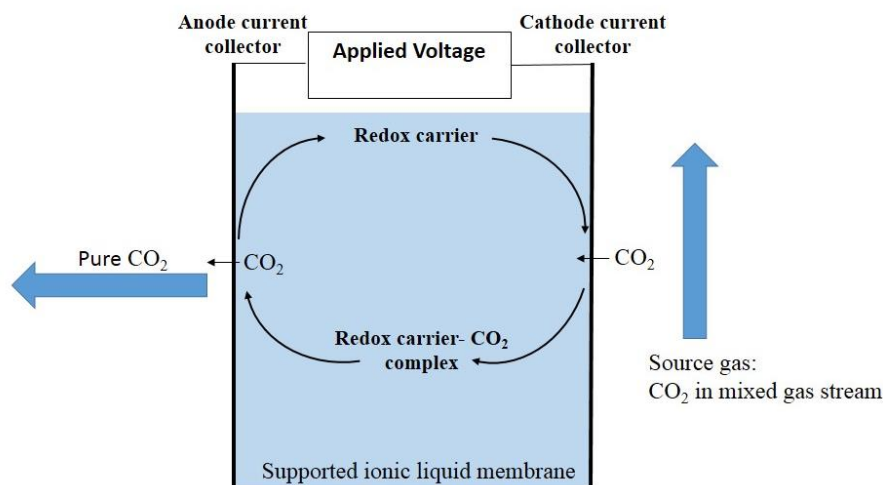


Figure 1. Facilitated Transport of CO₂

Unlike conventional membrane and absorption schemes, our approach selectively binds CO₂ via charge transfer mechanisms to redox carriers. This allows us to transfer the bound molecule through a membrane where it is subsequently released. With this approach, no regeneration step is required for this membrane allowing a continuous process to occur to separate and pressurize CO₂. This concept can be utilized in both a planar or tubular architecture. A schematic of the single tubular membrane electrode assembly (MEA) is shown in Figure 2 along with the relevant reactive processes for CO₂ separation.

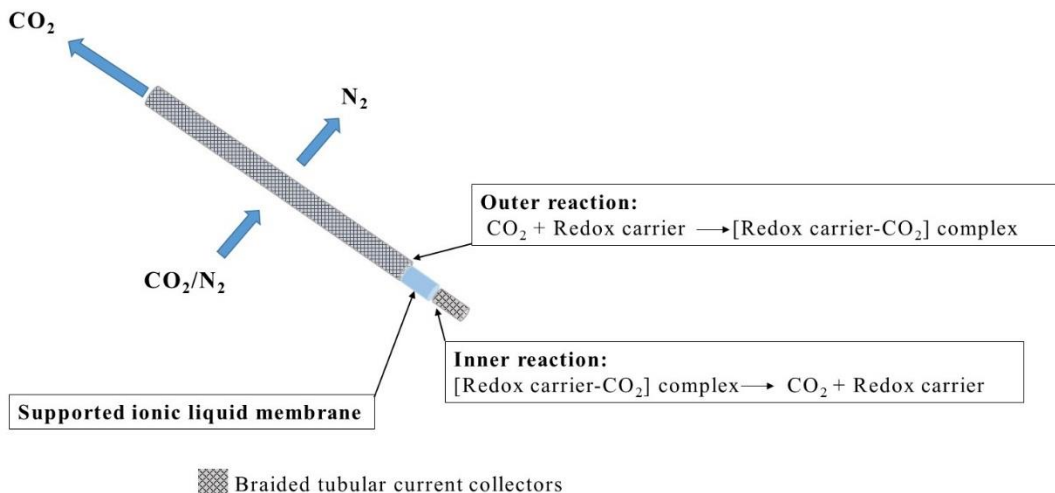


Figure 2. Tubular membrane-electrode assembly (MEA) Concept

An array of these tubular cells are then inserted into manifold covers that allow fluids to enter and leave through the interior flow tubes or through holes placed in the manifold as shown in Figure 3. Electrical connections are made from these manifold covers to either the interior porous metal flow through tubes or the external current collector wrapped around the outer electrode. The tubular cells are placed close together to allow sealing in the manifolds and for processing gases mixtures to flow over the tubes. Using the 2.79 mm diameter Nafion-based tubes, a multi-tubular assembly can be inserted into a cylindrical vessel with end-caps applied as shown in Figure 3. Similar hardware based on Reactive's multi-tubular array technology has been previously delivered to NASA incorporating the reactive chemistry for a high-pressure (2170 kPa) water electrolyzer for lunar and Martian applications.⁶

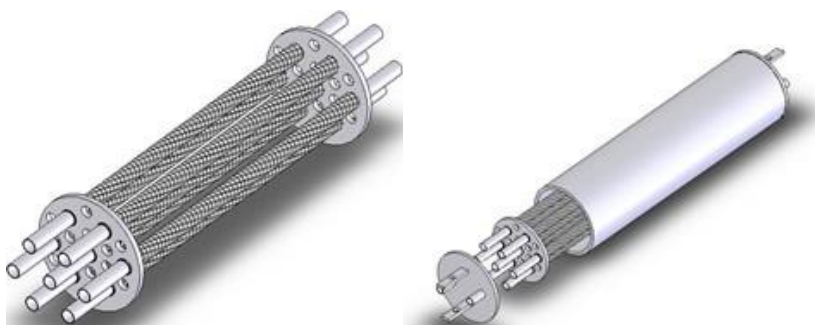


Figure 3. Reactive's multi-tubular array hardware: Multi-tubular cell arrangement in manifold covers (left) and reactive-separator cells in a containment vessel (right)

III. Membrane Fabrication and Evaluation Methods

We have prepared and evaluated two types of membranes for this application: 1) Nafion-based ionic liquid imbibed membranes and 2) ionic liquid-poly-vinylidene fluoride (IL-PVDF) composite gel membranes. Commercial planar and tubular Nafion ion-exchange membranes (Nafion 115, Nafion 112) were purchased from Sigma Aldrich and Permapure and pretreated to result in an H^+ form. Redox carriers (Alfa Aesar) were dissolved in 4-6 ml samples of commercially available ionic liquids (Aldrich) as received to result in saturated solutions. Nafion membranes were imbibed using a company proprietary method. A preparation method for IL-PVDF membranes has been reported by Jansen et al.⁷ This method was modified in the present work to include redox carrier species in the ionic liquid as well as to vary constituent concentrations and quantities to result in gel membranes of varying thickness and component ratios. In some instances, gel membranes were compressed in a heated hydraulic press to result in specific membrane thicknesses which range from 0.1-0.4 mm.

Membranes were evaluated in planar and tubular forms. A schematic of the test apparatus is shown in Figure 4 incorporating the planar test hardware. A photograph is shown in Figure 5. The planar cell has a 25 cm² active area and Teflon flow fields on both the anode and cathode sides. Current collectors comprised of platinum mesh or carbon fiber were pressed against each side of the membrane inside the cell. MEAs were evaluated by flowing pure carbon dioxide (or in separate experiments, a 1% carbon dioxide/99% nitrogen mixture) on one side of the single cell (cathode). Typical flow rates were 30 cc/min on both the anode and cathode. A DC power supply is used to apply a potential (0-4V) across the anode and the cathode and current is measured as reactive separation occurs. Outlet flow lines from the cell are directed to a commercial CO₂ analyzer (LiCOR) to give absolute concentration of CO₂ in the product stream and a gas chromatograph (SRI Multigas analyzer #2) to measure outlet composition ratios, thus confirming the reactive-separation process. As shown, helium (or nitrogen) is used to entrain the carbon dioxide in the anolyte to flow through the diagnostics and confirm CO₂ transport. The anolyte effluent stream has a pressure gauge and valving to allow CO₂ to build up pressure and confirm pressurization. A schematic of the single tubular MEA was shown earlier in Figure 2 along with the relevant reactive processes for CO₂ separation. Photographs of a single tubular MEA and a tubular reactor are shown in Figures 6 and 7. Typical 6-inch tubular MEAs have electrode active areas of 12 cm².

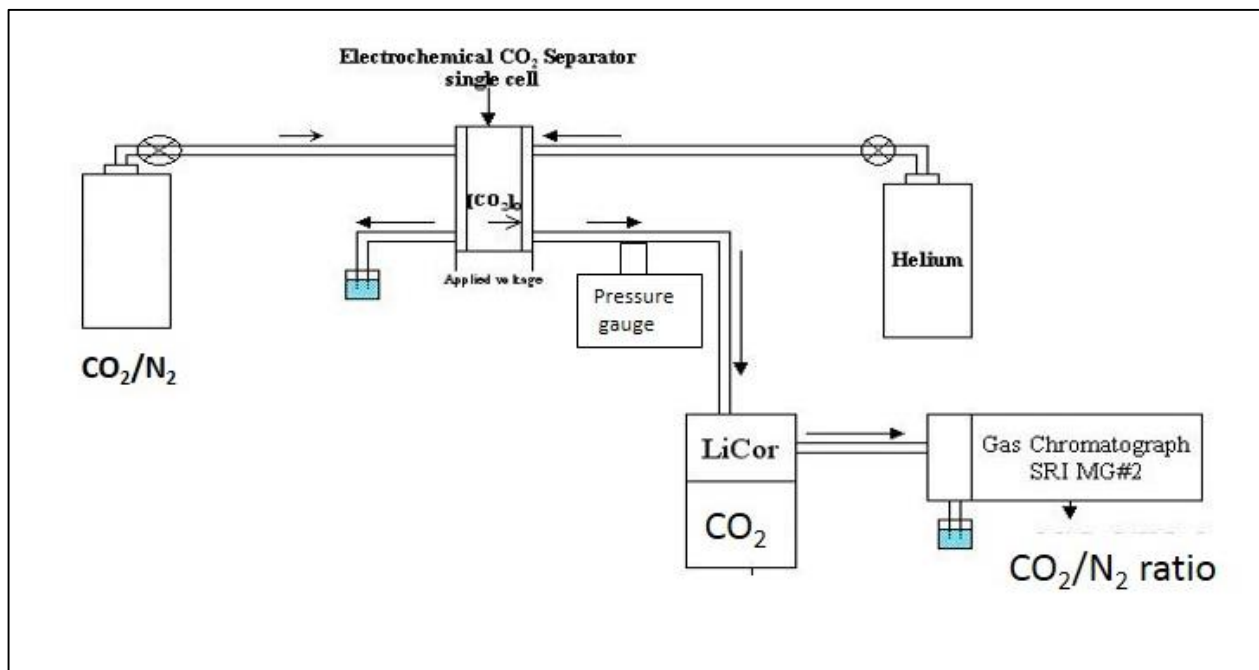


Figure 4. Single Cell Test Apparatus

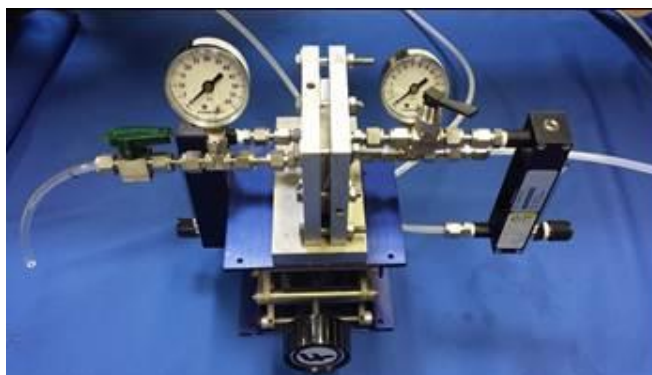


Figure 5. Photograph of Planar Membrane Test Apparatus



Figure 6. Photograph of Carbon Fiber Braided Tubular MEA



Figure 7. Photograph of a Single Tubular Reactor

We have evaluated both planar and tubular membranes using this hardware. The types of membranes are described in general terms in Table 2. Each category is representative of multiple experimental matrices designed to optimize the formulations. Some of the results from membranes have been previously reported.⁸ Some of those results are revisited below as it pertains to the motivation for the present work. Following that we report on new studies of membrane types highlighted in bold in Table 2.

Table 2. Membrane Categories under Development for CO₂ Collection and Pressurization

Redox Carrier/Ionic Liquid Formulation	Membrane support	Redox Carrier	Form
A	PVDF	1	planar
B	PVDF	2	planar
A	Nafion 115	1	planar
B	Nafion 115	2	planar
B	Nafion 112	2	planar
A	Nafion	1	tubular
B	Nafion TT-110	2	tubular

IV. Discussion of Earlier Work and Motivation for Current Studies

In an earlier paper⁸ we reported on our initial work involving Nafion-based and PVDF-based membranes incorporating different redox carriers to transport and pressurize carbon dioxide for ISRU applications. Both types of membranes demonstrated electrochemically modulated transport in planar tests, by monitoring the anode effluent using a CO₂ gas analyzer (LiCor).⁸ However we observed significant differences in stability and performance with component and membrane choice. These early data suggested that a PVDF-based membrane may have advantages in improving the rate of the process, so further studies on this type of membrane were warranted.

Also reported in the earlier paper, one redox carrier/ionic liquid formulation (A) was transitioned from planar to a tubular Nafion platform for pressurization studies. Some of the earlier data is reproduced here, as it informed the present work. A 6-inch tubular MEA was imbibed with formulation A and evaluated in the tubular reactor shown in Figure 7.

Pure CO₂ was introduced to the cathode at ambient pressure. With the anode sealed off (no gas flow) we applied 4 volts on the cell and monitored the pressure as well as the current. As shown in Figure 8, the pressure built up over time in two different trials. As shown, the rate of increase was about 0.26 psi/hr.

We also evaluated the cell performance to pressurize CO₂ starting at an elevated pressure. Figure 9 plots the pressure rise over time starting at a CO₂ gas pressure of about 58 psia. As Figure 9 shows, the initial rate of pressure rise was high (3.7 psi/hour) but decreased over time to about the same rate shown in Figure 8. Importantly, the fact that we observe a similar rate of pressurization starting at both low and high pressure in this experiment is very promising and implies that the rate is independent of the final pressure. One concern that we had in implementing this technology to pressurize carbon dioxide was that back diffusion at higher pressures would result in a lower net transport rate. The pressure is asymptoting with time in Figure 9 because of performance loss in the MEA evidenced by a large drop in current. At the end of the test we allowed the cell to stay stagnant at differential pressure overnight and did not observe any back diffusion into the catholyte.

These pressurization tests demonstrated the feasibility of the technology to collect and pressurize carbon dioxide using a supported ionic liquid membrane. The earlier studies also highlighted some of the issues which needed to be

improved to meet ISRU application needs. The CO₂ transport rate based on the planar studies of PVDF and Nafion membranes was low, ranging from 10⁻⁹-10⁻¹⁰ moles/CO₂/ hr/cm². At this rate, a scaled-up reactor would require a very large membrane area (100's of square meters) to meet a feed rate goal of 88 gr CO₂/hr. In addition, the transport rate was not stable: these membranes displayed a continuous loss in performance over time.

The objective of the more recent work was two-fold: 1) improve the transport rate and 2) improve the stability of the supported ionic liquid membranes. The current paper presents the status of that developmental effort which focused on planar membrane performance to improve the fundamental transport characteristics of the membranes.

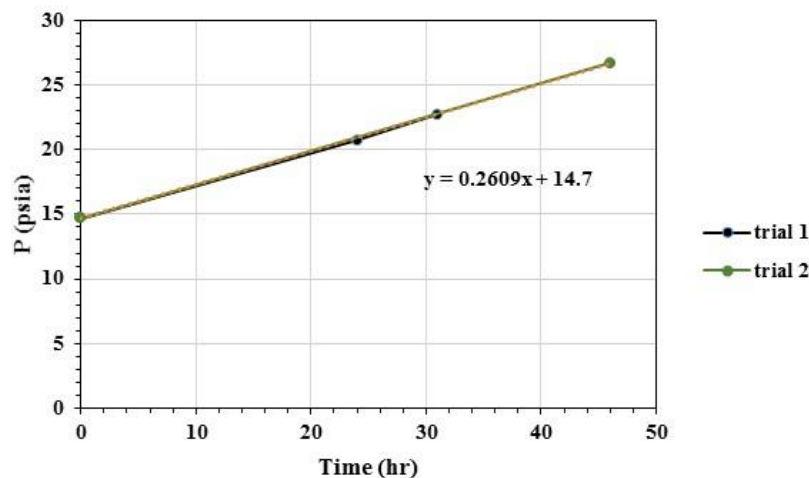


Figure 8. Single tubular reactor CO₂ collection and pressure build-up starting at atmospheric pressure

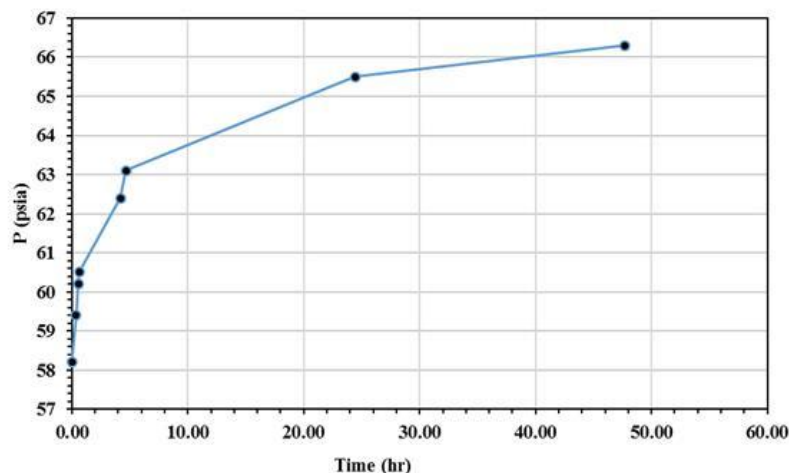


Figure 9. Single tubular reactor CO₂ collection and pressure build-up starting at 58 psia

V. Planar Membrane Performance

A. PVDF-based Membrane Performance

A photograph of an IL-PVDF membrane is shown in Figure 10. IL-PVDF membranes have been prepared using two different redox carriers A and B. Initial work suggested that Formulation B was the more promising recipe and recent work has focused on improving performance using this type of formulation in an IL-PVDF membrane. In our tests, the effluent of the permeate side of the single cell is monitored using LiCOR to measure the amount of CO₂ transported across the membrane and entrained in an inert gas as the voltage is turned on and off. Pure carbon dioxide was used as the feed gas. These tests were performed at atmospheric pressure on both sides of the membrane. The applied voltage across the cell results in an increase in the CO₂ gas concentration on the effluent side. In addition to the facilitated transport mechanism, transport is observed due to a dissolution/diffusion mechanism in the ionic liquid itself. This results in a constant background level of carbon dioxide.



Figure 10. Photograph of IL-PVDF membrane

Applying the voltage to the cell increases the CO₂ concentration over that of the background. Some of the variations to the membrane formulation we studied include: redox carrier concentration, ratio of IL to PVDF content, PVDF content, and thickness of the membrane. These factors were studied through a series of membrane fabrication matrices to synthesize duplicate membranes for testing in the single planar cell. Overall, trends were difficult to observe because of the variable performance we observed for any given membrane and in comparing duplicates. The most consistent performance was observed using the 0.25 mm thick membranes we prepared with the highest redox concentration in the ionic liquid. Figure 11 is the LiCOR data for this type of PVDF membrane monitoring the effluent of the single cell as the voltage is turned on and off. Based on the measured CO₂ concentration in the anode effluent and the carrier gas flow rate in these planar experiments, we calculate a facilitated transport rate of about 9×10^{-9} moles CO₂/min/cm² for the best PVDF membrane.

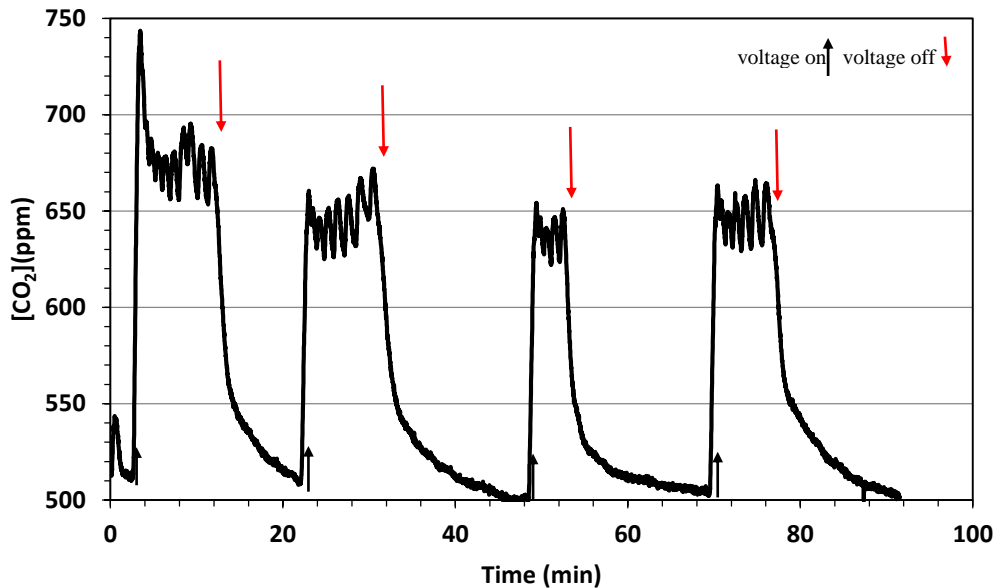


Figure 11. CO₂ concentration in anode effluent of planar cell as voltage is turned on and off. Applied voltage is 2.0 V. Membrane is IL-PVDF membrane imbibed with Formulation B. Feed gas is 100% CO₂

B. Nafion-based Membrane Performance

Earlier data using Nafion-based membranes focused on Formulation A. One issue with Formulation A is the solubility limit of the redox carrier in the ionic liquid which may be limiting the performance. More recently we modified the imbibing procedure to enable fabrication of Nafion membranes containing Formulation B, with increasing amounts of redox carrier. Figure 12 is the performance of a baseline membrane made with the lowest amount of redox carrier B. As shown, although the background CO₂ level is higher in this test, the increase in CO₂ concentration in the effluent with voltage is about a factor of 6 times greater than the PDVF membrane. Doubling the amount of redox carrier in the ionic liquid we observed additional improvements as shown in Figure 13. Here the CO₂ increased by almost 1000 ppm. This translates to a CO₂ transport rate of 7.2×10^{-8} moles CO₂/min/cm².

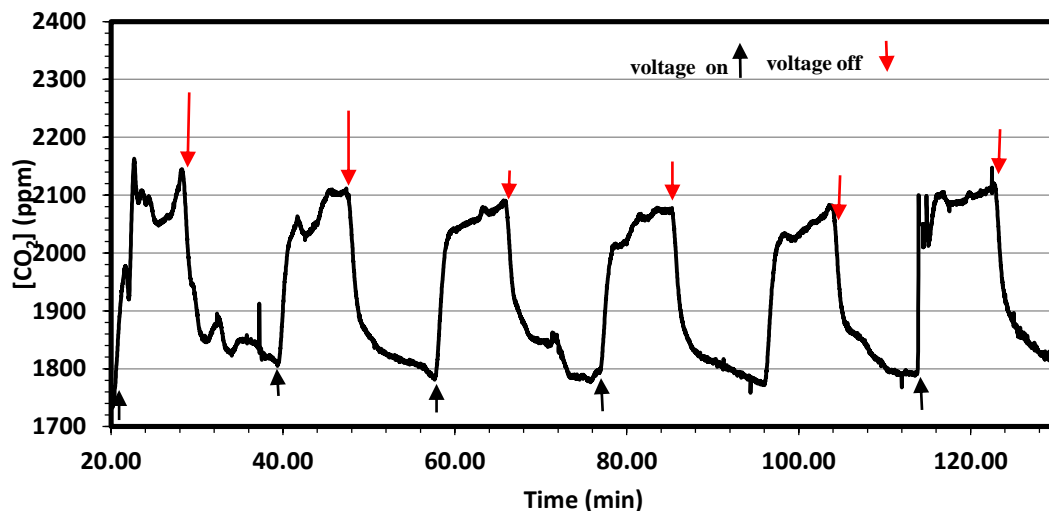


Figure 12. CO₂ concentration in anode effluent of planar cell as voltage is turned on and off. Applied voltage is 2.0 V. Membrane is Nafion 112 membrane imbibed with Formulation B with baseline redox carrier concentration. Feed gas is 100% CO₂

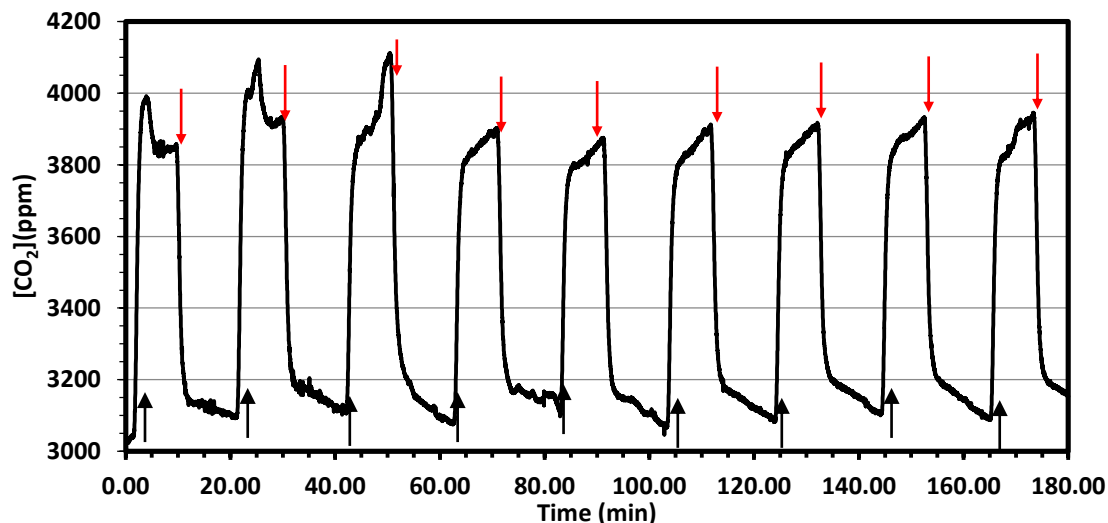


Figure 13. CO₂ concentration in anode effluent of planar cell as voltage is turned on and off. Applied voltage is 2.0V. Nafion 112 membrane imbibed with Formulation B with increased redox carrier concentration. Feed gas is 100% CO₂

Increasing the redox carrier concentration further did not result in additional performance improvements for the Nafion 112 membrane however. Figure 13 remains the best performance we have observed for CO₂ transport in a planar cell. This formulation was down-selected to transition to a tubular platform.

VI. Multi-tubular Reactor

In transitioning to the tubular architecture, we decided to build a multi-tube array, rather than a single tube reactor to evaluate both performance and multi-cell integration issues at the same time. We prepared 5 tubular MEAs for incorporation into a multi-tubular CO₂ separation reactor. In this work we used fabrication protocols to imbibe tubular Nafion membranes (Permapure TT-110) with the ionic liquid/redox carrier, braided interior anode and exterior cathode current collectors, 3-D printed manifolds/reactor components and used epoxy encapsulation to produce the CO₂ separation reactor.

The interior anode current collector for the tubular electrochemical cells is composed of braided platinum clad copper wire over a mandrel support. An internal support for the braided current collector is used to minimize contact resistance between the interior Nafion membrane surface and the anode current collector. The Nafion membrane is typically expanded in methanol and shrink fit over the mandrel/current collector before the membrane is imbibed with the ionic liquid/redox carrier. Figure 14 is a photograph of an imbibed tubular Nafion membrane fitted over the mandrel current collector.



Figure 14. Imbibed Tubular Nafion Membrane

After imbibing was completed, the exterior cathode current collector was braided over the Nafion membrane. The current cathode current collector design consists of 8 strands of carbon fiber braided over the Nafion membrane as shown in Figure 15.

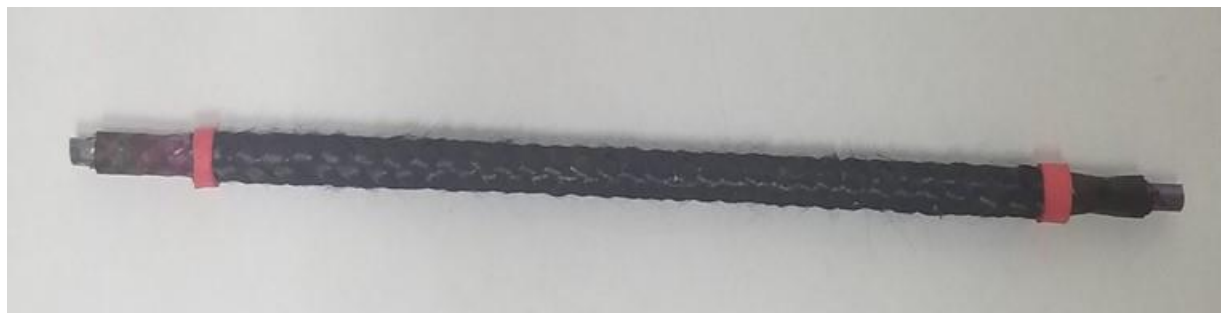


Figure 15. Electrochemical Cell

The internal mandrels of the five braided cells were then attached to a 3D printed manifold made of a flexible plastic as shown in Figure 16. Each mandrel was press fit into holes printed in the manifold component. Creating a manifold on each end of the 5 tube array allows for an inlet carrier gas to flow into one manifold and down each of the 5 cells where it is then collected and exits via a single outlet port. Figure 17 shows a manifold on each end of the 5 tube array. Electrical connections to each exterior cathode braid are done at each end of the tubes to minimize voltage loss down the length. The cells are connected electrically in parallel on the anode and cathode for each cell to operate at the same applied potential.

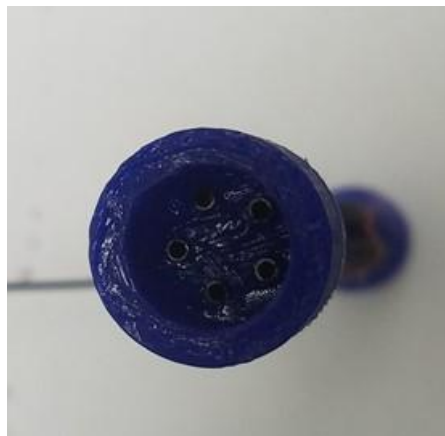
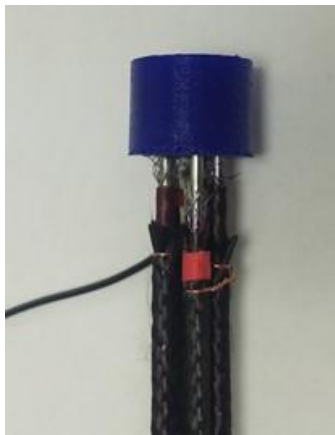


Figure 16. Interior Anode Manifold



Figure 17. 5-tube Array

The manifold was then placed inside a polycarbonate housing creating an exterior cathode annulus as shown in Figure 18. A 3-D printed cap was placed on the ends of each manifold creating a sealed manifold for the interior anode on each end of the 5 tube array. The cap has a single hole in which a 1/4" tube (0.635 cm) is inserted to enable inlet and outlet anode gas flow via a single tube out each end of the reactor. Inlet and outlet ports for CO₂ to the exterior cathode annulus are drilled and tapped in the side walls of the polycarbonate housing. Each end of the reactor is encapsulated inside a 3D printed mold using a high tech epoxy (Emerson and Cummings). The epoxy encapsulation technique fills the mold and the polycarbonate housing with epoxy, sealing the anode and cathode compartments from one another allowing for pressurization to occur without gas mixing. The epoxy seals the inlet and outlet tubes as well as the manifold and manifold cap. After the epoxy encapsulation steps the 5-tube CO₂ reactor is complete as shown in Figure 18. This reactor is approximately 18 cm long with a diameter of 2 inches. Figure 18 also shows the flow schematic of the reactor with a single inlet and outlet port for both the anode and cathode compartment. Electrical connections to each of the tubes is done via a single electrical wire. We have very recently begun testing the array and work is ongoing to evaluate performance metrics. Those data will be used to further refine the design of a scaled-up reactor to meet NASA's ISRU needs.

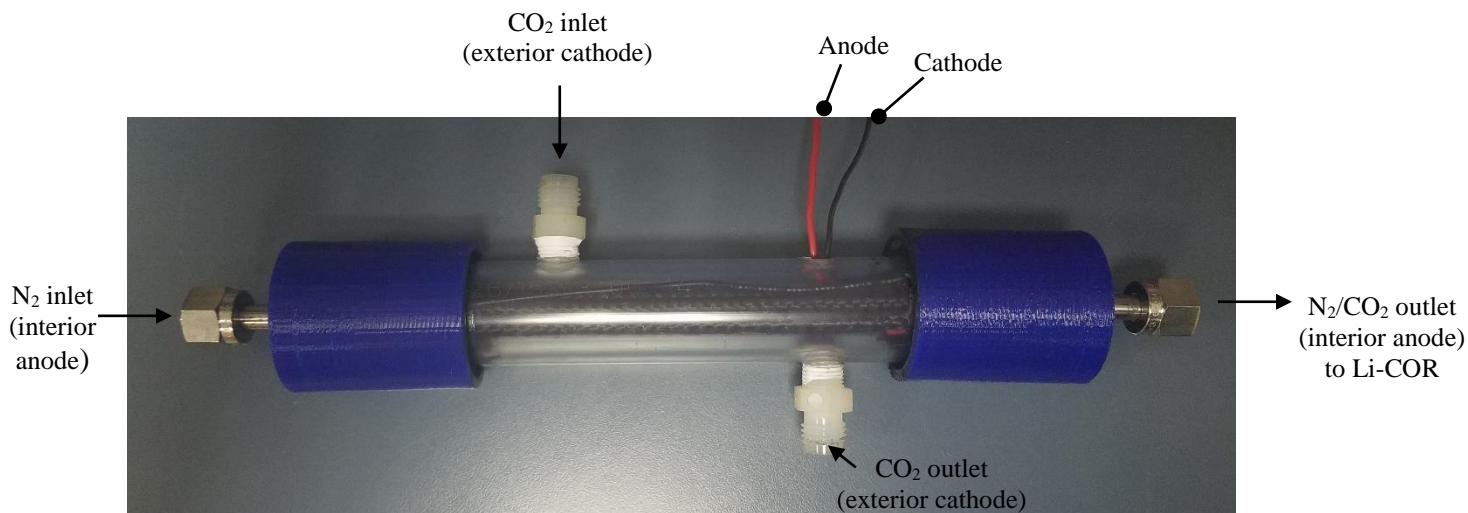


Figure 18. Multi-Tubular Reactor

VII. Conclusions

In this paper, we present the status of our work to develop a membrane-based approach to transport and pressurize carbon dioxide. We have evaluated Nafion-based and PVDF-based membranes incorporating different redox carriers. Both types demonstrated successful transport; however, we have observed significant differences in stability and performance with component and membrane choice. Current planar data indicates that a Nafion 112 membrane imbibed with IL-redox carrier formulation B has advantages in improving the CO₂ transport rate. The transport rate for this membrane was about 3x's higher than what we have previously reported.⁸ While this is a significant improvement, additional increases in transport need to be achieved for this technology to be scaled-up to achieve a CO₂ feed rate of 88 g/hr, meeting some ISRU applications. Going forward, further optimization is required in increasing the redox carrier concentration and decreasing the membrane thickness, both of which has been shown to improve performance.

Ultimately, we envision a tubular reactor design for the CO₂ separator to support either ISRU or air revitalization applications. In this study, we have successfully transitioned the planar formulations to a tubular architecture. A multi-tubular array has been built and is currently being tested. These tests will be used to update earlier pressurization data as well as to identify further developmental avenues for further improvements to this technology.

Acknowledgments

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