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Life Support Catalyst Regeneration Using Ionic Liquids and In Situ Resources

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Oxygen recovery from metabolic carbon dioxide is an enabling capability for long-duration manned space flight. Complete recovery of oxygen (100%) involves the production of solid carbon. Catalytic approaches for this purpose, such as Bosch technology, have been limited in trade analyses due in part to the mass penalty for high catalyst resupply caused by carbon fouling of the iron or nickel catalyst. In an effort to mitigate this challenge, several technology approaches have been proposed. These approaches have included methods to prolong the life of the catalysts by increasing the total carbon mass loading per mass catalyst, methods for simplified catalyst introduction and removal to limit the resupply container mass, methods of using in situ resources, and methods to regenerate catalyst material. Research and development into these methods is ongoing, but only use of in situ resources and/or complete regeneration of catalyst material has the potential to entirely eliminate the need for resupply. The use of ionic liquids provides an opportunity to combine these methods in a technology approach designed to eliminate the need for resupply of oxygen recovery catalyst. Here we describe the results of an initial feasibility study using ionic liquids and in situ resources for life support catalyst regeneration, we discuss the key challenges with the approach, and we propose future efforts to advance the technology.

Nomenclature

AR = Atmosphere Revitalization
CH_4 = Methane
CO = Carbon Monoxide
CO_2 = Carbon Dioxide
COR-CaTS = CO_2 Reduction Catalyst Test Stand
H_2 = Hydrogen
H_2O = Water
IL = Ionic Liquid
ISS = International Space Station
O_2 = Oxygen
RCFR = Regenerable Carbon Formation Reactor
RWGS = Reverse Water-Gas Shift
SmLPM = Standard milliLiters per Minute

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I. Introduction and Background

THE mid-2030's are currently targeted by NASA for the beginning of manned missions to the surface of Mars. These missions will invariably require highly reliable closed-loop life support systems to minimize material resupply and the corresponding launch and logistics costs. Heritage life support developed for the *International Space Station* (ISS) involves partially closed-loop architectures for both Water Recycling and Atmosphere Revitalization (AR). The AR system for ISS provides critical functions including oxygen generation, trace contaminant control, humidity and temperature control, and carbon dioxide (CO_2) removal. A Sabatier reactor system provides additional functionality designed to recover oxygen from metabolically-produced CO_2. The Sabatier reaction, as shown in Eq. 1, reacts CO_2 with hydrogen (H_2) to produce methane (CH_4) and water vapor. The water vapor is condensed, separated, cleaned by the Water Purification Assembly, and fed back to the Oxygen Generation Assembly to produce O_2 for the crew as shown in Eq. 2.

Sabatier Reaction $$CO_2 + 4H_2 \leftrightarrow 2H_2O + CH_4 \tag{1}$$
Water Electrolysis $$2H_2O \rightarrow 2H_2 + O_2 \tag{2}$$

The CH_4 product and uncondensed water vapor are vented overboard. When fully operational, the AR system is theoretically capable of ~54% recovery of O_2 from CO_2. This recovery is limited by the loss of reactant H_2 in the form of vented methane. A fully closed-loop AR life-support architecture (100% O_2 recovery) will require technology capable of converting CO_2 to elemental carbon and O_2, as shown in Eq. 3.

CO_2 Reduction $$CO_2 \leftrightarrow O_2 + C(s) \tag{3}$$

While this reduction can be completed via various pathways, the most common pathway explored for space flight has been via a combination of the Bosch process and water electrolysis (Eq. 2). The Bosch process, shown in Eq. 7, converts CO_2 into solid carbon and water through three reactions: the Reverse Water-Gas Shift (RWGS) reaction (Eq. 4), CO hydrogenation (Eq. 5), and the Boudouard reaction (Eq. 6).

RWGS $$CO_2 + H_2 \leftrightarrow H_2O + CO \tag{4}$$
CO Hydrogenation $$CO + H_2 \leftrightarrow H_2O + C(s) \tag{5}$$
Boudouard $$2CO \leftrightarrow CO_2 + C(s) \tag{6}$$
Bosch Process $$CO_2 + 2H_2 \leftrightarrow 2H_2O + C(s) \tag{7}$$

The Bosch process is a catalytic process producing carbon over iron, nickel, or cobalt catalyst at 400-650°C. The production of carbon during the process results in one of two situations. Either the carbon coats and fouls the catalyst, blocking reaction sites and effectively quenching catalytic reactions, or carbon is produced until pressure drop across the reactor becomes prohibitively high. The catalyst and carbon product have historically been discarded as a waste product resulting in considerable catalyst resupply mass. For Mars surface missions, this challenge may be partially alleviated through the use an *in situ* resource: the iron available in Martian regolith.

In 2012, Karr et. al. reported the results of iron and nickel extraction from a meteorite using ionic liquids (ILs).[1] ILs are organic salts that are liquid at ambient temperatures. They have virtually no vapor pressure, have low flammability, and can be extensively chemically modified to be task-specific. Moreover they are stable under extreme conditions, such as the vacuum and cold temperatures of space, are regenerable, and work at temperatures below 200°C. A mixture of an acidic IL known as betainium TFSI and a neutral IL electrolyte known as bmpyrr TFSI was used to co-extract iron and nickel from a Campo del Cielo meteorite. The iron and nickel were then successfully electroplated onto a carbon substrate from the IL, and the IL acid was regenerated in the process, which can then be used to process additional material. For a Mars surface mission, this same capability can potentially be used to extract iron and/or nickel from Martian regolith and prepare catalytic surfaces. Martian regolith is reported to contain iron compounds and nickel from meteorite accumulations, making use of this *in situ* resource both highly favorable and feasible.

In addition to extracting catalytic material from the surface of Mars, development of a catalyst that can be regenerated rather than resupplied will further reduce the need for catalyst resupply. Ionic liquids (IL's) are uniquely suited for this approach as well. When carbon forms on a metal surface via equations 5 and 6 above, it has been shown to form in one of three ways[2]. First, carbon can be deposited directly above (with respect to the bulk material) a metal nuclei and effectively push up all previously formed carbon. This is known as "base growth" because the catalyst stays at the base of the forming carbon. Second, carbon can form directly below a metal nuclei and effectively elevate the catalytic crystal above the bulk material. This is known as “tip growth.” Finally, carbon can form in all directions from a catalytic crystal. For situations where tip growth or multi-directional growth occurs, the catalytic metal ultimately becomes dispersed throughout the carbon product. Because IL’s can selectively extract iron and/or nickel from other minerals, it is theoretically possible to use an IL to extract the catalytic material from the bulk carbon product. Once extracted, the metal catalyst could be electroplated onto a desired substrate and reused in the carbon formation process. Because the extraction of the catalyst from the bulk carbon product is not expected to be 100% efficient, make-up catalyst from the Martian regolith could be used to supplement the lost catalyst over time. This potentially eliminates the need for catalyst resupply.

In 2015, internal NASA Marshall Space Flight Center research and development funds were secured to explore the feasibility of using ILs to reduce catalyst resupply in a closed-loop ECLSS AR system. There were six goals set for the project:

1. To demonstrate catalytic activity of iron (Fe) on a copper (Cu) support, electroplated using traditional methods
2. To demonstrate extraction of Fe from Fe-Carbon (C) product using IL
3. To demonstrate plating of Fe on Cu support, plated using IL
4. To demonstrate catalytic activity of Fe on Cu support, plated using IL
5. To demonstrate catalytic activity of IL-plated and regenerated Fe over several cycles

Below we describe the results of the initial feasibility study, we discuss the concept of oeprations for a Mars surface system, we discuss the key challenges with the approach, and we propose future efforts to advance the technology.

II. Materials and Methods

Four separate methods were required for feasibility testing: IL preparation, electroplating via both a traditional method and from ILs, carbon formation, and catalyst regeneration. Materials required for each of these efforts and description of procedures for these methods are provided below.

A. Materials

All wet chemicals for surface cleaning, IL preparation, and electroplating were purchased from Sigma-Aldrich (St. Louis, MO). Duocel® Cu foam was purchased from ERG Materials and Aerospace (Emeryville,CA) and was chosen as the substrate for all plating due to its inactivity for carbon formation reactions and its high electrical conductivity. Carbon monoxide (CO) gas was purchased from Matheson Tri-Gas (Basking Ridge, NJ). Hydrogen and nitrogen gases were purchased from Sexton Welding Supply, Inc. Iron-Carbon product from carbon formation reaction was produced from previous experiments during Bosch technology development. The iron source was Grade #0000 wound steel wool from Global Materials Technologies (Buffalo Grove, IL). Carbon was deposited on the wool via the method discussed in Section D below.

B. Ionic Liquids Preparation

Several acidic ILs were tested for this work. The first tests were conducted in IL N-butyl-N-methylpyrrolidinium hydrogen sulfate ([bmpyrr][HSO_4]). The hydrogen sulfate anion is the acidic species in the IL and has a pKa ≈ 2 in aqueous solution (a 1 mol/L aqueous solution has a pH ≈ 1). This IL was prepared by the reacting [bmpyrr][Br] (commercially available) with sulfuric acid and removing the hydrogen bromide by-product. Other acidic ILs tested included 1-ethyl-3-methylimidazolium hydrogen sulfate ([emim][HSO_4]), and N-methylpyrrolidinium hydrogen sulfate ([mpyrr][HSO_4]). In addition, an aqueous solution of the acid salt ammonium hydrogen sulfate [NH_4][HSO_4] was also tested (this is technically not a room temperature IL because it has a melting point of 125-150°C, which is just above the 100°C threshold). Lastly, a traditional iron plating solution consisting only of aqueous $FeSO_4$ was also been tested.

C. Electroplating

The raw ERG copper material was delivered as a 29.2cm length x 20.3 cm width x 2.54 cm thick block. The copper matrix was cut into a 4.6 cm diameter x 1.26 cm thick puck shape, cleaned for electroplating by rinsing with 30% nitric acid, and flushed with distilled water. A vacuum oven was used to dry each puck overnight. A pure copper wire electrode was inserted through the disc to make an electrical connection. The puck was placed in the cathode side of a 2-part glass electroplating chamber, as seen in Figure 1 below. The anode of the cell contained a platinium fuel cell membrane. An anion exchange membrane was placed in the center of the chamber to keep the anode and cathode solutions separated. The chamber was clamped together and filled from the top with degassed (argon purged) solutions. The anode side was then run with a slow flow of hydrogen gas. The cathode side was purged with argon during plating to prevent oxygen from attacking the fresh iron plating on the copper. A small reference electrode consisting of a cleaned platinum wire was fitted above the copper puck to monitor the voltage in an effort to stay within the electrochemical window of operation and to prevent break-down of the aqueous IL electrolyte solution. A voltage was applied to the chamber and the current monitored. The total current provides a suitable indictor of plating rate versus voltage applied to the chamber. The glass chamber approach has the advantage of providing a view of the iron plating on the copper. The copper puck was plated for 4 hours then rotated to plate the opposite side for a total plating time of ~8 hours. A traditional DC power supply was used to provide current to the system. Once plating was completed the fluids were drained and saved for future runs. The iron plated puck is washed thoroughly with de-gassed water to remove all traces of the electrolyte solution, and was then dried and held under vacuum until ready to be used for carbon formation testing.

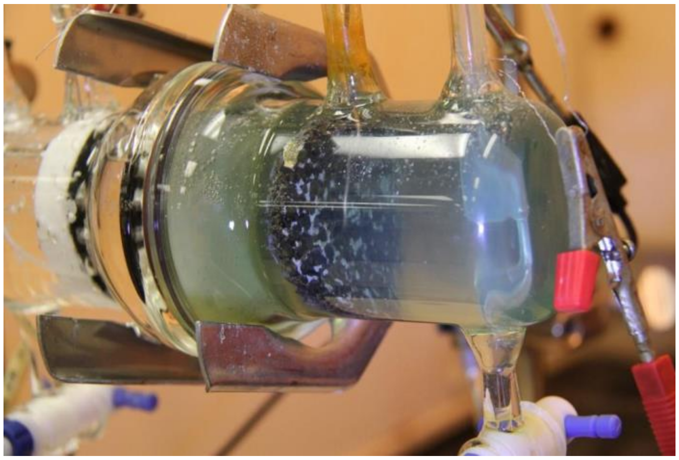

Figure 1. Copper Puck in IL Plating Cell.

D. Carbon Formation

Following electroplating, the Fe-Cu catalyst pucks were placed into a 46mm inner diameter quartz reactor tube (National Scientific, Inc, Quakertown, PA). Unifrax Fiberfrax S (Tonawanda, NY) insulation discs were cut to 46mm and installed on both inlet and outlet sides of the catalyst puck to promote gas distribution and to hold the puck in place. The reactor was installed in the CO_2 Reduction Catalyst Test Stand (COR-CaTS) at NASA's Marshall Space Flight Center. The COR-CaTS provides controlled gas feeds over a range of 10-1000 standard milliliters per minute (SmLPM) and all necessary valving, instrumentation, controls, and safety equipment to operate the test catalysts as desired. A Thermolyne F21100 tube furnace iss used to control temperature of reactor tubes.

Each reactor tube was heated to 500°C under 300 SmLPM UHP N_2. Once steady temperature was reported within the reactor, CO, H_2, and N_2 gas feeds were varied as shown in Table 1. Reactor pressure was maintained at 800 torr (absolute). At the completion of each test, the reactor was cooled to room temperature under 50 SmLPM N_2. The catalyst pucks were then removed from the reactor and stored in N_2-purged bags.

Table 1. Carbon Formation Feeds and Duration.

Test Point	H_2 Feed (SmLPM)	CO Feed (SmLPM)	N_2 Feed (SmLPM)	Duration (min)
1	0	300	20	36
2	30	300	20	24
3	100	300	20	20
4	300	300	20	32
5	600	300	20	28
6	30	300	20	Test Dependent

E. Substrate Regeneration

Iron plated copper substrates become coated with carbon deposits during the course of running the Bosch reaction, which foul the catalyst and interfere with the reaction. The substrate can be regenerated by heating the carbon coated substrate in an aqueous solution of an acidic IL for several hours. The IL acid reacts with the Fe and solubilizes it as Fe^{+2}, liberating hydrogen gas, which is collected using a metal hydride canister. This hydrogen is then used in the anode cell during the iron electroplating process. The carbon deposits do not react with the IL acid, but as the iron reacts they are removed from the copper substrate and become suspended in the solution and can be separated out by filtration and/or centrifugation. The remaining solution containing Fe^{+2} salts and the cleaned substrate are then ready for electroplating to regenerate the iron catalyst and the acidic IL.

III. Results

Five goals were targeted for this effort as described above. The results of each of these goals is provided below.

A. Carbon Formation on Traditionally Plated Puck

Previous and unrelated efforts with carbon formation catalysts had demonstrated a lack of activity on electroplated nickel, whereas other types of nickel were highly catalytic. There was a concern that electroplated Fe might similarly have different properties than other forms of Fe. Therefore, the first goal of the feasibility study was to demonstrate the catalytic activity of electroplated Fe. Additionally, once catalytic activity was demonstrated, the traditionally plated catalyst could be used as an inexpensive and easily reproducible control for further carbon formation experiments.

Microscope images of a traditionally plated puck both before and after carbon formation are shown in Figure 2. The top image shows a clean Fe surface demonstrated by the high light reflection off the surface. The bottom image shows high levels of carbon formation on the surface.

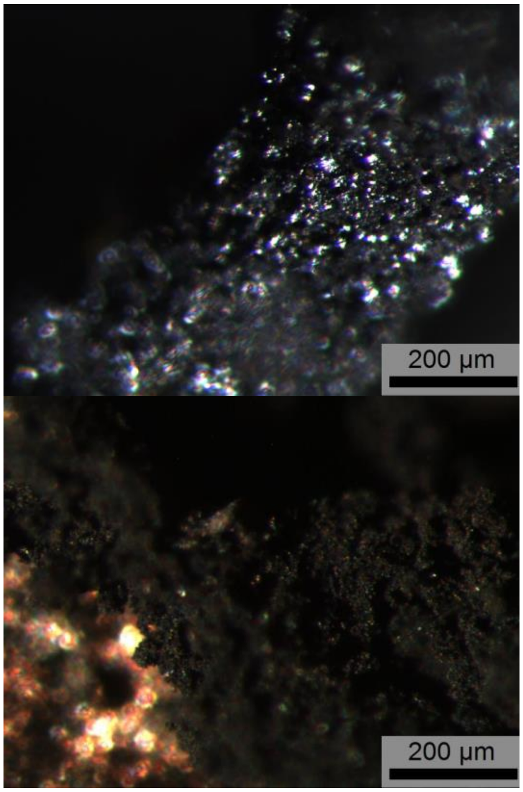

Figure 2. Traditionally-plated Fe-Cu puck before (top) and after (bottom) carbon formation reaction.

B. IL Extraction of Fe from Fe/Carbon product

Bosch catalyst development reported previously[3] demonstrated carbon formation on multiple catalysts, including iron wool. All of the samples from this testing are stored in the event they are needed for future analysis. To explore the feasibility of extracting iron from carbon-coated Fe source, one of these samples was removed from the archive. The sample showed considerable carbon formation over the “ball” for iron wool, as can be seen in Figure 4. The ball was “regenerated” per the procedure outlined in II.E using [bmpyrr][HSO_4 IL. All measurable iron was extracted from the Fe/C matrix. The remaining carbon and the Fe-full IL is shown in Figure 3.

Figure 3. Fe wool coated in carbon during previous Bosch catalyst development testing.

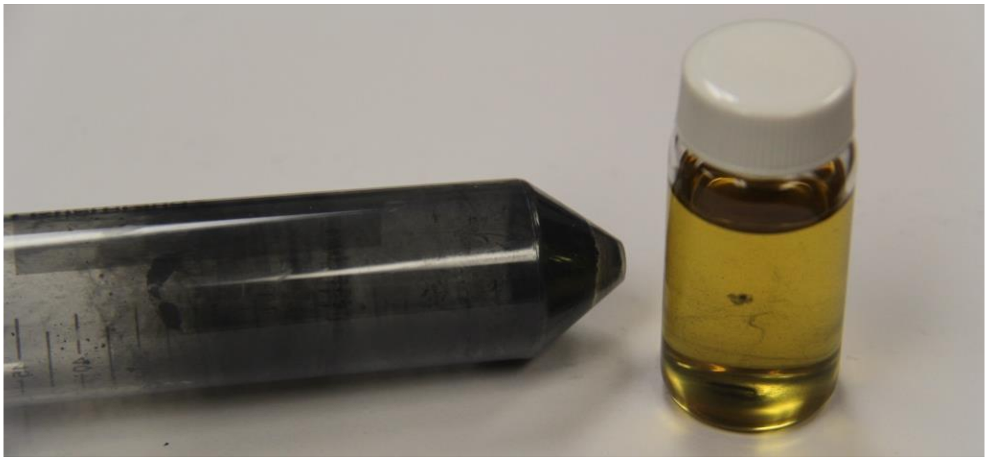

Figure 4. Carbon residue following IL extraction of Fe (left) and IL containing Fe (right).

C. Electro-plating of Fe on Cu substrate from various IL's

Three IL's,[bmpyrr], [mpyrr], and [emim] [HSO_4] were used to plate Fe onto Cu substrates. In addition, the acidic salt ammonium hydrogen sulfate, [NH_4][HSO_4], which melts at 125-150°C (just above the 100°C threshold for room temperature ILs) was also tested. Each of the plating efforts was successful, resulting in smooth and even radial coverage of Fe as determined using microscopy. Some axial variation in coverage was observed on each of the substrates. This was due to limitations of the electroplating process, particularly the use of DC current which is unable to "throw" the metal to locations further in axial distance from the electrodes. This axial variation was observed early in the project while using 2.54 cm thick pucks and was subsequently limited by cutting pucks in half for a total puck thickness of ~1.25cm. The use of a pulsed power supply is expected to result in more even coverage of the puck with the electroplated Fe.

D. Carbon Formation on Cu/Fe from IL's

Carbon formation was observed on all three IL-plated pucks tested. The highest rate of carbon formation was observed on the puck plated with bmpyr; however, the carbon was front-loaded onto the puck with respect to the gas flow. The second most carbon formation was observed on the puck plated with mpyrr, with significantly more uniform carbon formation throughout the catalyst. It is not clear if this difference in carbon coverage was due to electroplating differences, or due to differences in the IL used. Regardless, all pucks were demonstrated to be highly catalytic for the the carbon formation reactions. Figure 5 provides images of each of the pucks before and after carbon formation.

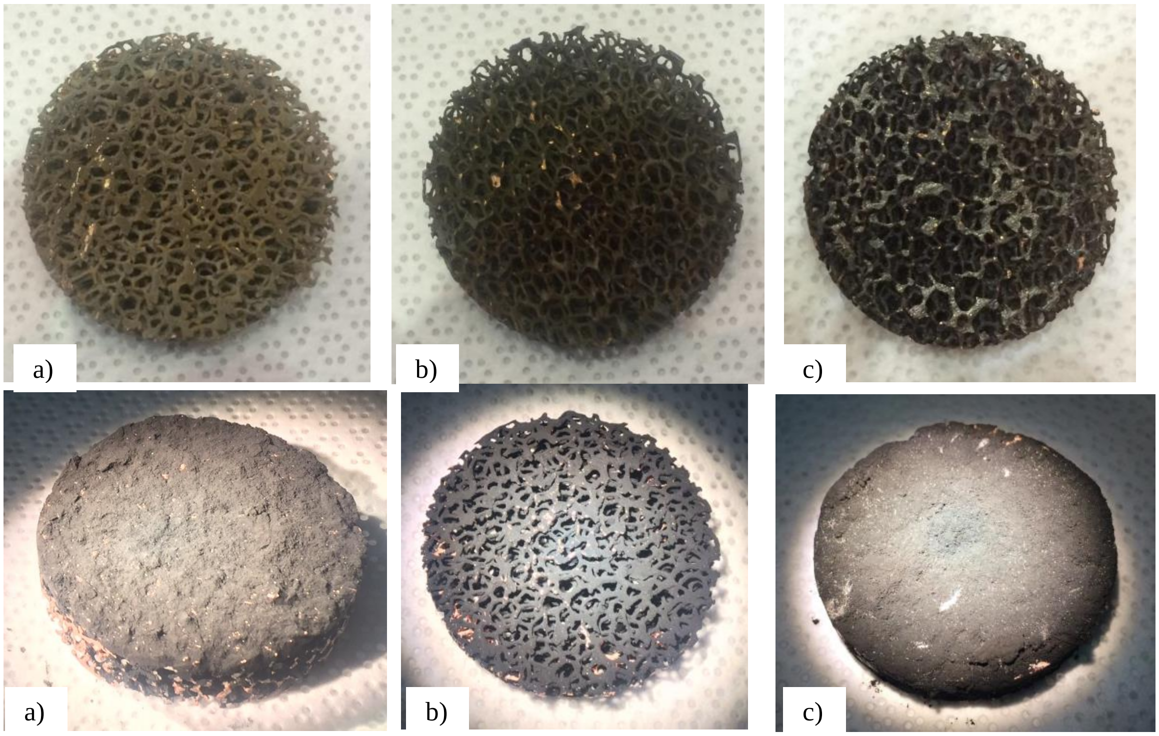

Figure 5. IL-plated pucks before (top) and after (bottom) carbon formation. *Plated using a) [bmpyrr], b) [emin], and c) [mpyrr].*

E. Multiple Cycle Demonstration

In order to have a fully regenerable system, the process of catalyst plating, carbon formation, and catalyst extraction must be feasible cyclically. To determine the feasibility of cyclic operation of this approach, a single Cu puck was plated with 0.88g of Fe solubilized using [bmpyrr][HSO$_4$]. Approximately 35mg of carbon was formed on the puck. A regeneration of the puck with the bmyprr IL was conducted resulting in a measured recovery of 0.89g of combined Fe and solid carbon. Given the error in measurements, the total recovered Fe in a single cycle was between 96.6 and 97.7%.

This initial test shows that cyclic regenation is feasible. However, the level of carbon formation prior to regeneration was relatively small. Ultimately, the process would target on the order of 1g of carbon per gram of catalyst plated between regenerations. Recovering >96% of the iron from a much denser carbon product will undoubtedly prove more challenging.

IV. Discussion

A. Discussion of feasibility based on results

Each of the goals targeted for this development effort were met. The data gathered demonstrates the feasibility of an IL-based regenerable catalyst carbon formation system for closed-loop life support. First, Fe was successfully electroplated onto Cu foam substrates using both a traditional approach and three IL's including [bmpyr], [mpyrr], and [emin] [HSO4]. Second, the electroplated Fe was shown to be catalytic from all sources, though there were variations in carbon formation rate between different pucks. It is not yet clear if these variations were due to the specific electroplating run and resulting deposition variations, the IL used for the electroplating and the potential variations in metal crystal structure, or the relative quantity of Fe plated on each surface. Third, Fe extraction using [bmpyrr] from a high carbon-content mixture was demonstrated using the Fe/C product from previous testing. Finally, cyclic operation of this approach is feasible and will ultimately greatly reduce catalyst resupply for a carbon formation reactor. However, it is clear from the data that while the catalyst resupply can be considerably decreased compared to historical Bosch technology, some catalyst resupply may be required. An added benefit of the IL approach is that *in situ* regolith on the surface of Mars may provide the necessary material for resupply, thereby eliminating the resupply from Earth.

B. System Concept for Mars Surface Mission

Carbon formation on a Mars surface mission will require operation of the electroplated catalyst at around 500°C or greater for optimum performance. Regeneration of the catalyst surface with ILs will occur at room temperature. The system will require gas reactant flow, liquid IL flow, and solid carbon production and control. Careful consideration must therefore go into the system concept to minimize complexity and to help identify the key challenge areas for development.

For the purposes of a life support architecture, an IL-based regenerable system would assume a Bosch process approach wherein all of the ISS functionality is used with the exception of the Sabatier reactor. This approach assumes that a two-stage Bosch approach is employed where a first stage reactor provides a syngas mixture to the second IL-regenerated carbon forming stage. Here we call this second stage the Regenerable Carbon Formation Reactor (RCFR).

One concept for the RCFR is shown in Figure 6. In this concept, a stack of clean copper pucks are packed into a reactor (A). Hydrogen gas is provided to the stack from a metal hydride (B). An ionic liquid containing iron is fed through the cathode while a voltage (V) is applied. Once the IL leaves stack, the Fe on the IL has been replaced with H+ and can be held in a reservoir (F) until needed for stack regeneration. The stack, now coated in Fe (C) is then subjected to the carbon formation reaction in the ECLSS AR system (D). Once the stack has been coated in carbon (E) and cooled, the IL containing H+ is fed to the stack. The IL extracts the Fe and C from the surface of the copper. Hydrogen gas is emitted from the stack and stored in a metal hydride (G). The IL containing the Fe and carbon is filtered (H) and stored in a reservoir (I) until needed to replate a clean copper stack (A). One or more pumps (J) are required to move the IL through the system.

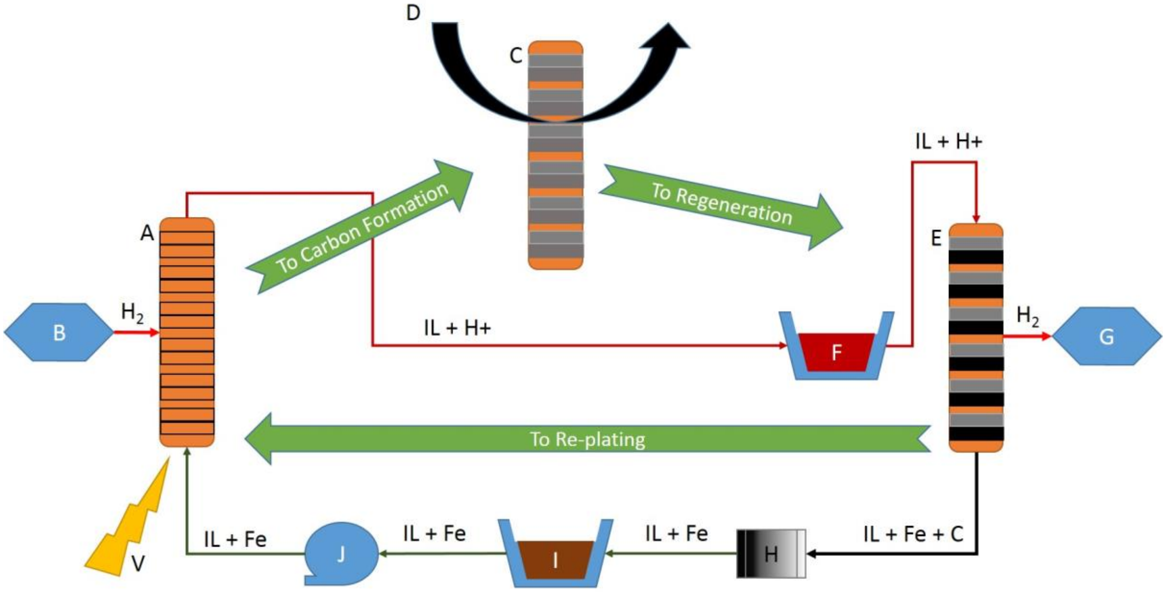

Figure 6. Concept for a Regenerable Carbon Formation Reactor based on the use of ionic liquids.

As shown, the system requires at least three stacks of substrate. At any given time, one is undergoing replating, one is in use as a carbon formation reactor, and one is in the process of regeneration. Given that the reactor must cool in order to be regenerated, a fourth stack may be necessary for continuous operation depending on the time needed for each step in the regenerable process. Additionally, metal hydrides (B) and (G) are cycled between the regeneration and re-plating processes as needed, resulting in a net zero requirement for H_2 resupply, other than inefficiency losses.

C. Key Challenges to Approach

Although the proposed IL-based regenerable CFR system feasibility has been demonstrated in bench-scale testing, there are several key challenges that will face this approach in application at mid-scale and full-scale operation. First, one of the most challenging aspects of bench-scale demonstration was achieving an even distribution of Fe plated on the Cu substrate. For a larger scale system, where Cu substrates are stacked on top of one-another, a single cathode and anode will be insufficient to evenly plate the entire substrate surface. The design of the stack will have to accommodate multiple cathodes and anodes and will likely also require a pulsed power supply to achieve adequate Fe plating. Second, the substrate stacks will have to first undergo electro-plating at room temperature, followed by carbon formation at high temperatures (≥450°C). Two options exist for implementation: cathode/anodes remain in the stack with the substrate/catalyst during carbon formation or the substrates must be installed in a plating sub-assembly containing cathodes/anodes then returned to a high temperature reactor housing. If the cathodes/anodes remain in the stack, all materials must be thermally robust at the desired carbon formation temperatures and they must not be catalytic for the carbon formation reactions. All materials in this case must also be capable of withstanding thermal cycling between regeneration steps. As an alternative, a separate plating sub-assembly may be necessary to achieve adequate performance. In this case, the reactor chamber would have to be opened and resealed numerous times. This may prove extremely challenging, especially given the need for very low leak rate systems such a life support system that will contain harmful gases such as H_2, CO, CH_4, and CO_2. Third, the carbon filtration system may prove very challenge. For a crew of four, 1.1 kg of carbon is produced each day. A five-year Mars mission will result in over 2000kg of carbon. The proposed system will require the carbon to be filtered from the IL product during regeneration. Achieving sufficient purity IL is essential to the success of replating and reuse of the IL material. Identifying a carbon removal system that is robust, highly efficient, and reusable with minimal resupply may prove extremely challenging. The fourth, and possibly the biggest challenge will be designing a stack that is sufficiently large for 4-CM carbon deposition rate and still be small enough to achieve reasonable power and cooling requirements for cyclic operation. Each time the system is regenerated, power will be required to heat the fresh reactor to temperature for carbon formation, the coolant system must be used to cool the reactor to prepare for regeneration, and power will be required to plate Fe on the clean substrate. The power required for operation can be limited by having long cycles between regenerations. However, to achieve this, the reactor must be large. A large reactor is significantly more difficult to adequately plate, thus causing a reduction in system efficiency. A careful balance will be required to optimize the regenerability and performance of the system while minimizing power and mass requirements.

V. Conclusions and Future Work

NASA's Technology Roadmaps outline various technology capabilities to help to meet the goals of NASA's Journey to Mars and designate each as either enabling or enhancing with respect to specific mission profiles. Enabling technology areas are of the most critical importance for mission success while Enhancing technologies provide desirable functions, but are not considered essential. For Mars surface missions, both CO_2 reduction (also called Oxygen Recovery) for life support[4] and oxygen production from *in situ* atmospheric CO_2 are designated as enabling capabilities[5] while extraction of regolith-bound metals and oxygen is designated as an enhancing capability[6]. The proposed approach to recovering oxygen from either metabolic CO_2 for from atmospheric CO_2 will provide a fully regenerable technique to achieve this goal by using IL's and *in situ* resources.

The data reported here demonstrates the feasibility of the proposed approach using ILs regenerability of a carbon formation reactor. However, much development is required to advance the technology to a competitive level with alternatives such as Sabatier, co-electrolysis, or Series-Bosch approaches. The next step to advancing this technology is first to determine the efficiency of cyclic regeneration for situations where high quantities of carbon are produced on the catalyst pucks. Second, determining the maximum carbon that can reasonably be plated on a substrate and still be regenerated will be critical as this will allow the system to be sized for a full 4-member crew. Third, a high efficiency filter system to remove product carbon from the IL material will be required to ensure long-term durability of the IL.

Acknowledgments

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