Methane Post-Processing and Hydrogen Separation for Spacecraft Oxygen Loop Closure

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State-of-the-art life support oxygen recovery technology on the International Space Station is based on the Sabatier reaction where only about half of the oxygen required for the crew is recovered from metabolic carbon dioxide (CO_2). The Sabatier reaction produces water as the primary product and methane as a byproduct. Oxygen recovery is constrained by both the limited availability of reactant hydrogen from water electrolysis and Sabatier methane (CH_4) being vented as a waste product resulting in a continuous loss of reactant hydrogen. Post-processing methane with the Plasma Pyrolysis Assembly (PPA) to recover this hydrogen has the potential to substantially increase oxygen recovery and thus dramatically reduce the logistical challenges associated with oxygen resupply. The PPA decomposes methane into predominantly hydrogen and acetylene. A purification system is necessary to purify hydrogen before it is recycled back to the Sabatier reactor. Testing and evaluation of acetylene removal systems and PPA system architectures are presented and discussed.

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Nomenclature

AR	=	Atmosphere Revitalization	*MRSHP*	=	Microwave Regenerative Sorbent-based Hydrogen Purifier
C_2H_2	=	Acetylene			
C_2H_4	=	Ethylene	*MTL*	=	Moderate Temperature Loop
C_2H_6	=	Ethane	*OGA*	=	Oxygen Generation Assembly
C	=	Constant	*P*	=	Pressure
CM	=	Crew Member	*PPA*	=	Plasma Pyrolysis Assembly
CDRA	=	Carbon Dioxide Removal Assembly	*R*	=	Universal Gas Constant
CH_4	=	Methane	*SA*	=	Stand Alone
CRA	=	Carbon Dioxide Reduction Assembly	*SBIR*	=	Small Business Innovation Research
H	=	Enthalpy	*SDU*	=	Sabatier Development Unit
H_2	=	Hydrogen	*SI*	=	Sustainable Innovations, Inc.
H_2O	=	Water	*SmLPM*	=	Standard millLiters Per Minute
HS	=	Hamilton Sundstrand	*T*	=	Absolute Temperature
Int	=	Integrated	*UHP*	=	Ultra High Purity
ISS	=	International Space Station	*UTAS*	=	UTC Aerospace Corporation
MSFC	=	Marshall Space Flight Center	*W*	=	Watts

I. Introduction

LIFE support is a critical function of any crewed space vehicle or habitat. Human life support systems on the *International Space Station (ISS)* include a number of atmosphere revitalization (AR) technologies to provide breathable air and a comfortable living environment to the crew. The Trace Contaminant Control System removes harmful volatile organic compounds and other trace contaminants from the circulating air. The Carbon Dioxide Removal Assembly (CDRA) removes metabolic carbon dioxide (CO_2) and returns humidified air to the cabin. Humidity is kept at comfortable levels by a number of condensing heat exchangers. The Oxygen Generation Assembly (OGA) electrolyzes water to produce oxygen for the crew and hydrogen (H_2) as a byproduct. A Sabatier reaction-based CO_2 Reduction Assembly (CRA) was launched to the ISS in 2009 and became fully operational in June 2011.

The CRA interfaces with both the OGA and CDRA. Carbon dioxide from the CDRA is compressed and stored in tanks until hydrogen is available from OGA water electrolysis. When the OGA is operational and there is CO_2 available, the CRA is activated and produces methane and water via the Sabatier reaction shown in Equation 1.

$$\text{Sabatier Reaction} \qquad\qquad CO_2 + 4H_2 \leftrightarrow CH_4 + 2H_2O \qquad\qquad \Delta H°_{rxn} = \text{-165 kJ/mol} \quad (1)$$
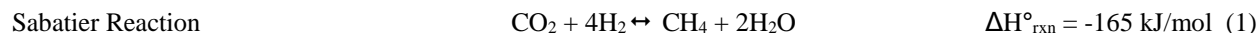
The water product is condensed out of the product stream, separated, and purified in the Water Processing Assembly before being recycled back to the OGA to be used to produce O_2 for the crew. Methane, saturated with water vapor at a dewpoint similar to the temperature of the ISS moderate temperature cooling loop that is used to cool the condensing heat exchanger, is vented to space as a waste product. The loss of H_2 in the form of vented CH_4 and uncondensed water vapor in the CH_4 stream limits the oxygen recovery to approximately 50% from metabolic CO_2.

Without the CRA, each Crew Member (CM) requires ~0.891 kg H_2O/day to be supplied from Earth to produce breathable oxygen via water electrolysis. The CRA can theoretically reduce this figure to ~0.459 kg/CM-day but that still equates to a total water resupply requirement of ~670 kg H_2O/year for a crew of four, just for breathable oxygen. To make long duration missions beyond Low Earth Orbit logistically feasible, greater oxygen recovery from metabolic CO_2 is needed. NASA is currently targeting technologies that achieve 75-90% O_2 recovery from metabolic CO_2[1].

One approach to achieve these higher recovery rates builds upon the ISS AR architecture and includes adding a methane post-processor to recover H_2 from CRA methane. NASA has been developing the Plasma Pyrolysis Assembly (PPA) to fill the role of a methane post-processor.[2,3,4,5,6,7,8] The PPA uses a magnetron to generate an H_2/CH_4 plasma targeting Sabatier CH_4 conversion to hydrogen and acetylene (C_2H_2) as shown in Eq. 2. Secondary reactions with CH_4, as shown in Equations 3-5, and reactions with residual water vapor as shown in Eqs 6-7, also occur in the PPA resulting in an effluent mixture containing H_2, unreacted CH_4, product C_2H_2, and trace quantities of H_2O, carbon monoxide (CO), ethylene (C_2H_4), ethane (C_2H_6), and solid carbon (C).

Targeted PPA Reaction	$2CH_4 \leftrightarrow 3H_2 + C_2H_2$	(2)
CH$_4$ Conversion to Ethane	$2CH_4 \leftrightarrow H_2 + C_2H_6$	(3)
CH$_4$ Conversion to Ethylene	$2CH_4 \leftrightarrow 2H_2 + C_2H_4$	(4)
CH$_4$ Conversion to Solid C	$CH_4 \leftrightarrow 2H_2 + C(s)$	(5)
CO Production	$C(s) + H_2O \leftrightarrow CO + H_2$	(6)
CO Production	$CH_4 + H_2O \leftrightarrow CO + 3H_2$	(7)

When H_2 recovered by the PPA is recycled back to the CRA, and the CRA is operated at a H_2:CO_2 ratio of 4.25, a theoretical O_2 recovery of >86% may be realized (assuming a respirator quotient of 0.92) from metabolic CO_2. This further reduces the water resupply requirement to ~0.18 kg/CM-day. However, before H_2 can be recycled to the CRA, C_2H_2 and other hydrocarbons must be removed from the gas stream to prevent fowling of the CRA catalyst. In 2015, four sub-scale hydrogen separation systems were delivered to NASA for evaluation. These included two electrolytic single-cell hydrogen purification cell stacks developed by Sustainable Innovations, LLC (SI), a sorbent-based hydrogen purification unit using microwave power for sorbent regeneration developed by Umpqua Research Company (Umpqua), and a $LaNi_{4.6}Sn_{0.4}$ metal hydride produced by Hydrogen Consultants, Inc. Evaluation of the SI cell stacks was reported in Ref. 2.

Evaluations have been conducted on the Umpqua and metal hydride hydrogen separators. The results of these evaluations, discussion of potential architecture options, and proposed future work are presented here.

II.   Hardware and Test Configuration

A. Hardware

1. Hydrogen Components, Inc. Metal Hydride

A $LaNi_{4.6}Sn_{0.4}$ metal hydride canister, shown in Figure 1, was purchased from Hydrogen Components, Inc. (Bailey, CO) with a maximum design pressure of 550 psig. The canister housing, made of stainless steel, was provided with a 500 psi relief valve to protect from over-

Figure 1. Hydrogen Components, Inc. Metal Hydride Canister.

pressurization. These canisters are typically utilized for hydrogen storage but by adding an additional inlet/outlet port (they usually only have one) this technology could be evaluated tor hydrogen separation.

2. Umpqua Microwave Regenerative Sorbent-based Hydrogen Purifier (MRSHP)

The Microwave Regenerative Sorbent-based Hydrogen Purifier (MRSHP), as seen in Figure 2, was the result of a Phase III Small Business Innovation Research (SBIR) development effort conducted by Umpqua Research Company of Myrtle Creek, Oregon. The result of the development effort was the delivery of a sub-scale hydrogen purifier that utilized molecular sieve 13X to carry out the separation between hydrogen and acetylene and employed the novel use of microwave power to heat the sorbent for regeneration. Extensive discussion of the development of the MRSHP may be found in Ref. 9.

Figure 2. Microwave Regenerative Sorbent-based Hydrogen Purifier (MRSHP).

B. Test Configurations

Both the metal hydride canister and the MRSHP were integrated with the 2nd Generation PPA. The PPA was operated at a 1-Crew Member (CM) rate of CH_4 production and an H_2:CH_4 ratio of 4:1. The reactor was maintained at ~52 torr with an applied microwave power of 550W. This resulted in a PPA effluent containing H_2, C_2H_2, unreacted CH_4, ethylene, and ethane. Operation in this manner was notably devoid of CO and water vapor. It should be noted that the metal hydride was tested only in this configuration. Adsorption cycles conducted with the MRSHP in this configuration are hereto described as "Dry" adsorption cycles. The gas leaving the separator was analyzed using a Hiden mass spectrometer.

The MRSHP was also operated with the PPA being fed CH_4 from a Sabatier Development Unit (SDU) designed by Precision Combustion, Inc. The SDU was operated such that 350 SmLPM CH_4 was produced with minimal unreacted CO_2. Water vapor content was maintained at a dewpoint of ~31°C. Additional hydrogen was fed to the PPA to maintain the overall 4:1 ratio of H_2:CH_4, and the PPA reactor was maintained at ~52 torr with an applied microwave power of 550W. Because the PPA was fed CH_4 containing water vapor, the resulting effluent contained all previously indicated components. Adsorption cycles conducted in this configuration are hereto described as "Wet" adsorption cycles. Gas leaving the separator was analyzed using a Hiden residual gas analyzer (RGA) mass spectrometer.

C. Methods

In the MRSHP, targeted sorbates for the microwave waveguide-based 13X sorbent media are the acetylene and ethylene byproducts present in the predominately hydrogen product stream exiting the PPA. Testing of the MRSHP involved adsorption runs (either "Wet" or "Dry") followed by a desorption run. Adsorption runs were performed with a constant feed from the PPA effluent equivalent to a 1-CM product rate while maintaining an MRSHP inlet bed pressure of ~720 torr. Each adsorption was allowed to continue until partial breakthrough of acetylene was observed.

Desorption of the 13X sorbent bed was accomplished using a microwave powered thermal desorption performed under vacuum. Desorption cycles were performed at a variety of microwave power levels and durations, as shown in Table 1. The goal for desorption

Table 1. Desorption conditions following each adsorption cycle.

Microwave Desorption #	Microwave Power Level (Watts)	Duration (Hours:Minutes)
1-Post Dry Adsorption	110	24:42
2-Post Dry Adsorption	135	16:2
3-Post Dry Adsorption	Varied	Long
1-Post Wet Adsorption	130	15:45
2-Post Wet Adsorption	130	15:00
3-Post Wet Adsorption	130	06:28
4-Post Wet Adsorption	130	06:45
5-Post Wet Adsorption	130	06:45

following the Dry runs and Wet runs 1 and 2, was to achieve complete desorption and subsequent recovery of the beds. The goal for desorption following Wet runs 3-5 was to examine the effect of cycle time on bed capacity for operation where the adsorption cycle was required to be equivalent in time to the desorption cycle.

III. Results and Discussion

The metal hydride hydrogen separator and MRSHP were tested at NASA MSFC in 2016 and early 2017. Both technologies were evaluated integrated with the PPA. In addition, the MRSHP was evaluated with the PPA supplied methane from the SDU. The results of this testing and a discussion of its relevance are provided below.

A. Metal Hydride Performance

The metal hydride showed no substantial capability to separate hydrogen from the PPA effluent at the test conditions. PPA effluent was supplied to the metal hydride at approximately ambient pressure. The composition of the flow stream leaving the metal hydride canister matched the inlet conditions to the limits of detection. To make a viable hydrogen separator the metal hydride would have had to capture all, or at least a substantial majority of the hydrogen out of the gas stream. Since the canister effectively captured none of the hydrogen it is not a viable candidate for the separation architecture.

For hydrogen storage applications these canisters are typically "charged" at 200 psig. The testing suggests that at ambient pressure the hydrogen capture occurs too slowly to be of value as a separator (slow kinetics). It is possible
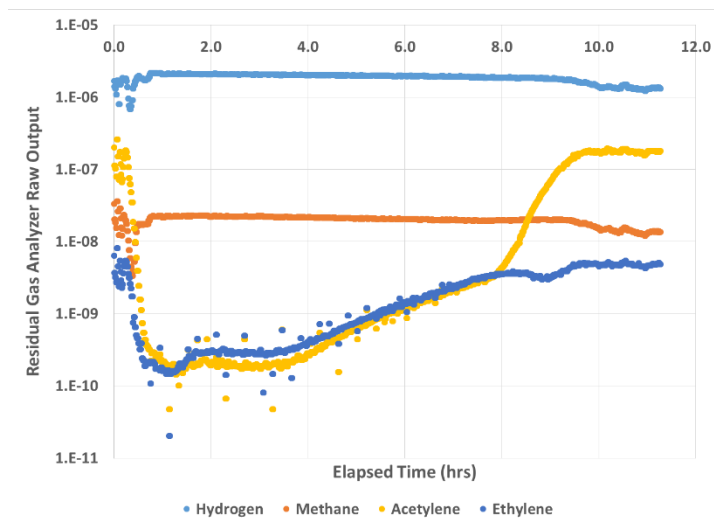
that there is a metal hydride other than $LaNi_{4.6}Sn_{0.4}$ that has a high affinity for hydrogen at the necessary conditions and is compatible with acetylene, but no further testing is planned at this time.

B. MRSHP Performance

The MRSHP was evaluated using Dry and Wet PPA effluent gas streams. The performance of the technology with these streams is reported below. Additionally, a discussion of the observed changed in acetylene capacity, characteristic adsorption curves, and overall performance is discussed.

1. MRSHP Dry Performance Results

A representative gas concentration curve for first three Dry adsorption cycles is shown in Figure 3. Elapsed time in hours is shown on the x-axis. The y-axis provides the residual gas analyzer raw output and can be interpreted as relative quantities of each gas component. Data from each run demonstrated relatively uniform and repeatable acetylene adsorptions up to four hours of adsorption. The third adsorption cycle had the distinction that it was continued for a total of just over 11 hours, vs. under six hours for the first two cycles, in order to capture complete break-through of the acetylene contaminant. Complete break-through of a particular contaminant is distinguished by a return to the influent gas concentration as measured in the sorbent bed effluent. Elevation above this

Figure 3. Third MRSHP dry run. *Data shown is representative of all three dry runs up to 4 hours of adsorption. Run 3 is the only run to exceed 4 hours of adsorption.*

level would represent a displacement of the contaminant from the bed that is generally induced by a more strongly adsorbed competitor. Partial break-through of C_2H_2 was observed at just under 3 hours of adsorption (hour 4 on the graph) and accelerated at just under 7 hours from baseline (hour 8 on the graph) before a full return to influent levels.

Desorption between each run was varied. However, gas analysis of desorption products, combined with very low outlet pressure at the end of each desorption suggested that complete desorption was achieved. The similar performance following each desorption also suggests that complete desorption was achieved.

2. MRSHP Wet Performance Results

All six wet adsorption cycles are shown in Figure 4. The x-axis provides the elapsed time for the run while the y-axis shows the normalized RGA response. This value was calculated by dividing the RGA response at a given time with the RGA response for that component prior to adsorption (100% of gas). The data from each run was shifted such that at 0.75 minutes into the run, the normalized RGA response for acetylene was 0.4. This was to provide a clearer comparison of the breakthrough curves. Note that due to the shift in data, the RGA response for Run 1, indicating complete adsorption of acetylene, was observed at 1 hour into the run. All other runs are compared based on this alignment of curves.
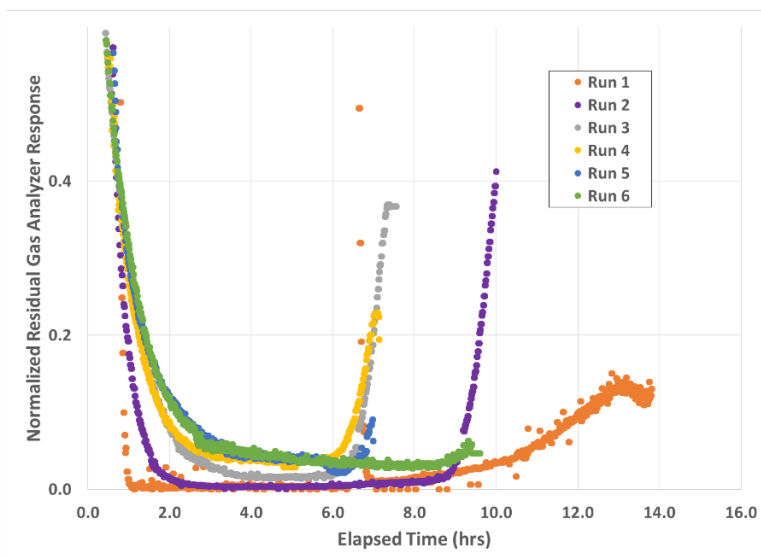

Figure 4. Combined adsorption breakthrough curves for Wet adsorption runs.

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The first Wet adsorption cycle shows a rapid return to low baseline concentration for acetylene. This is potentially due to the long duration, higher power and lower pressure desorption performed prior to this adsorption cycle as indicated in Table 1. Adsorption occurred over a 7.5-hour period until the beginning of acetylene breakthrough with subsequent gradual rise to accelerated breakthrough beginning just after 9 hours of adsorption. This clearly indicates a greatly restored capacity following the "super" desorption cycle preceding this adsorption cycle. The second Wet adsorption cycle showed a more gradual decrease to the baseline acetylene level, yet once to baseline, the time to initial breakthrough is around 5 hours, which is similar to that observed for the Dry adsorptions. However, continued breakthrough occurred much more rapidly than that observed in Run 1. This suggests a very sharp acetylene front within the bed. Subsequent runs showed a decreased capacity observed by initial breakthrough occurring approximately 5 hours after baseline (shown at hour 6 on the graph). However, a return to the longer breakthrough time was observed for the final run. The reason for the change in breakthrough time is not clear as conditions were not significantly different between runs. Relatively shorter desorption times explain the shorter breakthrough time for runs 3-5, but do not explain the higher capacity observed for run 6.

A second observation is the relative decrease in the time to reach baseline acetylene. Each subsequent run demonstrated a more gradual curve from the 0.75 hour mark. Although not confirmed, it is possible that the presence of CO_2 and water may be affecting this. All the Wet adsorption cycles show an elevated level of water vapor and CO in both the influent and effluent streams. This is to be expected due to the SDU product feed stream to the PPA containing residual CO_2 as well as H_2O at SDU effluent condenser saturation levels. Water vapor in particular is expected to have the greatest impact on 13X bed capacity due to its strongly held adsorptive properties on this molecular sieve, out-competing all other contaminants.

A third observation from the wet data was the change in adsorption performance of ethylene compared to that of acetylene. As seen in the Dry runs (Figure 3), ethylene and acetylene adsorption tracked with one another. However, in the Wet runs, there was a significant difference in adsorption and breakthrough between the two gases as seen in Figure 5. Acetylene clearly had a greater capacity than ethylene suggesting a higher affinity of 13X for the acetylene than the ethylene. For use in an oxygen recovery architecture, breakthrough of ethylene is just as important as acetylene since neither is desired to be recycled to the PPA.
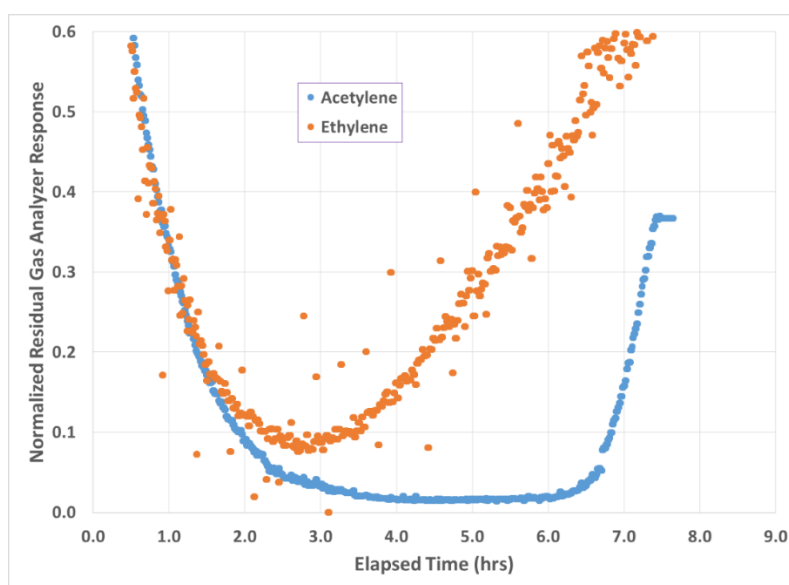

Figure 5. Wet adsorption run #4 contrasting ethylene and acetylene breakthrough curves.

3. MRSHP Bed Disassembly

Following the final Wet adsorption cycle a subsequent desorption was performed and the waveguide-based sorbent bed was removed from the MPRSHP prototype and the bed disassembled. The sorbent bed exit end piece was taken off to expose the 13X media which was then gradually removed. Darkened media was observed predominantly down the center of the bed as seen in the disassembly photos shown in Figure 6. This has been hypothesized to be due to the presence of carbon that formed from thermally decomposed acetylene. It is unclear as to when this carbon formed. It may be that a significant amount of carbon formed during the "super" desorption cycles that were executed between the last Dry adsorption and the first Wet adsorption, however there is no sign of this in the first Wet adsorption cycle which, as noted earlier, shows a fast drop to a very low baseline and greatly extended adsorption capacity. A closer review of all the data for the various desorption cycles may reveal elevated temperatures corresponding to elevated microwave power levels or perhaps certain exceptionally lengthy adsorption cycles. It is also possible that the carbon simply formed gradually over the course of the 10 cycles or alternatively during some preliminary adsorption/desorption experiments. In any case operation at lower power levels for the initial part of the desorption cycle could well be all that is required to mitigate this decomposition. The presence of carbon coating the 13X sorbent

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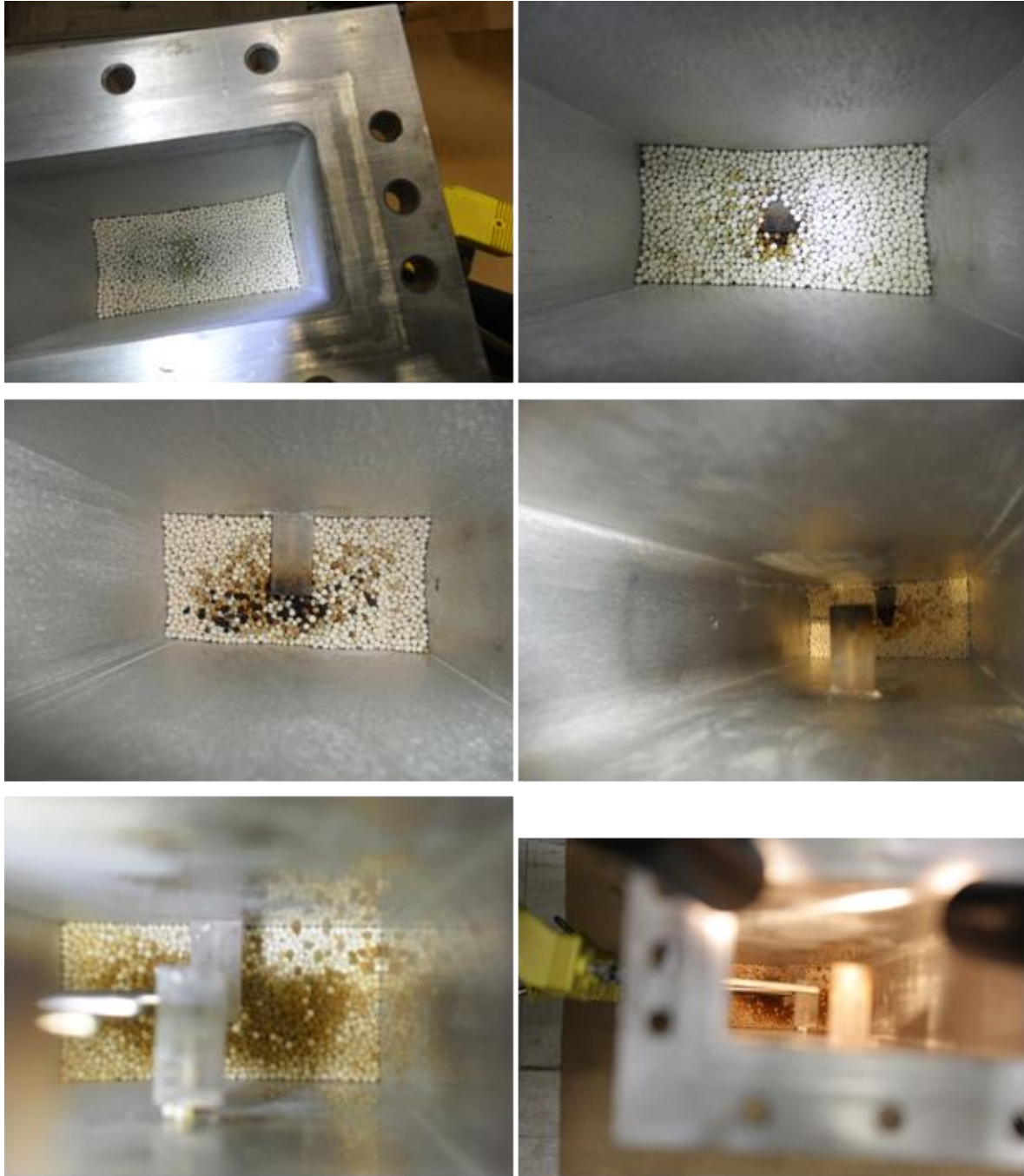
Figure 6. 13X sorbent media gradually removed from waveguide bed starting at the adsorption exit flow end (Top Left Photo) and progressing downwards (Left to Right Photos from Top Row to Bottom Row) to exposed 3rd antenna quartz sleeve (Bottom Right Photo).

media, predominantly down the center of the bed, may potentially explain the elevated baseline, with yet unchanged adsorption duration observed in the final five Wet adsorption cycles. If the carbon coating on the 13X media serves to block a significant portion of the adsorption surface area, then a reduced capacity for adsorption will clearly result. This reduced capacity media surrounded by restored capacity media existing down the length of the bed will effectively act as a bypass flow for the contaminant loaded hydrogen stream. As such, as the bypass flow valve is first turned to the sorbent bed as slow gradual drop to an elevated baseline would be expected since a portion of the influent stream survives to the end of the bed and mixes with the purified portion of the stream (assuming unmixed

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laminar flow conditions) and eventually results in an elevated baseline level. The time to breakthrough, however, can be expected to remain essentially unchanged since the fully restored perimeter media capacity remains unchanged and still sees the same influent contaminant level throughout adsorption and as such will require the same amount of time to reach capacity.

Despite a variety of anomalous operating fluctuations and the formation of carbon within the 13X media this set of sequential adsorption/desorption experiments has successfully demonstrated the feasibility of using microwave induced thermal-vacuum techniques to desorb contaminants from 13X sorbent media that had been earlier used to purify a hydrogen stream emanating from a PPA. The formation of carbon should be easily mitigated by operation at lower microwave power levels for the first part of the desorption cycle and then ramping to even higher power levels than those used in this testing for the latter part of the cycle. Alternatively, wider diameter quartz sleeves could be used to effectively further lower (via $1/r^2$ distance drop off) the intense microwave power flux; here a 50% increase in diameter would result in an additional 56% drop in intensity at the quartz sleeve/13X media boundary. Yet another approach would be to further distribute microwave power within the media thereby achieving lower local power levels by use of additional antennae (say 6 or 8 total vs. the 4 currently employed). Each of these approaches, and combinations thereof, as well as others as of yet unidentified would necessarily need to be explored in future advanced development work in which it would be logical to not only refine the bed design and desorption cycle for optimal restored cycle to cycle capacity with minimum time required for desorption, as touched on above, but to also investigate expanding (scaling) the 13X bed and more generally the MRSHP system design to accommodate a full 4-CM capacity PPA effluent.

IV. Architectural Options

Various architectures for hydrogen separation technologies to be used in conjunction with the PPA were presented and discussed in Ref. 2. The primary PPA system architecture currently being considered by NASA for integration with the ISS life support system, and ultimately for exploration systems, is shown schematically in Figure 7.
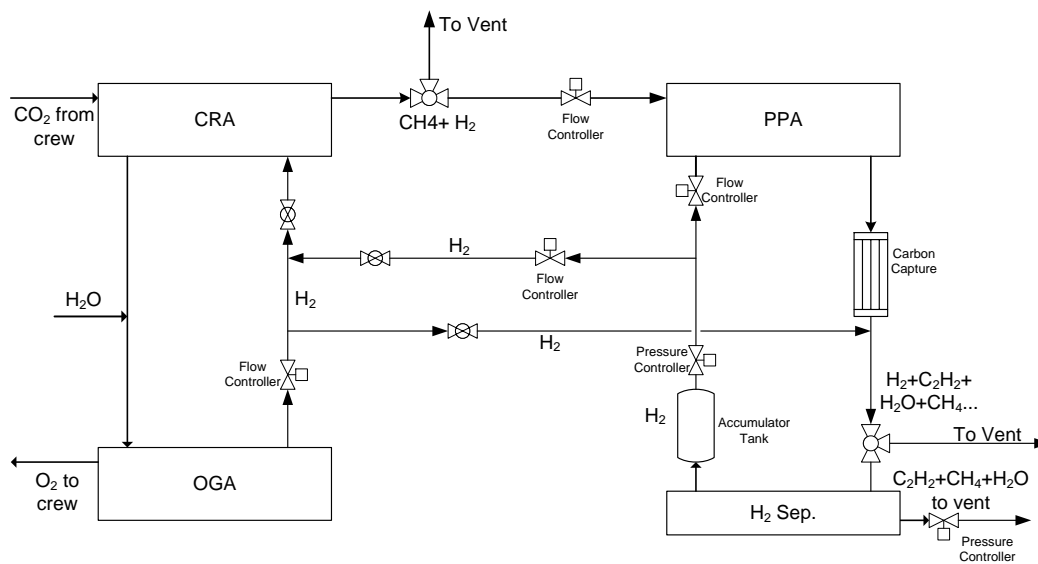

Figure 7: Proposed PPA system architecture.

This architecture reflects the use of an electrochemical cell stack to carry out the hydrogen separation step. While the MRSHP has demonstrated its potential as an effective hydrogen separator its use would add a number of complexities to the system architecture. The use of any zeolite sorbent-based system in an architecture similar to this would necessitate the use of an upstream desiccant to remove water vapor and a compressor, since the PPA reactor is expected to operate at 110 torr, to bring the feed stream up to a pressure where the sorbent can be effective and to charge an accumulator. The electrochemical cell stack has no issue with water vapor and could supply purified hydrogen at the necessary system pressure without the use of an additional compression step.

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A flight experiment on the ISS is the next logical milestone for demonstrating the PPA methane post-processing architecture but an ISS flight experiment presents a number of challenges. Included among these challenges are the necessarily tight coupling with other critical ECLSS, the additional volume of combustible hydrogen necessary for the accumulator, and the perceived risks of flying a high powered microwave induced plasma in a habitable volume.

The PPA could be flown and demonstrated by flying a supply of compressed methane and hydrogen; however, this would severely limit its operation time, raise questions about the behavior of the PPA system with the "real" methane mix coming from the Sabatier reactor, and pose its own challenges by necessitating the flight of large volumes of combustible gasses.

The accumulator volume and required system operating pressures are currently under investigation at NASA MSFC. A test is planned to start later this year to demonstrate and evaluate this architecture. Of primary interest is minimizing the accumulator volume and pressure. It is hoped that it proves to be possible to operate the system entirely at sub ambient (1 atm) pressure. This would mitigate any concerns of combustible gas leakage into the cabin environment since any leakage that occurred would push air into the system instead of pushing combustibles out.

There have been concerns expressed about crew exposure to microwave radiation. The PPA makes use of a substantial microwave power system to produce methane/hydrogen plasma. During normal processing at a 4-CM rate the PPA applies ~800W of microwave power to the plasma. However, because of the use of effectively sealed microwave system components the emitted microwave radiation from the PPA is substantially less than a cell phone during a call.

While there are a number of challenges associated with flying a PPA on the ISS they are largely manageable and the rewards of developing and demonstrating advanced ECLSS far outweigh the challenges. In addition to providing a demonstrated solution for oxygen recovery requirements for an exploration system architecture there are other benefits to a PPA flight demonstration, most notably a financial benefit. The cost of such a flight demonstration would not be insignificant but over a three year period of operation could save the ISS program over $83M (assuming PPA operation at a 4-CM rate and the current resupply cost of $68k/kg) in water resupply costs.

V.   Conclusion

Over the past two years four different hydrogen separation technologies (two Sustainable Innovations (SI) electrochemical cell stacks, the metal hydride, and the MRSHP; see Ref. 2 for discussion of the SI cell stack testing) have been evaluated at MSFC. While both the MRSHP and the SI cell stacks show the potential to meet the requirements for a hydrogen separator in a PPA system architecture, NASA's planned testing and development focuses on the use of electrochemical cells because of the simplified system architecture that they provide. A fully integrated test with the PPA, OGA, and CRA, including a hydrogen recycle loop, is planned for this year. While there are substantial challenges associated with it, a flight demonstration on the ISS is the next logical step for the PPA architecture. Not only would a flight demonstration help to substantially improve the maturity of AR ECLSS it also has the potential to save the ISS program over $83M in water resupply costs.

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