Extraction of Water from Martian Regolith Simulant via Open Reactor Concept

Andrew J. Trunek [1], Diane L. Linne [2], Julie E. Kleinhenz [3], Steven W. Bauman [4]

[1] NASA, Materials and Structures Division, 21000 Brookpark Rd, Cleveland OH, 44135; email: Andrew.J.Trunek@nasa.gov

[2] NASA, Propulsion Division, 21000 Brookpark Rd, Cleveland OH, 44135; email: Diane.L.Linne@nasa.gov

[3] NASA, Propulsion Division, 21000 Brookpark Rd, Cleveland OH, 44135; email: Julie.E.Kleinhenz@nasa.gov

[4] NASA, Materials and Structures Division, 21000 Brookpark Rd, Cleveland OH, 44135; email: Steven.W.Bauman@nasa.gov

ABSTRACT

To demonstrate proof of concept water extraction from simulated Martian regolith, an open reactor design is presented along with experimental results. The open reactor concept avoids sealing surfaces and complex moving parts. In an abrasive environment like the Martian surface, those reactor elements would be difficult to maintain and present a high probability of failure. A general lunar geotechnical simulant was modified by adding borax decahydrate ($Na_2B_4O_7 \cdot 10H_2O$) (BDH) to mimic the ~ 3 percent water content of hydrated salts in near surface soils on Mars. A rotating bucket wheel excavated the regolith from a source bin and deposited the material onto an inclined copper tray, which was fitted with heaters and a simple vibration system. The combination of vibration, tilt angle and heat was used to separate and expose as much regolith surface area as possible to liberate the water contained in the hydrated minerals, thereby increasing the efficiency of the system. The experiment was conducted in a vacuum system capable of maintaining a Martian like atmosphere. Evolved water vapor was directed to a condensing system using the ambient atmosphere as a sweep gas. The water vapor was condensed and measured. Processed simulant was captured in a collection bin and weighed in real time. The efficiency of the system was determined by comparing pre- and post-processing soil mass along with the volume of water captured.

Paper Content

I. Introduction

The concept of using local resources to reduce, supplement, and replace essential items required but not transported to an area of interest is not new. When those resources are tailored for the production of propellant and life-sustaining consumables, it is termed in-situ resource utilization (ISRU). NASA’s plans for human exploration of Mars have considered ISRU early on in mission development, Drake (2009). The Mars Design Reference Architecture 5.0 (DRA 5.0) called for the production of oxygen from the

Mars atmosphere as enabling technology, Drake (2010). However, the harvesting of water from soil for life support and fuel production was recognized as offering additional mission benefits, but considered too immature to be included in the baseline. Since that 2009 DRA report, additional robotic and orbital missions to Mars have greatly increased our knowledge about near surface water. It has been confirmed that globally, near surface regolith contains anywhere from 2-10 percent water by mass, Abbud-Madrid (2016), Mouginis-Mark (1987), Vaniman (2004). Thus, the focus on concepts for the extraction and harvesting of water from the surface of Mars, for its importance as fuel and life sustaining capabilities, has accelerated. Near surface is accepted to mean up to one meter in depth. This water is present is in the form of frost, ice, or bound to other materials like smectites (clays) and poly-hydrated sulfates (gypsum), and other hydrated minerals. Near-infrared spectroscopy mapping has confirmed the presence of H_2O and OH^- at the surface (~100 μm) from 3-13 percent as described by Audouard (2014). A recent planning study revisited the potential and challenges of water-based ISRU on Mars, Abbud-Madrid (2016), with a specific focus on harvesting the water in the near-surface materials like smectites. Smectites and clays are of interest as they tend to have a good amount of water content and fairly low decomposition temperature, Abbud-Madrid (2016). Therefore, it should be easier to exploit this resource versus other materials. This paper focuses on a unique method for water extraction from hydrated materials.

Even with the strong evidence for the presence of water, extraction of that resource is not trivial and is compounded by low temperatures and low atmospheric pressure, Leovy (2001). ISRU concepts for extraction and capture methodologies in a Martian environment need to be developed and evaluated. The Martian atmosphere, albeit thin at ~7 Torr, should be sufficient to support an open reactor design according to Linne (2016). While an open reactor might sacrifice in overall extraction efficiency, its simplicity may lead to a very robust system. An open reactor eliminates the requirement for reusable, high-temperature seals that must survive in a harsh, abrasive environment. In one embodiment, a reactor mounted on a rover would continually harvest water from the

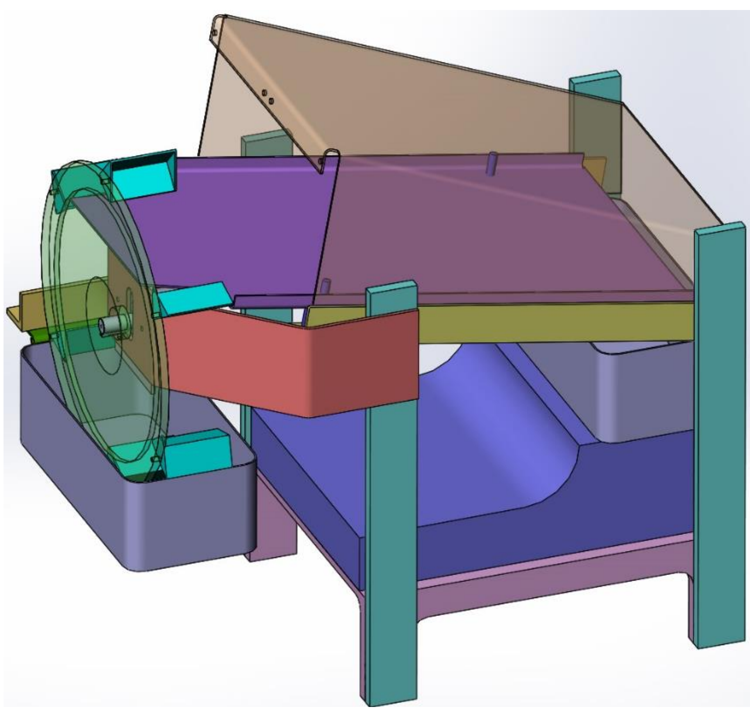

Figure 1: Working design model for open reactor concept.

Martian regolith as the rover/excavator roams the surface using the Martian atmosphere as a sweep gas. This paper discusses test results of the extraction of water from Martian regolith utilizing an open reactor design in partially simulated Martian environment, figure 1.

II. Experimental

General

The design of the test system focused on evaluation of several key concepts in a simulated Martian environment:

- Utilize the native atmosphere as a sweep gas for water vapor transport in an open reactor design
- Simulate the excavation of soil using a transverse bucket wheel
- Efficiently separate, heat, and transport soils employing a vibrating tray
- Evaluate the efficiency of a chilled condenser's ability to extract and collect the water vapor.

This ISRU concept employed an open reactor design that relied solely on the thin, low-pressure Martian atmosphere to transport (i.e., sweep gas) evolved water vapor to a chilled condensing system. A transverse bucket wheel was employed in the design to excavate soil and deposit it on a flat tray. While in actual operation the bucket wheel would be mounted on a moving excavator, to mimic this robotic approach in a static environment, simulant was loaded in a source bin that was continuously raised into the bucket wheel to provide a source of fresh simulant. Previous tests showed that a transverse bucket wheel excavator works well in low-gravity as the excavator wheels or tracks provide sufficient counter-force for the digging motion, Skonieczny (2011).

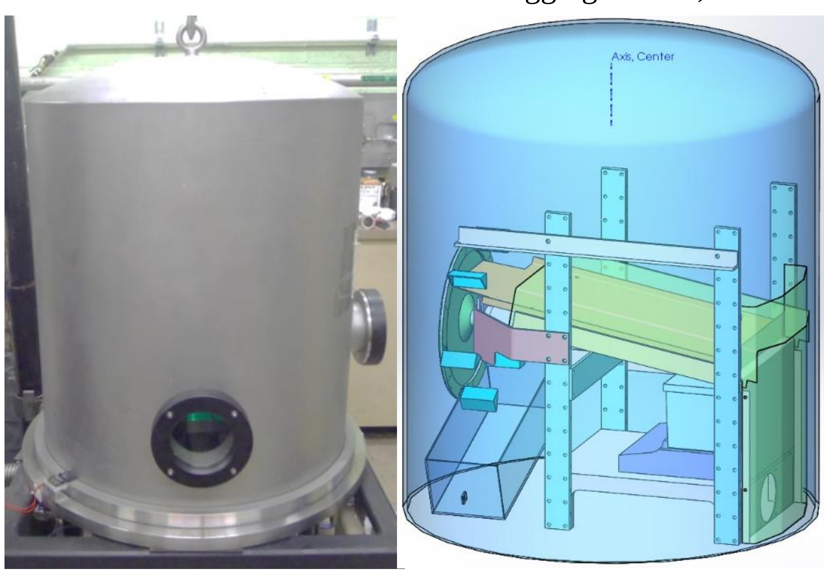

Figure 2: The MACS system with cover on and simulated image of the system. All ISRU runs were conducted in this system.

The transverse bucket wheel delivered the soil to an inclined (10°) tray where the soil was simultaneously heated, distributed, and transported to a collection bin. Water vapor baked out of the hydrated simulant was transported to an external condenser through a ducted fan configuration and collected. The Martian Atmospheric Chemistry Simulator (MACS) was used to maintain a Martian-like environment (7 Torr CO_2) for the water extraction demonstrations, figure 2.

Regolith Excavation and Depositing

The system was scaled to fit within the MACS system, which is 61 cm in diameter and 61 cm in height. A scaled bucket wheel utilized 3D-printed plastic scoops fastened to an aluminum hub, figure 3. The number of buckets, tilt angle of the buckets, and rotating speed of the wheel could all be varied to maximize initial soil distribution across the leading edge of the tilt tray. The scoop angle was set to achieve the most uniform distribution of soil onto the vibe tray during the upper crossing arc. Each scoop has a nominal capacity of 13 cm^3. The rotational speed of the bucket wheel was controlled by a variable speed motor with gear reduction (216:1) allowing for 0-12 RPM operation. Because this is a stationary system, and one of the concepts to be evaluated is the simulation of a rover excavating regolith, the soil source bin was moved instead. Tension springs moved the bin upwards into engagement with the bucket wheel, and continued this feed motion as the test progressed.

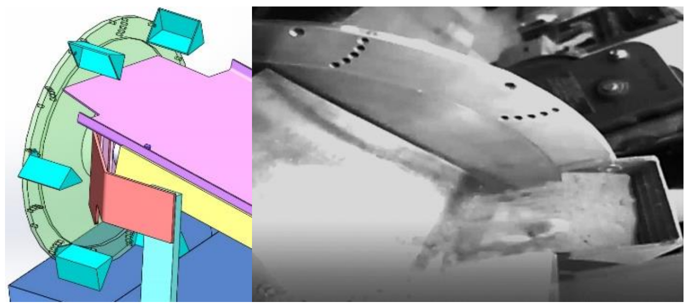

Figure 3: Bucket wheel design model and soil deposition during initial test setup.

Separation, heating and transport of regolith

The heart of the reactor is the inclined copper vibration tray (tilt tray), figure 4. Copper was chosen for its high thermal conductivity. The rectangular plate was configured such that the leading edge of the tray fit very close to the scoop wheel. This minimized loss of soil as it was dumped on the tilt tray from the bucket wheel. The tilt tray was supported by four springs that allow for vibratory motion. Vibration was generated by a single small DC motor turning an off-set weight. The combination of vibration, tilt angle, and gravity transported the soil down the length of the tilt tray to the collection bin. Rapid vibration helped distribute the soil. The depth of the soil varied, with most of the soil being less

Figure 4: Copper tilt tray with uniform soil distribution. Heaters are mounted on the backside.

than 0.5-cm thick as it travelled down the tray, although there were isolated regions where soil accumulated in little mounds and approached 1-cm in depth. The thin soil layer encouraged rapid heating of the regolith via conduction to maximize the heat transfer efficiency of the system.

Heating of the vibe tray and ultimately the soil was accomplished by two commercially available adhesive-backed electric resistance heaters attached to the underside of the tilt tray. High temperature insulation covered the heaters to minimize heat loss. The tilt tray temperature was set at 150 °C such that the soil on the tilt tray should reach the target temperature of 100 °C. After some experimentation in air, the tray angle was set at 10° to allow sufficient residence time for the soil to reach the 100 °C target temperature. At the end of the tilt tray, processed soil dropped into a collection bin where a load cell provided real time weighing of the soil.

Sweep gas and Condenser

A flow of simulated Martian atmosphere is directed over the heated soil particles to transport the liberated water vapor to the condenser. The condenser is a double walled tube; the water vapor flows through the outer tube, which is sealed to the chamber, while coolant flows through the inner wall. Condensed water collects in a drip line consisting of a graduated cylinder, figure 5. The dry gas is returned to the chamber to complete a closed loop, which is required to maintain the low Mars atmospheric pressure, figure 6. Two pressure transducers monitored the inlet and outlet pressures. The sweep flow is generated with two small fans located at the inlet and outlet of the condenser line. The first fan pulls the sweep gas down the tilt tray and pushes it into the condenser, while a second fan pulls the sweep gas through the condenser and pushes it back into the chamber. A shroud fits over the tilt tray to minimize the cross sectional area above the heated soil, increasing the efficiency of the sweep gas. As the soil drops into the collection bin, the moisture saturated sweep gas is ducted into the condenser line through the first fan. A small raised metal lip separates the soil path from the sweep gas. No filters were used to mitigate ingestion of dust into the condenser. However, the collected water was analyzed to assess filtration needs for future designs. The condenser was maintained at 0° C for all experimental runs.

Figure 5: External condenser collection tube. Scale assists with estimating volume. Graduated cylinder used for accurate volume measurements.

Hydrated Regolith Simulant

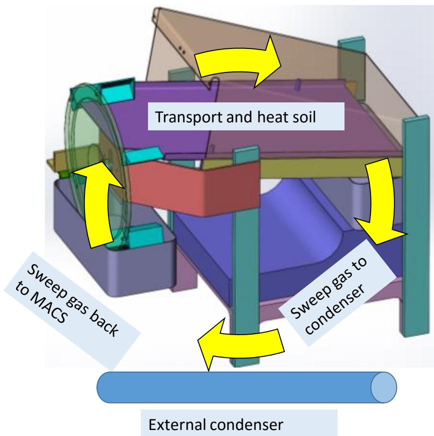

Figure 6: Circular sweep gas flow path.

Since this initial test program was a proof of concept, a low fidelity soil simulant was used. A good hydrated Mars regolith simulant for future testing is still being determined. Lunar regolith simulant GRC-3 was used for this testing since it was a readily available, low cost granular material. It has also been well characterized, He (2011), Zimmerman (2016). Borax decahydrate ($Na_2B_4O_7 \cdot 10H_2O$) (BDH) was added to the simulant to provide a hydrated mineral for water release. The heating system used in this testing could not achieve the temperatures needed to dehydrate the Mars hydrates in reference materials in Abbud-Madrid (2016). Borax was chosen because it contains a substantial amount of water and dehydrates at temperatures that can be reasonably attained in this proof of concept design.

Baseline measurements

Three baseline measurement tests were conducted. The first was to determine the maximum efficiency of the condensing system using liquid water. The second baseline measurement determined the efficiency of water removal from borax, while the third was to determine how much, if any, moisture might be contributed by the soil simulant GRC-3, e.g. from adsorbed water.

The condenser capability was tested using an initial volume of 68 ml of water. The liquid was placed in heated petri dish inside the MACS. The MACS pressure was reduced from ambient to ~7 Torr. The water temperature was monitored by thermocouple. In order to keep the water from freezing due to adiabatic cooling, heaters on the petri dish maintained the water temperature above 0 °C. Once the system attained 7 Torr the fans were turned on to direct water vapor to the condenser. This condition was maintained for a period of one hour and 58 ml of water was recovered in the condenser, leaving 10 ml unrecovered. It is assumed that most of the lost volume can be attributed to the pump down stage, where the vacuum pump would have expelled water vapor to the vent line. The efficiency of the system was calculated to be 85.3 percent.

The next baseline measurement focused on the efficiency of water extraction from BDH. The goal would be to reduce the BDH to anhydrous borax ($Na_2B_4O_7$). However, of the ten water molecules, two of the water molecules exist as hydroxyl groups and can only be liberated by molecular decomposition at much higher temperatures. Therefore, only eight loosely bound water molecules were targeted. BDH can be transformed to borax pentahydrate ($Na_2B_4O_7.5H_2O$) starting at temperatures of 59 °C, as described by Sahin (2002). BDH experiences a physical transformation as water is evolved from the molecule. It puffs up substantially with a noticeable increase in volume and decrease in density, figure 6. The change in physical appearance provides a good visual indicator if water is indeed being evolved through heating. The tray was blocked such that all of the BDH remained stationary on the tray for the duration of the

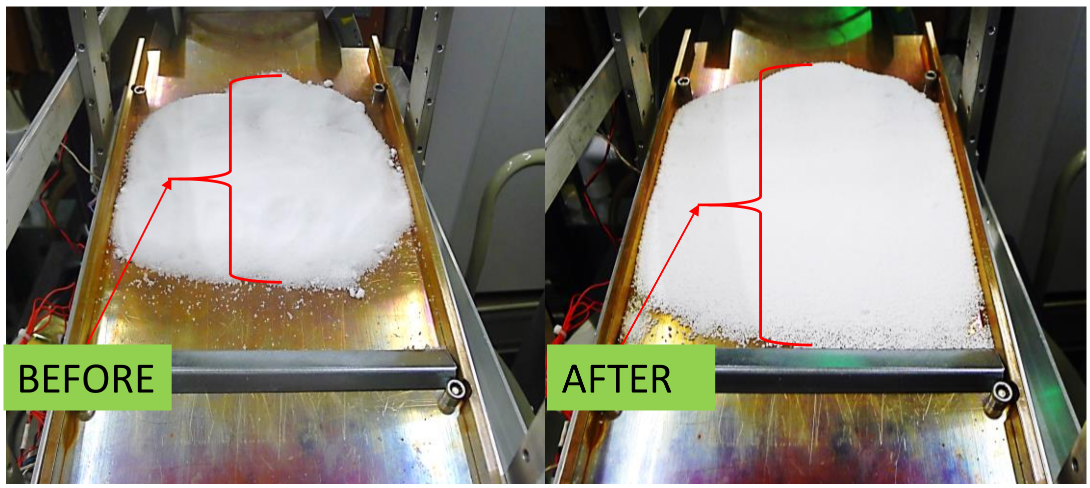

Figure 7: Changes to borax provide physical evidence of water extraction.

test. Once the system was evacuated down to 7 Torr pressure, the heaters and fans where turned on. The test was performed with 200 g of lab grade BDH, which consists of 75.6 g water available for release. The BDH was heated to the target temperature of 150 °C and maintained at a minimum of 150 °C for 21.8 min. The borax weighed 180 g, post run indicating that 20 g of H_2O, or 26.5 percent of the available water, had evolved. The condenser captured a total of 13.5 g of water. In this case the efficiency of the system is 67.5 percent and is in sharp contrast to the 85.3 percent attained with liquid water. The reason for the difference in efficiencies is undetermined.

The final baseline measurement was to determine the amount of water that might be evolved from the GRC-3 itself. Being a high surface area material it is possible water molecules may be attached to GRC-3 particulates. Because the GRC-3 material was not being stored in a dry box or other controlled environment, it had the potential to adsorb water molecules. GRC-3 is known to hold about 0.4 percent water by mass at standard room conditions. To duplicate the conditions that were used with the water extraction runs, 2200 g of GRC-3 was loaded into the source soil bin. The system was brought to the starting pressure of 7 Torr and 400 g of GRC-3 was processed at a tilt tray temperature of at least 150 °C. The condenser had almost no moisture in the collection tube and was estimated to be 8 drops. Therefore, any water contribution from the GRC-3 was considered negligible.

Experimental Runs

For each run, BDH was added to GRC-3 soil and thoroughly mixed to achieve a uniform distribution with a 4.2 percent water content by mass. All of the runs used 2000 g of GRC-s and 200 g of BDH. The simulant mixture was placed into the source soil bin and the system sealed and evacuated to 7 Torr. In order to replicate the Martian atmosphere CO_2 was introduced into the chamber at a flow rate of 100 sccm's beginning at atmospheric pressure and continuing until the set point pressure of 7 Torr is achieved. Once steady state conditions were achieved (7 Torr pressure, tilt tray ≥150°) the CO_2 flow and vacuum valves were closed. The bucket rotation was turned on with a rotation speed of 5 rpm, which at room conditions achieved a good distribution of soil on the heated tray to allow sufficient soil residence time to achieve desired heating. However, soil transport down the tilt tray was significantly faster under vacuum conditions so the soil temperature failed to reach the target temperature by at least 50 °C. A small amount (< 2 cc's) of water was liberated but volume was too small to measure accurately.

In the remaining runs the bucket wheel rotation was operated in a stop and go motion to regulate the amount of soil on the tilt tray and maintain a tray temperature of 150 °C or greater. Although the source soil bin is capable of holding more than 2 kg of material the bucket wheel's efficiency dropped off markedly as the soil was depleted, and therefore the remaining runs were terminated when approximately one-fourth of the loaded simulant was processed.

Results

Four runs were conducted with an average post-processed soil mass of 469 g of soil processed and an average of 8.3 cc of water captured. An accurate determination of the efficiency of the system was complicated by the difficulty in measuring the exact amount of soil and BDH processed, coupled with widely different results from baseline measurements. Assuming that the BDH was uniformly distributed within the GRC-3

Table 1: Potential water mass and recovery efficiency calculated from post processed mass only.

	Processed mass, g	Potential water, g	Water collected, g	Estimated Efficiency , %
	446	15.3	7	45.6
	488	16.8	9	53.6
	444	15.3	9	58.9
	497	17.1	8	46.8
Avg	468.8	16.1	8.3	51.2

and that the processed material was uniformly heated, the system efficiency based on post processed soil mass, could be calculated as shown in Table 1. A more accurate approach would be to calculate the total potential releasable water in the soil, and from that, the water extraction efficiency however this requires knowing the initial (unreacted) soil mass. An estimate was made for the initial soil mass by adding the mass of water captured to the post-processed soil mass, Table 2. This estimated initial soil mass does not account for water that was released but not captured in the condenser, and therefore has a bias error lower than the actual initial mass. Water extraction efficiency for the system, defined by dividing the captured water by the total potential water in the initial soil, is also listed in the table, and represents a slight positive bias from actual efficiency (bias error estimated to be less than 0.5 percent).

Table 2: Potential water mass and recovery efficiency assuming all water was recovered.

	Pre-Processed mass estimate, g	**Potential water , g**	**Estimated Efficiency , %**
	453	15.6	44.9
	497	17.1	52.6
	453	15.6	57.8
	505	17.4	46.1
Ave	477.0	16.4	50.3

Table 3: ICP-AES analysis of extracted water.

ICP-AES Analysis of extracted water					
Element	**concentration µg/mL**	**Element**	**concentration µg/mL**	**Element**	**concentration µg/mL**
Al	0.06	Al	0.06	P	0.1
B	3.2	Ca	17.71	S	1.0
Ba	0.044	Na	9.1	Si	6.3
Ca	17.71	Si	6.3	Sr	0.057
Cu	0.03	B	3.2	Ti	0.001
Fe	0.01	K	1.9	Mo	0.04
K	1.9	S	1.0	Cu	0.03
Mg	0.97	Mg	0.97	Fe	0.01
Mn	0.002	P	0.1	Mn	0.002
Mo	0.04	Sr	0.057	Ti	0.001
Na	9.1	Ba	0.044		

Future work to better calibrate the efficiency of the condensation system would allow a more precise calculation of initial mass and therefore overall system water extraction efficiency. The average water collection efficiency for the system was slightly better than 50 percent, with a range from 45 to 58 percent over the four runs. The water collected was analyzed by two separate methods; inductively coupled plasma atomic emission spectroscopy ICP-AES and x-ray photoelectron spectroscopy (XPS), which are shown in Table 3 and Table 4 respectively. There was excellent agreement between both methods. Calcium, sodium, silicon, potassium and boron were the primary elements detected. The absolute quantities differed as each method uses different techniques. However, the rankings of the elements were similar. ICP-AES did detect small amounts of other elements (Mg, Mn, Sr, S). In this study no filtering of the gas stream into the condenser was performed and it is likely microscopic dust particles from the GRC-3 is the major source of the elemental contamination.

Table 4: XPS analysis of extracted water

XPS Analysis of Evaporated Water on Glass Slide													
	Atomic Concentrations (at.%)												
Area	**O**	**C**	**Si**	**Na**	**Al**	**Mg**	**N**	**Ca**	**Cl**	**B**	**K**	**F**	**S**
Reference Glass 1	44	33	15	4.9	0.3	0.2	1.3	0.9	0.1	<.1	<.1		
Reference Glass 2	43	34	14	6.3	0.3	0.3	1.3	0.8	<.1	<.1	<.1		
Reference Glass 3	48	25	16	7	0.6	0.6	1.6	0.9	<.1	<.1	<.1		
Water Residue 1	45	32	11	5.1	0.4	0.4	2.6	1.4	0.6	1.4	0.3		
Water Residue 2	45	35	13	1.7	0.9	0.3	2.1	1.9	0.1	0.9	0.1		
Water Residue 3	40	37	10	4.5	0.5	0.3	2.2	0.8	0.3	1.6	0.1	2.4	0.4
Water Residue 4	47	31	16	2.2	0.5	0.2	2	0.8	0.1	0.4	0.1		

Conclusions

A set of experiments tested performance of a conceptual water extraction system for use on Mars hydrated regolith. The open reactor concept uses the Mars atmosphere as a sweep gas to transport evolved water vapor to a collection system. The tests have shown that at an atmospheric pressure of 7 Torr CO_2 there is sufficient molecular gas density to allow the native Martian atmosphere to be used as an effective sweep gas. The water recovery efficiency of the initial breadboard hardware system was approximately 50 percent, with greater efficiency possible upon design iteration. The system concept was predicated on sacrificing efficiency for the ruggedness offered by eliminating the need for high temperature seals in the dusty, abrasive environment.

With 50 percent collection efficiency as a lower bound for what is achievable, this concept shows considerable promise.

Further testing and design improvement will include refinement of the soil transport along the heated tray. The vibratory motion, the angle of the tray, the speed of the bucket wheel, and the bucket dimensions can be examined to increase residence time and soil distribution along the tray. During this test program the tray configuration actually transported the material too efficiently at low pressure, preventing the soil from being sufficiently heated. The fact that this tray configuration was selected based upon performance in one atmosphere of air and terrestrial gravity highlights the importance of testing these types of systems in relevant environments.

The condensed water exhibited a substantial amount of calcium, sodium, silicon and boron present. These elements give clear evidence of dust particle contamination of the sweep gas as it is being directed into the condenser. This information regarding contamination will feed into the design of the downstream ISRU systems including the water capture and clean up systems. Kleinhenz (2016) illustrates how a water extraction system like this one may be used in a larger Mars ISRU system.

Continued testing of this open reactor concept is on-going under the NASA Advanced Exploration Systems ISRU Technology Development Project. Currently the data presented here is feeding into modeling efforts, which will help identify parameter optimization and design improvements.

References

1. Abbud-Madrid, A., D.W. Beaty, D. Boucher, B. Bussey, R. Davis, L. Gertsch, L.E. Hays, J. Kleinhenz, M.A. Meyer, M.Moats, R.P. Mueller, A. Paz, N. Suzuki, P. van Susante, C. Whetsel, E.A. Zbinden, 2016, Report of the Mars Water In-Situ Resource Utilization (ISRU) Planning (M-WIP) Study; 90 p, posted April, 2016 at http://mepag.nasa.gov/reports.cfm.
2. Audouard, Joachim, et al. "Water in the Martian regolith from OMEGA/Mars Express." *Journal of Geophysical Research: Planets* 119.8 (2014): 1969-1989.
3. Drake, Bret G., editor, “Human Exploration of Mars Design Reference Architecture 5.0,” NASA-SP-2009-566, July 2009.
4. Drake, Bret G., Stephen J. Hoffman, and David W. Beaty. "Human exploration of Mars, design reference architecture 5.0." *Aerospace Conference, 2010 IEEE*. IEEE, 2010
5. He, Chunmei, Xiangwu Zeng, and Allen Wilkinson. "Geotechnical properties of GRC-3 lunar simulant." Journal of Aerospace Engineering 26.3 (2011): 528-534.
6. Kleinhenz, Julie, and Diane L. Linne. Preparation of a frozen regolith simulant bed for ISRU component testing in a vacuum chamber. National Aeronautics and Space Administration, Glenn Research Center, 2013.

7. Kleinhenz, J.E. and Paz, A. *An ISRU Propellant Production System to Fully Fuel a Mars Ascent Vehicle*. AIAA SciTech Forum 2017. American Institute for Aeronautics and Astronautics. AIAA-2017-0423
8. Leovy, Conway. "Weather and climate on Mars." *Nature* 412.6843 (2001): 245.
9. Linne, Diane L., et al. "Extraction and Capture of Water from Martian Regolith Experimental Proof-of-Concept." (2016).
10. Mouginis-Mark, Peter J. "Water or ice in the Martian regolith? Clues from rampart craters seen at very high resolution." *Icarus* 71.2 (1987): 268-286.
11. Sahin, Omer, and A. Nusret Bulutcu. "Dehydration behaviour of borax pentahydrate to anhydrous borax by multi-stage heating in a fluidized." Turkish Journal of Chemistry 26.1 (2002): 89-96.
12. Skonieczny, Krzysztof, et al. "Advantageous bucket-wheel configuration for lightweight planetary excavators." (2011).
13. Vaniman, David T., et al. "Magnesium sulphate salts and the history of water on Mars." *Nature* 431.7009 (2004): 663-665.
14. Zimmerman, Yuru Li. Developing a Lightweight Martian Simulant and a Miniature Triaxial Device for Lunar and Martian Soils. Diss. Case Western Reserve University, 2016.