EFFICIENT MICROWAVE APPROACHES FOR EXTRACTING WATER FROM HYDRATED MINERALS.

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Introduction: NASA is exploring ways to extract volatiles from lunar, Mars, and asteroid regolith. In particular, extracting water from Mars regolith is essential for sustaining a future human mission. We are evaluating various microwave methods for efficiently extracting water from hydrated minerals of interest to NASA.

Approach: A resonant microwave system [1] coupled to a volatile condensation unit shown in Fig. 1 has been used to quantify the amount of extracted water as a function of various operating scenarios for the hydrated minerals epsomite ($MgSO_4$) and magnesium perchlorate ($Mg(ClO_4)_2$).

A 200-W traveling wave tube was used to excite the transverse electric, TE103, resonant mode at 2.45 GHz in a WR340 waveguide cavity. A quartz tube containing the sample was positioned at an electric field maximum inside the cavity. During a microwave run, the dielectric properties of the sample changed as water was removed. A frequency tracker was used to stay on the shifting resonant frequency. The changes in the transmitted and reflected power, electric field strength, and resonant frequency in the cavity during a run were recorded using a custom LabVIEW program. The temperature of the surface of the sample was monitored using a Process Sensors Model SSSL-3ML-CF2 pyrometer having a temperature range 50 - 400°C. The quartz tube was connected to a glass manifold (shown in Fig. 1) and was pumped down to ~50 mTorr during a run to ensure ambient moisture was removed prior to microwave excitation. Any volatiles removed were condensed in liquid nitrogen cooled traps and immediately measured after the run.

The samples consisted of an alumina foam (40 ppi) substrate uniformly wash coated with the hydrated salt of interest. The wash coating was applied by making a 1.0 M solution of either $MgSO_4$ or $Mg(ClO_4)_2$, soaking the foam substrate in the solution until sufficiently wet and then drying at 30°C on Teflon. The loaded alumina foam samples were cylindrical in shape with approximate dimensions of r = 0.64 cm and L = 2.54 cm. To determine the amount of water extracted from a microwave run, the weight was measured for the empty alumina foam prior to wash coating and then again for the loaded alumina foam before and after each experiment. A loaded sample was also dehydrated thermally in order to experimentally verify the maximum potential water extraction. In order to better understand the effects of pressure on dehydration at 50 mTorr a "blank" run was conducted where the system was evacuated and the water was collected according to the normal protocol, however, no microwave radiation was used.

In each run, water was collected in two liquid nitrogen cooled traps. The first trap was downstream of the second trap and was the trap initially cooled in order to sufficiently differentiate the origins of the collected water. In addition, each trap was collected for an equal time, which was always for half the duration of the experiment.

Results: For epsomite, three runs were performed with 2 W of microwave radiation for 60 minutes and a single run with 4 W for 30 minutes. The isobaric experiment at 50 mTorr for 90 minutes resulted in an even split of water between the two cold traps totaling what equates to a single bound water from the epsomite. This result suggests that for the microwave runs, the hydrated epsomite has between 6 and 7 bound water molecules. Heating one of the loaded alumina foam samples in a furnace over night at 300°C thermally dehydrated the sample and the measured weight change agreed with the expected amount of water from the mass of epsomite. Assuming a starting point of 6 bound water molecules in the three microwave excited epsomite samples at 2 W, and averaging the measured extracted water, we determined that the microwaves extracted 65-70% of the bound water molecules. For the 4-W microwave run we extracted ~60% of the bound water.

In the case of magnesium perchlorate, an initial test was conducted at a 2-W microwave power level for 60 minutes and resulted in under 10% water extraction. The power was increased to 5 W and the experiment was repeated resulting in a slight increase in extracted

Fig. 1. Microwave/Condensation Apparatus

water but still ~10%. It was not until the power was increased to 10 W that a significant amount of water was extracted. However, the temperature began to rise after 10 minutes and approached the initial thermal dehydration temperature (75°C) and the power was reduced to 7 W for the remaining 50 minutes. This resulted in a water extraction of ~25%, however still less than the amount expected for the first two bound water molecules. From these initial magnesium perchlorate measurements, it appears that there is a power level between 7-10 W where the temperature could remain around 60-65°C while collecting the extracted water.

We plan to perform additional experiments on magnesium perchlorate samples to optimize microwave parameters to increase the amount of extracted water. In addition, we will perform similar microwave runs for gypsum to evaluate the effectiveness of microwaves to extract water from this hydrated mineral. Additional studies are planned with various percentages of hydrated materials mixed in a Martian regolith simulant to further define the optimum experimental conditions for extracting water in a more Martian-like environment.

Our results suggest that microwave excitation can be used to more efficiently extract bound water than conventional heating methods. This result could be a game changer in designing future Mars facilities for extracting water from hydrated minerals.

Microwave Space Approaches for Removing Bound Volatiles from Regolith: The coaxial cable approach directly transmits the microwave energy into the regolith. We have modeled and optimized such a coaxial cable having a fixed length of its center conductor that is imbedded in the regolith. By including a perforated ground plane and optimizing the length of the imbedded center conductor over 94% of the microwave energy will be transmitted into the regolith. Figure 2 shows a drawing of this approach that could be used in a space environment.
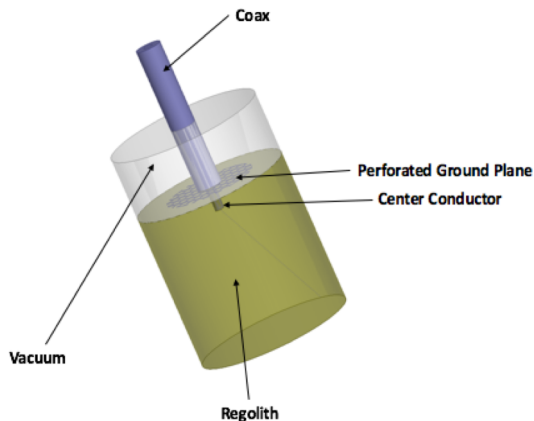

Fig. 2. Microwave Coaxial Cable Approach

The Ridge Waveguide approach is designed to constantly move across the surface of regolith to extract volatiles. The penetration of the microwave energy into the regolith is a function of the rate of sweeping along the regolith surface and energy applied. It would essentially be a microwave vacuum cleaner. For the ridge waveguide approach, over 97% of the microwave energy will be transmitted into the regolith by optimizing the dimensions of the ridges for the complex dielectric constant of the regolith. Figure 3 shows a drawing of how one of these ridge waveguides could be used to release volatiles from the regolith. By using several ridge waveguides side by side more volatiles could be extract per unit time.
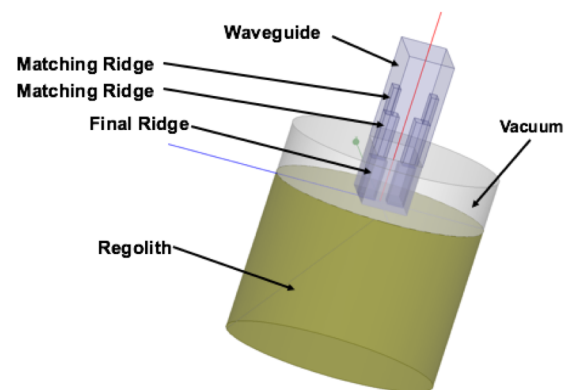

Fig. 3. Microwave Ridge Waveguide Approach

Which approach to use will depend on the method chosen to transmit microwave energy into the regolith [2]. The coax approach generates about twice the energy density in the regolith compared to the ridge waveguide approach. If the regolith will be excavated, it could be placed on a moving belt that moves around one or more microwave coaxial cables. The ridge waveguide approach has the advantage that the regolith does not need to be penetrated as is the case for the coaxial center conductor. One only needs to move the microwave ridge waveguide system along the surface of the regolith or move the regolith on a conveyor belt below the ridge waveguide to transmit energy into the regolith and extract volatiles.

References: [1] Barmatz, M., Steinfeld, D., Begley, S.B., Winterhalter, D., and Allen, C., (2011) *LPS XXXXII*, Abstract #1041. [2] Howe, A. S., Wilcox, B., Barmatz, M., and Voecks, B., (2016) Proceedings of the *Fifteenth Biennial ASCE Aerospace Division International Conference on Engineering, Science, Construction, and Operations in Challenging Environments* (Earth & Space 2016). Orlando, Florida, USA, 11 - 15 Apr 2016.