**NEW CRYOGENIC METHOD FOR COMBINING LUNAR REGOLITH SIMULANT AND FROZEN VOLATILES TO GENERATE ICY LUNAR SIMULATED REGOLITH.**

E. K. Lewis[1,2], C. L. Harris[2], S. Ghosh[2], C. L. Amick[2], C. A. Mantilla[2], K. K. Allums-Spencer[3] and J. W. Boyce[4], [1]Texas State University, ernest.k.lewis@nasa.gov, [2]Jacobs-JETS II, TX, [3]Jacobs/HX5, [4]NASA Johnson Space Center.

**Introduction:** There is a requirement within the lunar science and exploration communities to develop lunar simulants containing volatiles that are solids at the cryogenic temperatures found in the permanently shadowed regions (PSRs) [1], such as those found near the Lunar South Pole. Icy regolith simulants would be used for curation training, as well as for various research activities. One of the most critical aspects of developing a regolith simulant that is more physically and chemically like icy lunar regolith is minimizing any form of modification driven by elevated temperatures. Here we document our ongoing efforts to combine materials at ultralow temperatures, designed to minimize any chemical reactions or other physical changes during the production of the icy regolith.

Our goal is to document how one can create an essentially "unreacted" icy regolith that can serve as an effective "time zero" start point from which advanced curation research will proceed. This is done using commercial off-the-shelf equipment as much as possible, along with creating a custom spray plate that can be adapted to a wide variety of methods, all for the planetary simulant community.

This method creates crystals of various volatiles such as water, methanol, along with $CO_2$ ice and these components are combined with lunar regolith that is at -196°C temperature to avoid chemical reactions, and/or phase changes thus creating a more chemically relevant icy lunar regolith. The rules of chemistry stay the same regardless of location whether it's in the lab, or directly on the lunar surface, and therefore we aim for creating a more authentic icy lunar simulant in the laboratory by operating at ultralow temperatures.

It is also envisioned that this method will lead to advanced materials testing in the future. In addition, this method is directly applicable to prior missions such as LCROSS.[2]

**Equipment and Methods:** All tools and Lunar simulant start materials, such as NU-LHT-4M or LHS-1, are pre-chilled in Teflon flasks down to -196°C, along with all necessary tooling such as scoops and mixing bowl. The angled cryogenic spray block is also cooled to the same temperature. These components are placed into liquid nitrogen ($LN_2$) baths in the chemical hood to maintain ultracold temperature while performing tasks such as scooping, imaging, mixing, weighing, etc. The sprayer is not at $LN_2$ temperature; however, it is near the spray block, and this minimizes the volatile loss traversing from sprayer to condensation and the rapid temperature change (from ambient to cryogenic $LN_2$ temperature) can create low density mixable sheets of volatile ices.

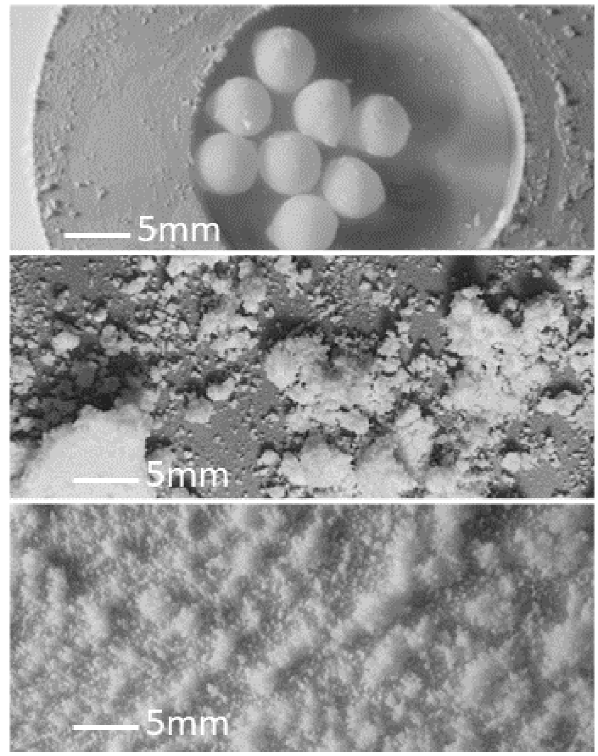


*Figure 1: Demonstrating the production of a wide range of controlled ice particle sizes such as large 5mm ice pellets (top panel), to mid-size sheets of low-density snow down (middle panel) to sub-100um ice crystals (bottom panel). Images are taken on cryogenic carriers at LN2 and are in the same visual scales. Low-temperature imaging provides ISRU characterization capability and limits melting effects.*

In the chemical hood, there are $LN_2$ cryo-baths as needed to maintain low temperatures of various components and chill plates. When a weighing measurement needs to take place, either the cryogenic spray block, or the mixing bowl can be placed on the scale determining the volatile mass dosed into the lunar simulant. The ability to quickly deliver a measured dose of icy regolith into the simulant is a key feature of this experimental procedure as that limits the losses and melting to within the feasible scope of this process.

Utilization of a standard mixer, with the mixing portion held at $LN_2$ temperature provides a means of combining multiple component ices dosed into a chosen lunar or planetary simulant. This method is generic to a wide variety of materials and volatiles because the ultracold temperatures avoid significant, if any, physical and chemical changes.

The volatiles like those in the PSR are sprayed onto an angled aluminum platform we call a cryo spray block. This has a known weight and the lower portion immersed within $LN_2$ with cooling fins to create a surface that causes immediate freezing of sprayed droplets. For deposition of volatiles, an air brush is moved around spraying onto the block creating a low-density ice sheet that can be transferred into the mixing bowl quickly and efficiently avoiding melting. The whole mixing bowl contents are held at $LN_2$ temperatures to create an all-cryogenic mixing environment.

**Ice Crystal Characterizations:** In the case of water, several different sizes of ice have been made and imaged as shown in Figure 1. In the top panel, spherical ice spheres (~5 mm) were produced by putting individual water droplets into $LN_2$. The middle panel uses spray deposition creating soft layers of ice. The bottom panel is from condensation which creates crystal sizes down to 100 μm crystals. This ability to design icy particle sizes as a function of volatiles provides an additional control handle into potential sample requests. For example, small crystals could be requested for ultrafine materials for ISRU testing and extraction of resources. Another example could be using larger 5 mm ice spheres for mobility testing within a PSR. This testing process is repeated for a wide variety of volatiles including but not limited to $CH_3OH$, $H_2S$/water, $NH_3$/$CH_3OH$, and in conjunction with $CO_2$ sprayer system. For characterization, we have a separate horizontal cold plate that is at $LN_2$ and a camera system above so that close particle sizes can be measured, and test images obtained during the process with limited melting.

The control of the deposition is such that the net weight is recorded and can be used to calculate the mass loading into the lunar or planetary simulant. This is repeated for a variety of compounds such as but not limited to water ($H_2O$), methanol ($CH_3OH$), etc. Carbon dioxide ($CO_2$) is added separately through a dry-ice generator. Future work will include $H_2S$ and $NH_3$ as well combined directly into regolith.

**Results:** Highly accurate, more chemically authentic icy lunar simulant has been made and a method is created for the lunar simulant community using standard laboratory available equipment. A variety of icy volatile crystals from millimeter in size down to sub-100 μm has been created and can be used to precision dose cryogenic lunar simulant with good control. All simulants and icy volatiles are held at temperatures below -150°C, limiting any side chemical reactions from occurring. The close-proximity of spray-capture results in approximately 95% transfer from sprayer to cryo spray block of volatile liquid for water for example, which will be important as the volatility of a particular solvent increases. The experimental setup can make simulant mixtures from around 400g, scalable up to 2kg at a time depending upon the available resources and space limitations.

**Conclusions:** Here we show that producing icy volatile simulants individually with a known crystal size then transferring the ice to a chilled lunar regolith simulant is an efficient method for creating high fidelity 'time-zero' lunar PSR simulants. These simulants will be relevant to research studies focused on lunar environments and beyond such as meteorite and asteroid sample returns along with meeting advanced curation testing requirements.

This experimental setup and method can create compositions with a higher precision than what is experimentally reported from excellent missions such as LCROSS. This means that we can test and replicate observations from missions within the laboratory through iteration and create a high degree of confidence when the precise mixture is found. This also means that a wide variety of icy volatile mixtures can be tested prior to missions involving the lunar south pole PSR regions. This method provides a wide variety of scalable icy volatile lunar simulant systems when a known combination is found to be favorable for PSR testing within the lunar simulant community.

**Acknowledgments:** Authors would like to acknowledge support from the NASA Johnson Space Center ARES XI Curation and Research Departments, as well as support from SSERVI Ice-Five-O project 80NSSC20M0027, along with lunar simulant provided by the NASA ARES Simulant Development Laboratory (SDL).

**References:** [1] Brown H.M., Boyd A.K., Denevi B.W., Henriksen M.R., Manheim M.R., Robinson M.S., Speyerer E.J., Wagner R.V., *Resource potential of lunar permanently shadowed regions*, Icarus, Volume 377, 2022, 114874, ISSN 0019-1035, https://doi.org/10.1016/j.icarus.2021.114874 [2] Colaprete, A., et al., *Detection of Water in the LCROSS Ejecta Plume*, Science 330, 463-468 (2010). DOI: 10.1126/science.1186986