Lunar Oxygen Production and Metals Extraction Using Ionic Liquids

Matthew Marone, Mercer University, Macon, GA
Mark Steven Paley, AZ Technology, Huntsville, AL
David N. Donovan Marshall Space Flight Center
Laurel J. Karr, Marshall Space Flight Center, Huntsville, AL

LEAG Meeting
Houston, TX Nov. 18, 2009
There are two main strategies for exploring the Moon. Both begin with a few short sorties to various sites to scout the region and validate the lunar landing and ascent systems. In one strategy, the next step would be to build a base. Over many missions, a small colony of habitats would be assembled, and explorers would begin to live there for many months, conducting scientific studies and prospecting for resources that could be used as fuel. In the other strategy, sorties would continue to different sites, spending weeks and then months at each one. More equipment would have to be brought on each trip, but more diverse sites would be explored and in greater detail.
Buzz Aldrin described the Moon as “magnificent desolation”
In Situ Resource Utilization (ISRU)

Desolate but not without resources

Si  Al  Mg  Ca  O
Ti  Fe
Desolate, but not without resources
<table>
<thead>
<tr>
<th>Oxide</th>
<th>Conc. (mean of 3)</th>
<th>Std. Dev.</th>
<th>Lunar Soil 14163*</th>
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<td>LOI</td>
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<tr>
<td>Total</td>
<td>99.65</td>
<td></td>
<td>99.8</td>
</tr>
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</table>

**JSC-1: A NEW LUNAR SOIL SIMULANT**

Achieving NASA’s goal of establishing a sustainable manned lunar outpost will require the extraction of oxygen from lunar regolith in a safe manner that minimizes consumables and energy. Methods include:

Molten Oxide Electrolysis – Requires High Temperature and Refractory Materials

Hydrogen Reduction – Low Yield of Oxygen per Unit Mass of Regolith (1%-5%)

Chemical Beneficiation—Requires Launching, Storing, and Handling of Hazardous, Corrosive, and/or Toxic Reagents
Our approach for extracting oxygen is based on the ability of an acid to react with a metal oxide and produce water. A simple acid-base reaction

Hydrochloric acid and iron oxide react to form iron chloride and water

$$6\text{HCl} + \text{Fe}_2\text{O}_3 \rightarrow 2\text{FeCl}_3 + 3\text{H}_2\text{O}$$
For Lunar exploration, conventional acids are too hazardous.

Launching such materials and using them in a confined environment represent major safety issues.
We Use Ionic Liquids Instead of Conventional Acids

Ionic Liquids are a new class of materials that can be engineered at the molecular level for a particular task. Ionic Liquids have very low vapor pressures and low flammabilities.

They are molten salts with melting points near room temperature. Safe “green” (is that gray on the Moon?) chemicals.

Excellent chemical and thermal stability. Even in hard vacuum.
Experimental Approach:

1) Solubilization of regolith in IL medium to convert metal oxides to water and metallic ions.

2) Electrolysis of water produced to generate oxygen and hydrogen.

3) Regeneration of IL medium by electrolysis of hydrogen by-product using hydrogen gas electrode.
Dissolves Regolith Below 200°C in Ionic Liquid Acid
Solubilization Experiments with JSC-1

Several IL acid systems have been prepared in our laboratory and tested for their abilities to solubilize JSC-1. Initial experiments consisted of taking small (1g) samples of JSC-1 and heating them with aqueous solutions of the ILs for 24 hours. Aqueous solutions were used instead of the neat (anhydrous) ILs so that solubilization would take place at a reasonable rate. Water is needed at the start of the reaction in order to ionize the acid and to act as a stabilizing ligand by coordinating to the metallic ions in the solution. In a closed system this initial amount of water would be recycled.
Solubilization Experiments with JSC-1

For the acids we have tested, the weak IL acids showed solubilization efficiencies around 50%, whereas the strong IL acids showed efficiencies around 70%. This result is not unexpected since stronger acids would be expected to react more effectively than weak acids.

A control experiment using sulfuric acid also showed an efficiency of 70%. This is not surprising since in aqueous solution the strong IL acids are of approximately the same strength as sulfuric acid.
Movie of JSC-1 Dissolving in IL at 165C
JSC-1 was not designed to be a mineralogical simulant. Apollo samples would be better, but our work is destructive. Small samples of Lunar meteorites are easy to obtain. Dar al Gani 400 is a well characterized meteorite whose composition is representative of the Lunar Highlands.
<table>
<thead>
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<th>Oxide</th>
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<td>K$_2$O</td>
<td>0.1</td>
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</table>

Lunar Oxygen Production Using Ionic liquids
Yield 70-80% of theoretical amount of water that can be produced from metal and silicon oxides.
Distill Aqua Luna from Solubilized regolith
Electrolysis with IL Electrolyte

Pt Electrode with $O_2$ Bubbles
Metal salts in spent IL
Regeneration of Ionic Liquid

Because of up-mass costs, it is critical that the IL medium can be regenerated so that the process is economically viable.

During solubilization of the lunar regolith, only the $\text{H}^+$ of the IL acid medium is consumed; the “frame” of the IL and the counter-anion should remain intact. Hence regeneration of the medium essentially consists of re-protonating the IL.

This can be accomplished by electrolysis using a hydrogen gas electrode (a blackened Pt electrode in a hydrogen atmosphere) to oxidize the hydrogen by-product from the water electrolysis step to $\text{H}^+$ at the anode. At the cathode, the metallic ions in solution become reduced to the free metals.:
Ionic Liquid Regeneration Experiments

It is critical that the spent IL acid medium can be regenerated. As a 1\textsuperscript{st} step a preliminary experiment was carried out to ascertain whether the IL survives the regolith solubilization process and can be re-protonated.

1g of JSC-1 was reacted with aqueous IL acid, and the insoluble residue was filtered out. The spent acid was run down an ion-exchange column (H\textsuperscript{+} form), the eluent was collected, and the water removed. The mass of recovered IL acid was approximately 97\% of the starting mass, indicating that the IL does indeed survive the solubilization conditions.

This solubilization experiment was then repeated 4 more times using the recovered IL acid with fresh JSC-1 (1g); in each case the recovery yield was 95 - 98\%. These results are very encouraging because they show that the IL can, in principle, be regenerated multiple times. The next (and most technically challenging) step in this research is to conduct regeneration (re-protonation) experiments using a hydrogen gas electrode.
Hydrogen from Electrolysis is Used to Reduce Metals

Cu Flakes
Conclusions

- Initial results indicate that ionic liquids are promising media for the extraction of oxygen from lunar regolith.

- IL acid systems can solubilize regolith and produce water with high efficiency.

- IL electrolytes are effective for water electrolysis, and the spent IL acid media are capable of regeneration.
NASA Marshall Space Flight Center ISRU Lab

- Laurel J. Karr (MSFC)
- Mark Steven Paley (AZ Technologies)
- Matt Marone (Mercer University)
- David N. Donovan (MSFC)
- Peter Curreri (MSFC) - Molten Oxide Electrolysis

- William Kaukler (UAH)
- Ed Ethridge (MSFC)

Lunar Water Extraction