An Evaluation of Lunar Simulant and Meteorite as a Proxy for Lunar Regolith for In Situ Resource Utilization Experiments

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Introduction: Water is one of the most critical resources required to enable long term space exploration. Lunar water could not only provide the life support needs of crew members, but its constituents hydrogen and oxygen can also be used as rocket propellant. The use of local resources such as water is known as in situ resource utilization (ISRU). Arguably, there are water ice deposits at the lunar poles. However, the majority of these ice deposits are thought to be in areas known as permanently shadowed regions (PSRs). These PSRs experience temperatures as low as 30 K and, therefore, accessing water-ice deposits in these regions will be technologically challenging. There are other potential ways of producing water on the Moon, such as through hydrogen reduction of iron-oxide-bearing minerals. Hydrogen can reduce iron-oxides such as ilmenite in an equilibrium reaction when heated to temperatures of at least 900°C [1] as in eq. 1:

$$\text{FeO}_x + x\text{H}_2 \leftrightarrow \text{Fe} + x\text{H}_2\text{O} \quad (1)$$

A hydrogen reduction experiment is to be performed on the lunar surface using the ProSPA instrument on-board Luna-27 in 2025 [2]. A breadboard of ProSPA was successfully used to produce water from the hydrogen reduction of ilmenite and the procedure was optimized [3]. ProSPA is expected to land in a highlands-like region which is likely to be ilmenite-poor. However, iron-bearing silicates such as pyroxene and olivine can also be reduced by hydrogen, although at much lower efficiencies than ilmenite [4].

A handful of previous studies have successfully produced water from lunar minerals [e.g. 1,5]. However, there is currently a resurgence in ISRU studies and Apollo soils are not available for routine large-scale destructive ISRU experiments. Therefore, lunar simulants are being used as a proxy for lunar materials [e.g. 6,7]. However, certain requirements must be met by a simulant to realistically replicate the behavior of lunar material for each type of extraction technique. In this study a highland simulant, a lunar meteorite, and two Apollo soils were reacted in a ProSPA breadboard to determine the feasibility of the reduction experiment on lunar soils and to identify any key differences between the samples that should be considered when performing ISRU studies.

Methodology: The ProSPA instrument is static in that it does not utilize a flow of carrier gas to remove reaction products from the reaction site. Instead, samples are reacted in a furnace which operates in a closed system that contains hydrogen. Produced water is condensed on a cold finger, and the resultant pressure gradient created ensures the diffusion of water away from the reaction site and towards the cold finger. Consequently, the equilibrium reaction can continue forwards and reduction can continue.

A breadboard model (ISRU-BDM) of the ISRU-relevant components of ProSPA was built at The Open University. Samples of ~45 mg were reacted in a furnace at 1000 °C for 4 hours in the presence of ~420 mbar of hydrogen, while the cold finger was set to ~80 °C [3]. Quantification of the yield was determined by pressure changes within the system.

Materials: Four different materials were reacted in the ISRU-BDM. The FeO content of each material is shown in Table 1, along with the estimated maximum ilmenite content, as derived from the TiO₂ content.

Lunar Simulant: NU-LHT-2M is the chosen simulant which aims to replicate some of the characteristics of Apollo 16 highland soils [8]. The simulant contains only trace amounts of ilmenite so it will be used to represent the ‘worse-case-scenario’ material that could be expected on the lunar surface at the Luna-27 landing site. The NU-LHT-2M was also doped with 10% ilmenite (‘Sim & Ilm’ in Table 1) to understand how beneficiation would influence the yields.

Lunar Meteorite: Northwest Africa 12592 is classified as a fragmental breccia and chosen as a representative of the bulk lunar regolith at feldspathic lunar highlands terrain, albeit with no reported ilmenite [9]. To eliminate the effects of iron-oxide weathering products, some of the meteorite samples were treated with EATG which is commonly used to remove secondary iron-oxides [10].

Apollo Soils: 10084 is an Apollo 11 mare soil [11]. With high ilmenite concentrations of up to 14.33 wt.%, 10084 was considered suitable for an initial reduction experiment. If 10084 produced measurable yields of water, then a series of follow up experiments on 60500 were planned. The sample 60500 is from the Apollo 16 highland soils and has low ilmenite content of up to 1.14 wt.% [12]. The two Apollo samples analysed in this study represent two very different compositions of the major lunar terrains from which we currently have samples.

Results: The pressure rise from the release of water from the cold finger was used to quantify the yields. Yields are shown in Table 1 as the wt.% oxygen pro-
duced. The 1σ uncertainty was derived from the three repeats for each experiment. The results for a pure ilmenite sample are also shown, as taken from [3]. All samples were successful in producing water, with those containing high ilmenite contents producing the highest yields.

**Table 1** Yields from the reduction of lunar simulant and samples, as compared to pure ilmenite

<table>
<thead>
<tr>
<th>Sample</th>
<th>FeO (wt.%)</th>
<th>Est. Ilmenite (wt.%)</th>
<th>Avg. yield (wt. % O₂)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ilmenite</td>
<td>45.0</td>
<td>95</td>
<td>3.43±0.14</td>
</tr>
<tr>
<td>NU-LHT-2M</td>
<td>3.59</td>
<td>0.7</td>
<td>0.29±0.04</td>
</tr>
<tr>
<td>Sim &amp; Ilm</td>
<td>7.73</td>
<td>10.13</td>
<td>0.89±0.04</td>
</tr>
<tr>
<td>NWA 12592 - treated</td>
<td>3.89</td>
<td>0.32</td>
<td>0.07±0.02</td>
</tr>
<tr>
<td>NWA 12592 - untreated</td>
<td>0.08±0.01</td>
<td></td>
<td></td>
</tr>
<tr>
<td>10084</td>
<td>15.81</td>
<td>14.33</td>
<td>0.94±0.03</td>
</tr>
<tr>
<td>60500</td>
<td>5.53</td>
<td>1.14</td>
<td>0.18±0.02</td>
</tr>
</tbody>
</table>

Scanning electron microscope (SEM) images were obtained from a random selection of grains of each unreacted and reacted sample. Evidence of reduction was identified in all samples by the appearance of the pure iron (Fe⁰) blebs in ilmenite, pyroxene, and olivine grains.

**Discussion:** A comparison of ilmenite content with yield is shown in Fig. 2 for the samples used in this work and other lunar or lunar-like samples reacted at 1000 °C with hydrogen in previous studies [1,5,6,7]. It should be noted that the results from literature were obtained from fluidized systems with various H₂ pressures. Generally, the more FeO rich a material, the higher the O₂ yield. However, if the FeO is bound mostly in ilmenite, it would produce higher yields. Therefore, ilmenite content is a more accurate predictor of yield of oxygen than FeO content alone. For example, the doped simulant (Sim & Ilm) contains less than half the FeO content of the Apollo 11 soil, however has similar ilmenite contents. Consequently, the doped simulant produces almost as much water as the lunar soil.

There was no significant difference between the un-treated and treated meteorite sample, suggesting minimal weathering products present that would influence the reaction. The meteorite samples produced particularly low yields, probably because they contain no ilmenite, but also because they have a larger average grain size than the other materials, a consequence of the manual crushing of the sample (i.e., smaller area/volume ratio compared to lunar soils). Larger grain sizes limit the movement of reactants and products to and from the reaction site.

Most lunar soils are a mixture of feldspathic and mafic components. As a result, ilmenite is often likely to be present as a component in lunar soils [6]. Also, finer soils contain more ilmenite than larger size fractions [13]. Thus, fine soils, even those found in more highland-like regions, are likely to produce higher yields than some lunar rocks, such as the genesis rock, that contains very little FeO and no ilmenite [14].

**Conclusions:** Ilmenite content is a good indicator for potential oxygen yields in hydrogen reduction experiments and should be considered when using simulants as a proxy for lunar material. Lunar simulant or crushed meteorite containing relatively low ilmenite contents would be recommended to represent the likely soils that will be sampled by ProSPA. As grain size also significantly influences reaction rates, the grain size distribution of any proxy material should also be representative of the expected lunar material at the proposed landing site.

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