FLUXED MELTING FOR RAPID REGOLITH DIGESTION.

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Introduction: We present a way to pre-treat regolith, by fluxing it with sodium and melting it to glass, that allows it to be almost completely dissolved in hot hydrochloric acid. This regolith digestion allows the included metals and silica to be extracted using a solar powered closed-loop process.

Motivation: Use of in-situ materials can transform our ability to explore space [1]. As we boot up an industrial ecosystem from scratch, we have a chance to rethink how and what we produce, enabling new sustainable technologies that can be used both on or off Earth. In building up infrastructure and habitats, there is an enormous need for useful bulk industrial elements such as iron, aluminum, magnesium, and calcium. On Earth, these materials are produced from concentrated deposits, but in space we don't have evidence that concentrated deposits even exist, so we work with ordinary regolith dust available everywhere.

Many regolith separation and beneficiation processes have been proposed [2]. Metal oxides dissolve in acid, while silicates dissolve in strong alkali, which makes regolith difficult to dissolve completely. Traditional mining beneficiation such as floatation uses specialized reagents. Analytic rock acid digestion requires large quantities of acids that can volatilize silica such as HF, which is complex to recycle [1]. Electrostatic and triboelectric separation, like magnetic separation, operates on individual grains which can be heterogeneous in composition. Magnetic separation is high throughput and useful with highlands anorthosite, but not effective on mare basalt or mixed impact glass.

Approach: We propose a sodium-fluxed melting pretreatment for regolith, followed by digestion in hot hydrochloric acid. Melting transforms slow-dissolving crystalline minerals into leachable amorphous glass.

Using a sodium flux allows easier melting of a variety of regolith simulants, and much more rapid acid attack on the resulting obsidian glass. The mechanism by which sodium causes rapid glass degradation in liquids, known as 'crizzling' [3], seems to be dissolution of the Si-O-Na bonds in the glass, via either ionic exchange with H^+ or via acid-base reaction of non-bridging oxygens. We observed bubbles (hydrogen?) during regolith glass dissolution in HCl.

Any sodium-containing compound should work as a flux, and we have measured NaOH and Na_2CO_3 work about equally well. KOH was much less effective, and $Ca(OH)_2$ was counterproductive. A sodium to regolith ratio of 10-20% w/w seems to work rapidly, while 5%

works incompletely, particularly with basaltic regolith. We have tested this approach on Exolith Labs [4] LHS-1 lunar highlands simulant (primarily anorthosite, giving excellent speed), LMS-1 lunar mare simulant (primarily basalt, slower), and MGS-1 Mars global simulant; as well as a local basalt dust we call NP-1.
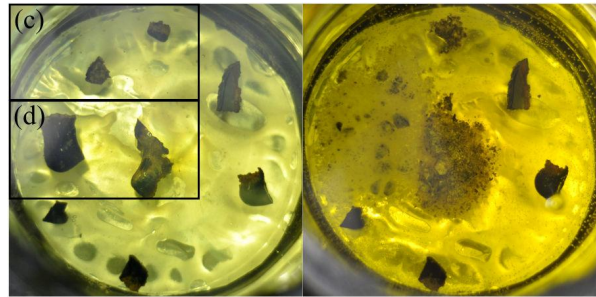
(a) Before acid treatment (b) After 3 hr in 50°C HCl

Figure 1. Melted regolith simulants (a) before and (b) after HCl acid leaching. Note how fluxed melted regolith (c and d) dissolves almost completely in acid.

The high speed of acid digestion of this sodium fluxed glass is shown in Figure 1. Group (c) was fluxed with 10% of a calcium/sodium flux; group (d) was fluxed with 10% sodium carbonate flux; the other samples were melted without flux. Groups (c) and (d) are LHS-1 and LMS-1 from left to right. The remaining four unfluxed glass samples did not dissolve or even degrade: clockwise from the top right are 4x-remelted LMS-1, NP-1, LMS-1, and LHS-1.

Throughput: Solar melting is rapid, taking only seconds with the fine powder that is the top layer of lunar regolith, making glass beads a few mm thick. Using a 0.5 square meter fresnel lens concentrating sunlight to a 10mm diameter spot, we measured melting throughput at 0.75-2.0 grams per minute for raw regolith (for LHS-1 and LMS-1 respectively), and 1.6-2.2 grams per minute using 10% sodium carbonate flux, an increased throughput because flux lowers the melting point. This corresponds to a melting rate of about 0.2 kg/hour per square meter of solar collecting area, an energy of 18 MJ/kg melted regolith, or about 50 tonnes/year for a 8 meter diameter concentrator. Throughput may be higher with insulated vacuum operation, reflecting radiated heat during melting, or preheating by dropping the hot melted beads onto cold incoming regolith. Detailed experiments remain to be done to determine the best way to feed regolith for efficient melting, but melting and cooling regolith on a

bed of regolith dust seems to eliminate crucible wear due to hot glass contact.

Melting Options: Fluxed regolith can be melted with concentrated solar, microwave energy, electrical arc or inductive heating. The simplest is concentrated solar, which couples strongly to the black melted regolith glass, using either large mirrors or a Fresnel lens collector. Mirrors may be inflatable or constructed from local materials, and much lighter than Earth given the moon's lower gravity and lack of wind. However, a thin film Fresnel lens (currently 0.2 kg/m^2) can incorporate the required focal curvature into the lens pattern, allowing launch as a space efficient rolled-up tube and deployment in a mass efficient flat tensile plane structure. Either approach needs sun tracking and robustness to UV, dust, and micrometeorites. Valuable volatiles in the regolith could be captured as they boil out during melting, requiring a high temperature pressure vessel with an optical window that may be challenging to keep clean and cooled.

Microwave melting [5] would allow melting without sunlight, though low-iron regolith may not couple strongly to microwaves until molten. In our tests at 2.4GHz, a 0.5g magnetite "sparkplug" allowed regolith to melt in under 1 minute, but melting remained confined to the one hotspot. A susceptor kiln [6] is reported to melt more uniformly, but required over 80 MJ/kg of electricity for melting regolith.

Acid Leaching: The central observation of this paper is that regolith melted with sodium flux rapidly dissolves in acid. The dissolution rate is faster with higher acid concentration and temperature [7]. At 1 bar, 33% HCl produces corrosive vapors at 50°C, requiring a closed glass vessel and fume hood. Higher pressures allow higher temperatures without boiling, so trade-offs exist between operating pressure, temperature, acid concentration, sodium flux use, regolith type, and leaching time. A test of 10g LMS-1 melted to glass with 2g NaOH flux dissolved almost completely white on the surface in 100mL of 33% HCl in 1 hour at 70°C, so even basaltic regolith can be processed using enough flux and heat. Leaching fully melted fluxed glass for 24 hours produces a bright white crumble with no dark glass remaining inside.

After acid leaching, a fragile white precipitate remains which is primarily silica (and likely some calcium/titanium/zirconium contaminants). This silica rapidly converts to sodium silicate when baked with NaOH, and can be filtered and precipitated as silica gel with HCl. Silica extraction (such as for making glass or silicon) is much faster than for raw regolith.

In the orange solution after acid leaching we have a mix of metal chlorides, from which we can precipitate metal oxides via incremental pH neutralization using any alkali. Titanium dioxide should precipitate around pH 0 by oxidizing the solution, though we have not observed this. Near-quantitative amounts of iron and aluminum precipitate as hydroxides before pH 6 as a voluminous fluffy red mud; the alumina can be washed out with a strong base to recover these two useful metals individually for electrolytic processing (taking about 15 MJ/kg for iron and 50 MJ/kg for aluminum). After filtering, calcium and magnesium can be precipitated as white hydroxides above pH 10, though yields are low in our tests indicating incomplete dissolution, possibly as calcium silicate. What remains is a clear brine containing sodium, chloride, and any leftover ions picked up from the regolith (K, Li, etc).

Recycling: In space, all reagents must be either brought from Earth at great expense, extracted from in-space resources, or recycled. Regolith is effectively unlimited on the lunar surface. If we use sodium hydroxide as both the flux and alkali neutralizer, these sodium ions should all end up in the brine remaining after metal precipitation. From this NaCl brine, HCl and NaOH can be regenerated using a brine electrolysis method such as chlor-alkali, electrodialysis, or direct electrolysis, which are well-known industrial scale processes using under 8 MJ/kg of electricity [8].

Sodium is present in lunar plagioclase at up to 2% by weight [9], and so careful recovery and recycling may actually yield excess sodium for consumptive use such as float glass. Chlorine has only been detected at 20-200 ppm in lunar samples, so will need resupply from Earth unless recycling is excellent, or deposits are found. If we use large quantities of wash water to recover these ions from the various process precipitates, wash water can be converted to brine and clean water via either freeze-distillation using heat radiators, or vapor distillation using solar heating.

Conclusions: Solar melting regolith with sodium flux produces an acid-soluble glass that can be used to separate the elements in the regolith, and could be used as the first step in bootstrapping a civilization off Earth. Work is needed to quantify the extracted materials, but melting unlocks metals in crystalline minerals or impact glass, and acid leaching allows the separation of pure silica and a variety of metal oxides.

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