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Electrostatic Beneficiation of Lunar Simulant

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Abstract—Electrostatic beneficiation of lunar regolith is a method allowing refinement of specific minerals in the material for processing on the moon. The use of tribocharging the regolith prior to separation was investigated on the lunar simulant MLS-1 by passing the dust through static mixers constructed from different materials; aluminum, copper, stainless steel, and polytetrafluoroethylene (PTFE). The amount of charge acquired by the simulant was dependant upon the difference in the work function of the dust and the charging material. XPS and SEM were used to characterize the simulant after it was sieved into five size fractions (> 100 μm, 75-100 μm, 50-75 μm, 50-25 μm, and < 25 μm), where very little difference in surface composition was observed between the sizes. Samples of the smallest (< 25 μm) and largest (> 100 μm) size fractions were beneficiated through a charge separator using the aluminum (charged the simulant negatively) and PTFE (charged positively) mixers. The mass fractions of the separated simulant revealed that for the larger particle size, significant unipolar charging was observed for both mixers, whereas for the smaller particle sizes, more bipolar charging was observed, probably due to the finer simulant adhering to the inside of the mixers shielding the dust from the charging material. Subsequent XPS analysis of the beneficiated fractions showed the larger particle size fraction having some species differentiation, but very little difference for the smaller size. Although MLS-1 was made to have similar chemistry to actual lunar dust, its mineralogy is quite different. On-going experiments are using NASA JSC-1 lunar simulant. A vacuum chamber has been constructed, and future experiments are planned in a simulated lunar environment.

I. INTRODUCTION

Any future lunar base and habitat must be constructed from strong dense materials in order to provide for thermal and radiation protection. It has been proposed that lunar soil may meet this need, and sintering of full-scale bricks has been accomplished using lunar simulant [1]. However, in these experiments whole lunar dust as-received was used. Beneficiation of ores to an industrial feedstock grade may be more efficient. Lunar regolith has high concentrations of aluminum, silicon, calcium, magnesium, iron, manganese, sodium, and titanium oxides. Refinement or enrichment of specific minerals in the soil before it is chemically processed may be more desirable as it would reduce the size and energy requirements required to produce the virgin material and it may significantly reduce the process' complexity. Successful separation of lunar soil has been reported in an electrostatic separator where the refinement of ilmenite, up to 60% from other metal-bearing agglutinates, was achieved [2]. However, this was performed using a high-voltage electrode and in a nitrogen environment. To improve yield in a lunar environment, a vertical free-fall separator design was suggested due to the lower gravity on the moon [2,3].

With the method of electrostatic beneficiation described herein, powders are tribocharged by contact with materials of a different composition. Particles in the powder are charged with positive or negative polarities, depending upon their composition relative to the charging material. These charged particles can then undergo electrostatic separation in an electric field based upon their charge-to-mass ratio (Q/M). The efficiency of separation is dependant upon the powder's bulk and surface composition, as well as the fineness of the powder. Triboelectric separation of coal from minerals, quartz from feldspar, phosphate rock from silica sand, and phosphorous and silica from iron ore have been successfully achieved in laboratory experiments and pilot plant studies [4]. The fine granular regolith and lunar environment are ideal for triboelectrification and electrostatic separation, the lack of moisture prevents the regolith grains from sticking together, and the lower gravitational pull increases separation of the charged particles, all of which enhance mineral segregation.

Two lunar simulants MLS-1 and NASA JSC-1 were chosen for this study. However, in this paper, only the preliminary results for MLS-1 are reported. Both simulants were developed to replicate as close as possible the chemistry of lunar soil brought back from the Apollo missions [5,6]. They are principally basalts, containing phases of plagioclase, pyroxene, olivine, and ilmenite [5,6]. Lunar regolith is a powdery dust with a mean grain size range of 45 – 800 μm and characteristics similar to that of silty sand. It has low electrical conductivity and dielectric losses, permitting the accumulation of electrostatic charge [7].

The MLS-1 and JSC-1 were sieved to different grain size fractions (> 100 μm , 75-100 μm , 50-75 μm , 50-25 μm , and < 25 μm) and each fraction was analyzed using X-ray Photoelectron Spectroscopy (XPS), Secondary Electron Microscopy (SEM), and Electron Dispersive Spectroscopy (EDS) to determine mineral surface and bulk composition, speciation, and size distribution. The information obtained from these techniques was utilized

to help understand how different surface characteristics affect charge transfer during triboelectrification. An electrostatic separator was designed and tested and consists of two electrically conducting plates, angled towards each other such that the spacing between them decreases linearly. This allows highly charged particles to be collected at the top and particles with lower charge or mobility to be collected near the bottom (higher E-field); providing for more optimum collection efficiency. High voltages ranging from 5 to 30 kV of opposite polarity can be applied to each plate to attract the oppositely charged particles. The separator was designed to be used in both earth's atmosphere and under vacuum conditions.

Four static mixers of different materials were explored for the triboelectrification process; namely aluminum, copper, stainless steel, and polytetrafluoroethylene (PTFE). These materials were selected because they offer a wide variation in work functions (aluminum 4.28 eV, copper 4.65 eV, stainless steel 5.04 eV, and PTFE 5.75 eV) [8]. The difference between the work function of each material and the simulant influences the charge obtained by the grains. The resulting charge-to-mass ratio was expected to lead to variations in mineral separation and allow for optimization of the process. In this study, the static mixers were only used in ambient conditions to determine which material transferred the greatest amount of charge to the lunar simulant.

A vacuum chamber capable of reaching 8×10^{-7} torr pressure has been built, and a charge separator and powder tribocharging and dispersion system to work at this low pressure are currently under construction. The results using the NASA JSC-1 and separation data of both simulants taken at high vacuum will be reported later.

II. EXPERIMENTAL

XPS analysis of the sieved fractions of MLS-1 were conducted on a Kratos XSAM 800 Spectrometer at a background pressure of 1×10^{-9} torr, using a Mg K α ($h\nu = 1253.6$ eV) x-ray source. The x-ray beam used was 150 W and 4 - 6 mm in diameter. The collected data were referenced to the C1s peak to 284.6 +/- 0.5 eV. Wide survey scans were collected from 0 - 1100 eV at a pass energy of 80 eV in 1 eV steps with a 50 ms dwell time to determine overall elemental composition. The relative atomic concentrations of the detected elements were calculated and normalized to 100% using sensitivity factors supplied by the instrument manufacturer from known certified standards. The individual element spectra were converted to VAMAS ASCII format and imported to a computer where the peak curve fitting was performed using XPS International SDP v.4.1 data reducing software.

SEM images and EDS spectra of the sieved fractions were taken on a Zeiss EVO50 microscope equipped with an Oxford INCS EDS system. The SEM images were taken at magnifications of x100 at a beam energy of 5 kV. The EDS spectra were taken on individual grains within each size fraction at a beam energy of 20 kV.

To determine the amount of charge (Q/M) accumulated by the MLS-1 grains when contacted by various materials, the simulant was placed into a sealed powder cell where dry air at 15 psi and a flow rate of 6 l/min was

introduced. The simulant was blown by the air through different static mixers, constructed of aluminum, copper, stainless steel, and PTFE where it was collected in an electrometer-grounded faraday pail. The faraday pail was constructed with a removable inner section so the accumulated dust could be weighed. The simulant was blown through the various spiral-path static mixers for 30 seconds and the air flow turned off. The charge reading was allowed to steady before the inside section of the faraday pail was removed to determine the mass of dust. A minimum of five runs were performed for each static mixer. Between runs, the static mixer and faraday pail were cleaned thoroughly to remove any residual dust. For the separation experiments, the faraday pail was replaced with the charge separator. Figure 1 shows the charge separator set-up where the lunar simulant was passed through the static mixer into the charge separator chamber.

The charge separator was constructed of two copper-clad plates of dimensions 6" x 12" inclined at 4 degrees separated at the top by 3 inches. This geometry increases the forces acting on the particles as they move downward inside the separator. Particles with a lower charge then have an opportunity to be collected at the bottom of the plates. With parallel-positioned plates, lower charge particles might not be captured at all. The theory and design of this separator has been described elsewhere [9]. The plates are powered by positive and negative 30 kV Glassman MJ series 15 W regulated DC modules. For the separation experiments in this study, the plates were run at +15 kV and -15 kV. The dust accumulated on each plate and on the filter paper in the collection box at the bottom of the plates was weighed to determine the mass-fraction separated. Samples of the simulant collected on each plate were then analyzed by XPS.

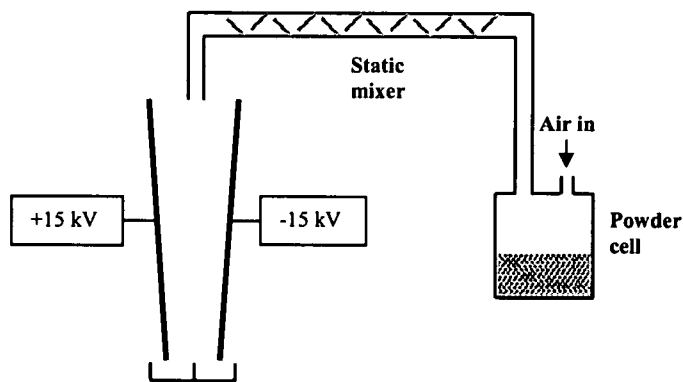


Fig. 1. Charge separator set-up in ambient.

III. RESULTS AND DISCUSSION

Figure 2 shows the SEM images for the five sieved fractions of the MLS-1 simulant. Spherical, as compared to ragged edged particles, were observed in each fraction, which suggests compositional differences between particles in each sieved fraction. A representative XPS spectrum for the simulant is presented in Figure 3 indicating the detected elements. The relative atomic concentration for each size fraction is presented in Table 1. Unfortunately, magnesium, a component of MLS-1, was not detected as the Mg2s and Mg2p peaks were too small and overlap with the Al2p and Si2p peaks. The mean relative atomic concentrations when converted to weight %, for all five size fractions, were very close to that reported for the bulk composition (within +/- 5%) [5]. It must, however, be emphasized that the sensitivity factors used to quantify the XPS data are for pure elements and not oxide forms, and therefore exact comparisons will have an inherent error. It was observed that there was little compositional variation between the sieved fractions except for a small increase in the carbon and silicon concentrations and the appearance of nitrogen for the smaller fractions.

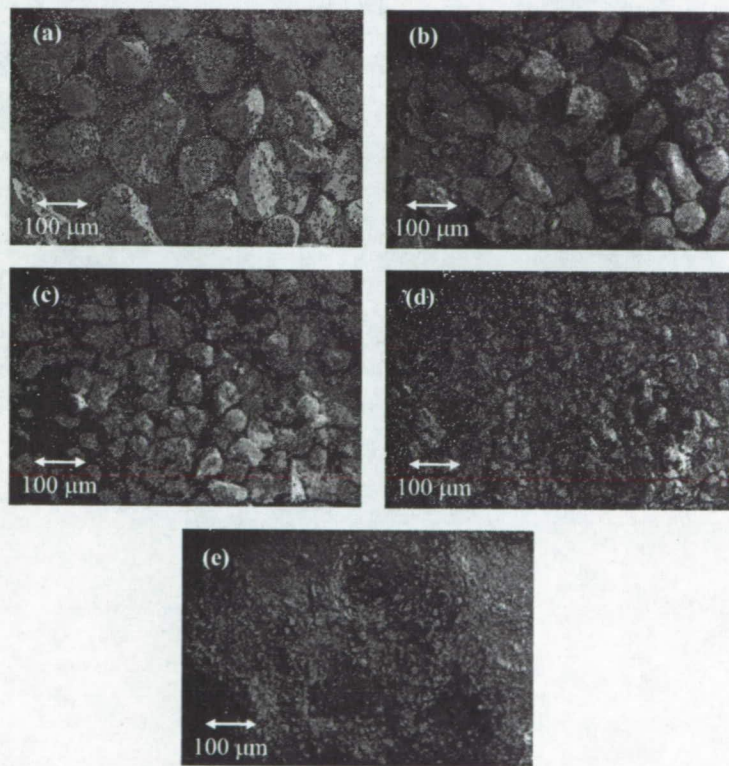


Fig 2. SEM images of the sieved fractions of the MLS-1 lunar simulant. (a) >100 μm , (b) 75-100 μm , (c) 50-75 μm , (d) 25-50 μm , and (e) <25 μm . x 100 mag.

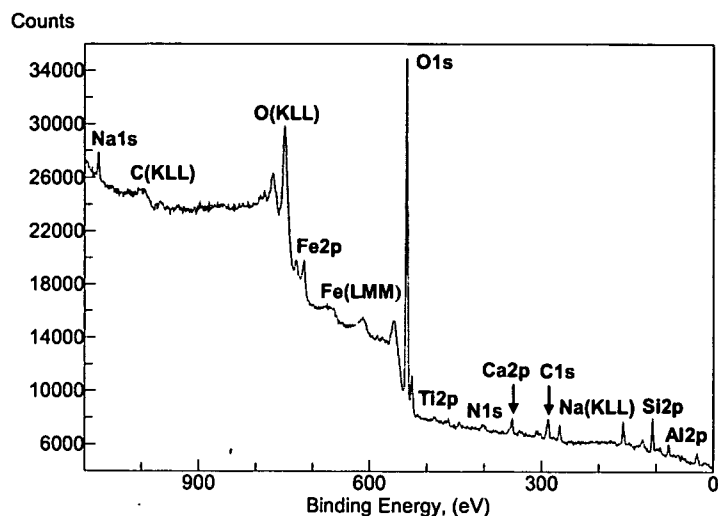


Fig. 3. XPS spectrum of MLS-1 lunar simulant.

TABLE 1. MLS-1 LUNAR SIMULANT SURFACE COMPOSITION MEASURED BY XPS [RELATIVE ATOMIC %] AS A FUNCTION OF SIEVED SIZE FRACTION.

Size range	O	C	Si	Al	Na	Fe	Ca	Ti	N
< 25 μm	59.7	13.1	12.6	5.6	3.2	3.0	1.6	0.6	0.6
25-50 μm	61.7	12.1	11.2	5.5	3.1	3.8	1.5	0.5	0.6
50- 5 μm	60.7	11.5	11.0	5.3	3.1	4.7	1.5	0.5	1.7
75-100 μm	64.8	11.2	10.3	4.7	2.5	4.7	1.3	0.3	-
> 100 μm	64.1	11.4	9.8	5.1	3.4	4.0	1.5	0.8	-

In the representative EDS spectrum for the simulant (Fig. 4), possible potassium and magnesium were additionally detected, but again, very little variation in composition was observed between the sieved fractions. EDS is a bulk composition analytical technique and therefore differences between the bulk composition as detected by EDS and the surface composition as detected by XPS are expected. For the most efficient tribocharging using static mixers, the particles must impact the baffles or spirals within the mixers and not simply contact deposited layers that may accumulate inside the mixer from finer powders. The flow must be sufficiently turbulent to re-suspend the larger particles that are deposited.

Impact charging is more efficient for larger particles, as smaller particles are likely to follow laminar flow, avoiding contact with the static mixer surface.

To determine the maximum charge that could be transferred to the simulant, the larger sieved fraction was used ($>100\ \mu\text{m}$). The results of the tribocharging experiments are presented in Figure 5. Against PTFE, the dust charged positively, while for the three metals, it charged negatively, in direct correlation between the acquired charge on the dust and the work function of the charging material. From the plot, the work function of the lunar simulant was estimated to be $\sim 5.4\ \text{eV}$. Sternovsky *et al.* [10] determined a work function of $5.8\ \text{eV}$ for JSC-1 lunar simulant in the $125\text{--}150\ \mu\text{m}$ range, but had also determined in earlier work that the effective work functions of Al_2O_3 and SiO_2 were $5.25\ \text{eV}$ and $5.5\ \text{eV}$ [11], which are the major components of MLS-1, so there appears to be good agreement. The large variation in acquired charge for each material is most likely due to irregular particle shapes, which allows charging to occur only on localized spots. Variation in the acquired charge also may be caused by the number of contacts the larger particles have with the charging material. Based on this data, beneficiation of the <25 and $>100\ \mu\text{m}$ size fractions was attempted using the aluminum and PTFE static mixers. The separated positive and negative mass fractions were weighed before samples were analyzed by XPS. The results are presented in Table 2.

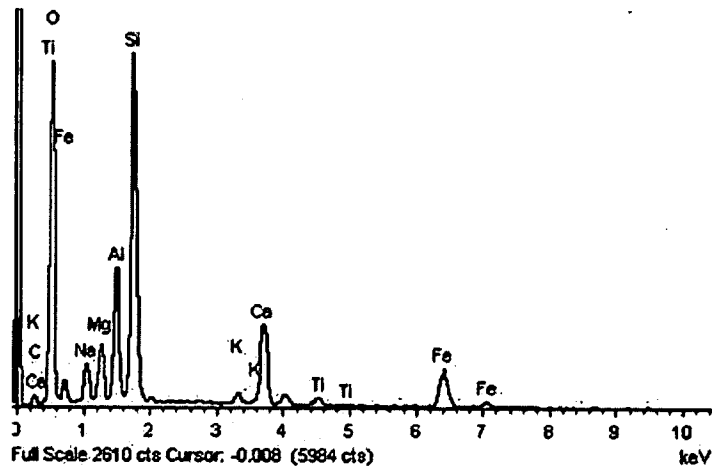


Fig 4. EDS spectrum of MLS-1 lunar simulant grain.

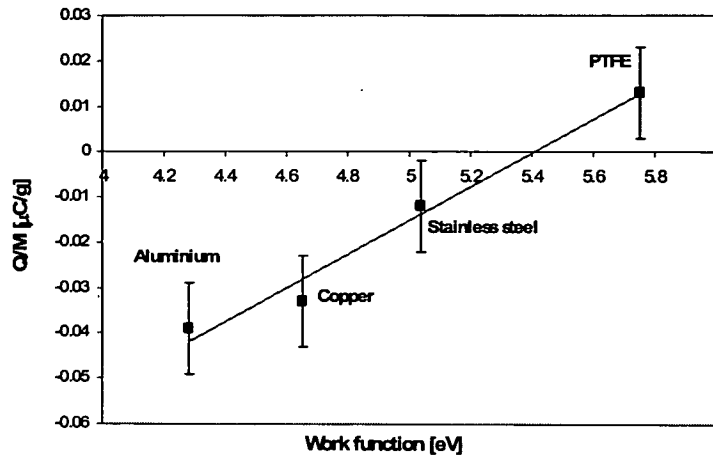


Fig. 5. Acquired charge-to-mass (Q/M) of MLS-1 lunar simulant as a function of the work function of the charging material; Al (4.28 eV), Cu (4.65 eV), SS (5.04 eV), and PTFE (5.75 eV).

TABLE 2. MASS FRACTIONS [%] AND ELEMENTAL COMPOSITIONS [RELATIVE ATOMIC %] FOR TWO MLS-1 LUNAR SIMULANT SIEVED FRACTIONS [<25 AND >100 MICRON] TRIBOCHARGED AGAINST ALUMINUM AND PTFE.

Charger	Plate	Mass %	O	C	Si	Al	Na	Fe	Ca	Ti	N
Al < 25 μm	+ ve	70	61.2	14.2	10.2	7.3	3.4	2.4	1.2	0.2	-
Al < 25 μm	- ve	30	56.6	19.8	9.2	6.9	3.2	2.3	1.0	0.3	0.7
PTFE < 25 μm	+ ve	52	62.6	10.6	12.0	6.1	4.3	2.4	1.5	0.6	-
PTFE < 25 μm	- ve	48	60.4	12.6	12.0	7.12	3.1	2.9	1.5	0.5	-
Al > 100 μm	+ ve	84	65.0	6.3	13.7	5.0	5.1	2.8	1.7	0.2	0.3
Al > 100 μm	- ve	16	64.1	8.1	13.0	5.5	4.1	2.9	2.1	0.1	-
PTFE > 100 μm	+ ve	14	65.4	5.1	14.3	5.4	4.2	3.5	1.8	0.3	-
PTFE > 100 μm	- ve	86	62.4	9.1	12.5	6.9	4.1	2.8	2.0	0.4	-

The mass fractions for the >100 μm particle size fractions showed that for the aluminum mixer, the simulant acquired a predominant negative charge (84%) compared to 16% positive, while for the PTFE mixer, it acquired a predominant positive charge (86%) compared to 14% negative. For the < 25 μm size fraction, although the simulant acquired a similar greater

negative charge (70%) for the aluminum mixer, the mass fractions were even for the PTFE mixer. This result was most likely due to the finer particles quickly coating the inside of the PTFE mixer and the remaining aliquot of simulant being charged against itself, resulting in bi-polar charging. The major chemical difference between the larger and smaller particle sizes is the significantly higher concentration of carbon in the smaller size fraction, and a higher concentration of silicon in the larger fraction, which affects the charge efficiency of the materials. There was no statistically significant compositional difference however, between the separated fractions for each size and charging material, which was expected. PTFE has one of the highest work functions of most materials, and so any simulant coming into contact with it will charge positively. This was observed by the high amount of simulant that was collected on the negative plate for the $>100\ \mu\text{m}$ size fraction. Similarly for aluminum, which has one of the lowest work functions, most simulant that comes in contact with it will charge negatively and migrate towards the positive plate, as also observed for the $>100\ \mu\text{m}$ size fraction.

To achieve separation of the various minerals by tribocharging, a material with a work function between that of the different minerals is required. The minerals in the lunar soil are predominantly silicates (feldspar), with various amounts of pyroxene, olivine and ilmenite. Although MLS-1 was made to have an approximate chemical composition to that of lunar soil, it has been suggested that the mineralogy does not reflect that of lunar conditions and is perhaps not a representative test material for tribocharged beneficiation [12]. For this reason, the newer NASA JSC-1 lunar simulant will also be tested. As described above, the surface chemistry of MLS-1 is similar to lunar soil, but the mineralogy is not, and hence mineralogy analysis of the separated simulant needs to be performed. Further separation experiments, including the copper and stainless steel tribochargers, on all sieved size fractions are also planned. Future work will focus on optimizing the tribocharging method in both ambient and vacuum conditions and using mineralogy analysis to determine mineral separation effectiveness.

IV. CONCLUSION

Lunar simulant MLS-1 was successfully and consistently tribocharged in air using static, spiral mixers of various materials. A direct correlation between the work function of the simulant and that of the charging material was established. Significant unipolar charging as well as positive and negative charging against PTFE and aluminum, respectively, was observed for the simulant size fraction $>100\ \mu\text{m}$. This was most likely due to the larger particle size not sticking to the internal walls of the static mixers as was observed for the smaller ($< 25\ \mu\text{m}$) size fraction. The twin plate separator proved successful in separating simulant by charge with full recovery of the material. It is predicted that the lower gravity on the moon will assist in the separation due to the longer fall times. As there is no atmosphere on the moon, stronger electric fields can also be used for more efficient beneficiation. The preliminary data presented herein indicates that tribocharging lunar simulant followed by a twin plate charge

separator is potentially a viable method for mineral separation and enrichment of lunar soils.

ACKNOWLEDGMENTS

The authors would like to thank the NASA Kennedy Space Center Spaceport Engineering and Technology Directorate for Center Director Discretionary Funding for this research.

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