Extracting Respirable Particles from Lunar Regolith for Toxicology Studies

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ABSTRACT

The Lunar Airborne Dust Toxicity Assessment Group (LADTAG) is working to determine the permissible limits for exposure to lunar dust. This standard will guide the design of airlocks and ports for EVA, as well as the requirements for filtering and monitoring the atmosphere in habitable vehicles and other modules. Rodent toxicity testing will be done using the respirable fraction of actual lunar soils (particles with physical size of less than 2.5 micrometers). We are currently separating this fine material from the coarser material that comprises >95% of the mass of each soil sample. Sieving is not practical in this size range, so a new system was developed for this task. Collection and separation efficiencies are tracked as development and tests proceed. LADTAG's recommendation for permissible exposure limits will be delivered to the Constellation Program in 2010.

LADTAG RESEARCH

During the Apollo missions, crewmembers were briefly exposed to dust in the lunar module, brought in after extravehicular activity. When the lunar ascent module returned to micro-gravity, the dust that had settled on the floor now floated into the air and caused eye discomfort and occasional respiratory symptoms. When the dust was bothersome, the crew donned their helmets and waited for the air revitalization system to remove the dust.

Because our goal is to set an exposure standard for 6 months of episodic exposure to lunar dust (presumably after each EVA), the brief astronaut exposures of a few days are not conclusive. Based on experience with industrial minerals such as sandblasting quartz, an exposure of several months may cause serious damage, while a short exposure may cause none.

WHY RESPIRABLE PARTICLES MUST BE SEPARATED PRIOR TO TESTING

The equipment that is used for inhalation toxicology studies is designed to accommodate particles of respirable size. Larger particles mixed in with the smaller ones would block the flow lines used in the animal tests, and the tests would not be directly comparable with published data on other kinds of respirable material, used in silicosis and nanoparticle toxicity studies. Thus it is necessary for us to separate out the respirable fraction of lunar dust (particles less than 2.5 μm diameter) from a bulk sample of lunar regolith. We build on the initial results reported by (Liu, Schnare et al. 2008) on dry separation of the respirable fraction of lunar soil.

DRY SEPARATION IS REQUIRED

Surface activation of lunar dust: Lunar dust *in situ* is subjected to several processes that would cause grain surfaces to become chemically reactive, increasing its toxicity. Lunar dust is the product of repeated fracturing and comminution from meteorite impacts. This produces fractured surfaces that are likely to have unsatisfied chemical bonds. Pressure pulses from impact can produce shock effects, dislocations, and phase changes in the grain surface material. In addition, individual particles are subject to intense UV radiation which is energetic enough to break additional bonds and thereby increase the potential reactivity of grain surfaces. Regolith grains are also subject to particulate radiation from solar wind and solar flare events which can damage the surface and near-surface regions down to several 10s or even 100s of nanometers. The result is that the dust grain surfaces and outermost regions may have enhanced reactivity with human tissue. The detailed characteristics of sub-micrometer lunar dust are only poorly known, and this is the size range of particles that are of greatest concern.

In addition, lunar dust contains metallic nano-size iron. This is due largely to vaporization of the soil induced by micro-meteorite impact, with subsequent deposition of the vapors as impact glass filled with myriads of nanophase metallic Fe grains (3-33 nm). Some native iron may also be formed by hydrogen-reduction (solar-wind protons) of this same melt. The effect of this nanophase iron on human tissue and cells is yet to be determined; such material does not occur on Earth and humans have not developed any specific evolutionary ability to deal with it.

Our previous work has shown that lunar dust particles are likely to be highly reactive (Wallace, Taylor et al. 2009). Grinding of lunar simulant can create a similar reactivity, which is greatly reduced ("passivated") over the course of a few hours by contact with humidity and atmospheric oxygen (Wallace, Taylor et al. 2009). As our objective is to determine the native reactivity of lunar dust, we must minimize any changes in reactivity caused by the size separation processing. Because of this potential strong reaction with water, and possibly other fluids, the separation of lunar dust must be done either in vacuum or in a dry inert gas atmosphere. We have chosen to perform all separation procedures in an ultrapure dry nitrogen atmosphere.

DUST SEPARATION SYSTEM

Historically, there have been two main reasons to separate particles—either to measure the particle size distribution, or to clean a gas stream. Both of these motivations have resulted in mechanisms that can be used to separate particles for inhalation toxicology studies. However, because lunar dust is a priceless national treasure, we cannot afford to use a method that results in loss of material. Our dust separation system incorporates some of the traditional methods, while preventing the dust from being contaminated or changed in reactivity properties, and also minimizing losses. The system as it operates inside a nitrogen cabinet is shown in Figure 1, and shown schematically in Figure 2.

Sieving was Not Included: Among the methods that use particle separation for measurement of particle size distribution; sieving is a well-known example (Allen 2003). Techniques exist for both dry and wet sieving, with wet sieving having the advantage of producing a cleaner particle separation (McKay, Fruland et al. 1974). Sieving is not feasible for our purposes, however, because the smallest separation practical with a sieve is 10 μm.

Figure 1. Dust separation system inside a nitrogen glove box at NASA Johnson Space Center's lunar sample curatorial facility.

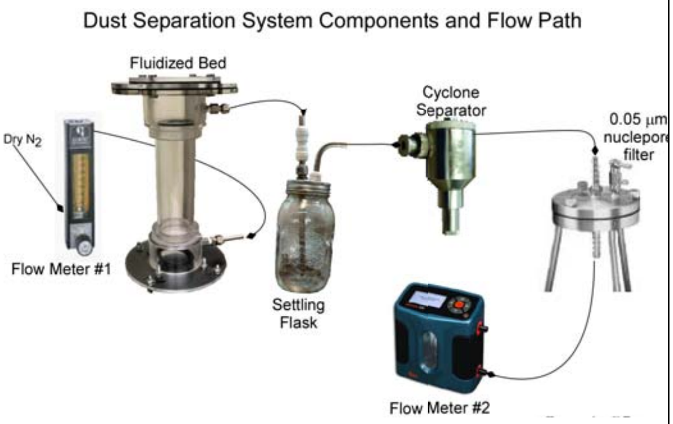

Figure 2. Dust Separation System components and flow path.

Because dry sieving is not very effective at releasing the respirable dust particles that are adhered to larger grains, we selected a fluidized bed as the first stage in our system. The fluidized bed thus becomes an enhanced elutriation system (Allen 2003).

Fluidized Bed: Fluidized beds are most commonly used for chemical processing (Kunii and Levenspiel 1991), but the principal of operation makes them a useful option for a dust separation system. The "bed" is the mass of particulate material which is contained by the system. Fluidization refers to the fact that gas (or liquid) passes through the material from below, causing the particulate matter to behave like a fluid. The gravitational pull on the particles is offset by the fluid drag that pulls them upward, and the particles remain in a semi-suspended condition.

Our fluidized bed was developed at JSC for use as an *in-situ* resource utilization (ISRU) demonstration unit. Modifications to the original component include the addition of a cup-shaped filter holder at the bottom, which prevents material from being caught in the corner space between the bottom of the bed and its floor. Previous experience had shown us that these "dead" spots would eventually lead to channeling, in which fluidization stops and the gas forms a channel to the top of the bed (Cooper 2008). The modification has resulted in continuous fluidization behavior for the entire duration of four hours used in separation runs.

Settling Flask/Impactor: Following the fluidized bed is a settling flask, in which the input is directed via a tube to the bottom of the flask. The settling flask presents a large volume to the air stream of the separation system. The sudden expansion of size at the flask reduces the speed of the dust-filled airstream and thus heavier particles will settle out. Smaller particles will remain in the air stream and flow around the tube. These smaller particles then drift upwards in the air stream to the top of the flask, and are carried out to the next component—the cyclone.

Cyclone: A cyclone separator consists of a cylindrical shell with a tangential inlet through which dusty gas enters, an exit pipe for discharging the processed gas, and a conical base for discharge of oversized particles. A dual vortex is created inside the cyclone because of its geometry, and this separates coarse soil from fine dust. The main vortex spirals downward and carries most of the coarser dust particles. The inner vortex, created near the bottom of the cyclone, spirals upward and carries finer dust particles (Stairmand 1951). The cut size (the size of particle that is passed through the exit pipe) is a function not only of the design of the cyclone, but also of the gas flow rate. Because gravity plays only a small part, a non-intuitive result is obtained in which lower flow rates release larger particles through the exit, while higher flow rates pass smaller particles.

Filter: The membrane filter is the final component in the system, and is the point at which the product dust is collected. Filters are one of the most efficient methods of separating dust from a gas stream, with collection efficiencies of more than 99% for very fine particulates.

Cellulose or paper filters cannot be used because (a) it is difficult to remove the dust from them after collection; and (b) scraping material from this type of filter results in fibers in the dust. Instead we use a membrane filter with a pore size of 0.05 μm. We have found these filters to be robust—a single filter is used throughout a four-hour system operation.

In spite of their high efficiency in capturing dust, filters do not perform well in separating various particle sizes from one another—all particles of almost any size are trapped on the filter. This is why the filter is used only at the end of the separation system, after we have removed the oversize particles from the gas stream.

WORKING WITH ACTUAL LUNAR SOIL

Obtaining actual lunar soil for testing: Because there is no simulant that mimics every property of lunar soil, it is necessary to use lunar material for our final test. Available lunar simulants do not take into account the possible effects of nanophase iron (Wallace, Jeevarajan et al. 2009), solar wind impingement, or galactic cosmic ray bombardment. Tests on fresh lunar soil from the lunar surface would be ideal. Lacking this, Apollo lunar samples that have been carefully preserved in nitrogen are the most realistic experiment that we can make.

It is essential that the samples we acquire have not been subjected to fluids or to any other contamination. We identified three highland samples currently in the Returned Sample Vault that should yield sufficient material to complete the studies we have planned. The Returned Sample Vault houses samples that have been issued to various researchers and then returned (as per requirements). These samples are somewhat easier to obtain (compared to pristine lunar material) from the Curation and Analysis Planning Team for Extraterrestrial Materials (CAPTEM), who reviews requests for lunar samples (Allen 2008).

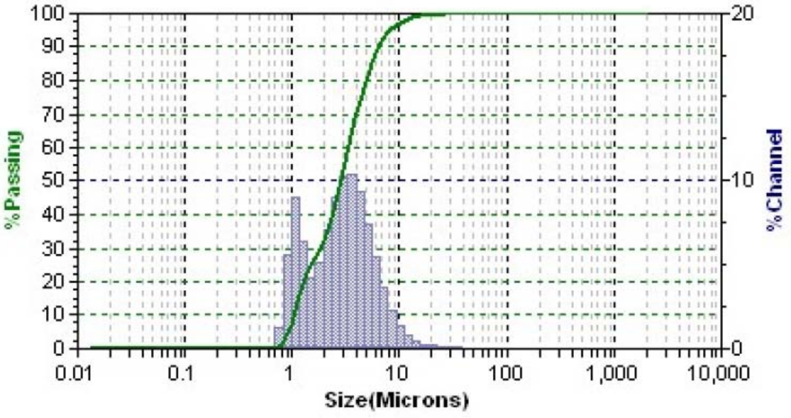

Figure 3. Particle size distribution of ground JSC-1A. Samples were ground with a Sturtevant 2" Micronizer™ jet mill. Median grain diameter = 2.855 μm; mean = 3.46 μm, standard deviation = 2.083 μm.

Amount of lunar soil needed to obtain sufficient dust for our study: Based on published data, we estimate that 1-3% by mass of typical mature lunar soils are in the respirable size range. To perform a set of statistically valid rodent exposure experiments, at least 23 grams of respirable dust is required. If all of this material were derived from size fractionation of native lunar soil, about 1.15 kg would be required as starting material. This much lunar soil is simply not available from the present Apollo collection. We therefore had to modify our test protocol to tests using a more limited amount of natural respirable lunar dust, with additional tests using lunar dust produced by grinding of coarser material down to the appropriate size. Our modified protocol would therefore require about 5.5 gm of natural lunar dust and 17.5 gm of ground dust.

We have evaluated a number of methods for grinding lunar soil and have chosen a jet-mill, in which the soil is entrapped in intersecting gas (pure, dry nitrogen) streams which cause the material to be impacted and ground by collisions with itself. Contamination by non-lunar grinding media is minimized by this process. By using ground dust (Figure 3) in conjunction with natural dust (Figure 4), we have reduced our requirement for lunar soil from 1.15 kg to 260 grams. Because this amount is still considerably larger than most lunar soil allocations, it was necessary for us to assure CAPTEM that (a) we have done all prerequisite proving of the system; and (b) the experiments outlined above are both necessary and sufficient to recommend an exposure limit for crews on the lunar surface. CAPTEM approved our request and the testing will be done using the 260 gm allocation. After first extracting the respirable native dust from this total allocation, a sufficient amount of the remaining coarser dust will be ground to produce the total requirement of approximately 23 gm of respirable dust.

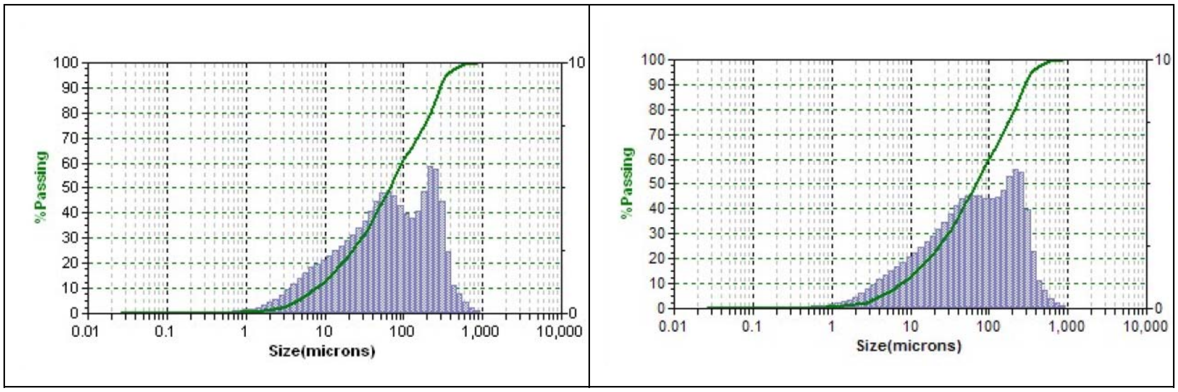

Figure 4. Particle size distribution of lunar soil 10084,2005 measured with the Microtrac™ laser diffraction instrument. The graphs represent two subsamples, each approximately ten milligrams. In these measurements, 2.08% and 1.58% respectively of the volume of the (sub millimeter) sample is 2.0 μm or smaller.

To validate the estimate of 1-3% of lunar soil in the respirable fraction, we measured the particle size distribution of lunar soil 10084,2005 with a Microtrac™ laser diffraction particle size analyzer (Figure 4). Our results, along with those of previous workers (Duke, Woo et al. 1970; King, Butler et al. 1971; Basu, Wentworth et al. 2001), are shown in Table 1. Although all of the measurements resulted in a median diameter between 55.1 and 67.96 μm, there is a much larger spread of values for the mean diameters. Our mean diameters for these measurements are significantly larger than previous sieve results, suggesting that some sample agglomeration was occurring during the diffraction-based analysis (McKay, Cooper et al. 2009). However, the less-than-ten μm fraction in our two measurements accounts for 18.3% and 22.2% of the total volume of the sample—a higher value than previously measured by sieving. In any case, our measurements show that this lunar sample has on the order of 1.8% by volume respirable (2.5 μm or smaller) dust, validating our original estimate that 1-3% would be in this size range.

From Table 1 it can be seen that the lunar simulant JSC-1A has so little material in the respirable size range that it is difficult to perform meaningful separation tests with it. After some initial testing with JSC-1A to establish proof of principle, we began using JSC-1AF, a standardized, more fine-grained subset produced from JSC-1A (James Carter, pers. communication). The amount of material in the less-than-10 μm and less-than-2 μm bins of JSC-1AF more closely resembles the grain size distribution of the finer fractions of lunar soil 10084.

Table 1. Measurements of subsamples of Apollo 11 lunar soil 10084, and comparison with simulants.

Source	**Sub-sample No.**	**Median**	**Mean**	**Less Than 10 μm**	**Less Than 2 μm**
Duke et al. (1970)	79	61.64	85.38	6.4 %	n.d.
King et al. (1971)	79	55.67	52.0	9.2%	n.d.
Basu et al. (2001)	n.d.	55.1	51	14.2%	n.d.
Recent #1 (2007)	2005	66.49	117.0	18.3%	2.08%
Recent #2 (2007)	2005	67.96	115.5	22.2%	1.58%
JSC-1A	*n/a*	*95.2*	*145.0*	*3.2%*	*0.05%*
JSC-1AF	*KA0706,33*	*24*	*28*	*12.4%*	*1.86 %*

CONCLUSIONS

We have developed a method for extracting respirable dust (<2.5 μm) from Apollo lunar soils. This method meets stringent requirements that the soil must be kept dry, exposed only to pure nitrogen gas, and must conserve and recover the maximum amount of both respirable soil and coarser soil. In addition, we have developed a method for grinding coarser lunar soil to produce sufficient respirable soil for animal toxicity testing while preserving the freshly exposed grain surfaces in a pristine state. Grain size analyses of Apollo 11 soil 10084 by a laser diffraction technique shows that this soil contains roughly 2% by mass in the respirable grain size, in agreement with our prior estimate. While useful for other purposes, lunar simulant JSC-1A does not contain sufficient mass in the finest fraction (<10 μm) to provide a good analog for testing our size separation method. Similar measurements of lunar simulant JSC-1AF show that it is a suitable simulant in the finest size fractions and is appropriate for our uses.

The method that has been developed to separate lunar dust of respirable size can be used for many other purposes, and can be modified to handle larger or smaller quantities of material. The system could also be modified to collect a different size fraction. Follow-on uses for this system include lunar ISRU for hydrogen extraction (Carter 1984) and terrestrial waste clean-up methods (Cuero and McKay 2007).

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