Beneficiation of Stillwater Complex Rock for the Production of Lunar Simulants

D.L. Rickman
Marshall Space Flight Center, Huntsville, Alabama

C. Young
Metallurgical and Materials Engineering, Montana Tech, Butte, Montana

D. Stoeser
USGS, Denver, Colorado

J. Edmunson
Jacobs ESSSA Group, Huntsville, Alabama

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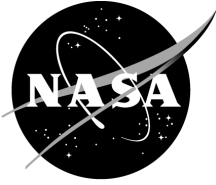
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LIST OF ACRONYMS AND SYMBOLS

Al	aluminum
Ca	calcium
CMC	carboxymethyl cellulose
Fe	iron
H_2O	water
Mg	magnesium
MLA	mineral liberation analysis
Na	sodium
O	oxygen
OH	hydroxyl ion or radical
RE	Rare-Earth (magnetic belt)
SEM	scanning electron microscopy
SMC	Stillwater Mining Company
TM	Technical Memorandum
USGS	United States Geological Survey
WHIMS	Wet, High-Intensity Magnetic Separator
XRD	x-ray diffraction
XRF	x-ray fluoresence

TECHNICAL MEMORANDUM

BENEFICIATION OF STILLWATER COMPLEX ROCK FOR THE PRODUCTION OF LUNAR SIMULANTS

1. INTRODUCTION

Simulation of the lunar regolith is technically problematic once the utility of milling a single terrestrial source is exceeded. In addition to particle textures, there are several compositional reasons for this. For example, high calcium plagioclase, which is common on the Moon, is rare in bulk terrestrial rocks. The ratios of specific minerals and their exact compositions, as found in the lunar regolith, are not found in terrestrial sources. Most terrestrial rocks contain significant amounts of associated hydroxyl or water bearing mineral phases, whereas these are absent or rare on the Moon. Minerals, such as quartz, clays, albite, zeolites, and amphiboles, are common in terrestrial sources, but these minerals and their associated fabrics are not useful and may be significantly detrimental in emulating lunar prototypes.

One way to address such problems in the creation of a simulant is to separate terrestrial source rocks into constituent mineral fractions. This is a technology that has been developed for many centuries[1] and is essential for the mining industry. For simulant production, this would permit extraction of desired mineral phases from different source rocks into mineral separates while removing undesired minerals. Each mineral separate would be a feedstock to subsequent production of a needed simulant. The result would be easier manufacturing of higher fidelity simulant.

The separation of a rock into its constituent minerals is a complex scientific and engineering problem. It combines economic factors, details of chemistry, applications of physics, and the specifics of the rock being processed. Therefore, for every mineral one wishes to extract from a specific rock, it becomes necessary to test possible separation techniques. This Technical Memorandum (TM) addresses a series of separation tests using source rocks from the property of the Stillwater Mining Company (SMC), specifically their operations near Nye, Montana. Other tests using dry magnetic separation are reported in Rickman et al.[2]

2. STILLWATER COMPLEX

A good general overview of the Stillwater Complex is by McCallum.[3] The Complex is a classic example of a layered mafic intrusive and formed by processes similar in many respects to those that formed the lunar crust.[4,5] For this reason, a large body of literature exists that compares various aspects of Stillwater petrology, mineralogy, and geochemistry to that of primary lunar crustal rocks (e.g. Raedeke and McCallum[6] and Salpas et al.[7]). In its well defined layers, the Stillwater Complex contains many rock types similar to the lunar upper crust (see fig. 1) which makes them a good resource for feedstock in the manufacture of lunar regolith simulants. Many of the Stillwater rock types are exposed as outcrops, but only a few are accessible from roads. Other types come from the activities of the SMC at their Nye and East Boulder mines as they excavate the J-M Reef material. The J-M Reef is a narrow zone (<3 m wide) located in the lower banded series in which extremely fine-grained, platinum group minerals are present in sulfides. This sulfide zone is the ore that is mined and processed for economic gain. Millions of tons of ore are processed annually. First, the rock is ground such that most of the particles are smaller than 75 μm, a very fine sand. Then mineral separation is achieved using froth flotation. The valuable, platinum-bearing, sulfide minerals report to the floated fraction and are thus concentrated. The gangue minerals, the nonfloating fraction, are disposed of as waste in the mine or to the tailings. In addition, waste rock must also be removed by mining in order to access the ore. This rock is discarded into piles referred to as waste dumps. The bulk of both the tailings and the waste dumps is a plagioclase-rich, noritic gabbro.

Lunar regolith simulant can be produced from not only the outcrops that are accessible from nearby roads, but also from the mill tailings and waste dumps produced by SMC. Each of these resources are discussed in the ensuing sections.
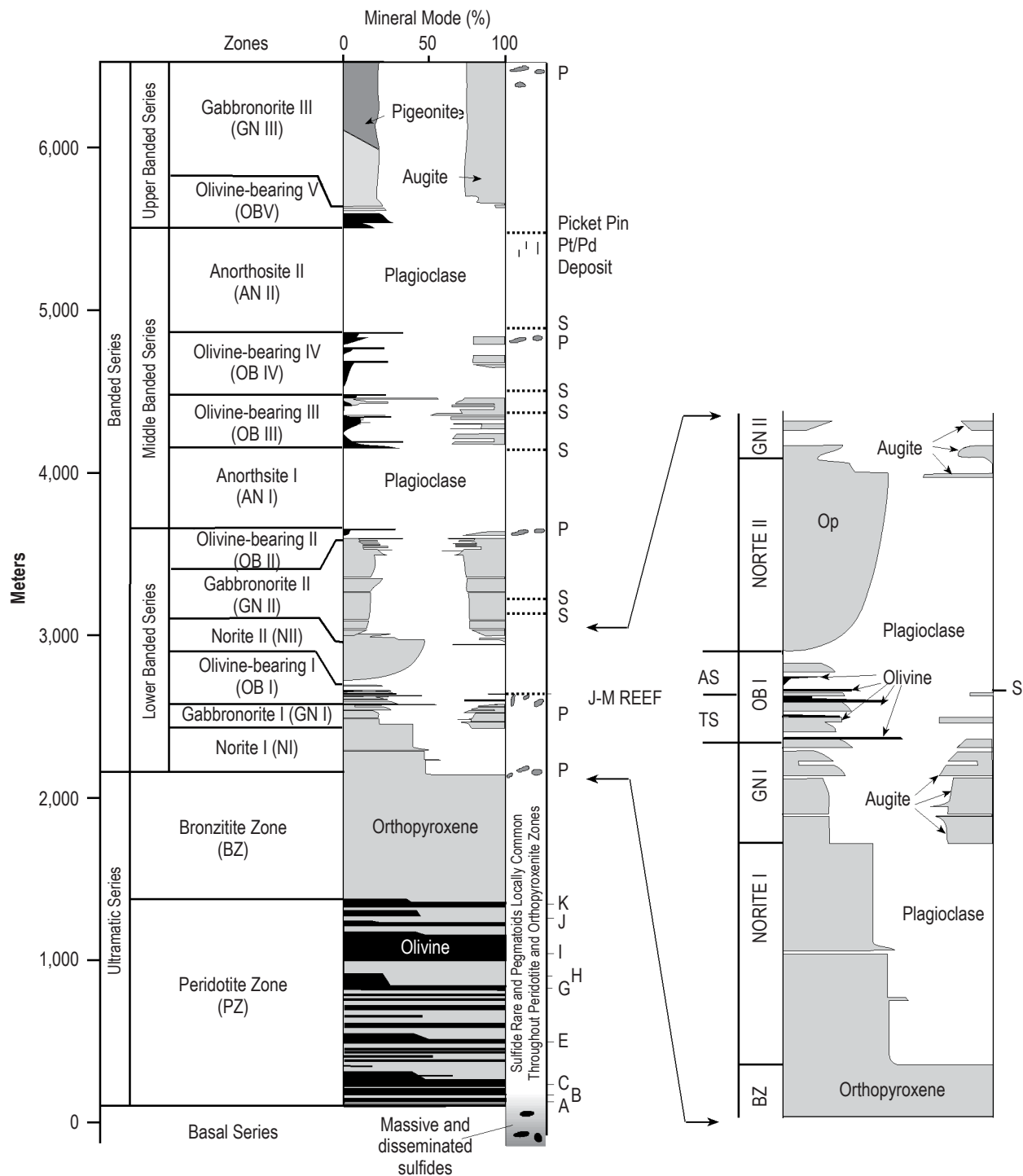
Figure 1. Composite stratigraphic section from Boudreau[8] showing zones and modal mineral stratigraphy (cumulus minerals only): S denotes sulfide-enriched zones, A–K denotes the major chromites, P denotes podiform concentrations of sulfide of limited lateral extent, and J-M Reef marks the platinoid-rich zone being mined.

2.1 Waste Dumps

Rock in the waste dumps is predominantly norite, but also contains gabbronorite, clinopyroxene gabbro, altered troctolite, anorthosite, and orthopyroxenite. The abundance of each rock type varies almost hourly, depending on the development of the mine. The dump material varies from very fresh to very altered. Polovina et al.[9] note that, in their sample of seven drill cores, hydrothermal alteration near the J-M Reef varied from 10% to 90%. Alteration minerals identified by D. Stoeser and S. Wilson of the USGS in waste dump rock consisted mainly of hydrated silicates including chlorite, albite, epidote, zoisite, talc, sericite, amphibole, serpentine, magnetite, stilbite, and calcite. As hydrothermal alteration in the J-M Reef rock is spatially associated with and probably related genetically to sulfide deposition, it has received some attention in the geological literature. McCallum gave brief statements on alteration in various parts of the Complex.[3] Any bulk use of dump materials will unavoidably include a significant amount of low-grade altered rock. A study has been initiated to characterize the variability of the waste dumps, but this has not yet been completed. Only modest amounts of high-quality material can be hand-picked off the dumps; doing so is difficult due to a thick layer of rock dust covering the surfaces of all stones.

2.2 Outcrops

Useful outcrop sources for feedstock are primarily restricted to exposures in road cuts and float below outcrops. A comparatively good road, shown in figure 2, provides access to the Mountain View chromite mine, which is owned by the Mouat family. The road crosses the anorthosites of the Middle Banded Series, the Lower Banded Series, and provides access to the Bronzitite and Peridotite Zones of the Ultramafic Series. Cuts along this road have been repeatedly used for two simulant feedstocks, a norite and an anorthosite. Rock available at and above the Mountain View main portal have also been used for simulant development. These include harzburgite, orthopyroxenite, and chromite ore.
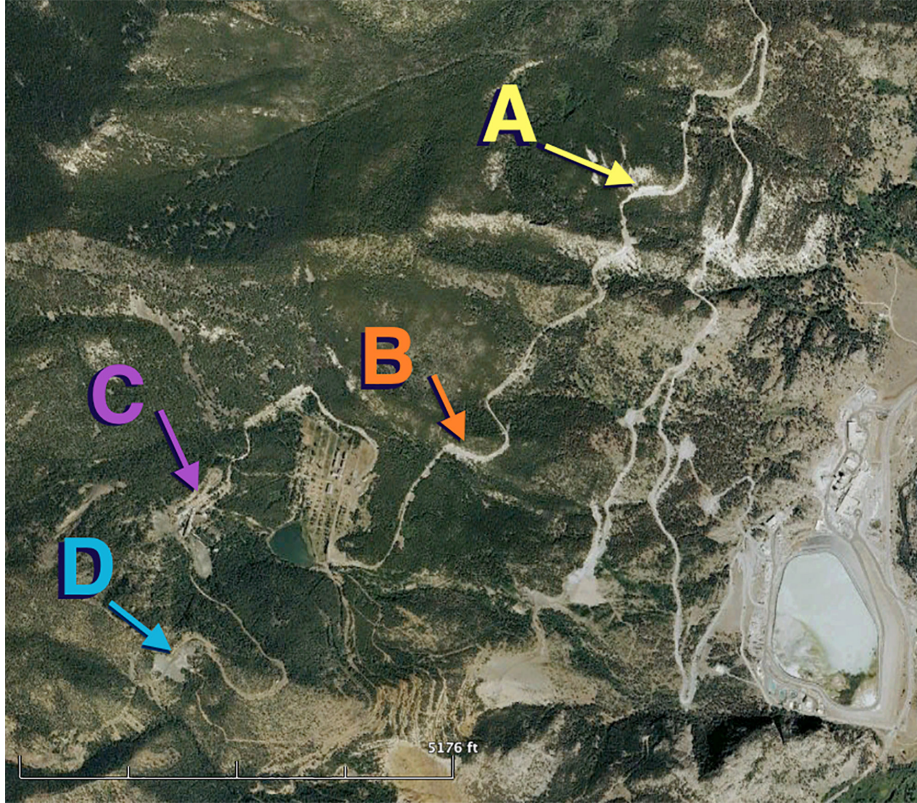
Figure 2. Surface sample locations of material used in simulant development: A–anorthosite, B–road norite, C–harzburgite, and D–othropyroxinite. Chromite ore was collected from bulk sample piles adjacent to point C (image © 2013 Google and USDA Farm Service Agency).

The material informally termed 'road norite' was obtained in a road cut below the Mountain View Mine, at 45.390208°, –109.893324°. Road norite is relatively unaltered by hydrothermal processes (see fig. 3). However, the amount of alteration and the ratios of the major minerals varies within the road cut. Careful selection of individual rocks from this site by senior geologists involved in the simulant development project provided the single largest constituent in the NU-LHT series of simulant prototypes.
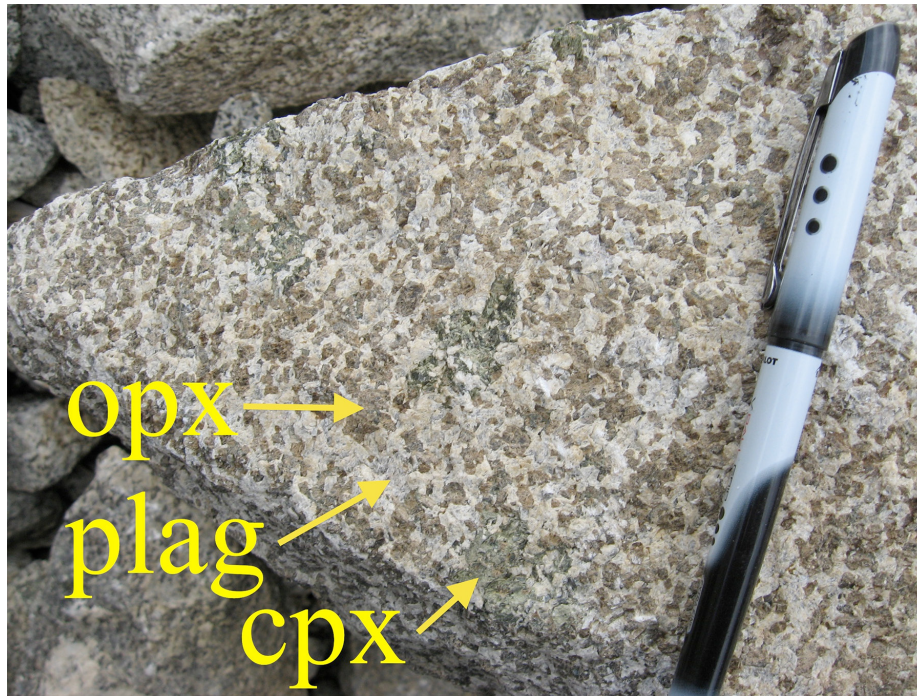
Figure 3. Hand sample of the road norite in a lightly weathered surface. The light-colored material composing ~60% of the rocks is plagioclase with a composition of ~An_{85}. The medium brown crystals are orthopyroxenes; the rich green masses are clinopyroxene. Rickman et al. listed typical compositions of the pyroxenes.[2]

Table 1 gives an analysis of road norite from hand-picked samples.[2] The ratio of the two pyroxene minerals (opx/(opx + cpx)) is 87.3. The minerals quartz, albite, calcite, biotite, epidote, and a potassium feldspar were found in an analysis of the simulant NU-LHT-2M;[10] although not reported in table 1, their likely source is the road norite. These six minerals, as well as the chlorite and talc, are loosely considered to have originated by hydrothermal alteration and their abundance varies noticeably within the road cut.

Table 1. Mineralogy of the road norite from hand-picked samples (after Rickman et al.[2]).

Mineral		Fitted Composition	Wt.%
Family	Specific Mineral		
Plagioclase	Bytownite	$(Ca_{0.86}Na_{0.14})(Al_{1.86}Si_{0.14})Si_2O_8$	75.0
Orthopyroxene	Enstatite	$Mg(Ca_{0.054}Mg_{0.946})(Si_2O_6)$	18.4
Clinopyroxene	Diopside (Augite)	$CaMgSi_2O_6$	2.7
Chlorite	Clinochlore IIb	$Mg_{4.54}Al_{0.97}Fe_{0.46}Mn_{0.03}(Si_{2.85}Al_{1.15}O_{10})(OH)$	1.2
	Talc 2M	$Mg_3(OH)_2(Si_4O_{10})$	1.9
	Hornblende	$Na_{0.9}K_{0.4}Ca_{1.6}Mg_{2.8}Fe_{1.4}Ti_{0.5}Al_{1.4}(AlSi_6O_{23}(OH))$	0.8

The locations used for the harzburgite, orthopyroxenite, chromite ore, and anorthosite are also shown in figure 2. The harzburgite came from 45.383269°, –109.907372°. The orthopyroxenite came from 45.388833°, –109.905966°. The anorthosite came from 45.398638°, –109.885209°. The harzburgite was used in NU-LHT-1M, providing olivine, pyroxene, and chromite. For NU-LHT-2M, commercially available olivine was used, as it was essentially pure olivine instead of the harzburgite.[11] The orthopyroxenite was not used after NU-LHT-1M, as the norite provided sufficient pyroxenes for the target simulant composition and the commercial olivine contains lower amounts of alteration minerals. Further, the ratio of clinopyroxene to orthopyroxene in the orthopyroxenite is even further from the targeted ratio than the ratio that exists in the norites. Because additional chromite was not needed in the simulants, the chromite collected from the bulk sampling dumps was not used.

Stillwater anorthosites were used in all of the NU-LHT series simulants. Much of it came from one road cut, indicated in figure 2 as anorthosite and shown in figure 4. The anorthosite consists of approximately 90%–95% plagioclase with the remainder of the primary (meaning original) minerals in the rock being poikolitic clinopyroxene (augite) up to 5 or 6 cm in diameter (see fig. 5). Where present, the clinopyroxenes enclose earlier cumulate plagioclase crystals. The plagioclase was quite homogeneous in composition with an An content of ~78. Locally within this site, there are planes of intense alteration (see fig. 6). All samples larger than ~10 cm on a side have at least some hydrothermal alteration. But careful hand selection yields a feedstock that is estimated to be better than 95% pure primary minerals.

Figure 4. Road cut in the anorthosite I zone from above the Stillwater Mine. This was used as a feedstock for LHT-1M and -2M. Dark rock to the left of the contact is troctolite and further to the left, the more blocky rock, is a zone of norite. Note the reddish material in the anorthosite is one of many alteration mineral assemblages (localized along fractures) in the anorthosite.
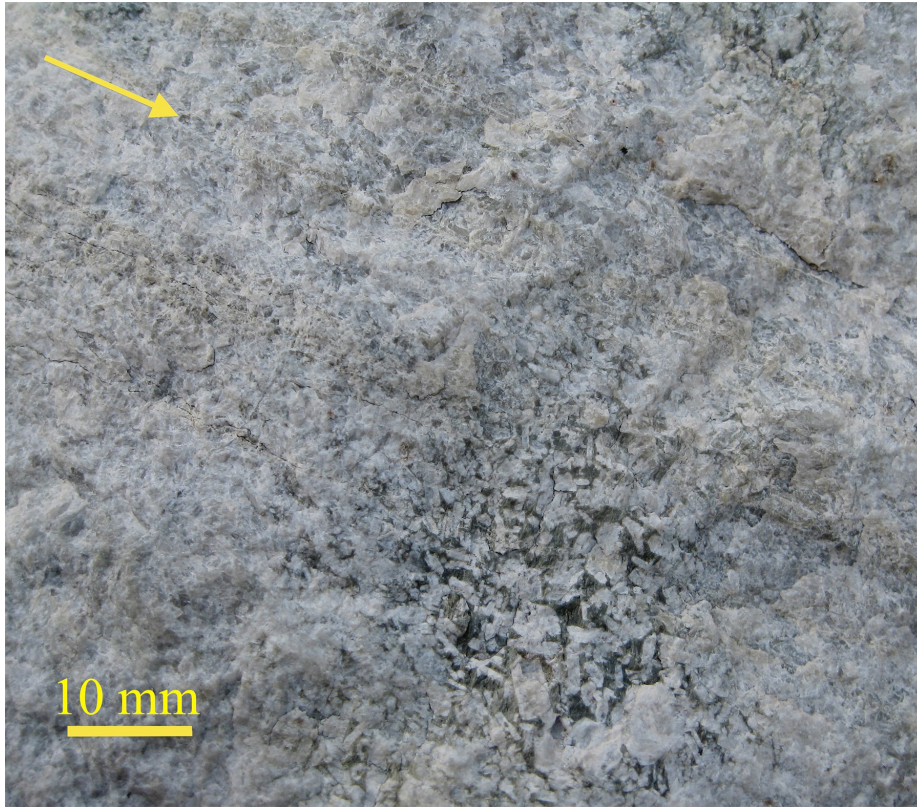
Figure 5. Detail of anorthosite in road exposure. Dark green in the lower center is clinopyrox-
ene, which has grown around euhedral plagioclase crystals. The yellow arrow points
along a fracture line on which there is alteration. In this photo there are at least five
such fracture lines, which are almost parallel.

Figure 6. Alteration zones in anorthosite. Two subparallel zones of intense hydrothermal alteration, 30–40 cm wide, can be seen running from lower left to middle right.

2.3 Tailings

The Stillwater tailings from the mill is a waste material commonly called mill sand. It is the lithic residuum left over from processing the Stillwater ore after the valuable minerals are extracted. The tailings piles of SMC contain several million tons of mill sand. The mill sand is a homogeneous, fine-grained sandy material as illustrated in figure 7 and shown in table 2. Because of the nature of the ore deposit and the mixing inherent in the mining and milling processes, the mill sand is relatively consistent in its composition and size distribution, which is surprising for a geologic material. As shown in table 3, the mill waste has the composition of a feldspathic, leuco-gabbroic norite that contains more normative magnesium (Mg)-iron (Fe) silicate minerals than typical lunar highlands (65% normative plagioclase compared to an average of 75% for the highlands at the Apollo 16 site). Because its parent rock is from the ore zone, it contains more alteration minerals than typical of the lower norite zone as a whole (see fig. 8 and table 4). As the mill sand is similar to lunar highlands in chemical composition and mineralogy, it has been used as the feedstock for making the pseudo-agglutinate and glassy components of the LHT simulants.
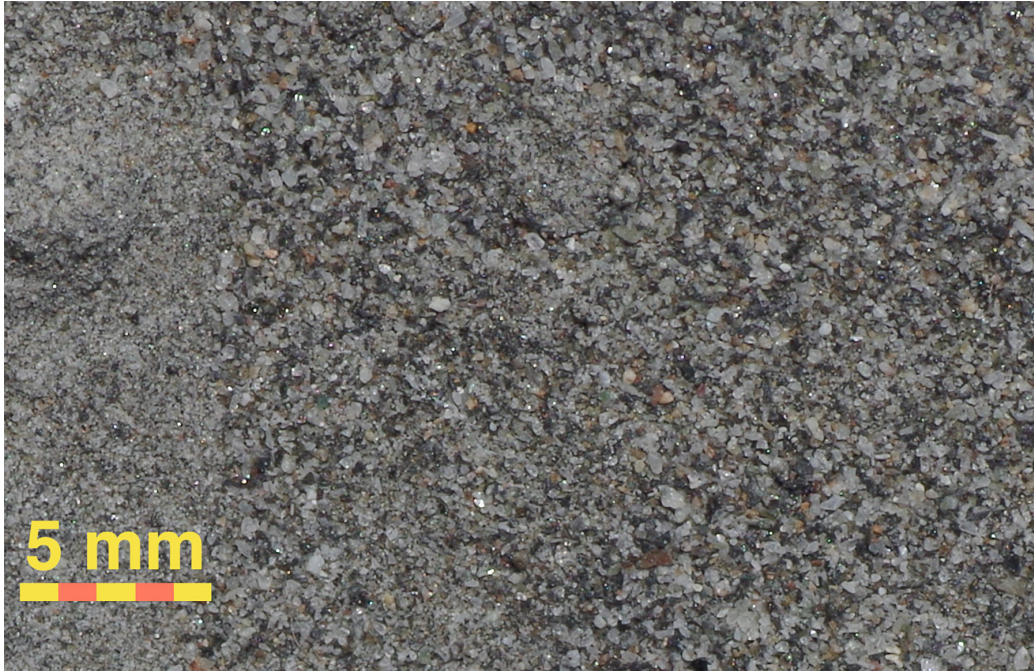
Figure 7. Stillwater mill sand. The material is susceptible to sorting by particle size, which is evident along the left edge of the image.

Table 2. Grain size analysis of Stillwater mill waste by dry sieving.

Mesh Size	Interval (µm)	Cumulative % Total
24	>701	–
30	701–595	–
80	595–180	4.04
100	180–150	7.70
120	150–125	13.15
200	125–75	61.39
325	75–44	82.37
325	<44	100.00
–	–	—

Table 3. Chemical composition and normative mineral calculation for mill waste feedstock used for LHT-1M; average of 12 analyses.

Major Element Chemistry		CIPW Normative Mineralogy		
Oxide	Wt.%	Mineral	Cation%	Wt.%
SiO_2	48.19	or	0.72	0.73
TiO_2	0.12	ab	8.81	8.43
Al_2O_3	22.29	an	55.05	55.87
Fe_2O_3	–	Total plag	64.58	65.03
MnO	0.09	en	86.2	86.9
MgO	9.82	di	6.46	6.51
CaO	13.03	hy	23.71	22.71
Na_2O	1.00	Total px	30.17	29.22
K_2O	0.12	opx:cpx	77.23	78.22
P_2O_5	0.07	ol	3.22	2.93
FeO_t	5.26	mt	1.72	2.42
Total	100.18	il	0.16	0.23
		ap	0.15	0.17
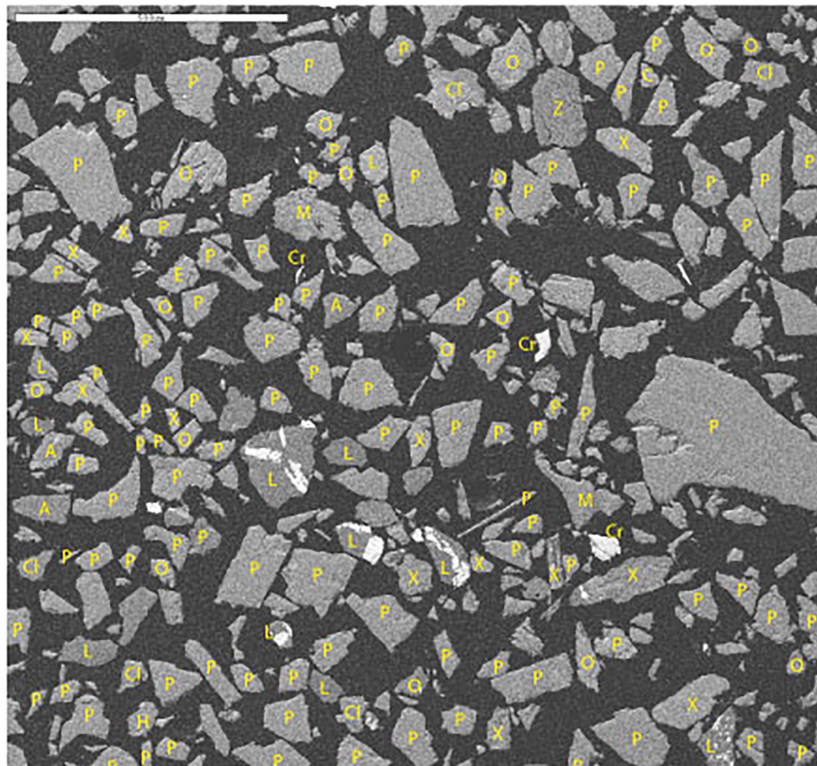
Figure 8. SEM backscatter image of mill sand (P–plagioclase, A–albite, M–mixed plagioclase, and alteration products, Z–zoisite, O–orthopyroxene, X–clinopyroxene, L–olivine ± Fe-oxide, Cr–chromite, Cl–chlorite, E–epidote, H–hornblende?, and C–calcite (white bar = 500 µm)).

Table 4. Reconnaissance SEM-based modal mineralogy of the mill sand based on 172 grain EDS x-ray spectrum mineral identifications.

Material	Count	%
Primary minerals:		
Plagioclase	108	63
Orthopyroxene	20	12
Clinopyroxene	14	8
Olivine (± Fe-oxide blebs)	11	6
Chromite	3	2
Total primary	156	91
Secondary minerals:		
Albite	4	2
Mixed plagioclase and alteration minerals	2	1
Zoisite	1	0.6
Fe-epidote	1	0.6
Chlorite	6	4
Amphibole	1	0.6
Calcite	1	0.6
Total secondary	16	9.4
Total	172	100

Although the Stillwater Complex is a virtual smorgasbord of potential simulant materials, rock that can be accounted as simulant feedstock reserves is limited. (Reserves are geologic material that is legal, economical, and technically feasible to extract.) For example, the Complex above the anorthosite I zone is not accessible by road. Feedstock material can only be obtained by hand-picking float blocks, inactive mine waste piles, and from road cuts. The road norite location has been estimated to hold no more than 30 tons of high-quality material that is safe to remove by hand. None of the feedstock resources are greater than ~50 tons without heavy equipment and appropriate engineering. (Resources are geologic material that might become economically valuable to extract.) The exceptions to the above are the SMC's mine dump and mill waste piles, which are private property.

2.4 Petrographic Textures

While the abundance of alteration minerals is important, the distribution of the minerals and the interfaces between the minerals are equally important when attempting to extract specific minerals from rocks. In many cases, features within individual crystals, such as fractures, are also important. Collectively, these and related features are referred to as textures. Although at least one nanoscopic texture is known to exist in Stillwater rock, specifically the presence of nanoscopic needles of primary magnetite in primary plagioclase,[12] optical examination of planar sections through

whole rock is the standard way to evaluate textures. For Stillwater rocks, the appropriate technique is to make petrographic thin sections, which are slices of rock approximately 27 mm × 45 mm × 30 μm thick mounted on glass slides. When prepared to this thickness, light is readily transmitted through the sample and important textural features can easily be seen.

Figures 9–12 are thin sections chosen to illustrate textural features common in the Stillwater rocks. Of specific emphasis are the textures of the alteration minerals. In these images, plagioclase is nearly colorless, clinopyroxene is a green, and orthopyroxene varies, having shades of brown with grey or pink tones. The ratios between plagioclase, clinopyroxene, and orthopyroxene are not significant for this TM. The exact identity of the alteration minerals seen in figures 10–12 has not been established by qualitative methods. It is common for geologists to assign an identity based on judgment, pending positive identification, should it be necessary.

Figure 9 is typical of essentially unaltered rock. The individual crystals are approximately equant and the interfaces are relatively simple. The bulk of the mass of each mineral is in crystals with a limited size range, approximately to 2–3 mm. Also note the lack of opaque material, which would appear black in these images. In detail, other primary textures can be observed in Stillwater rock, but to the nonigneous petrologist the different textures share the characteristics of being dominated by relatively equant crystals having a limited range in crystal size and simple crystal to crystal boundaries.
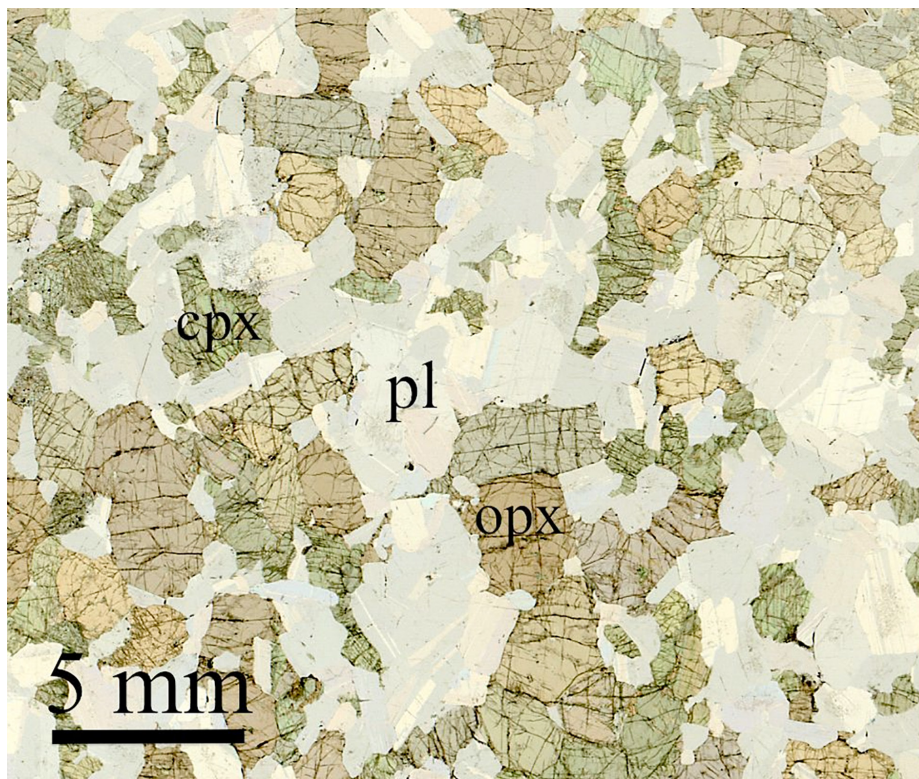
Figure 9. Relatively unaltered rock from Stillwater. This sample shows textures typical of Stillwater rock. See text for discussion. Section id: STW3.

In figure 10, a patch of alteration at A has completely replaced the plagioclase. Minerals typical in such alteration include sericite, albite, calcite, epidote, and chlorite. Note the lack of alteration in the remaining plagioclase. Alteration of the pyroxenes is extensive. The perimeters of the crystals are extensively replaced and the bodies of several are deeply corroded by alteration. Opaque minerals, probably magnetite, have precipitated in crystallographically-controlled patches inside the original pyroxenes. A secondary crystal of biotite has developed at B.
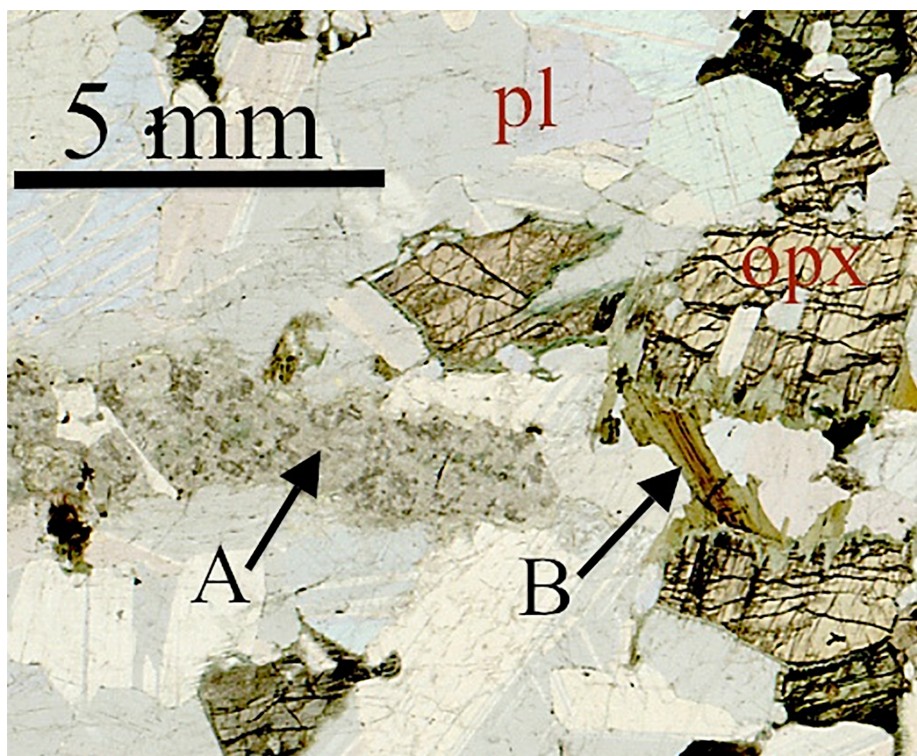
Figure 10. Locally complete replacement of plagioclase by alteration minerals without significant alteration of the remaining plagioclase. See text for discussion. Section id: STW12.

In figure 11, much of the alteration is clearly associated with fractures. At A, magnetite has formed and other magnetite deposition can be seen in patches. Magnetite is localized by the crystallography of the host pyroxene in the upper right of the image. Contacts between crystals are common foci for the diffusion of hydrothermal alteration. This is apparent at and around B. At C, two parallel fractures through the plagioclase contain what may be epidote.
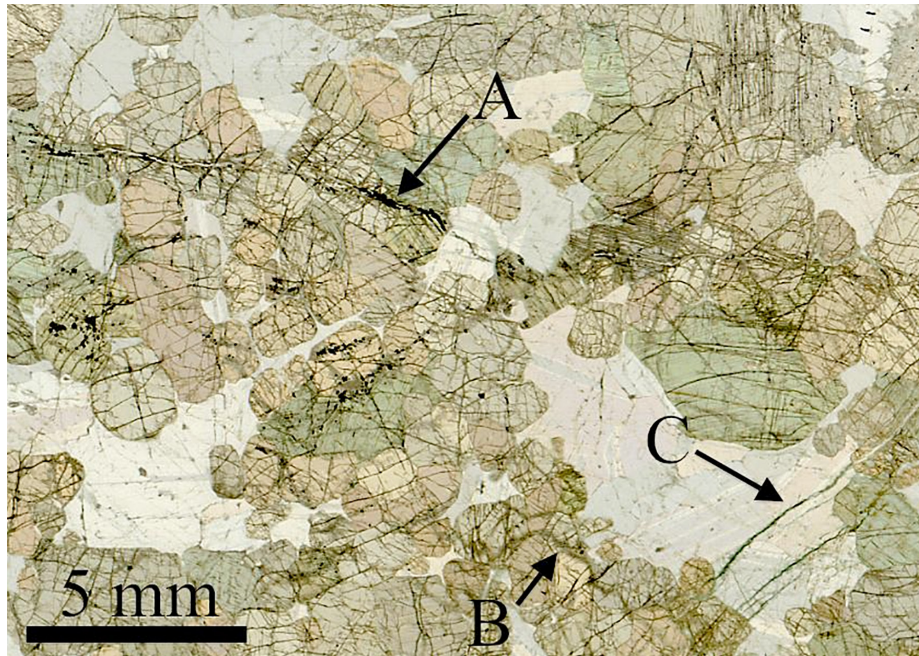
Figure 11. Alteration localized along fractures and at crystal boundaries. See text for discussion. Section id: STW11.

Pervasive chloritic alteration is seen in figure 12. At A, the chlorite is extensively replacing the orthopyroxene and magnetite has precipitated along crystallographic boundaries of the host pyroxene. Other patches of magnetite, which is black in this section, can be seen along boundaries of the plagioclase crystals, especially at triple junctions. Patches of chlorite are seen throughout the plagioclase, for example at B, not just at crystal boundaries. The plagioclase in this rock has an approximate formula of $(Ca_{0.85},Na_{0.15})(Al_{1.85}Si_{2.15})O_8$. Chlorite is a family of minerals, which are generally not discriminated in thin section. A common and likely chlorite has an approximate formula $(Mg_5Al)(AlSi_3)O_{10}(OH)_8$. As the host plagioclase does not contain magnesium, that element has been transported through the plagioclase by the hydrothermal fluids.
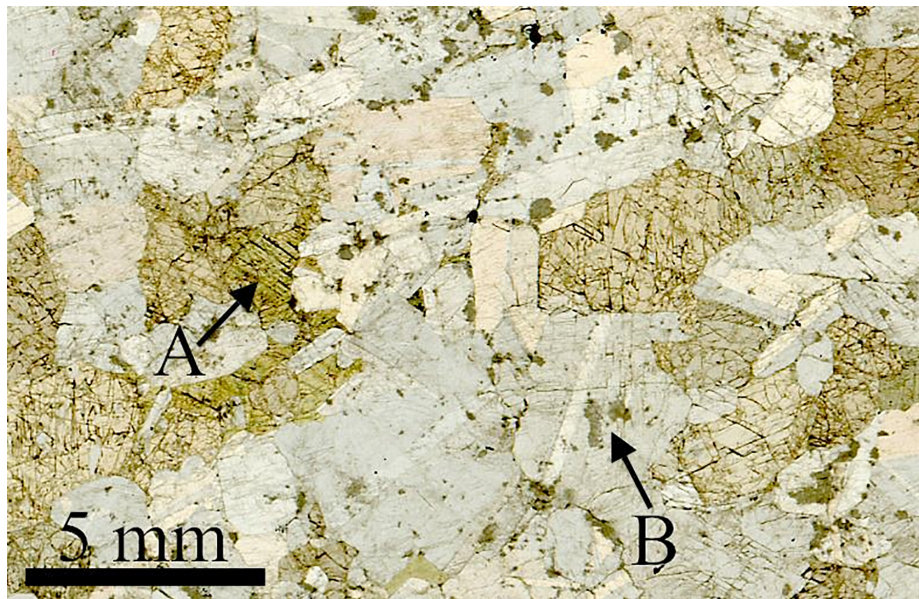
Figure 12. Disseminated chloritic alteration. See text for discussion. Section id: ST21B.

2.5 Analysis

The lunar regolith is dominated by high calcium plagioclase, clinopyroxenes, orthopyroxenes, olivine, and glasses made dominantly from these minerals. Suitable olivine can be purchased commercially. Plagioclase can be purchased, but its calcium content is not as high as that of the best material from the Stillwater Complex. For simulants, the calcium content of the plagioclase is important in many cases, as it affects the melting point of the simulant and changes the chemistry of the resulting melt. There are no commercial sources for the pyroxenes.

All of the Stillwater material contains some alteration minerals, often to excessive levels. However, by hand selection, it is possible to get rock with <5% total secondary minerals. But the selection process requires some skill, training, and experience. The tonnage available by this approach, without heavy equipment, proper engineering, and permits, is in the tens of tons.

The waste dumps and the mill tailings of SMC's Nye, Montana, operations contain millions of tons of material. This material, which is dominantly high calcium plagioclase, orthopyroxene, and clinopyroxene, has variable amounts of hydrothermal alteration minerals as secondary minerals. Almost all of these secondary minerals are objectionable in simulants of lunar regolith for several reasons. A partial list of secondary minerals observed in the Complex is given in table 5. The list in table 5 is not exhaustive and the relative abundances indicated are estimates. Different zones of the Complex definitely have different secondary minerals and the abundances of the secondary minerals will also vary substantially.

Table 5. Secondary minerals reported in the Stillwater Complex. Abundance of the minerals decreases from group 1 to group 2 to group 3. The sulfides are primary and locally secondary minerals. As these are strongly associated with the precious metal content being mined, they do not commonly show in either the waste pile or the milling tailings.

Group 1	Group 2	Group 3		Sulfides
Chlorite	Calcite	Apatite	Phrenite	Chalcopyrite
Albite	Epidote	Biotite	Serpentine	Pentlandite
Talc	Hornblende	'Clay'/sericite	Sphene	Pyrrhotite
	Magnetite	Ferropargasite	Stilbite	Pyrite
	Quartz	FeO_x	TiO_2 species	
	(Clino-) Zoisite	K-feldspar	Tremolite	
		Phlogopite		

Experimentation is required to establish whether or not the plagioclase, clinopyroxenes, and orthopyroxenes can practically be recovered from the waste dumps or mill sands. Using either resource, is it practical to separate the plagioclase, orthopyroxene, and clinopyroxene from each other and to simultaneously remove any or all of the secondary minerals? If the separations are possible, what are the purities, retention versus loss ratios, and the costs per ton of processing?

Of particular note is the realization that successful separation techniques developed for the various Stillwater materials can almost certainly be adopted for use with other feedstocks. This could be very useful. For example, the clinopyroxene to orthopyroxene ratio in the Stillwater materials generally is significantly <1; in other words, enstatite is much more common than augite in the Stillwater rocks. Much of the lunar regolith, especially the mare, has values >1.[13] If the pyroxene ratio is important to the performance of the simulant, to reproduce a desired lunar ratio using Stillwater rock would likely require processing to remove and dispose of orthopyroxene from the Stillwater feedstock. Further, the Stillwater pyroxenes tend to have at least some secondary minerals.

There are alternative sources. One could make pyroxene separates from two different sources such that, when the separates are combined, they yield the equivalent of lunar regolith. Specifically, the Bushveld Complex of South Africa has a much better clinopyroxene to orthopyroxene ratio for

simulant production. Much of the Bushveld rock is also free of any alteration mineralogy. Bushveld rock with a desirable pyroxene ratio is commonly imported into this country under several trade names, including Impala Black, Nero Impala, Rustenburg Granite, and Rustenburg Gabbro. This means the source rock is readily accessed by heavy equipment and inexpensively shipped internationally. However, the plagioclase in the Bushveld rock has significantly less calcium compared to both lunar and Stillwater modes, which is known to be important in many uses of simulants. Similarly, techniques developed for segregating plagioclase from the pyroxene in any of the Stillwater feedstocks would permit using Bushveld pyroxenes with Stillwater plagioclase, for example.

3. SEPARATIONS

3.1 Background

In order to make separations, the constituents being sought or to be removed must first be identified. The goal is to then identify differences in the physical and chemical properties of those constituents. Separations can only be made if differences exist, and separation becomes easier as the differences increase. In this regard, tables 6 and 7 were developed. Table 6 compares the major minerals in lunar regolith to the mineralogy of the road norite outcrop and mill tailings. Because neither resource contains significant, unaltered olivine, an olivine separate cannot be made. As mentioned previously, an olivine separate can also be obtained simply by purchasing a commercially available commodity, as long as it meets the necessary chemical and mineralogical specifications. Table 7 lists the various properties of the minerals and differences show that, for example, a plagioclase separate could be made first by magnetic separation and then the two pyroxenes separated by electrostatic separation to make clinopyroxene and orthopyroxene separates. In addition, based on differences in density, gravity separation could also be used to make a plagioclase separate; however, experience suggests that magnetic separation should work better. This does not mean that gravity separation will not be considered but rather does not have as high of a priority for testing.

Table 6. Characteristic wt.% abundance of major minerals in lunar regolith compared to road norite outcrop and mill tailings from SMC's Nye operations.

Mineral	Formula	Outcrop	Tailings	Regolith
Plagioclase	$(Na,Ca)(Si,Al)_4O_8$	75	63	75
Orthopyroxene	$(Mg,Fe,Ca)(Mg,Fe,Al)Si_2O_6$	18.4	12	10
Clinopyroxene	$(Ca,Na)(Mg,Fe)(Si,Al)_2O_6$	2.7	8	5
Olivine	$(Mg,Fe)_2SiO_4$	–	6	10

Table 7. Properties of minerals in the road norite and tailings materials.

Mineral		Density	Conductor	Magnetic	Hydrophobic
Family	Specific				
Plagioclase	Anorthite	2.8	N	N (Dia)	N
Orthopyroxene	Enstatite	3.2	N	Y (Para)	N
Clinopyroxene	Augite	3.2	Y	Y (Para)	N
Olivine	Forsterite	3.3	N	Y (Para)	N
	Talc	2.8	N	N (Non)	Y

Finally, although not present on the Moon, talc is included in table 7 because it is present in the two SMC resources and is functionally significant in mineral separation. Talc is naturally hydrophobic and can be separated from other silicate minerals by flotation. The processing of the ore by SMC is designed to float the platinum-bearing sulfide minerals; thus, if talc also floats, it would significantly dilute the concentrate. To force the talc to report to the tailings, SMC adds carboxymethyl cellulose (CMC) to their flotation circuit. This chemical is a talc depressant which thereby prevents it from floating.

A major advantage of using mill tailings is that material preparation by additional comminution is minimal, if at all. However, because the mill tailings are already slurried, only wet processing technologies can be considered, thus avoiding expensive dewatering processes to enable dry processing. In addition, it is important to consider process reagents, like CMC, that might be present in the mill tailings as a result of SMC's milling process for extracting the valuable precious metal bearing sulfide minerals. By comparison, the road norite outcrop requires comminution. However, because this material is dry, both wet and dry separations can be considered.

The effectiveness of typical separations is normally measured by grade and recovery where grade refers to the purity of separates and recovery refers to the amount of separate being produced. Both quantities can be measured with a variety of analytical and characterization tools which, in this case, included x-ray fluoresence (XRF), x-ray diffraction (XRD), and scanning electron microscopy (SEM)/mineral liberation analysis (MLA), depending on the separate being examined. These tools are commonly used in mineral analysis with SEM/MLA being a recent innovation that has revolutionized the mineral processing industry. SEM/MLA is a state-of-the-art technology that uses the greyscale, back-scattered images from the SEM and correlates them to their energy dispersive x-ray spectra to determine what minerals they contain, then gives each mineral a representative or 'false' color, and quantifies them accordingly to produce an MLA micrograph.

For this study, the various separators that were used were all located at Montana Tech and included a (dry) Electrostatic Separator, (dry) Rare-Earth (RE) 25,000 Gauss Magnetic Belt, (dry) Multi-Element 2500-8000 Gauss Magnetic Drum, (wet) Electromagnetic 15,000 Gauss Drum, Wet High-Intensity Magnetic Separator (WHIMS) 20,000 Gauss Magnetic Carousel, and a 2-L batch flotation cell. The flotation cell is from Wemco and all other separators were from Eriez.

Outcrop materials for the work reported here were hand selected from the outcrop by D. Stoeser and D. Rickman as part of a larger collection effort. The rock was then broken as needed with a sledge hammer to 100% passing 4 inches, then dry crushed to 100% passing ½ inch with a 5-inch laboratory jaw crusher, and then to 100% passing approximately 1/10 inch with a 6-inch laboratory double-roll crusher. It was then either wet or dry ground with 6 inch ×10 inch laboratory batch ball mills to 80%, passing either 600 μ (Tyler 28 Mesh) or 45 μ (Tyler 325 Mesh). Due to equipment constraints, feed to the WHIMS had to be <45 μ. Flotation tests were conducted on both size fractions. In some cases, experiments were conducted on fractions sized by sieving. Tests were conducted on as-received wet mill tailings. Size measurements via sieve analysis showed the materials to be 80% passing 45 μ (Tyler 325 Mesh), which concurs with the results presented in table 1.

3.2 Road Norite

Figure 13 shows the MLA micrograph of the nonmagnetic fraction produced from the outcrop material using the dry RE Magnetic Belt Separator. Results indicate that a plagioclase product results with an assay of 97.74% plagioclase and 1.29% pyroxene. These results are comparable with the results reported in Rickman et al.[2] When this fraction was size separated, XRF analysis of the coarse fractions were found to assay 27.1% Si, 20.0% Al, 15.0% Ca, 1.1% Na, and 0.8% Fe with the balance being 36.00% O, whereas the fine fractions assayed 19.2% Si, 16.6% Al, 12.4% Ca with no Na and Fe present, and the balance being 51.8% O. The differences are attributed to the separation being dependent on particle size as well as liberation. Figure 14 confirms that incomplete liberation likely caused the grade of the plagioclase product to not be pure. In this case, region 1 of the locked particle contains Fe and is presumed to be paramagnetic in spite of reporting to the nonmagnetic fraction. By comparison, region 2 does not contain Fe. Finally, XRD analysis of this product suggests the plagioclase is bytownite.

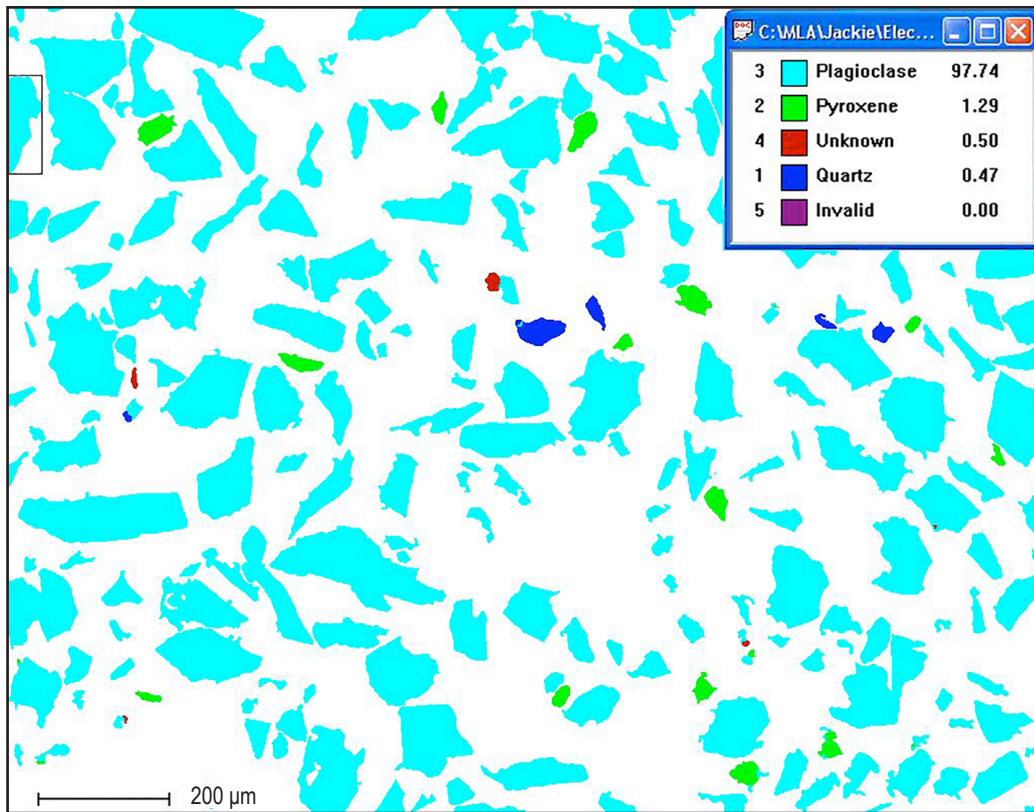Figure 13. MLA micrograph of the nonmagnetic product made by dry processing outcrop material with the RE Magnetic Belt Separator.
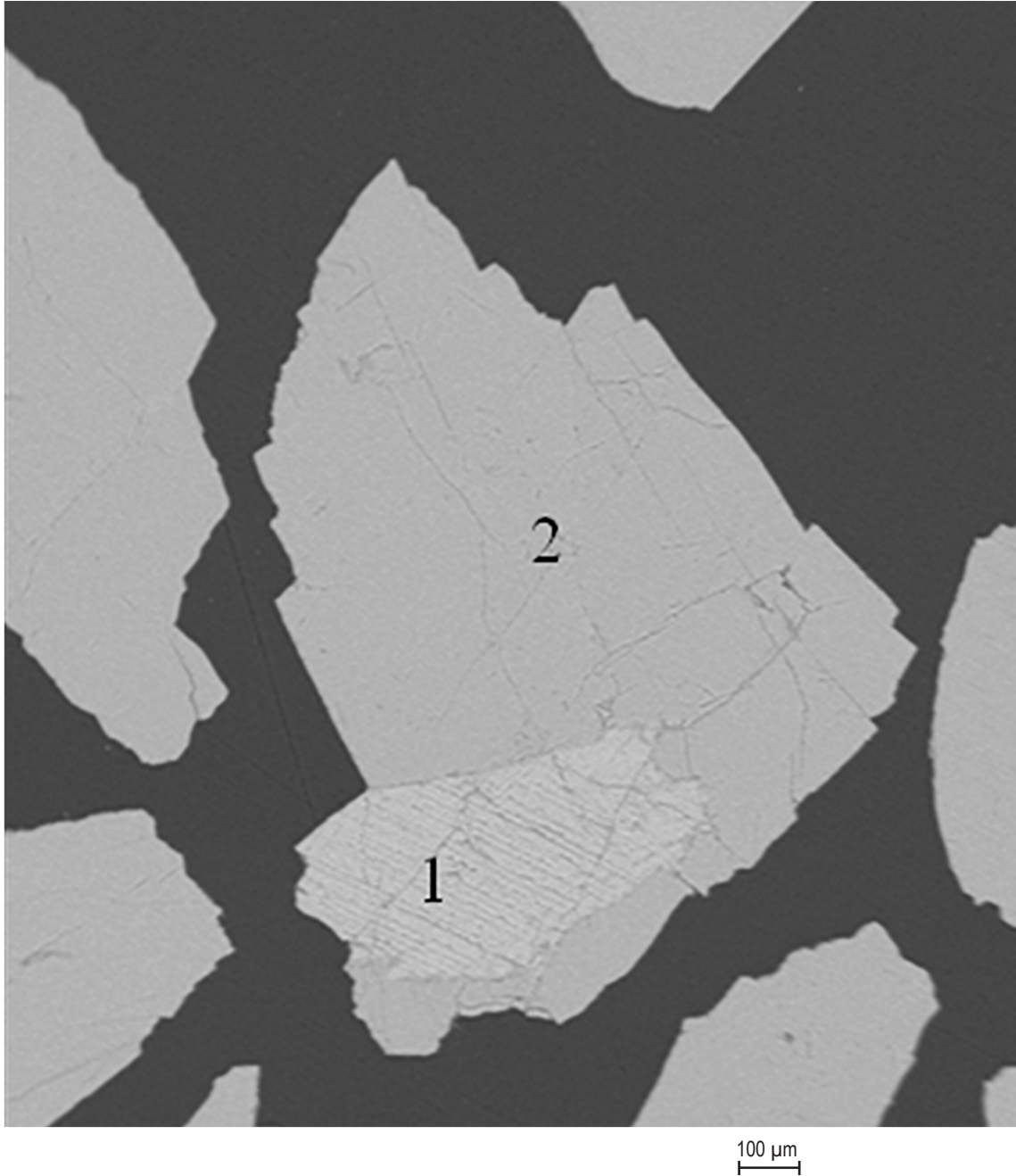
Figure 14. SEM micrograph illustrating a locked particle in the nonmagnetic product made by dry processing outcrop material with the RE Magnetic Belt Separator. Region 1 contains Fe and is considered magnetic whereas region 2 does not and is considered nonmagnetic.

By comparison, figure 15 shows the MLA micrograph of the magnetic fraction produced with the dry RE Magnetic Belt. In this case, the grade was 34.94% plagioclase and 64.83% pyroxene. This product clearly needs upgrading. Figure 15 demonstrates locking occurs as indicated by the boxed-in particle; however, reducing the particle size further by additional grinding did not improve the separation with the dry RE Magnetic Belt such that even the nonmagnetic fraction was not improved. Comparing figures 14 and 15 suggests that locking affects the magnetic fraction more and that the dry RE Magnetic Belt Separator does not process fine materials as well as coarse materials. When the feed was sized, results showed that the weight fraction reporting to the magnetic product was consistently between 63%–68% in agreement with an independent study; however, when the feed size was <150 μ, recovery fell to 50%, and when the feed size was <75 μ, separation was poor (see table 8).
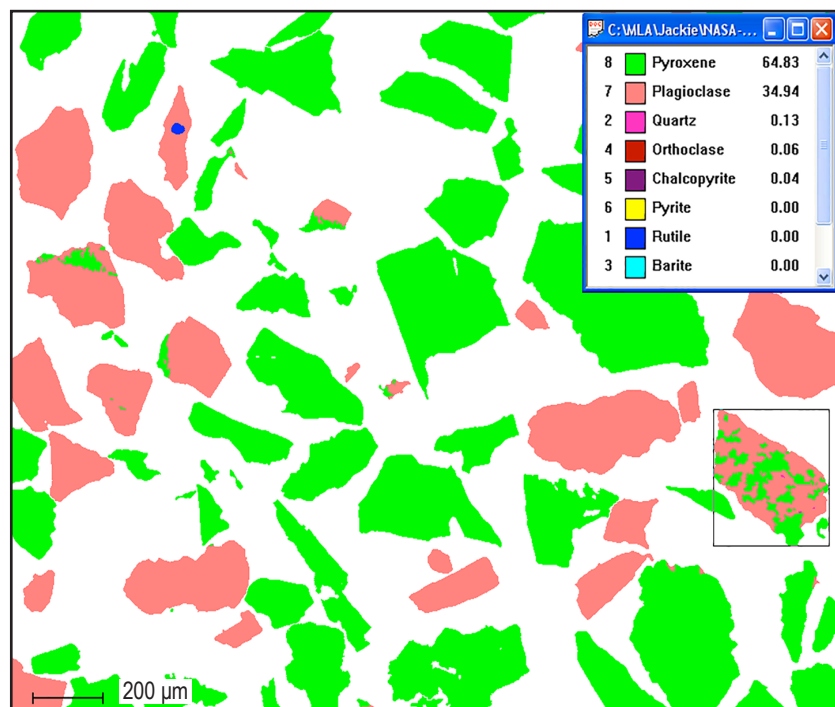
Figure 15. MLA micrograph of a product produced by dry processing outcrop material with a RE Magnetic Belt Separator.

Table 8. Recovery of road norite outcrop material at various size fractions by RE Magnetic Belt Separation.

Size (μm)	Feed (g)	Mag Con (g)	Weight Recovery (%)
–1,000 / 600	564.0	380.0	67.4
–600 / 300	665.1	453.3	68.2
–300 / 150	429.9	274.8	63.9
–150 / 75	240.3	117.4	48.9
–75	Too Fine		

Electrostatic separations were performed to process outcrop material as well as both the magnetic and nonmagnetic products from the dry RE Magnetic Belt Separator. Results were negative in all cases. Therefore, further electrostatic separations were not performed in subsequent experiments.

A dry RE Magnetic Belt Separation was repeated and the resulting nonmagnetic product was wet ground to 80% passing 45 μ and prepared for flotation. The slurry was adjusted to pH 9 with lime and floated in three stages each for 5 minutes with stage 1 employing only Dow Froth to separate talc, and with stages 2 and 3 employing both Dow Froth and Cytec Aero 704 Fatty Acid Promotor to separate any remaining talc and other hydrated silicates. In this reverse flotation strategy, the unfloated fraction is the plagioclase concentrate which was found to measure effectively 100% anorthite.

The magnetic product from the dry RE Belt separation was also wet ground for liberation purposes and then processed with the wet High Intensity Magnetic Carousel. The nonmagnetic fraction from this WHIMS separation step was then subjected to the same flotation procedure as just described. In this case, a plagioclase concentrate was found to have a grade of 89% anorthite. Combining the two products will yield an overall plagioclase concentrate near 94% anorthite which will be more than acceptable as a separate.

With most of the talc and plagioclase removed, the magnetic fraction from the WHIMS separation step should be predominantly pyroxene. Table 7 suggests it will contain orthopyroxene and clinopyroxene in a 3:1 ratio along with various secondary mafic minerals. All that needs to be done is purify the product and either remove some orthopyroxene or add clinopyroxene from another separate to obtain the 2:1 ratio in lunar soil. Work continues in this regard as well as to determine an analytical method for precisely measuring the pyroxene content. This work is being done by Frank Asirifi, MS candidate in Metallurgical and Materials Engineering, Montana Tech.

3.3 Mill Tailings

Mill tailings from the SMC's Nye Concentrator in Montana were also examined. Because the material was already 80% passing 45 μ, it was first processed with the wet Electromagnetic Drum Separator to remove any ferromagnetic material that may be in the material including mill scale. This protects the WHIMS which is used next to process the nonferromagnetic product from the Electromagnetic Drum Separator. The WHIMS magnetic fraction should contain predominantly pyroxene but XRD and SEM/MLA analyses were not reliable. As noted above, a precise analytical method is being developed. However, table 7 suggests the product will contain orthopyroxene and clinopyroxene in a 0.5:1 ratio. It is conceivable that this product could be combined with that produced from the road norite outcrop material such that the 2:1 ratio in lunar soil is obtained. Work also continues in this regard as well as to try and separate the pyroxene minerals.

The WHIMS nonmagnetic fraction was then treated by flotation in order to remove talc. However, for this to work, it was first going to be necessary to destroy the CMC used at the Nye Concentrator as a talc depressant. The slurry was found to be at pH 8.5 and was left as is. Flotation was conducted in five stages, each for 5 minutes. Stages 1–4 used only Dow Froth; stage 5

used Cytec Aero 704 Fatty Acid Promotor and Dow Froth. However, prior to stages 2, 3, 4, and 5, hydrogen peroxide was added and allowed to react for 5, 20, 40, and 3,000 minutes, respectively. The resulting plagioclase concentrate was also found to be free of talc and other hydrated silicates and also measured 100% anorthite.

3.4 Flowsheet Design

Based on the information gathered, the flowsheets shown in figures 16 and 17 would be used to process the road norite outcrop and mill tailings, respectively. The road norite material is dry comminuted and beneficiated with a RE Magnetic Belt Separator. Resulting nonmagnetic materials are then wet ground and processed by flotation to remove naturally hydrophobic, hydrated minerals such as talc. A second stage of flotation is conducted to remove any remaining talc but requires the use of a collector. Removing talc in two stages in this manner minimizes reagent consumption which helps minimize overall expenses. Likewise, concentrator material was first separated with a Wet Drum Separator to remove the strong magnetic fractions. The remaining material was processed with WHIMS and cleaned by flotation albeit CMC in the mill tailings had to first be destroyed. It is important to point out that figure 17 is incorporated into figure 16; consequently, they could be operated together.
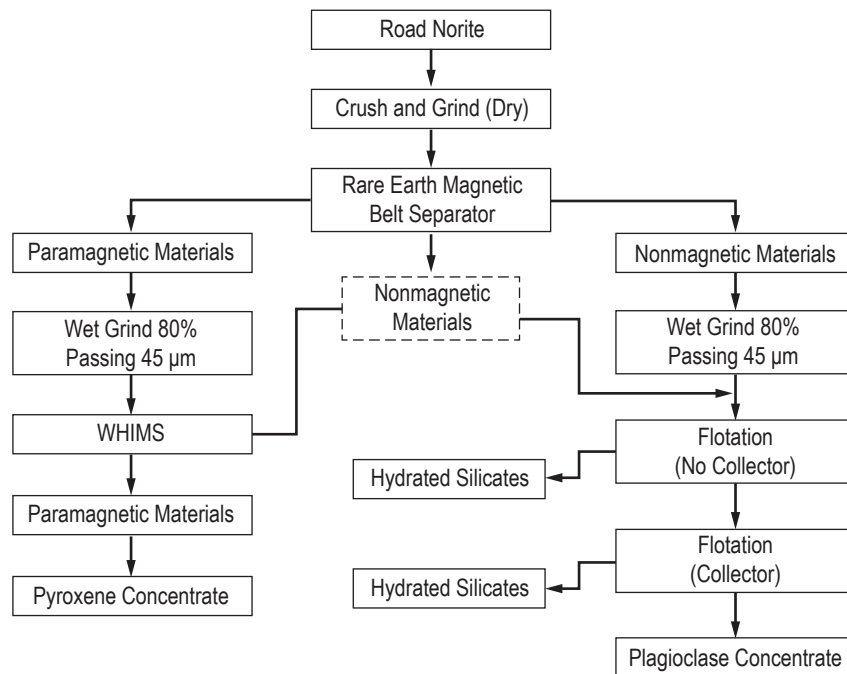

Figure 16. Potential flowsheet that was developed for processing road norite outcrop material.
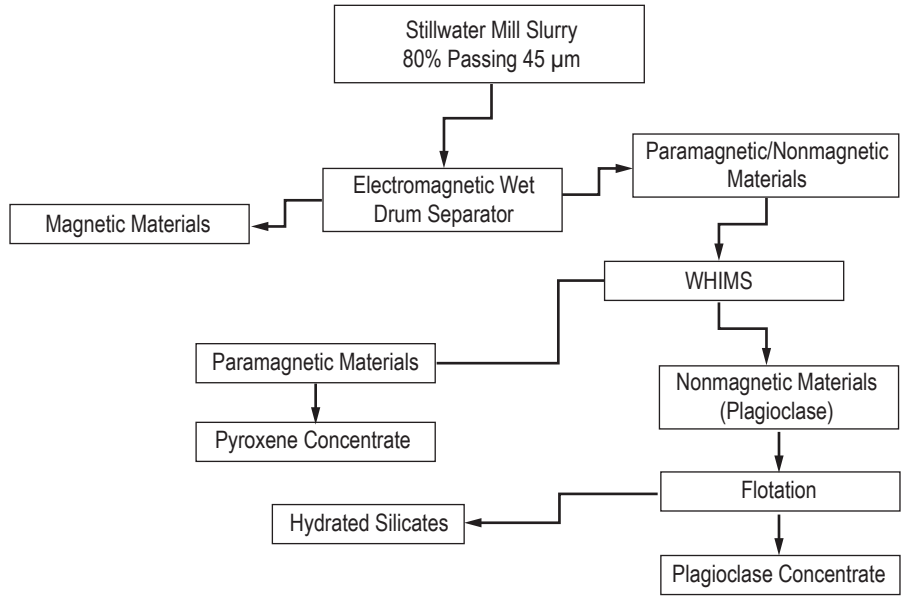
Figure 17. Potential flowsheet that was developed for processing the wet mill tailings material produced at the Nye Concentrator.

4. CONCLUSIONS

It has been previously established that a high purity plagioclase product could be obtained from the road norite.[2] Work reported here shows the same. Work here shows this can also be achieved using mill tailings. More than 6 tons of high calcium plagioclase concentrate could, in principle, be obtained from every 10 tons of feedstock. The availability of such pure, high calcium plagioclase would be a significant asset in any attempt to manufactured high-quality lunar simulants. By demonstrating successful plagioclase recovery from mill tailings, the need to have Ph.D. senior geologists hand pick rocks from a limited outcrop is obviated. Further, the reserve of road norite is clearly limited unless heavy equipment is brought in.

This study is an attempt at using mineral processing technologies to make simulant reserves from two Earth resources, namely the road norite outcrop and mill tailings from SMC's Nye Concentrator in Montana. These resources contain three of the four silicate minerals that lunar soil contains in both high and pure amounts. Based on promising results obtained from a series of tests, flowsheets were developed showing how these resources could be processed and made into 'separates' of (1) high calcium plagioclase and (2) orthopyroxene/clinopyroxene with the thought that they would be combined later to make simulant. Although both of these Earth resources also contain olivine, it was not present in significant amounts to produce a separate. Therefore, a different resource will be needed to do that and will also be the subject of a future study.

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14. ABSTRACT

The availability of pure, high calcium plagioclase would be a significant asset in any attempt to manufacture high-quality lunar simulants. A suitable plagioclase product can be obtained from materials obtained from the Stillwater Complex of Montana. The access, geology, petrology, and mineralogy of the relevant rocks and the mill tailings are described here. This study demonstrates successful plagioclase recovery from mill tailings produced by the Stillwater Mine Company. Hydrogen peroxide was used to remove carboxymethyl cellulose from the tailing. The characteristics of the plagioclase products are shown and locked grains are identified as a limit to achievable purity. Based on the experimental results, flowsheets were developed showing how these resources could be processed and made into 'separates' of (1) high calcium plagioclase and (2) orthopyroxene/clinopyroxene with the thought that they would be combined later to make simulant.

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IS20
George C. Marshall Space Flight Center
Huntsville, Alabama 35812