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2 **The Spectral Characteristics of Lunar Agglutinates: Visible–Near-Infrared**
3 **Spectroscopy of Apollo Soil Separates**

4 **Chanud N. Yasanayake^{1,2}, Brett W. Denevi¹, Takahiro Hiroi³, Brad. L. Jolliff⁴, Anna C.**
5 **Martin¹, Annabelle L. Gao^{3,5}, Margaret L. Zhang^{6,7}, Lucas M. Bloom^{8,9}, and Samuel J.**
6 **Lawrence¹⁰**

7 ¹Johns Hopkins University Applied Physics Laboratory. ²Johns Hopkins University. ³Brown
8 University. ⁴Washington University in St. Louis. ⁵Marriotts Ridge High School. ⁶University of
9 North Carolina at Chapel Hill. ⁷Mount Hebron High School. ⁸University of Alabama. ⁹Severna
10 Park High School. ¹⁰NASA Johnson Space Center.

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12 Corresponding authors: Chanud N. Yasanayake (chanud@jhu.edu), Brett W. Denevi
13 (brett.denevi@jhuapl.edu)

14
15 **Key Points:**

- 16 • We measured reflectance spectra of agglutinates and non-agglutinates separated from six
17 lunar soils of varying composition and maturity.
- 18 • Spectral comparisons suggest that spectral reddening of mature soils is expressed mainly
19 via agglutinates (for the size fractions measured).
- 20 • These findings reframe our understanding of the relative spectral roles of agglutinates and
21 soil grain rims in weathered lunar soils.

22 Abstract

23 The lunar surface evolves over time due to space weathering, and the visible–near-infrared
24 spectra of more mature (i.e., heavily weathered) soils are lower in reflectance and steeper in
25 spectral slope (i.e., darker and redder) than their immature counterparts. These spectral changes
26 have traditionally been attributed to the space-weathered rims of soil grains (and particularly
27 nanophase iron therein). However, understudied thus far is the spectral role of agglutinates—the
28 agglomerates of mineral and lithic fragments, nanophase iron, and glass that are formed by
29 micrometeoroid impacts and are ubiquitous in mature lunar soils. We separated agglutinates and
30 non-agglutinates from six lunar soils of varying maturity and composition, primarily from the
31 125–250 μm size fraction, and measured their visible–near-infrared reflectance spectra. For each
32 soil, agglutinate spectra are darker, are redder, and have weaker absorption bands than the
33 corresponding non-agglutinate and unsorted soil spectra. Moreover, greater soil maturity
34 corresponds to darker agglutinate spectra with weaker absorption bands. These findings suggest
35 that agglutinates (rather than solely the space-weathered rims) play an important role in both the
36 darkening and reddening of mature soils—at least for the size fractions examined here.
37 Comparisons with analog soils suggest that high nanophase iron abundance in agglutinates is
38 likely responsible for their low reflectance and spectrally red slope. Additional studies of
39 agglutinates are needed, both to more comprehensively characterize their spectral properties
40 (across size fractions and in mixing with non-agglutinates) and to assess the relative roles of
41 agglutinates and rims in weathering-associated spectral changes.

42

43 Plain Language Summary

44 In scientific study of the Moon, one key focus is surface processes: how do physical and
45 chemical properties of the Moon’s surface change over time due to weathering (e.g.,
46 bombardment by micrometeoroids and by particles from the Sun)? Such investigations provide
47 valuable insights into the Moon’s history (such as the ages of impact craters) that are often
48 deduced from measurements of reflected light; as a soil is weathered it reflects light differently,
49 which manifests visually as a progressive darkening of the soil. This phenomenon had primarily
50 been attributed to weathering-associated development of rims on individual soil grains, but in
51 this work we explored an alternative cause: soil particles known as agglutinates (misshapen,
52 vesicular agglomerates of mineral fragments, iron, and glass that form due to weathering
53 processes). We isolated agglutinates of six soil samples from the Moon and measured how they
54 reflect light. We find that they reflect light in patterns reminiscent of how the Moon’s surface
55 does when weathered. These findings suggest that agglutinates play a more important role than
56 previously thought in determining the light-reflecting properties of the Moon’s surface, thus
57 warranting greater and more nuanced consideration in future studies of how the Moon’s surface
58 changes over time.

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64 1 Introduction

65 The lunar surface evolves over time, gradually changing as it endures a continual
66 bombardment by micrometeoroids and the solar wind—chief among processes that are
67 collectively known as space weathering. These weathering processes impart observable changes
68 onto lunar soil that alter how it reflects light. At visible–near-infrared (Vis–NIR) wavelengths,
69 mature (i.e., heavily weathered) lunar soils are lower in reflectance, have a steeper (redder)
70 spectral slope, and have weaker crystal field absorption bands than their less-weathered
71 counterparts (e.g., Adams & McCord, 1970, 1971a, 1971b, 1973; Pieters et al., 1993; Taylor et
72 al., 2001a, 2001b). It is an ongoing effort dating back many decades to link these spectral
73 changes to physical and chemical changes in the lunar soil and, in turn, link the physical and
74 chemical changes to specific causative processes.

75
76 The physical and chemical changes that have the largest spectral effects include the
77 comminution of rocks into a fine particulate, the conversion of crystalline minerals into less
78 ordered and glassy materials, and changes in the oxidation state of iron (e.g., the reduction of
79 ferrous iron (FeO) to metallic iron (Fe⁰)) (e.g., Hapke, 2001; Lucey et al., 2006; Pieters & Noble,
80 2016). All three of these processes are observed at the individual lunar soil (regolith) particle
81 level. A single regolith particle is on average ~70 μm in size (Carrier, 2003) for mature soils, the
82 result of long-term impact bombardment and regolith gardening. Each particle in a mature soil
83 typically has an amorphous rim or surficial coating up to ~200 nm thick (Keller et al., 2021).
84 This rim can either be due to damage to the crystal structure caused by energetic charged-particle
85 radiation (largely sourced from the Sun, i.e., the solar wind), or can be depositional, from
86 condensation of impact-generated vapor or solar-wind ion sputtering (see overview in Denevi et
87 al., 2023). While differences in grain size and crystallinity have modest effects on spectral
88 reflectance, the depositional rims have been the focus of many studies because they contain
89 small (<10 nm) spherules of iron (Keller & McKay, 1993, 1997) that increase in abundance with
90 maturity and cause substantial changes in reflectance (e.g., Hapke, 1973; Hapke et al., 1975;
91 Cassidy & Hapke, 1975; Hapke, 2001; Pieters et al., 1993). This population of small iron grains
92 is often called nanophase iron (npFe or npFe⁰) or submicroscopic iron (SMFe). While this iron is
93 typically reduced, metallic iron (Fe⁰), it can be found in a range of oxidation states (Fe⁰, Fe²⁺, or
94 Fe³⁺) (Keller and Clemett 2001; Thompson et al., 2016; Burgess and Stroud, 2017; Burgess and
95 Stroud, 2018), so in this work we use the terms nanophase iron or npFe (rather than npFe⁰).

96
97 While the depositional rims containing npFe have been the most recent focus of attention
98 in terms of causing spectral changes (e.g., Pieters et al., 1993; Christoffersen et al., 1996; Keller
99 et al., 2021), another soil particle plays an important role: agglutinates. Agglutinates are
100 misshapen, vesicular agglomerates of mineral and lithic fragments, bound together by glass
101 (Figure 1). They form when an impact event melts regolith, which incorporates clasts of other
102 regolith grains and releases trapped solar wind volatiles to create vesicles (Basu et al., 2002 and
103 references therein). Agglutinates range in size from ~tens of microns to mm-scale, and in mature
104 soils they can comprise up to 60% of the soil volume (McKay et al., 1991). Their abundance and
105 the fact that they are so visually dark suggests that agglutinates have a substantial influence on
106 spectral reflectance. Initially their dark brown appearance was attributed to the glass itself (e.g.,
107 Conel & Nash, 1970; Nash & Conel, 1973; Adams & McCord, 1971a), but subsequent work
108 showed that glass melted in a vacuum is not inherently low in reflectance (Wells & Hapke,
109 1977). Instead, the spectral properties of agglutinates are strongly affected by their substantial

110 population of npFe. This iron is found within the flash-melted glass (Figure 1). Some of the npFe
111 may be from the npFe-rich rims on soil grains that have been incorporated into the melt. It is not
112 known whether melting/vaporization alone results in the creation of additional npFe via the
113 reduction of FeO to Fe (with O lost as a volatile species; e.g., Hapke, 1975, 2001; Keller &
114 McKay, 1993, 1997) or whether implanted solar wind H⁺ aids in the reduction process (forming
115 OH⁻ or H₂O; Housley et al., 1972, 1974; Morris, 1977, 1980; Taylor & Cirlin, 1985; Denevi et
116 al., 2023).

117
118 However it is formed, the abundance of npFe in agglutinates is so high that agglutinates
119 can be separated magnetically from most other soil components (e.g., Adams & McCord, 1973;
120 Via & Taylor 1976a, 1976b), and the npFe spans a much larger range of sizes than in
121 depositional rims (mean size 120 nm vs. 3 nm; James et al., 2002, Keller & Clemett, 2001),
122 possibly because npFe grains grow with repeated thermal shocking (Thompson et al., 2017). The
123 spectral effects of npFe are now known to vary with abundance and size (Noble et al., 2007);
124 greater abundance and larger size can both lead to lower reflectance and a shallower spectral
125 slope. Large grains (greater than ~40 nm; sometimes referred to as microphase Fe) are thought to
126 “darken without reddening” and agglutinates have often been described as only lowering the
127 overall reflectance of a soil, without changing its spectral slope due to their large average npFe
128 grain size (e.g., Britt & Pieters, 1994; Keller et al., 1998; Noble et al., 2007; Pieters & Noble,
129 2016).

130
131 Although agglutinates are a major component of mature soils, their spectra have not been
132 extensively studied. In fact, agglutinate reflectance spectra have only been published for a single
133 lunar soil, the high-titanium Apollo 11 sample 10084 (Pieters et al., 1993; Keller et al., 1998).
134 The lack of in-depth spectral studies of agglutinates is somewhat surprising, until one learns just
135 how tedious it is to separate agglutinates from lunar soil (see Section 2.2). Still, given the dearth
136 of comprehensive studies of agglutinate spectral properties thus far, there remain many open
137 questions regarding the basic nature of these particles. For example, do all lunar agglutinates
138 have comparable spectra, or do they differ significantly based on the composition of the regolith
139 from which they were produced? And, while it is well-documented that agglutinates increase in
140 abundance as soils are progressively weathered (e.g., Taylor et al., 2001a, 2001b, 2010), do the
141 agglutinate particles themselves evolve with increasing maturity? What is the overall spectral
142 contribution of agglutinates vs. depositional rims to lunar spectra?

143
144 Here we have extended the existing characterization of agglutinates and contributed to
145 answering these questions by measuring the spectral properties of (primarily 125–250 μm)
146 agglutinate and non-agglutinate particles isolated from six different Apollo lunar soil samples:
147 14259, 15041, 61141, 62231, 67461, 79221. We have similarly measured each soil after sieving
148 (e.g., to 125–250 μm) but prior to sorting into agglutinate and non-agglutinate separates, and we
149 henceforth refer to this soil as the unsorted soil. By comparing the reflectance spectra of
150 agglutinates, non-agglutinates, and unsorted soils of varying composition and maturity, we seek
151 to contribute to the effort to understand how space weathering proceeds on the Moon and how to
152 interpret spectral changes related to maturity.

153

154 **2 Methods**

155 2.1 Lunar soil selection

156 In order to characterize agglutinates, we selected six lunar regolith samples (<1 mm
 157 fines) for this study: 14259, 15041, 61141, 62231, 67461, and 79221 (Table 1). These samples
 158 were chosen for their wide range in soil composition and maturity, and because they have been
 159 extensively characterized by previous studies in terms of mineralogy, chemistry, maturity, and
 160 spectral properties (Taylor et al., 2001a, 2001b, 2010; Morris, 1978; Pieters et al., 2002).

161
 162

Soil	Soil type	Provenance ^a	FeO (wt%) ^b	TiO ₂ (wt%) ^b	I _S /FeO ^c	Maturity	Agglutinate abundance (%) ^d
67461	Low-Fe highlands	Fillet of boulder, rim of North Ray Crater	4.2	0.4	25	Immature	25
61141	Low-Fe highlands	30m from rim of Plum Crater	4.8	0.6	56	Submature	50
62231	Low-Fe highlands	Rim of Buster Crater	4.9	0.6	91	Mature	50
14259	Moderate-Fe nonmare	Top 1cm of soil, 100m west of lunar module	9.5	1.8	85	Mature	61
15041	Low-Ti mare	Top of trench dug near Station 8	14.2	1.8	94	Mature	51
79221	High-Ti mare	Top 2cm of trench dug near Van Serg Crater	14.0	6.4	81	Mature	47

163

164 **Table 1.** Lunar regolith samples examined in this study.

165 ^aMeyer (2005); reports for soils 67461, 61181, 62231, 14259, 15030, 79221. ^bTaylor et al.
 166 (2001a, 2001b, 2010); weight percent in <45 μm size fraction. ^cMorris (1978); I_S/FeO for <250
 167 μm size fraction. ^dTaylor et al. (2001a, 2001b, 2010); modal (volume) abundance of agglutinitic
 168 glass in the 20–45 μm size fraction.

169

170 The range in lunar soil composition is represented by samples that are of comparable
 171 maturity, but that originate from low- and high-titanium mare (15041 and 79221, respectively),
 172 nonmare (14259), and highlands (62231) regions. This range of compositions expands beyond
 173 previous published work, which focused only on agglutinates from a high-titanium mare region
 174 (sample 10084, in Pieters et al., 1993).

175

176 The suite of Apollo 16 soils (67461, 61141, and 62231) represent the range of soil
 177 maturity found on the Moon. The maturity of lunar samples is commonly quantified as I_S/FeO:
 178 the ferromagnetic resonance intensity (I_S) of the soil's <250 μm size fraction normalized to its
 179 FeO concentration (Housley et al., 1973, 1974, 1975; Morris, 1976, 1978). The normalization of

180 I_S to FeO is necessary because the I_S signal is produced by nanophase iron (npFe) particles in the
181 diameter range of 4–33 nm (Housley et al., 1976, Morris, 1980), and the concentration of npFe in
182 the soil depends both on the degree of surface exposure (which reduces Fe^{2+} in the soil to Fe^0) as
183 well as on the concentration of FeO in the soil (which is the source of the Fe^{2+}). By normalizing
184 I_S to the FeO concentration in the soil, the maturity of soils of differing composition can be
185 compared (Housley et al., 1973, 1974, 1975).

186

187 Lunar soils are categorized based on I_S/FeO value as immature ($I_S/FeO < 30$), submature
188 ($30 < I_S/FeO < 60$), or mature ($I_S/FeO > 60$). Sample 62231, like the samples used here from
189 Apollo 14, 15, and 17, is mature (Table 1). Sample 61141 is submature and 67461 is immature.
190 All three samples are from the highlands, although each is from the rim of a different crater at
191 the Apollo 16 landing site, allowing for an assessment of if/how agglutinate spectral properties
192 vary with maturity.

193

194 Note that the I_S/FeO values cited in Table 1, from Morris (1978), are based on
195 measurements of a broader size fraction ($<250 \mu m$) than the size fraction of the separated
196 agglutinates (125–250 μm). This means that the I_S/FeO values may not *directly* correspond to the
197 physical properties of the soils in our study, but they are still a valid tool for comparing soils to
198 one another and considering *relative* differences in maturity, under the assumption that, for any
199 two soils, a soil with greater I_S/FeO value for the $<250 \mu m$ size fraction also has greater I_S/FeO
200 value for any other size fraction.

201

202 2.2 Separating agglutinates

203 In past work, a variety of methods have been used to isolate agglutinates from regolith
204 samples. Separation of particles based on magnetic susceptibility is effective to some degree, as
205 demonstrated by Adams and McCord (1973), since the iron-metal-rich agglutinates tend to be the
206 most magnetic particles in the soil. However while magnetic separation can yield an agglutinate-
207 rich separate, it may contain 10–20% non-agglutinates that are highly magnetic while excluding
208 agglutinates that are less magnetic (ranging from $<5\%$ to $>20\%$ of the remnant soil) (Via &
209 Taylor, 1976a). Pieters et al. (1993) bypassed this issue in their study of sample 10084 by hand
210 picking individual agglutinates, which, while time-intensive, presumably produced a purer
211 agglutinate separate.

212

213 In this work, we used two different methods of separating agglutinates and compared
214 their effectiveness. In both cases we first isolated the 125–250 μm size fraction of each regolith
215 sample by sieving and then rinsing with ethanol to remove any clinging fines. This size fraction
216 was the smallest we found to be practicable to manipulate with tweezers (as static causes smaller
217 particles to jump and stick to tools). For each sample we started with an initial mass of 2 g, with
218 the 125–250 μm size fraction constituting $\sim 15\%$ of the total sample mass. We obtained two 2-g
219 splits of sample 62231, in order to test both agglutinate separation methods on the same material
220 (see below). We also requested a second 2-g split of sample 67461 because the agglutinates
221 make up a smaller fraction of this immature sample, and the mass of agglutinates separated from
222 the initial split was too small for the acquisition of reflectance spectra with the setup used.

223

224 The first agglutinate separation method was manual, similar to the method of [Pieters et al.](#)
225 [\(1993\)](#), wherein we selected individual agglutinate particles from the sample using tweezers. To
226 do so, we poured a small portion of the sieved regolith sample into a dish. We then observed the
227 sample under a binocular microscope and identified agglutinates based on their appearance:
228 irregular shape, vesicular, typically brown in color, and a surface texture that varies from rough
229 to glassy, interspersed with mineral fragments ([Figure 2](#)). All other particles were deemed “non-
230 agglutinates”. We took care to gently grip the agglutinates with the tweezers, as the friable
231 particles were liable to break apart if gripped too strongly, and moved them to a separate
232 container. These particles constituted the manually separated agglutinates, and we sorted through
233 the remainder of the soil until the point of diminishing returns (i.e., continued searching revealed
234 few additional agglutinates). This remainder of the soil was the non-agglutinates. We refer to this
235 first separation method as the manual method.

236
237 We note that there is some ambiguity in whether a given particle is an agglutinate or not.
238 Because agglutinates include mineral and lithic fragments bound by impact melt glass, there are
239 some regolith particles that appear to be, e.g., largely a mineral fragment with only a small
240 portion of the particle having the characteristic shiny surface and irregular shape that results from
241 impact melt glass. There are also a number of particles that may best be described as dark, glassy
242 melt breccias, and that appear to contain a smaller portion of glass and have angular shapes.
243 These particles have sometimes been referred to as “dark matrix breccias” or lumped together
244 with agglutinates as “fused soil components” (e.g., [Adams & Charette, 1975](#); [Vaniman & Papike,](#)
245 [1977](#)). Thus, when performing the manual separation, we took pains to be consistent in what was
246 included as an agglutinate and what was excluded as a non-agglutinate ([Figure 2](#)).

247
248 The second separation method began with magnetic separation. We poured the sieved
249 regolith sample into a Frantz magnetic separator where the particles flowed down a chute (25°
250 forward angle, 15° side angle) past a magnet. The magnet current was held at 0.5 A for all
251 samples except the immature sample 67461, which required a stronger current of 1.0 A to
252 effectively separate agglutinates. We used vibration pulses of the feed and chute to ensure that
253 most particles fell into the two bins at the end of the chute, and we considered everything that
254 went down the chute to be the low-agglutinate remnant (i.e., [Figures 3A, 3D](#) combined with
255 [Figures 3B, 3E](#)). The most magnetic particles were held suspended in the chute by the magnet;
256 after setting aside the low-agglutinate remnant we gathered the magnetically suspended particles
257 after slowly reducing the current to zero. These particles constituted our agglutinate-rich separate
258 ([Figures 3C, 3F](#)). We found that this agglutinate-rich separate contained ~15–45% agglutinates
259 (poorer than the 80–90% purity obtained by [Via and Taylor \(1976b\)](#) using a hand magnet) while
260 the low-agglutinate remnant contained >90% non-agglutinates (comparable to the 80–95% purity
261 of [Via and Taylor \(1976b\)](#)). We then employed the manual separation method to improve the
262 purity of the agglutinate-rich separate, both removing any non-agglutinates from the separate and
263 also recovering agglutinates from the low-agglutinate remnant to leave just non-agglutinates
264 ([Tables S1, S2](#)). We refer to this second separation method as the magnetic–manual method.

265 Note that the magnetic separation step in our magnetic–manual method differs from that
266 used by [Adams and McCord \(1973\)](#). While we poured our regolith samples down the chute of
267 the magnetic separator, they poured their samples down an ethanol-filled pipette attached to their
268 magnetic separator. We initially tried their method, but had difficulty achieving successful
269 separation; the process was time-intensive, the soil particles tended to get stuck on the pipette

270 valve, and the agglutinate-rich separate yielded by this method still included a substantial
271 number of non-agglutinates (particularly, dark, glassy melt breccia particles). Given these
272 difficulties of using the pipette, we opted for a simpler magnetic separation step using the
273 magnetic separator chute instead of a pipette, as already described.

274
275 The magnetic–manual method does not resolve all ambiguity in determining what is an
276 agglutinate or not. The current selected for the magnet affects which regolith particles end up on
277 either side of the dividing line, and the existence of the intermediate population of regolith
278 particles (Figures 3B, 3E) suggests again there is not a clear cut-off. Further, because the
279 magnetic separation is imperfect, one must still manually inspect and decide on whether or not
280 each particle is an agglutinate. This is unfortunate as it is this manual separation step that makes
281 both methods time-intensive (taking tens of hours to separate out ~50 mg of agglutinate
282 particles). However the magnetic–manual method was still noticeably faster than the manual
283 method; by initially concentrating agglutinates via the magnetic separation step, far fewer
284 particles needed to be separated during the manual separation step.

285
286 The two separation methods were independently applied to soil 62231. This yielded two
287 agglutinate separates: one separated using the manual method and one using the magnetic–
288 manual method. Measurements of these two 62231 agglutinate separates, identical except for
289 sorting method, allow us to compare the effectiveness of the two separation methods.
290

291 2.3 Collecting reflectance spectra

292 We collected reflectance spectra of the samples at the Reflectance Experiment
293 Laboratory (RELAB) at Brown University (Milliken et al., 2016). A bidirectional spectrometer
294 (Figure S1, Table S3) was used to gather Vis–NIR spectra from 0.32–2.55 μm at a sampling
295 interval of 10 nm (under ambient environmental conditions) and an FT-IR spectrometer was used
296 for 2–25 μm at a sampling interval of 4 cm^{-1} (in a dry-air purged environment). In this work we
297 focus solely on the Vis–NIR reflectance spectra, but the FT-IR data are available to interested
298 readers (see Open Research section). Twenty samples were measured: unsorted soil, magnetic–
299 manual separated agglutinates, and magnetic–manual separated non-agglutinates from each of
300 the six samples, as well as manually separated agglutinates and non-agglutinates from soil
301 62231. The spectra were gathered with an incidence angle of 30° and an emission angle of 0°
302 (i.e., a phase angle of 30°).

303
304 For the bidirectional spectrometer measurements, the sample was placed in a black
305 Teflon-coated sample dish that rotated as the measurement was taken, averaging out any
306 rotational asymmetries in the sample. Nearly all measurements were made using a 5 mm
307 diameter sample dish holding ~14–19 mg of sample mass and illuminated by a 9 mm diameter
308 beam (Table S4 and Figure S2). For a few samples, additional measurements were taken in a
309 larger 9 mm diameter dish (Figure S3) or using a narrower 4 mm diameter beam to test whether
310 altering these parameters substantially altered the measured spectra (they generally did not, as
311 described in Text S1 and Figures S4–S8).

312
313 Spectra were collected of each sample and of a Spectralon calibration target at the same
314 geometry over four distinct wavelength regions defined by the source lamps and detectors (Table

315 S3). The ratio of sample measurement to Spectralon measurement was recorded as the sample
 316 reflectance spectrum, and the four spectra for the different wavelength regions were stitched
 317 together to form a single, continuous spectrum. A correction was then applied to this spectrum to
 318 account for Spectralon's nonideal behavior; it is not perfectly reflective at all wavelengths of
 319 interest (with absorption bands beyond $\sim 2.14 \mu\text{m}$) and it is not a perfectly Lambertian surface
 320 (Yang et al., 2019; Bruegge et al., 2001; Zhang et al., 2014).

321
 322 Although we are primarily interested in how the spectral properties of lunar soils differ
 323 by separate type (unsorted, non-agglutinates, agglutinates) and by maturity, the reflectance
 324 spectra obtained at RELAB could also be affected by additional factors related to the
 325 measurement process: the use of a depolarizer on the illumination source, the size of the sample
 326 dish, the width of the illumination beam, and the specific soil particles that end up at the
 327 measured sample surface (i.e., sample heterogeneity). We examined each of these factors
 328 individually (see details in Text S1) and found that all were small compared to the largest source
 329 of variation: sample heterogeneity. Because the volumes of our agglutinate and non-agglutinate
 330 separates were small, we typically used a 5 mm dish, and thus the surface area of a soil sample
 331 prepared for spectral measurement consisted of a layer of only a few hundred soil particles. We
 332 collected spectra of each sample multiple times, with the sample emptied from and returned to
 333 the sample cup between each measurement to randomize the soil particles present on the surface.
 334 We found that this was the dominant source of variability in the spectra (as seen in the spread in
 335 individual spectra in Figure 9).

336

337 2.4 Characterizing reflectance spectra

338 Following conventions used for Moon Mineralogy Mapper (M^3) data (Mustard et al.,
 339 2011; Nettles et al., 2011), we characterize the reflectance spectra in terms of their spectral
 340 contrast and spectral slope using four parameters: integrated 1 μm band depth, integrated 2 μm
 341 band depth, continuum ratio, and albedo (Table 2). We define these parameters in a manner
 342 similar to the M^3 analyses, but with adjustments to wavelengths to better match the features in
 343 our spectral data.

344

345 The 1 and 2 μm integrated band depths are measures of spectral contrast, defined here as

346

$$IBD_{1\mu m} = \sum_{n=0}^{75} 1 - \frac{R(770 + 10n)}{R_c(770 + 10n)} \quad \text{and} \quad IBD_{2\mu m} = \sum_{n=0}^{93} 1 - \frac{R(1570 + 10n)}{R_c(1570 + 10n)}$$

347

348 where $R(\lambda)$ refers to reflectance at a given wavelength λ , $R_c(\lambda)$ is the continuum reflectance
 349 (defined as a straight line across the absorption band) at wavelength λ , and n is the number of
 350 wavelength intervals to be integrated over. Wavelength is specified here by the starting
 351 wavelength (770 or 1570 nm) and the wavelength interval of the spectral data (10 nm). In other
 352 words, the integrated 1 μm band depth is calculated from spectral data for 770–1520 nm, while
 353 the integrated 2 μm band depth is calculated similarly for 1570–2500 nm.

354

355 The continuum ratio is a measure of spectral slope and is defined here as R_{1550}/R_{750}
 356 (the ratio of reflectance at 1550 nm and at 750 nm, on either side of the 1 μm band). The albedo

357 parameter is a measure of overall visible–near-IR brightness and is defined as R1550, since 1550
358 nm is a wavelength at which the spectral influence of the 1 and 2 μm absorption bands is
359 minimal.
360

361 **3 Results**

362 3.1 Sorted particles

363 3.1.1 Agglutinate abundance

364 We estimate agglutinate abundance as the mass percentage of an agglutinate separate
365 relative to the mass of the soil from which it was separated (i.e., the 125–250 μm size fraction of
366 the given soil) (Figure 4). Note that this metric does *not* account for incorrectly sorted particles
367 within a grain-size separate (Table S2). In comparing these agglutinate abundances to soil
368 maturity (as given by I_S/FeO values from Morris (1978)), mature soils generally have more
369 agglutinates. This is as expected, since agglutinates are a product of the weathering processes
370 that create mature soils. Notably, though, soil 15041 ($I_S/\text{FeO} = 94$; 41% abundance) has a
371 substantially higher agglutinate abundance than the other soils, even compared to the similarly
372 mature soil 62231 ($I_S/\text{FeO} = 91$; 13% abundance).

373
374 We also see a notable difference in agglutinate abundance for 62231 between the sample
375 that was separated by the magnetic–manual method (62231.58) versus the sample that was
376 manually separated (62231.52). A comparison of these separation methods is presented in
377 Section 3.1.2, but, in short, this discrepancy in agglutinate abundance is likely due to the lower
378 effectiveness of the manual separation method: the manual separation method results in
379 substantial contamination of the (nominally) agglutinate separate with non-agglutinate particles,
380 and this additional mass artificially inflates the calculated agglutinate abundance (see estimated
381 purity of the agglutinate and non-agglutinate separates in Table S2).

382

383 3.1.2 Impact of sorting method (manual vs. magnetic–manual)

384 We compare the effectiveness of the two sorting methods—manual and magnetic–
385 manual—using microscopic images of the agglutinate separate yielded by each method (Figure
386 5). Most of the particles in both agglutinate samples show the telltale markers of an agglutinate:
387 irregular shape, vesicularity, a brown color, and a surface texture that varies from rough to
388 glassy, interspersed with mineral fragments. However, we see that not every particle in either
389 separate is an agglutinate, indicating that neither was perfectly sorted.

390

391 To quantify how successful each sorting method was, we calculate the sample purity (i.e.,
392 the ratio of correctly sorted particles to total particles) by counting the number of agglutinate
393 particles and non-agglutinate particles in microscope images where a representative sample of
394 ~ 50 particles are present (not shown). We find that the magnetic–manual sorting method yielded
395 a higher-purity 62231 agglutinate separate than the manual method did (95% vs. 75%). We
396 similarly calculate the purity of the 62231 non-agglutinate separates resulting from the two
397 methods and find both to have a purity of about 90%. We also calculate purities for the other
398 samples (all magnetic–manual separated), which yields purities comparable to those for the

399 magnetic–manual separated 62231: on average about 95% agglutinate purity, 85% non-
400 agglutinate purity.

401
402 Based on these observations we conclude that the magnetic–manual method is more
403 effective than the manual method at producing a pure agglutinate separate. Therefore, in the
404 following sections we include only results from the magnetic–manual separated 62231 sample
405 when discussing soil 62231, omitting the manually separated sample data (available in
406 [Supplemental Material: Text S2, Figures S9 and S10](#)).

407

408 3.1.3 Variations in agglutinate and non-agglutinate appearance with composition 409 and maturity

410 Four of the agglutinate samples—14259, 62231, 15041, and 79221—have comparable
411 maturity (I_S/FeO values ranging from 81 to 94), but differ in composition ([Table 1](#)). As expected
412 based on their iron and titanium content, the non-agglutinate particles are visually distinct based
413 on composition, ranging from an overall brighter appearance for the highland soil to darker for
414 the ilmenite-rich high-Ti mare soil ([Figure 6](#)). Although the agglutinates from all of these soils
415 appear morphologically similar, there is also a noticeable darkening of the particles in going
416 from highlands to non-mare to mare soil ([Figure 7](#)). Hence, despite vitrification and a large
417 population of opaque npFe, agglutinate appearance and reflectance are not the same from site to
418 site, but depend on local composition.

419

420 There is also a substantial difference in agglutinate appearance across soils 67461, 61141,
421 and 62231 ([Figures 8A, 8C, 8E](#)). These soils are similar in composition owing to their shared
422 highlands origin at the Apollo 16 landing site, so differences in appearance are primarily a result
423 of differing maturity: soil 67461 is immature ($I_S/FeO = 25$), 61141 is submature ($I_S/FeO = 56$),
424 and 62231 is mature ($I_S/FeO = 91$). The morphology of the agglutinates is generally comparable
425 across the three soils, but the agglutinates of the immature soil 67461 appear to have a more
426 abundant population of high-reflectance clasts visible at their surface (see [Figures 8A vs. 8C and](#)
427 [8E](#)). Overall, the agglutinates from the more mature soils 61141 and 62231 are noticeably darker
428 by eye than the agglutinates from the immature soil 67461 (a qualitative observation that is
429 quantified in [Section 3.2](#)). The trend of darkening with maturity also holds true for the non-
430 agglutinates from each soil ([Figures 8B, 8D, 8F](#)).

431

432 3.2 Vis–NIR reflectance of agglutinates and non-agglutinates

433 Here we compare reflectance properties of the 125–250 μm agglutinate and non-
434 agglutinate separates by examining the reflectance spectra ([Figures 9, 10](#)) as well as the
435 calculated spectral parameters defined in [Section 2.4](#) ([Table 2, Figure 11](#)). We similarly analyze
436 reflectance spectra of 75–125 μm separates in [Section 3.2.3](#), in which we consider the effects of
437 particle size. Although we calculated four spectral parameters—integrated 1 μm band depth,
438 integrated 2 μm band depth, continuum ratio, and albedo—the two band depth parameters are
439 highly correlated ([Table 2](#)). Therefore, we can adequately characterize the spectra with only
440 three parameters, and we omit mention of the integrated 2 μm band depth hereafter.

441

442 Note that the reflectance of each 125–250 μm unsorted soil, at any given wavelength,
443 should be intermediate to that of the 125–250 μm agglutinate and non-agglutinate separates
444 (since the agglutinate and non-agglutinate separates together constitute the unsorted soil).
445 However, this is only true for two soils: 67461 and 62231. The other four soils have wavelength
446 regions where the mean agglutinate and non-agglutinate spectra are both lower in reflectance
447 than the mean unsorted soil spectrum (Figure 9). As described in Section 2.3 (and discussed in
448 greater detail in Text S1), we explored the causes of variance among spectra collected multiple
449 times of the same sample, and found it was dominantly the result of heterogeneity within a soil
450 and the small number of grains seen by the spectrometer at a time. This was recognized as
451 differences in reflectance when the sample was emptied from and then returned to the sample
452 dish, thereby randomizing which particles were on the surface. In particular, the largest
453 variations were observed for the absolute reflectance of the unsorted and non-agglutinate spectra,
454 which are inherently more heterogeneous (e.g., Figure 2B), whereas the agglutinate spectra were
455 highly consistent (more uniformly dark particles, e.g., Figure 2A). To convey this variance,
456 Figure 9 shows not only the *average* spectrum for each sample, but also the *individual* spectra
457 that constitute each average.
458

459 3.2.1 General spectral patterns

460 While the reflectance spectra of each soil's agglutinate and non-agglutinate components
461 exhibit nuances related to maturity and composition (discussed below), there are general spectral
462 patterns that are present across all six soils. Marked differences in continuum slope, absorption
463 band strength, and albedo distinguish agglutinates and non-agglutinates from each other and
464 from the unsorted soil.
465

466 In general, the spectral slope is predicted moreso by the separate type (agglutinate, non-
467 agglutinate, unsorted) than by the composition of the bulk soil (highlands, nonmare, low-Ti
468 mare, high-Ti mare) (Figure S11). For every soil, agglutinates are reddest in spectral slope
469 (continuum ratios of 1.47–1.64), non-agglutinates are bluest (1.11–1.24), and unsorted soils are
470 intermediate (1.13–1.39) (Table 2, Figures 10, 11).
471

472 The 1- and 2- μm absorption bands, due to FeO-bearing minerals and glass, also display
473 variations by separate type (Table 2, Figure 11). For each soil, the agglutinates have weaker
474 absorption bands than both the unsorted soil and non-agglutinates, with the integrated 1 μm band
475 depth for agglutinates being on average 25% (13–41%) smaller than for the unsorted soil and on
476 average 41% (23–58%) smaller than for non-agglutinates (Table 2).
477

478 For each soil, the albedo parameter is comparable for the non-agglutinate and unsorted
479 separates (Table 2, Figure 10), but lower for the agglutinates: the albedo parameter for
480 agglutinates is on average 19% (10–38%) less than for the unsorted soil and on average 20%
481 (10–38%) less than for non-agglutinates (Table 2). Moreover there is a distinct impact of soil
482 composition on this parameter; it distinguishes the separates of the highlands (Apollo 16) soils
483 from the separates of the nonmare (Apollo 14), low-Ti mare (Apollo 15), and high-Ti mare
484 (Apollo 17) soils, as visually evident in the two-parameter space of albedo vs. integrated 1 μm
485 band depth (Figure 11).
486

487 These three parameters—continuum ratio, integrated 1 μm band depth, and albedo—
 488 broadly differentiate the reflectance spectra of agglutinates, non-agglutinates, and unsorted soil.
 489 Notably, in the two-parameter space of continuum ratio vs. integrated 1 μm band depth, the
 490 agglutinate spectra form a cluster distinct from the unsorted and non-agglutinate spectra due to
 491 their high continuum ratio (red slope) and small integrated 1 μm band depths (weak absorption
 492 bands) (Figure 11). The non-agglutinate and unsorted clusters are not as well-separated, and their
 493 overlap is attributable to the effects of soil maturity: the immature 67461 soil has non-agglutinate
 494 and unsorted separates that are close together in this space (i.e., they are similar in both
 495 continuum ratio and integrated 1 μm band depth).
 496
 497

Separate Type	Soil Name	Spectral Parameter			
		Int. 1 μm Band Depth	Int. 2 μm Band Depth	Continuum Ratio	Albedo
Unsorted	67461	4.45	5.11	1.13	0.29
	61141	3.06	2.17	1.28	0.16
	62231	3.38	2.59	1.18	0.18
	14259	3.75	4.79	1.34	0.10
	15041	4.89	4.91	1.39	0.10
	79221	4.23	3.51	1.33	0.09
	Average	3.96	3.85	1.27	0.15
Non-agglutinates	67461	4.32	5.55	1.11	0.29
	61141	3.61	2.91	1.19	0.16
	62231	3.47	3.04	1.13	0.19
	14259	4.91	7.18	1.22	0.10
	15041	10.07	10.88	1.24	0.10
	79221	6.54	5.59	1.23	0.09
	Average	5.49	5.86	1.19	0.15
Agglutinates	67461	3.31	1.74	1.55	0.18
	61141	2.42	0.88	1.64	0.14
	62231	1.99	0.76	1.55	0.14
	14259	3.28	2.49	1.60	0.09
	15041	4.25	3.59	1.47	0.08
	79221	2.80	1.41	1.56	0.08
	Average	3.01	1.81	1.56	0.12

498 **Table 2.** Spectral parameters calculated for the mean reflectance spectra of the 125–250 μm
 499 unsorted, non-agglutinate, and agglutinate separates. Integrated band depths are defined over
 500 770–1520 nm (for the 1 μm band) and 1570–2500 nm (for the 2 μm band). Continuum ratio is

501 defined as R1550/R750. Albedo is R1550. See [Section 2.4](#) for more detailed definitions of these
502 parameters.
503

504 3.2.2 Effects of varying maturity

505 The three Apollo 16 soils have similar highlands composition ([Taylor et al., 2010](#)) and
506 thus their spectral differences ([Figure 12](#)) are largely attributable to variations in maturity.
507 Namely, we see notable differences in albedo and absorption band strength—and minor
508 differences in continuum slope—that set the immature soil (67461) separates apart from those of
509 the submature (61141) and mature (62231) soils. While previous studies have presented similar
510 results to those detailed below for unsorted soils (e.g., [Taylor et al., 2001a, 2010](#); [Noble et al.,](#)
511 [2001](#)) here we also explore the spectral changes related to maturity for the non-agglutinate and
512 agglutinate separates, as well as their relative spectral contributions to the unsorted soil.

513
514 The spectrum for each immature separate has a higher albedo than the corresponding
515 submature and mature separate ([Figure 11](#), [Figure 12 left](#)), which aligns with the visually
516 brighter appearance of the immature particles ([Figure 8](#)). This higher albedo is most striking for
517 the unsorted soils (albedo parameter of 0.29 for immature vs. 0.16 for submature and 0.18 for
518 mature) and for the non-agglutinates (0.29 vs. 0.16 and 0.19), and evident to a lesser extent for
519 the agglutinates (0.18 vs. 0.14 and 0.14) ([Table 2](#)). Agglutinates are thus low in albedo regardless
520 of the level of maturity of the associated soil, but the albedo of non-agglutinate separates
521 decreases substantially from immature to mature. We note that for the non-agglutinates, it
522 appears that there is a larger fraction of moderate-albedo breccia fragments in the submature and
523 mature samples than in the immature sample ([Figure 8](#)). These dark, glassy melt breccias were of
524 higher abundance in the moderately magnetic particles of each sample ([Figures 3B, 3E](#)).

525
526 For each separate type (i.e., unsorted, non-agglutinates, agglutinates), the absorption
527 bands are stronger (i.e., the integrated 1 μm band depth is greater) for the immature sample than
528 for the submature and mature samples ([Figures 11, 12](#)). For the unsorted separates, the immature
529 sample's integrated 1 μm band depth is 45% greater than for the submature sample and 32%
530 greater than for the mature sample. For the non-agglutinates these band depth differences are
531 more modest, with the immature sample's band depth being 20% and 25% greater than for the
532 submature and mature samples, respectively. For the agglutinates these band depth differences
533 are much larger, with the immature sample's band depth being 37% and 67% greater than for the
534 submature and mature samples, respectively.

535
536 The continuum slope shows the smallest differences attributable to maturity ([Figures 11,](#)
537 [12](#)). For the unsorted separates, the immature sample's continuum ratio is 12% less than for the
538 submature sample and 4% less than for the mature sample. For the non-agglutinates these
539 continuum ratio differences are smaller, with the immature sample's band depth being 7% and
540 2% less than for the submature and mature samples, respectively. For the agglutinates these
541 continuum ratio differences are even smaller, with the immature sample's continuum ratio being
542 6% and 0% less than for the submature and mature samples, respectively. Thus, while the
543 agglutinates overall have a substantially redder slope than the non-agglutinates, this red slope is
544 largely consistent among immature, submature, and mature agglutinates.

545

546 For the submature (61141) soil, we observe a peculiarity in the spectra. One might expect
 547 the submature separates to have spectral characteristics intermediate to those of the immature
 548 and mature soil separates. Yet the spectra for the submature separates have steeper continuum
 549 slopes and lower albedos than the corresponding spectra for the immature and mature soils (one
 550 exception: the submature agglutinates have an albedo comparable to that of the mature
 551 agglutinates). These results for the submature soil are in contrast to expectations based on
 552 maturity, but they are congruent with the spectral measurements of [Taylor et al. \(2010\)](#), who
 553 observed spectra of some size fractions of submature soil 61141 to be lower in reflectance than
 554 the corresponding spectra of the more mature soil 62231 (unlike spectra of another submature
 555 soil (67481) and another mature soil (64801), which were higher in reflectance than the
 556 corresponding spectra of 62231; see Figure 5 in [Taylor et al., 2010](#)). Given these observations,
 557 the unexpected spectral properties of soil 61141 seem to be characteristic of that soil itself (rather
 558 than being representative of submature agglutinates, non-agglutinates, or unsorted soil in
 559 general). Therefore the spectral differences we observe between separates of submature soil
 560 61141 and of mature soil 62231 do not necessarily have broader implications for how the
 561 spectral properties of agglutinates and non-agglutinates change as a given soil matures.
 562

563 3.2.3 Effects of particle size

564 For sample 62231, we explored one additional variable: particle size. Differences in
 565 particle size are associated with changes in composition and reflectance (e.g., [Taylor et al.,](#)
 566 [2001a, 2001b, 2010; Noble et al., 2001](#)), and the reflectance spectra of smaller particle size
 567 separates (i.e., 10–20 and 20–45 μm) tend to be most similar to the bulk soil ([Pieters et al., 1993;](#)
 568 [Fischer, 1995](#)). This motivates an analysis of spectral properties for smaller particle sizes, but our
 569 magnetic–manual method of concentrating agglutinates is only practical for particle sizes larger
 570 than $\sim 125 \mu\text{m}$, as smaller particles cling to tweezers and other tools. Thus we performed a
 571 magnetic-only separation on the 75–125 μm size fraction of sample 62231. Unfortunately,
 572 without changes to procedure (e.g., using the ethanol-filled pipette method of [Adams and](#)
 573 [McCord \(1973\)](#), as described in [Section 2.2](#)), magnetic separation of even smaller size fractions
 574 is not feasible—particles cling to the magnetic separator and do not flow easily down its chute.
 575

576 The magnetic separation yielded three distinct 75–125 μm separates: the agglutinate-
 577 rich/highly magnetic particles (hereafter referred to as *aggl-rich (high-mag)*), the low-
 578 agglutinate/moderately magnetic particles (*low-aggl (mid-mag)*) and the no agglutinate/least
 579 magnetic particles (*no aggl (low-mag)*) ([Figure 3](#)). Recall that these same three separates were
 580 yielded by magnetic separation of the larger 125–250 μm size fraction as well, but in that case
 581 we combined the low-aggl (mid-mag) and no aggl (low-mag) separates and then manually sorted
 582 them into the agglutinate and non-agglutinate categories. In contrast, for the 75–125 μm size
 583 fraction we have no subsequent manual separation, so we retained the three magnetic groups.
 584 This discrepancy complicates direct comparisons between the 75–125 μm and 125–250 μm
 585 separates, but we can still draw some comparisons, as follows.
 586

587 The 75–125 μm aggl-rich (high-mag) separate is comparable to the 125–250 μm
 588 agglutinate separate, but has lower purity of agglutinates: it is missing some less-magnetic
 589 agglutinates and includes some highly magnetic non-agglutinates. Yet when we consider the
 590 spectra for these separates (the dotted yellow and dotted dark yellow spectra in [Figure 13](#)), we

591 find that they are almost identical in both absolute and normalized reflectance, despite the
592 differences in particle size and purity between these samples. We might have expected the 75–
593 125 μm agglutinates to have higher reflectance than the 125–250 μm agglutinates based on their
594 smaller particle size as well as their lower purity (i.e., a higher percentage of the brighter non-
595 agglutinate particles).

596
597 The 75–125 μm low-aggl (mid-mag) and no aggl (low-mag) separates (dashed medium
598 blue and light blue spectra in [Figure 13](#)), if mixed together, would be most comparable to the
599 125–250 μm non-agglutinate separate (dashed dark blue). However this mixture would have
600 lower purity of non-agglutinates, as it would be missing highly magnetic non-agglutinates and
601 would include some less magnetic agglutinates. When we consider the spectra for these
602 separates, we find that the 75–125 μm low-aggl (mid-mag) is similar in absolute reflectance to
603 the 125–250 μm non-agglutinates, albeit with lower reflectance at wavelengths shorter than 2.0
604 μm . Meanwhile the 75–125 μm no-aggl (low-mag) absolute reflectance is far greater at all
605 wavelengths than for all of the other separates. The normalized reflectance spectra show a
606 different pattern, where the 75–125 μm no-aggl (low-mag) separate and the 125–250 μm non-
607 agglutinates have a similar slope (that is also similar to the 125–250 μm unsorted soil), while the
608 75–125 μm low-aggl (mid-mag) has a redder slope (similar to the 75–125 μm unsorted soil).

609
610 Looking to the unsorted soil spectra (solid gray and black spectra in [Figure 13](#)), we see
611 that the 75–125 μm spectrum is almost identical to the 125–250 μm spectrum at the UV and
612 visible wavelengths, but has higher reflectance and a redder slope in the near-infrared. This
613 matches the spectral patterns observed for the same size fractions of soil 10084 by [Pieters et al.](#)
614 (1993).

616 4 Discussion

617 4.1 Defining an agglutinate

618 Agglutinates constitute a key component of lunar soils, both in terms of abundance (in
619 mature soils) as well as spectral impact. Yet there is ambiguity in how the literature defines what
620 is and isn't an agglutinate. Here we discuss the nuances of defining an agglutinate and how
621 differing definitions of agglutinates impact comparison of our results to those of prior agglutinate
622 work.

623
624 In an abstract, conceptual sense, agglutinates are agglomerates of mineral and lithic
625 fragments bound together by impact-generated glass. However in practice it is challenging to
626 distinguish individual soil particles as agglutinate or non-agglutinate. For example, is a particle
627 still an agglutinate if it contains a minimal, but nonzero amount of agglutinitic glass? Should the
628 distribution of the glass within the particle also be considered (e.g., if the particle is composed of
629 an agglutinitic glass-rich half fused with a glass-free mineral fragment half)? There is no clear
630 agreement on this matter, with the literature of agglutinate analyses using a myriad of definitions
631 of an agglutinate.

632
633 Some studies, as ours, have identified agglutinate particles on the basis of visual
634 appearance ([McKay et al., 1972](#); [Heiken & McKay, 1974](#); [Basu et al., 1982](#)). This approach is

635 effective for particles that are clearly agglutinate or non-agglutinate, but relies on the judgment
636 of the observer for the numerous cases that are difficult to classify (e.g., distinguishing
637 agglutinates from dark, glassy melt breccias). In our study an additional element of variance is
638 introduced to visual identification by our preprocessing step (i.e., our magnetic separation step
639 that concentrated agglutinates), as one's perception of whether a given particle is or isn't an
640 agglutinate may be influenced by characteristics of the surrounding particles (e.g., one's
641 threshold for "agglutinate" may be more stringent when the sample's particles are difficult to
642 categorize and require a high level of scrutiny). In contrast to the visual identification approach,
643 other studies have used quantitative definitions based on particle chemistry and morphology
644 (Simon & Papike, 1981; Taylor et al., 1996, 2001a, 2010). For example, in Taylor et al.'s study
645 of the 90–150 μm size fraction of mare soils, agglutinates were defined as particles with 30–80%
646 Al-rich glass and $>100 \mu\text{m}^2$ total void (vesicle) area (Taylor et al., 1996).

647
648 Further complicating any definition of agglutinates is their evolution over time. Exposure
649 of surface soils to space weathering processes not only increases agglutinate abundance, but also
650 alters the composition, spectral properties, and appearance of the agglutinates themselves. We
651 observed this phenomenon in the Apollo 16 soils of varying maturity, with the agglutinate grains
652 from more mature soils being significantly darker in appearance owing to their higher iron metal
653 content. However these observations were only for Apollo 16 (highlands) soils, so what remains
654 to be seen is how this evolution path of maturing agglutinates varies according to the
655 composition of the bulk soil.

656
657 The soils studied here were chosen because they have been characterized by previous
658 studies in terms of mineralogy, chemistry, maturity, and spectral properties (Taylor et al. 1996,
659 2001a, 2001b, 2010; Basu et al., 1982; McKay et al., 1972; Heiken & McKay, 1974; Simon &
660 Papike, 1981; Morris, 1978; Pieters et al., 2002). By comparing our agglutinate abundance
661 results to those of prior studies (Figure 14), which differ in agglutinate definitions, we can assess
662 how this ambiguity in defining agglutinates can impact measurements of their abundance. For
663 the soils we studied, we calculated agglutinate abundance as a weight percentage based on the
664 masses of the agglutinate and non-agglutinate separates. McKay et al. (1972), Heiken and
665 McKay (1974), and Basu et al. (1982) counted the percentage of agglutinate particles. Simon and
666 Papike (1981) and Taylor et al. (1996) gathered modal abundance (i.e., a volume percentage) of
667 agglutinate particles. The Lunar Soil Characterization Consortium (Taylor et al., 2001a, 2010)
668 also gathered modal abundance, but agglutinitic glass was distinguished based on its chemistry at
669 the sub-particle level (glass within an agglutinate was included and mineral fragments within an
670 agglutinate were excluded, rather than categorizing whole particles as agglutinate or non-
671 agglutinate); impact glasses were also lumped with agglutinitic glass.

672
673 Yet despite these differences in how agglutinate abundance is measured, we see a fairly
674 consistent overall trend of decreasing abundance for larger size fractions (barring the data for soil
675 15041 from Basu et al. (1982), where there isn't a clear correspondence between agglutinate
676 abundance and particle size). Our agglutinate abundance results for the 125–250 μm soils match
677 this trend, but are noticeably on the low end of agglutinate abundance. These low values may
678 indicate a difference in particle mass density between agglutinates and non-agglutinates, as we
679 measured abundance based on mass whereas others used the number or volume of agglutinate
680 particles. Another possible cause may be differences across studies in how often non-agglutinate

681 particles were incorrectly counted as agglutinates and vice versa (particularly when there are
682 similarities in visual appearance and magnetic properties, as for dark, glassy melt breccias).
683

684 4.2 New insights into agglutinate reflectance spectra

685 The agglutinate spectra presented here provide new insights into the spectral variability
686 of agglutinates with respect to soil composition and maturity. These soil characteristics appear to
687 control spectral albedo and band depth (Figure 11). In terms of albedo—measured here as 1550
688 nm reflectance—the Apollo 16 (highlands) agglutinate spectra are all higher in albedo than the
689 Apollo 14 (non-mare), 15 (low-Ti mare), and 17 (high-Ti mare) agglutinate spectra.
690 Furthermore, among the Apollo 16 agglutinate spectra, albedo (and band depth) decreases with
691 increasing soil maturity. However, albedo and band depth do not distinctly separate the
692 agglutinate spectra from the non-agglutinate and unsorted spectra; there is overlap in the range of
693 these parameter values for the agglutinate spectra versus for non-agglutinate and unsorted
694 spectra.

695
696 The patterns in albedo and band depth highlight the complexity of agglutinate spectral
697 properties and can inform future spectral modeling of lunar soils, an area in which agglutinates
698 have historically been modeled simplistically for lack of comprehensive spectral data. For
699 example, some work has simply treated agglutinates as glass, ignoring the effects of their npFe
700 (e.g., Warell & Davidsson, 2010). Some have used the agglutinate spectrum of Pieters et al.
701 (1993) as representative of all agglutinates, despite its high-titanium mare composition (e.g., Li
702 & Li, 2011). Many efforts have used empirical parameters to represent agglutinates (Shkuratov
703 et al., 1999; Clark et al., 2001; Hapke, 2001; Poulet et al., 2002; Poulet & Erard, 2004; Lawrence
704 & Lucey, 2007; Denevi et al., 2008; Nimura et al., 2008). This current work has shown, though,
705 that agglutinate spectra vary substantially across soils of different bulk composition, highlighting
706 that the bulk soil properties must be taken into account for accurate spectral modeling of
707 agglutinates.

708
709 Moreover, agglutinate spectra vary with maturity—at least for the specific immature,
710 submature, and mature soils of this study—suggesting the existence of an agglutinate “life cycle”
711 over which its spectral properties change due to progressive weathering (more specifically due to
712 changes in npFe, such as the increasing npFe abundance indicated by increasing I_S/FeO values of
713 the bulk soil). However, since the soils of different maturities studied here were all (Apollo 16)
714 highlands soils, we cannot yet surmise how this agglutinate life cycle might vary across soils of
715 different composition—will non-highlands agglutinates show different maturing trends, perhaps
716 due to differences in the bulk soil’s iron abundance? It may also be enlightening to study other
717 highland soils to better characterize the robustness of these maturing trends (particularly since
718 the submature soil 61141’s properties may be atypical, such as having a lower albedo than the
719 more mature soil 62231). These are areas for future investigation via study of other immature
720 and submature lunar soils.

721
722 A parameter in which the agglutinate spectra *are* generally comparable is spectral
723 slope—quantified in this work as the continuum ratio of 1550 nm reflectance to 750 nm
724 reflectance. Unlike albedo and band depth, this continuum ratio distinctly separates the

725 agglutinate spectra from the non-agglutinate and unsorted spectra, which all have smaller
726 continuum ratios (Figure 11).

727
728 The spectral slopes of the agglutinate spectra also provide new insight into a question that
729 has persisted in the literature of lunar soils: what causes the characteristic spectral changes
730 (darkening and reddening) of maturing soils? Historically these changes have primarily been
731 attributed to two sources—agglutinates and depositional rims—both of which form and
732 accumulate in the lunar soil as it is exposed to space weathering processes. Agglutinates are
733 typically considered to darken rather than redden the unsorted spectra due to the large npFe
734 within agglutinate particles (e.g., Britt & Pieters, 1994; Keller et al., 1998; Noble et al., 2007;
735 Pieters & Noble, 2016). Meanwhile, reddening is typically attributed to the small npFe found in
736 the rims of all weathered particles (both agglutinates and non-agglutinates) (e.g., Hapke, 1973;
737 Hapke et al., 1975; Cassidy & Hapke, 1975; Hapke, 2001; Pieters et al., 1993). Yet the
738 agglutinate spectra in this study are all quite steep (red) in slope (Figure 10), suggesting that it is
739 the agglutinates (and potentially their depositional rims), rather than the npFe-rich rims of non-
740 agglutinates, that primarily contribute to reddening of the unsorted soil spectra. These results
741 reframe our understanding of the relative spectral roles of agglutinates and rims: agglutinates
742 play a greater role in weathering-associated spectral reddening than previously thought.

743
744 Admittedly we cannot isolate this reddening impact of agglutinates with further
745 granularity—to what extent are these spectral effects attributable to an agglutinate grain's own
746 depositional rim versus its interior? Given that rims can vary in microstructure and chemistry
747 (Keller & McKay, 1997), it is conceivable that the depositional rims that form on agglutinates
748 are fundamentally different from those that form on non-agglutinates, leading to spectral
749 reddening that is attributable to the agglutinate component of the soil, but is driven specifically
750 by the rims of the agglutinates.

751
752 To better understand why agglutinates have such steep spectral slopes despite the large
753 sizes of npFe grains within them, we compared the agglutinate and non-agglutinate spectra to
754 spectra of npFe in silica gel from Noble et al. (2007). These npFe spectra represent a range of
755 average npFe sizes (8 nm, 15 nm, 35 nm, and 40 nm) and weight percentage abundances. Here
756 we consider only the normalized spectra, focusing on how well the agglutinate and non-
757 agglutinate spectra match the npFe spectra in terms of spectral slope (Figure 15).

758
759 The closest spectral matches for both agglutinates and non-agglutinates are npFe with
760 average sizes of 15 nm and 35 nm. However, npFe abundance is a critical parameter in fitting the
761 spectra as well. The agglutinate and ~15 nm npFe spectra match best for npFe abundance of
762 about 0.13 wt%. For matching the agglutinate and ~35 nm npFe spectra, neither the 0.02 wt%
763 nor the 0.20 wt% spectra are truly good fits, with the former being too shallow in slope and the
764 latter too steep. Therefore one might expect an intermediate abundance (between 0.02 wt% and
765 0.20 wt%) of ~35 nm npFe to produce a spectrum that fits the continuum slopes of the
766 agglutinate spectra. In contrast the non-agglutinate spectra are fit best by smaller abundances of
767 ~15 and ~35 nm npFe (about 0.07 wt% and 0.02 wt%, respectively) (Figure 15). These spectral
768 comparisons suggest similar average npFe sizes within both the agglutinates and non-
769 agglutinates, but greater npFe abundance in the agglutinates. Given that increasing npFe

770 abundance in silica gel yields a steeper spectral slope (Figure 15), the abundance of npFe appears
771 to play a key role in causing the steep spectral slope of agglutinates.

772

773 An important caveat here is that this spectral comparison does not provide a constraint on
774 the *size distribution* of npFe within agglutinates, which can also have a spectral impact (e.g.,
775 Lucey & Riner, 2011). The range of npFe sizes can be quite wide for agglutinates—larger npFe
776 is typically found within the agglutinate particle interior, while smaller npFe spherules occur in
777 the particle rims. James et al. (2002) estimated an average npFe size within agglutinates of 120
778 nm with a standard deviation of 20 nm, while Keller and Clemett (2001) estimated an average
779 npFe size of 3 nm in the depositional rims (of all lunar soil particles, not only agglutinates).
780 However these estimates themselves have uncertainties; the scanning electron microscopy used
781 by James et al. is limited in its ability to resolve—and therefore tends to underestimate—the
782 smallest sizes of npFe (<10 nm), while the transmission electron microscopy used by Keller and
783 Clemett images smaller spatial areas and therefore has greater difficulty sampling a
784 representative number of particles (sampling ~1300 npFe particles compared to the ~9600 of
785 James et al.).

786

787 These spectral comparisons motivate future work on the spectral impact of npFe within
788 agglutinates. While npFe size is an important parameter, npFe abundance plays a prominent role
789 as well. Moreover, Arnaut et al. (2021) found that the density and distribution of npFe particles,
790 rather than just their size or abundance, may have distinct effects on spectral slope due to
791 interparticle interactions when the npFe particles are arranged in layers or clusters. The spectral
792 impact of npFe is complex, with nuances that have yet to be fully understood. Future work
793 analyzing SEM and TEM image data of individual agglutinate grains can elucidate this matter.

794

795 4.3 Contribution of agglutinates to bulk reflectance spectra

796 Our new insights into the spectral properties of agglutinates come with an important
797 caveat: they only strictly hold for the 125–250 μm size fraction that we studied. However for
798 applications such as spectral modeling of lunar soils, one would rather understand agglutinate
799 spectral signatures as they exist in situ, across a range of size fractions and mixed within the bulk
800 soil. Therefore we still have two lingering questions: (1) how well do the 125–250 μm size
801 fraction spectra (investigated in this study) represent the spectral properties of bulk agglutinates
802 (across size fractions), and (2) how well does spectral mixing of agglutinates and non-
803 agglutinates in the 125–250 μm size fraction translate to the same spectral mixing in the bulk
804 soil? While definitively answering these questions will require further study, here we discuss
805 what we know based on the findings of this study and the existing literature.

806

807 First, we consider how bulk agglutinate spectra may compare to the 125–250 μm
808 agglutinate spectra gathered in this study. Recall that the 125–250 μm size fraction was chosen
809 for this study owing to practical considerations (feasibility of manual separation) rather than any
810 particular spectral relevance. In fact, Fischer (1995) found smaller particle size separates (10–20
811 and 20–45 μm)—rather than larger size fractions like 125–250 μm —to be most spectrally similar
812 to the bulk (<1 mm) soil. If this pattern holds true within the agglutinate portion of the soil as
813 well, one expects smaller agglutinates to better represent the spectral properties of agglutinates as
814 a whole.

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This spectral dominance of smaller size fractions motivated our analysis of the 75–125 μm size fraction of soil 62231, which aimed to identify patterns of spectral changes as particle size decreases (Section 3.2.3). Although comparisons between the 75–125 μm and 125–250 μm size fractions are complicated by their different separation methods—the former magnetic-only and the latter magnetic–manual—our results suggest that agglutinates for the two size fractions are spectrally quite similar.

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Given the spectral similarity between the 75–125 μm and 125–250 μm agglutinates, can we assume similar agglutinate spectra for smaller size fractions? Our evidence from just two size fractions seems too limited to draw any strong conclusions. However there is some evidence that agglutinates of varying size fractions are at least similar in composition (per the findings of Taylor et al. (2001a, 2001b, 2010) that agglutinitic glass composition is relatively invariant to particle size across the <10 μm , 10–20 μm , and 20–45 μm size fractions). Yet even if agglutinate composition (and associated spectral properties) is invariant to particle size, the particle size itself will influence the spectrum (i.e., smaller particles tend to have higher reflectance).

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Second, let's consider how spectral mixing of agglutinates and non-agglutinates might differ for the bulk soil versus for the 125–250 μm size fraction. We get some insight into this from our analysis of the 75–125 μm and 125–250 μm size fractions of soil 62231. For these two size fractions, the unsorted soil spectra are comparable at visible wavelengths, but diverge at longer wavelengths due to the 75–125 μm spectrum's redder near-IR slope (Figure 13). This redder slope in the smaller size fraction may be due to increased agglutinate abundance (as this abundance tends to increase for smaller size fractions; Figure 14). If so, we would expect the unsorted soil spectra for even smaller size fractions to be comparable to our unsorted spectra at visible wavelengths, but even redder at longer (near-IR) wavelengths. We would then expect similar of the bulk soil spectra, since smaller size fractions best spectrally represent the bulk.

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Striking, though, is how closely the 75–125 μm unsorted spectrum is tracked by the spectrum of the low-aggl (mid-mag) separate (Figure 13). This separate consists of moderately magnetic particles, including many dark, glassy melt breccias and a very small number of agglutinates (Figures 3B, 3E). We did not consider this separate in our other analyses, where all non-agglutinates were lumped together, yet it appears that these moderately magnetic particles may warrant further study as an important contributor to the unsorted soil spectrum.

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The highly influential work of Pieters et al. (1993) also considered the relative contributions of agglutinates and space-weathered rims to the bulk spectra of weathered soils. In that work, agglutinates were also separated from two size fractions (250–500 μm and >500 μm) of sample 10084. The spectra of these agglutinates were compared to spectra of the <250 μm fraction of bulk soil and smaller size fractions, and the fact that these agglutinates were not as red was interpreted to mean that the agglutinates were not the cause of the bulk soil's red spectral slope. However, the 250–500 μm agglutinates were redder than the >500 μm agglutinates, and these agglutinates were not compared to like size fractions.

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860

To test whether the finest fraction of soils (<25 μm) was strongly affected by agglutinates, Pieters et al. measured the reflectance spectrum of an unsorted 45–75 μm size

861 separate from an agglutinate-rich soil, ground down to a size of $<25\ \mu\text{m}$, and found that it was
862 too bright and its slope was shallower compared to the $<25\ \mu\text{m}$ soil fraction. This spectral
863 difference was attributed to the physical difference in the ground sample: its particles had
864 exposed interiors, minimizing the spectral impact of the particle rims. This result, combined with
865 other work suggesting that agglutinate abundance decreases with decreasing particle size
866 (Labotka et al., 1980; Simon et al., 1981), led to the argument that the rims of soil grains in the
867 finest fraction are responsible for the spectral changes associated with space weathering, rather
868 than agglutinates. However, grinding would expose fresh mineral fragments from within the
869 agglutinates, in which glass constitutes only $\sim 30\%$ of the non-vesicle area (Baker et al., 2020).
870 Thus, this ground soil is likely not an appropriate test of the nature of the spectral properties of
871 the finest fraction of agglutinates. Moreover, since that time we now know that agglutinates
872 increase in abundance with decreasing particle size rather than decrease (Figure 14 and
873 references therein), and now, we also know that agglutinates cause spectral reddening. Thus,
874 agglutinates may indeed play an important role in defining the spectra of the finest fraction of
875 soils, and the relative contributions of space-weathered rims and agglutinates to the overall bulk
876 spectra of mature soils remains an important open question.

877
878 These questions regarding the in situ spectral signature of agglutinates could be clarified
879 with further work on separating smaller size fractions of agglutinates. However this would
880 require a different agglutinate/non-agglutinate separation methodology than the one presented
881 here, as we noted that the fine particles ($<75\ \mu\text{m}$) do not easily flow down the magnetic separator
882 chute used in our method. An alternative method that may work for such fine particles is
883 described by Adams and McCord (1973), wherein soil was passed through a column of ethanol
884 attached to a magnetic separator, yielding agglutinate separates from a $<250\ \mu\text{m}$ lunar soil
885 sample. Yet, as described in Section 2.2, this method has its own unique challenges to overcome,
886 including the unintended separation of highly magnetic non-agglutinates alongside the
887 agglutinates.

888

889 4.4 Unaddressed questions on non-agglutinates

890 This work's primary focus has been on the agglutinate portion of lunar soils. To simplify
891 discussion we have referred to the remainder of the soil as the "non-agglutinates", but this catch-
892 all term belies the heterogeneity of these particles and their resulting spectral complexities—
893 itself a valuable topic of study for better understanding the reflectance spectra of bulk lunar soils.
894 Although a thorough spectral characterization of the many types of non-agglutinates is beyond
895 the scope of this work, our findings provide insights into and provoke questions on this subject
896 that we broach here as a starting point for future work.

897

898 In particular, questions arise from the soil separates we obtained from magnetically
899 separating the $75\text{--}125\ \mu\text{m}$ size fraction of the mature soil 62231 (Figure 3). The no aggl (low-
900 mag) separate is far brighter than the unsorted soil it was derived from (Figures 3A, 13), which
901 suggests that, if the particles in this separate have space-weathered rims, these rims do not have a
902 strong spectral impact on the bulk soil. On the other hand the low-aggl (mid-mag) separate is
903 much darker (Figures 3B, 13) and is spectrally similar to the unsorted soil (Figure 13). What
904 makes these two separates—both primarily consisting of non-agglutinates—so visually distinct?

905 What are the features of the non-agglutinate particles in the low-aggl (mid-mag) separate that
906 make it so dark in appearance?

907
908 Whereas the no aggl (low-mag) particles are largely homogeneous, each composed of
909 uniformly high-reflectance mineral fragments (Figures 3A, 3D), the low-aggl (mid-mag)
910 particles appear to contain a large population of more heterogeneous particles—dark, glassy melt
911 breccias—that are intermediate in reflectance (Figures 3B, 3E). In our manual separation, most
912 of these particles would not have been included as agglutinates because they have more angular
913 margins and less glassy texture (see Figure 7). Further study of this population of particles within
914 the low-aggl (mid-mag) separate is needed to determine the nature of their low reflectance and
915 their significance for understanding space weathering processes. Characterizing the spectral
916 properties of these particles and the abundance and nature of their npFe could provide greater
917 insight into how regolith evolves as it is weathered.

918

919 **5 Conclusions**

920 By isolating and characterizing agglutinates from a suite of lunar soils, we aimed to
921 develop a comprehensive understanding of agglutinate properties across the lunar surface. The
922 six soils in the suite represent the diversity of the sampled sites, spanning both a range of soil
923 compositions (highlands 62231, non-mare 14259, low-Ti mare 15041, high-Ti mare 79221) and
924 of soil maturities (immature 67461, submature 61141, mature 62231). While we primarily
925 studied the 125–250 μm size fraction of these soils, we have also considered the impact of
926 particle size by characterizing 62231 agglutinates from the 75–125 μm size fraction as well.

927

928 Regarding methodology, we find that separating out 125–250 μm agglutinates is most
929 effective using a two-step magnetic–manual separation technique rather than either magnetic
930 separation or manual separation alone. This magnetic–manual method yielded sample purities of
931 ~95% for agglutinate separates and ~85% for non-agglutinate separates. However the manual
932 part of this method makes it both time-intensive and limited to large particles; magnetic
933 separation alone may still be the more appropriate method for some applications, such as
934 investigations of fine (<125 μm) particles, and there is certainly still unexplored potential for
935 improving the magnetic separation method (e.g., by optimizing the magnetic separator settings).

936

937 Notably, the reflectance spectra of the agglutinate/non-agglutinate separates and the
938 unsorted soil show similar spectral patterns across all six soils: for each soil the agglutinate
939 spectrum is redder in slope than the unsorted spectrum from the same soil while the non-
940 agglutinate spectrum has a shallower (bluer) slope. Additionally, each agglutinate spectrum is
941 lower in overall reflectance and has weaker absorption bands than the corresponding non-
942 agglutinate and unsorted soil spectra. The consistent pattern in spectral slope is particularly
943 intriguing, suggesting that it is the agglutinates (rather than the non-agglutinates) that contribute
944 to reddening of the unsorted soil—at least for the studied size fractions of 125–250 μm and 75–
945 125 μm .

946

947 Among the Apollo 16 soils of varying maturity, we find that the agglutinates from the
948 immature soil are noticeably higher in reflectance and have weaker absorption bands than the
949 agglutinates from submature and mature soils, but they do not differ substantially in spectral

950 slope. This novel finding suggests the existence of an agglutinate “life cycle”; the weathering of
951 a lunar soil entails not only an increase in agglutinate abundance, but also alteration of the
952 physical and spectral properties of said agglutinates.

953

954 The npFe within weathered rims of soil grains and agglutinates is generally considered
955 the driver of reddening and darkening of lunar soils, with smaller npFe (<10 nm) primarily
956 reddening and larger npFe (>40 nm) primarily darkening (Noble et al., 2007). Based on
957 comparison to spectra of npFe in silica gel from Noble et al. (2007), we find that the *abundance*
958 of npFe may be particularly relevant as well for agglutinate-associated reddening, with the best
959 spectral matches between agglutinates and npFe being at higher npFe abundances than the best
960 matches between non-agglutinates and npFe. However this analysis does not constrain the
961 distribution of npFe sizes within each agglutinate particle. Future studies of the agglutinate
962 separates may provide a more robust estimate of npFe abundance and size distribution, providing
963 greater context for the agglutinate spectral characterization presented in this study.

964

965 Our work here begins to illustrate the spectral complexities of lunar agglutinates. In
966 particular, our findings suggest that agglutinates play a different spectral role in the weathering
967 of lunar soils than previously thought, contributing not only to the darkening of mature soils, but
968 also to their reddening. Yet there is still much to be done to comprehensively characterize the
969 spectral properties of lunar agglutinates, and numerous avenues of future work are motivated by
970 the myriad questions that remain. For instance, how might agglutinate spectra differ across grain
971 size fractions, beyond the 125–250 μm size fraction that we primarily focused on in this study
972 (and particularly for the smaller size fractions that may better represent the spectral properties of
973 the bulk soil)? How do agglutinate and non-agglutinate particles spectrally mix, and how do non-
974 linearities in this mixing process impact the spectrum of the bulk soil? And, complicating the
975 debate on the spectral role of agglutinates versus the space-weathered rims on soil grains, to what
976 extent can the spectral properties of agglutinates be attributed to space-weathered rims on the
977 agglutinate grains themselves? Perhaps these questions and more will be answered by future
978 studies of lunar agglutinates, further refining our understanding of the lunar surface and its
979 weathering over time.

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989 Acknowledgments

990
991 We thank Ben Wing, Karen Stockstill-Cahill, and Karl Hibbitts (Johns Hopkins University
992 Applied Physics Laboratory) for initial assistance with spectral characterization of the lunar soil
993 samples. We also thank the two reviewers Tim Glotch and Bruce Hapke, as well as Carle Pieters
994 and Michelle Thompson, for thoughtful feedback that improved the manuscript.

995 This work was primarily funded by NASA LDAP grant 80NSSC17K0418 to co-author BWD.
996 The contributions of co-authors ALG, MLZ, and LMB were facilitated by the ASPIRE program
997 (APL's Student Program to Inspire, Relate, and Enrich).
998 RELAB is a multiuser facility supported by NASA grants.
999

1000 1001 Open Research

1002
1003 Materials used in and produced by this work can be located as follows.
1004

1005 Soil samples:

1006 Apollo lunar soils were obtained from the NASA Johnson Space Center. The sample IDs for the
1007 soils are: 67461.168, 67461.45, 61141.5, 62231.52, 62231.58, 14259.136, 15041.47, 79221.158.
1008

1009 Reflectance spectra:

1010 All spectral data gathered for this study (of agglutinate/non-agglutinate separates and of unsorted
1011 soils) can be downloaded from the RELAB Spectral Database within the PDS Geosciences Node
1012 Spectral Library (<https://pds-speclib.rsl.wustl.edu/search.aspx?catalog=RELAB>). The Specimen
1013 IDs are LS-BWD-[xxx], where [xxx] ranges from 140–165 (e.g., LS-BWD-140).

1014 These spectra are also available directly from the RELAB Spectral Database
1015 (<https://sites.brown.edu/rehab/rehab-spectral-database/>).
1016

1017 The spectra of nanophase iron in silica gel from [Noble et al. \(2007\)](#) can be downloaded from the
1018 RELAB Spectral Database within the PDS Geosciences Node Spectral Library ([https://pds-
1019 speclib.rsl.wustl.edu/search.aspx?catalog=RELAB](https://pds-speclib.rsl.wustl.edu/search.aspx?catalog=RELAB)). The Specimen IDs are SN-CMP-[xxx],
1020 where [xxx] is 016, 019, 021, 023, 027, 032, 037–040, 045–050, 054–060, 062, 064, 066, 068,
1021 069, 074–129, 139–145 (e.g., SN-CMP-016). Note that in this study we plotted the silica gel
1022 spectra measured over 300–2600 nm (rather than the 300–880 nm spectra that are also found
1023 under the same Specimen IDs).

1024 These spectra are also available directly from the RELAB Spectral Database
1025 (<https://sites.brown.edu/rehab/rehab-spectral-database/>).
1026

1027 Software:

1028 Microscope images were edited (contrast stretching for Figure 1; white balancing for Figures 2,
1029 5–8) and all figures were formatted using Adobe Photoshop.

1030 Data analysis and plotting were done using Python v3.9.16 ([Python Software Foundation, 2022](#))
1031 with additional packages matplotlib v3.7.1 ([Hunter, 2007](#)), pandas v1.5.3 ([The pandas
1032 development team, 2023](#)), numpy v1.23.5 ([Harris et al., 2020](#)), and tabulate v0.8.10 ([Astanin,
1033 2022](#)).
1034

1035 **References**

- 1036
- 1037 Adams, J. B., & Charette, M. P. (1975). Effects of maturation on the reflectance of the lunar
1038 regolith: Apollo 16 — A case study. *The Moon*, 13(1), 293–299.
1039 <https://doi.org/10.1007/BF00567521>
- 1040 Adams, J. B., & McCord, T. B. (1970). Remote sensing of lunar surface mineralogy:
1041 Implications from visible and near-infrared reflectivity of Apollo 11 samples. In
1042 *Geochimica et Cosmochimica Acta Supplement, Volume 1. Proceedings of the Apollo 11*
1043 *Lunar Science Conference held 5-8 January, 1970 in Houston, TX. Volume 3: Physical*
1044 *Properties. Edited by AA Levinson. New York: Pergamon Press, 1970., p. 1937 (Vol. 1, p.*
1045 *1937).*
- 1046 Adams, J. B., & McCord, T. B. (1971a). Alteration of Lunar Optical Properties: Age and
1047 Composition Effects. *Science*, 171(3971), 567–571.
1048 <https://doi.org/10.1126/science.171.3971.567>
- 1049 Adams, J. B., & McCord, T. B. (1971b). Optical properties of mineral separates, glass, and
1050 anorthositic fragments from Apollo mare samples. In *Lunar and Planetary Science*
1051 *Conference Proceedings (Vol. 2, p. 2183).*
- 1052 Adams, J. B., & McCord, T. B. (1973). Vitrification darkening in the lunar highlands and
1053 identification of Descartes material at the Apollo 16 site. In *Lunar and Planetary Science*
1054 *Conference Proceedings (Vol. 4, p. 163).*
- 1055 Arnaut, M., Wohlfarth, K., & Wöhler, C. (2021). The interaction between multiple nanophase
1056 iron particles changes the slope of lunar reflectance spectra. Presented at the EPSC2021,
1057 Copernicus Meetings. <https://doi.org/10.5194/epsc2021-770>

- 1058 Astanin, S. (2022, June 21). Tabulate (Version 0.8.10) [OS Independent]. Retrieved from
1059 <https://github.com/astanin/python-tabulate>
- 1060 Baker, A. E., Jolliff, B. L., Carpenter, P., Yasanayake, C. N., & Denevi, B. W. (2020). Lunar
1061 Agglutinate Glass Composition and Implications for Agglutinate Formation (Abstract P054-
1062 0016). Presented at the AGU Fall Meeting 2020. Retrieved from
1063 <https://agu.confex.com/agu/fm20/meetingapp.cgi/Paper/765987>
- 1064 Basu, A., McKay, D. S., Griffiths, S. A., & Nace, G. (1982). Regolith maturation on the earth
1065 and the moon with an example from Apollo 15. In *Lunar and Planetary Science Conference*
1066 *Proceedings* (Vol. 12, pp. 433–449).
- 1067 Basu, A., Wentworth, S. J., & McKay, D. S. (2002). Heterogeneous agglutinitic glass and the
1068 fusion of the finest fraction (F3) model. *Meteoritics & Planetary Science*, 37(12), 1835–
1069 1842. <https://doi.org/10.1111/j.1945-5100.2002.tb01167.x>
- 1070 Britt, D. T., & Pieters, C. M. (1994). Darkening in black and gas-rich ordinary chondrites: The
1071 spectral effects of opaque morphology and distribution. *Geochimica et Cosmochimica Acta*,
1072 58(18), 3905–3919. [https://doi.org/10.1016/0016-7037\(94\)90370-0](https://doi.org/10.1016/0016-7037(94)90370-0)
- 1073 Bruegge, C., Chrien, N., & Haner, D. (2001). A Spectralon BRF data base for MISR calibration
1074 applications. *Remote Sensing of Environment*, 77(3), 354–366.
1075 [https://doi.org/10.1016/S0034-4257\(01\)00214-0](https://doi.org/10.1016/S0034-4257(01)00214-0)
- 1076 Burgess, K., & Stroud, R. (2017). Glassy with a Chance of Nanophase Iron: Space Weathering
1077 of Lunar Soil as Observed with Aberration-Corrected Scanning Transmission Electron
1078 Microscopy. *Microscopy Today*, 25(3), 32–39. <https://doi.org/10.1017/S1551929517000372>

- 1079 Burgess, K. D., & Stroud, R. M. (2018). Coordinated Nanoscale Compositional and Oxidation
1080 State Measurements of Lunar Space-Weathered Material. *Journal of Geophysical Research:*
1081 *Planets*, 123(8), 2022–2037. <https://doi.org/10.1029/2018JE005537>
- 1082 Carrier, W. D. (2003). Particle Size Distribution of Lunar Soil. *Journal of Geotechnical and*
1083 *Geoenvironmental Engineering*, 129(10), 956–959. [https://doi.org/10.1061/\(ASCE\)1090-
1084 0241\(2003\)129:10\(956\)](https://doi.org/10.1061/(ASCE)1090-0241(2003)129:10(956))
- 1085 Cassidy, W., & Hapke, B. (1975). Effects of darkening processes on surfaces of airless bodies.
1086 *Icarus*, 25(3), 371–383. [https://doi.org/10.1016/0019-1035\(75\)90002-0](https://doi.org/10.1016/0019-1035(75)90002-0)
- 1087 Christoffersen, R., McKay, D. S., & Keller, L. P. (1996). Microstructure, chemistry, and origin of
1088 grain rims on ilmenite from the lunar soil finest fraction. *Meteoritics & Planetary Science*,
1089 31(6), 835–848. <https://doi.org/10.1111/j.1945-5100.1996.tb02117.x>
- 1090 Clark, B. E., Lucey, P., Helfenstein, P., Bell Iii, J. F., Peterson, C., Veverka, J., et al. (2001).
1091 Space weathering on Eros: Constraints from albedo and spectral measurements of Psyche
1092 crater. *Meteoritics & Planetary Science*, 36(12), 1617–1637. [https://doi.org/10.1111/j.1945-
1093 5100.2001.tb01853.x](https://doi.org/10.1111/j.1945-5100.2001.tb01853.x)
- 1094 Conel, J. E., & Nash, D. B. (1970). Spectral reflectance and albedo of Apollo 11 lunar samples:
1095 Effects of irradiation and vitrification and comparison with telescopic observations.
1096 *Geochimica et Cosmochimica Acta Supplement*, 1, 2013.
- 1097 Denevi, B. W., Lucey, P. G., & Sherman, S. B. (2008). Radiative transfer modeling of near-
1098 infrared spectra of lunar mare soils: Theory and measurement. *Journal of Geophysical*
1099 *Research: Planets*, 113(E2). <https://doi.org/10.1029/2007JE002929>

- 1100 Denevi, Brett W., Noble, S. K., Christoffersen, R., Thompson, M. S., Glotch, T. D., Blewett, D.
1101 T., et al. (2023). Space Weathering At The Moon. *Reviews in Mineralogy and*
1102 *Geochemistry*, 89(1), 611–650. <https://doi.org/10.2138/rmg.2023.89.14>
- 1103 Fischer, E. M. (1995). *Quantitative compositional analysis of the lunar surface from reflectance*
1104 *spectroscopy: Iron, aluminum, and model for removing the optical effects of space*
1105 *weathering* (Ph.D.). Brown University, United States -- Rhode Island. Retrieved from
1106 <https://www.proquest.com/docview/304164546/abstract/BA6999E74DEA4539PQ/1>
- 1107 Hapke, B. (1973). Darkening of silicate rock powders by solar wind sputtering. *The Moon*, 7(3),
1108 342–355. <https://doi.org/10.1007/BF00564639>
- 1109 Hapke, B. (2001). Space weathering from Mercury to the asteroid belt. *Journal of Geophysical*
1110 *Research: Planets*, 106(E5), 10039–10073. <https://doi.org/10.1029/2000JE001338>
- 1111 Hapke, B., Cassidy, W., & Wells, E. (1975). Effects of vapor-phase deposition processes on the
1112 optical, chemical, and magnetic properties of the lunar regolith. *The Moon*, 13(1), 339–
1113 353. <https://doi.org/10.1007/BF00567525>
- 1114 Harris, C. R., Millman, K. J., van der Walt, S. J., Gommers, R., Virtanen, P., Cournapeau, D., et
1115 al. (2020). Array programming with NumPy. *Nature*, 585(7825), 357–362.
1116 <https://doi.org/10.1038/s41586-020-2649-2>
- 1117 Heiken, G., & McKay, D. S. (1974). Petrography of Apollo 17 soils. In *In: Lunar Science*
1118 *Conference, 5th, Houston, Tex., March 18-22, 1974, Proceedings. Volume 1.(A75-39540*
1119 *19-91) New York, Pergamon Press, Inc., 1974, p. 843-860. (Vol. 5, pp. 843–860).*
- 1120 Housley, R. M., Grant, R. W., & Abdel-Gawad, M. (1972). Study of excess Fe metal in the lunar
1121 fines by magnetic separation Mössbauer spectroscopy, and microscopic examination. In
1122 *Lunar and Planetary Science Conference Proceedings (Vol. 3, p. 392).*

- 1123 Housley, R. M., Cirlin, E. H., Paton, N. E., & Goldberg, I. B. (1974). Solar wind and
1124 micrometeorite alteration of the lunar regolith. In *Lunar and planetary science conference*
1125 *proceedings* (Vol. 5, pp. 2623–2642).
- 1126 Housley, R. M., Cirlin, E. H., Goldberg, I. B., Crowe, H., Weeks, R. A., & Perhac, R. (1975).
1127 Ferromagnetic resonance as a method of studying the micrometeorite bombardment history
1128 of the lunar surface. In *Lunar and Planetary Science Conference Proceedings* (Vol. 6, pp.
1129 3173–3186).
- 1130 Housley, R. M., Cirlin, E. H., Goldberg, I. B., & Crowe, H. (1976). Ferromagnetic resonance
1131 studies of lunar core stratigraphy. In *Lunar and Planetary Science Conference Proceedings*
1132 (Vol. 7, pp. 13–26).
- 1133 Housley, R. M., Grant, R. W., & Paton, N. E. (1973). Origin and characteristics of excess Fe
1134 metal in lunar glass welded aggregates. In *Proceedings of the Lunar Science Conference,*
1135 *vol. 4, p. 2737* (Vol. 4, p. 2737).
- 1136 Hunter, J. D. (2007). Matplotlib: A 2D Graphics Environment. *Computing in Science &*
1137 *Engineering*, 9(3), 90–95. <https://doi.org/10.1109/MCSE.2007.55>
- 1138 James, C. L., Letsinger, S. L., Basu, A., Wentworth, S. J., & McKay, D. S. (2002). Size
1139 Distribution of Fe₀ Globules in Lunar Agglutinitic Glass, 1827. Presented at the Lunar and
1140 Planetary Science Conference.
- 1141 Keller, L. P., & Clemett, S. J. (2001). Formation of Nanophase Iron in the Lunar Regolith, 2097.
1142 Presented at the Lunar and Planetary Science Conference.
- 1143 Keller, L. P., Wentworth, S. J., & McKay, D. S. (1998). Space Weathering: Reflectance
1144 Spectroscopy and TEM Analysis of Individual Lunar Soil Grains. In *Lunar and Planetary*
1145 *Science Conference* (p. 1762).

- 1146 Keller, Lindsay P., & McKay, D. S. (1993). Discovery of Vapor Deposits in the Lunar Regolith.
1147 *Science*, 261(5126), 1305–1307. <https://doi.org/10.1126/science.261.5126.1305>
- 1148 Keller, Lindsay P., & McKay, D. S. (1997). The nature and origin of rims on lunar soil grains.
1149 *Geochimica et Cosmochimica Acta*, 61(11), 2331–2341. [https://doi.org/10.1016/S0016-](https://doi.org/10.1016/S0016-7037(97)00085-9)
1150 [7037\(97\)00085-9](https://doi.org/10.1016/S0016-7037(97)00085-9)
- 1151 Keller, Lindsay P., Berger, E. L., Zhang, S., & Christoffersen, R. (2021). Solar energetic particle
1152 tracks in lunar samples: A transmission electron microscope calibration and implications for
1153 lunar space weathering. *Meteoritics & Planetary Science*, 56(9), 1685–1707.
1154 <https://doi.org/10.1111/maps.13732>
- 1155 Labotka, T. C., Kempa, M. J., White, C., Papike, J. J., & Laul, J. C. (1980). The lunar regolith-
1156 Comparative petrology of the Apollo sites. In *Lunar and Planetary Science Conference*
1157 *Proceedings* (Vol. 11, pp. 1285–1305).
- 1158 Lawrence, S. J., & Lucey, P. G. (2007). Radiative transfer mixing models of meteoritic
1159 assemblages. *Journal of Geophysical Research: Planets*, 112(E7).
1160 <https://doi.org/10.1029/2006JE002765>
- 1161 Li, S., & Li, L. (2011). Radiative transfer modeling for quantifying lunar surface minerals,
1162 particle size, and submicroscopic metallic Fe. *Journal of Geophysical Research: Planets*,
1163 116(E9). <https://doi.org/10.1029/2011JE003837>
- 1164 Lucey, P., Korotev, R. L., Gillis, J. J., Taylor, L. A., Lawrence, D., Campbell, B. A., et al.
1165 (2006). Understanding the Lunar Surface and Space-Moon Interactions. *Reviews in*
1166 *Mineralogy and Geochemistry*, 60(1), 83–219. <https://doi.org/10.2138/rmg.2006.60.2>

- 1167 Lucey, P. G., & Riner, M. A. (2011). The optical effects of small iron particles that darken but do
1168 not redden: Evidence of intense space weathering on Mercury. *Icarus*, 212(2), 451–462.
1169 <https://doi.org/10.1016/j.icarus.2011.01.022>
- 1170 McKay, D. S., Heiken, G. H., Taylor, R. M., Clanton, U. S., Morrison, D. A., & Ladle, G. H.
1171 (1972). Apollo 14 soils: Size distribution and particle types. In *Lunar and Planetary Science*
1172 *Conference Proceedings* (Vol. 3, p. 983).
- 1173 McKay, D. S., Heiken, G., Basu, A., Blanford, G., Simon, S., Reedy, R., et al. (1991). The lunar
1174 regolith. *Lunar Sourcebook*, 567, 285–356.
- 1175 Meyer, C. (2005). Lunar Sample Compendium. *NASA STI/Recon Technical Report N*, 6.
1176 Retrieved from <https://curator.jsc.nasa.gov/lunar/lsc/>
- 1177 Milliken, R. E., Hiroi, T., & Patterson, W. (2016). The NASA reflectance experiment laboratory
1178 (RELAB) facility: Past, present, and future. In *47th Annual Lunar and Planetary Science*
1179 *Conference* (p. 2058).
- 1180 Morris, R. V. (1976). Surface exposure indices of lunar soils-A comparative FMR study. In
1181 *Lunar and Planetary Science Conference Proceedings* (Vol. 7, pp. 315–335).
- 1182 Morris, R. V. (1977). Origin and evolution of the grain-size dependence of the concentration of
1183 fine-grained metal in lunar soils-The maturation of lunar soils to a steady-state stage. In
1184 *Lunar and Planetary Science Conference Proceedings* (Vol. 8, pp. 3719–3747).
- 1185 Morris, R. V. (1978). The surface exposure (maturity) of lunar soils-Some concepts and Is/FeO
1186 compilation. In *Lunar and Planetary Science Conference Proceedings* (Vol. 9, pp. 2287–
1187 2297).
- 1188 Morris, R. V. (1980). Origins and size distribution of metallic iron particles in the lunar regolith.
1189 In *In: Lunar and Planetary Science Conference, 11th, Houston, TX, March 17-21, 1980,*

- 1190 *Proceedings. Volume 2.(A82-22296 09-91) New York, Pergamon Press, 1980, p. 1697-*
1191 *1712. (Vol. 11, pp. 1697–1712).*
- 1192 Mustard, J. F., Pieters, C. M., Isaacson, P. J., Head, J. W., Besse, S., Clark, R. N., et al. (2011).
1193 Compositional diversity and geologic insights of the Aristarchus crater from Moon
1194 Mineralogy Mapper data. *Journal of Geophysical Research: Planets*, 116(E6).
1195 <https://doi.org/10.1029/2010JE003726>
- 1196 Nash, D. B., & Conel, J. E. (1973). Vitrification darkening of rock powders: implications for
1197 optical properties of the lunar surface. *Moon*, 8, 346–364.
1198 <https://doi.org/10.1007/BF00581729>
- 1199 Nettles, J. W., Staid, M., Besse, S., Boardman, J., Clark, R. N., Dhingra, D., et al. (2011). Optical
1200 maturity variation in lunar spectra as measured by Moon Mineralogy Mapper data. *Journal*
1201 *of Geophysical Research: Planets*, 116(E9). <https://doi.org/10.1029/2010JE003748>
- 1202 Nimura, T., Hiroi, T., & Pieters, C. M. (2008). An improved scheme for modeling the reflectance
1203 spectra of space-weathered regoliths. *Earth, Planets and Space*, 60(4), 271–275.
1204 <https://doi.org/10.1186/BF03352791>
- 1205 Noble, S. K., Pieters, C. M., Taylor, L. A., Morris, R. V., Allen, C. C., McKAY, D. S., & Keller,
1206 L. P. (2001). The optical properties of the finest fraction of lunar soil: Implications for space
1207 weathering. *Meteoritics & Planetary Science*, 36(1), 31–42. [https://doi.org/10.1111/j.1945-](https://doi.org/10.1111/j.1945-5100.2001.tb01808.x)
1208 [5100.2001.tb01808.x](https://doi.org/10.1111/j.1945-5100.2001.tb01808.x)
- 1209 Noble, S. K., Pieters, C. M., & Keller, L. P. (2007). An experimental approach to understanding
1210 the optical effects of space weathering. *Icarus*, 192(2), 629–642.
1211 <https://doi.org/10.1016/j.icarus.2007.07.021>

- 1212 Pieters, C. M., Fischer, E. M., Rode, O., & Basu, A. (1993). Optical effects of space weathering:
1213 The role of the finest fraction. *Journal of Geophysical Research: Planets*, 98(E11), 20817–
1214 20824. <https://doi.org/10.1029/93JE02467>
- 1215 Pieters, Carle M., & Noble, S. K. (2016). Space weathering on airless bodies. *Journal of*
1216 *Geophysical Research: Planets*, 121(10), 1865–1884.
1217 <https://doi.org/10.1002/2016JE005128>
- 1218 Pieters, Carlé M., Stankevich, D. G., Shkuratov, Yu. G., & Taylor, L. A. (2002). Statistical
1219 Analysis of the Links among Lunar Mare Soil Mineralogy, Chemistry, and Reflectance
1220 Spectra. *Icarus*, 155(2), 285–298. <https://doi.org/10.1006/icar.2001.6749>
- 1221 Poulet, F., & Erard, S. (2004). Nonlinear spectral mixing: Quantitative analysis of laboratory
1222 mineral mixtures. *Journal of Geophysical Research: Planets*, 109(E2).
1223 <https://doi.org/10.1029/2003JE002179>
- 1224 Poulet, F., Cuzzi, J. N., Cruikshank, D. P., Roush, T., & Dalle Ore, C. M. (2002). Comparison
1225 between the Shkuratov and Hapke Scattering Theories for Solid Planetary Surfaces:
1226 Application to the Surface Composition of Two Centaurs. *Icarus*, 160(2), 313–324.
1227 <https://doi.org/10.1006/icar.2002.6970>
- 1228 Python Software Foundation. (2022, December 6). Python 3.9.16 documentation (Version
1229 3.9.16). Retrieved from <https://docs.python.org/release/3.9.16/>
- 1230 Shkuratov, Y., Starukhina, L., Hoffmann, H., & Arnold, G. (1999). A Model of Spectral Albedo
1231 of Particulate Surfaces: Implications for Optical Properties of the Moon. *Icarus*, 137(2),
1232 235–246. <https://doi.org/10.1006/icar.1998.6035>
- 1233 Simon, S. B., & Papike, J. J. (1981). The Lunar regolith: comparative petrology of the Apollo
1234 and Luna soils. In *Lunar and Planetary Science Conference* (Vol. 12, pp. 984–986).

- 1235 Simon, Steven B., Papike, J. J., & Laul, J. C. (1981). The lunar regolith-Comparative studies of
1236 the Apollo and Luna sites. Petrology of soils from Apollo 17, Luna 16, 20, and 24. In *Lunar*
1237 *and Planetary Science Conference Proceedings* (Vol. 12, pp. 371–388).
- 1238 Taylor, L. A., & Cirlin, E. H. (1985). A review of ESR studies on lunar samples. In M. Ikeya &
1239 T. Miki (Eds.), *ESR Dating and Dosimetry* (pp. 19–29). Tokyo: IONICS.
- 1240 Taylor, L. A., Patchen, A., Taylor, D.-H. S., Chambers, J. G., & McKay, D. S. (1996). X-Ray
1241 Digital Imaging Petrography of Lunar Mare Soils: Modal Analyses of Minerals and Glasses.
1242 *Icarus*, 124(2), 500–512. <https://doi.org/10.1006/icar.1996.0226>
- 1243 Taylor, L. A., Pieters, C. M., Keller, L. P., Morris, R. V., & McKay, D. S. (2001a). Lunar Mare
1244 Soils: Space weathering and the major effects of surface-correlated nanophase Fe. *Journal*
1245 *of Geophysical Research: Planets*, 106(E11), 27985–27999.
1246 <https://doi.org/10.1029/2000JE001402>
- 1247 Taylor, L. A., Pieters, C., Keller, L. P., Morris, R. V., McKAY, D. S., Patchen, A., & Wentworth,
1248 S. (2001b). The effects of space weathering on Apollo 17 mare soils: Petrographie and
1249 chemical characterization. *Meteoritics & Planetary Science*, 36(2), 285–299.
1250 <https://doi.org/10.1111/j.1945-5100.2001.tb01871.x>
- 1251 Taylor, L. A., Pieters, C., Patchen, A., Taylor, D.-H. S., Morris, R. V., Keller, L. P., & McKay,
1252 D. S. (2010). Mineralogical and chemical characterization of lunar highland soils: Insights
1253 into the space weathering of soils on airless bodies. *Journal of Geophysical Research:*
1254 *Planets*, 115(E2). <https://doi.org/10.1029/2009JE003427>
- 1255 The pandas development team. (2023, January 19). pandas-dev/pandas: Pandas (Version v1.5.3).
1256 Zenodo. <https://doi.org/10.5281/zenodo.7549438>

- 1257 Thompson, M. S., Zega, T. J., Becerra, P., Keane, J. T., & Byrne, S. (2016). The oxidation state
1258 of nanophase Fe particles in lunar soil: Implications for space weathering. *Meteoritics &*
1259 *Planetary Science*, 51(6), 1082–1095. <https://doi.org/10.1111/maps.12646>
- 1260 Thompson, M. S., Zega, T. J., & Howe, J. Y. (2017). In situ experimental formation and growth
1261 of Fe nanoparticles and vesicles in lunar soil. *Meteoritics & Planetary Science*, 52(3), 413–
1262 427. <https://doi.org/10.1111/maps.12798>
- 1263 Vaniman, D. T., & Papike, J. J. (1977). The Apollo 17 drill core-Modal petrology and glass
1264 chemistry/sections 70007, 70008, 70009. In *In: Lunar Science Conference, 8th, Houston,*
1265 *Tex., March 14-18, 1977, Proceedings. Volume 3.(A78-41551 18-91) New York, Pergamon*
1266 *Press, Inc., 1977, p. 3161-3193. (Vol. 8, pp. 3161–3193).*
- 1267 Via, W. N., & Taylor, L. A. (1976a). Agglutinate Formation: Is Chemical Fractionation
1268 Involved? In *Lunar and Planetary Science Conference (Vol. 7).*
- 1269 Via, W. N., & Taylor, L. A. (1976b). Chemical aspects of agglutinate formation-Relationships
1270 between agglutinate composition and the composition of the bulk soil. In *Lunar and*
1271 *Planetary Science Conference Proceedings (Vol. 7, pp. 393–403).*
- 1272 Warell, J., & Davidsson, B. J. R. (2010). A Hapke model implementation for compositional
1273 analysis of VNIR spectra of Mercury. *Icarus*, 209(1), 164–178.
1274 <https://doi.org/10.1016/j.icarus.2009.11.037>
- 1275 Wells, E., & Hapke, B. (1977). Lunar Soil: Iron and Titanium Bands in the Glass Fraction.
1276 *Science*, 195(4282), 977–979. <https://doi.org/10.1126/science.195.4282.977>
- 1277 Yang, Y., Li, S., Milliken, R. E., Zhang, H., Robertson, K., & Hiroi, T. (2019). Phase Functions
1278 of Typical Lunar Surface Minerals Derived for the Hapke Model and Implications for

1279 Visible to Near-Infrared Spectral Unmixing. *Journal of Geophysical Research: Planets*,
1280 124(1), 31–60. <https://doi.org/10.1029/2018JE005713>

1281 Zhang, H., Yang, Y., Jin, W., Liu, C., & Hsu, W. (2014). Effects of Spectralon absorption on
1282 reflectance spectra of typical planetary surface analog materials. *Optics Express*, 22(18),
1283 21280–21291. <https://doi.org/10.1364/OE.22.021280>

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1302 **Figure Captions**

1303

1304 **Figure 1.** Scanning electron microscope images of agglutinates from the 125–250 μm size
 1305 fraction (contrast stretched for clarity). **Top row:** whole grains from two soils ((**A**) 67461, (**B**)
 1306 79221) illustrate their three-dimensional structure as misshapen agglomerates of glass, mineral,
 1307 and lithic fragments. **Bottom row:** polished carbon-coated grain mounts from two soils ((**C**)
 1308 67461, (**D**) 79221) highlight the vesicular internal structure.

1309

1310 **Figure 2.** Microscope images of (**A**) magnetically separated 125–250 μm agglutinates from
 1311 sample 62231 and (**B**) the corresponding non-agglutinates. The images have been color corrected
 1312 to approximate appearance under white light.

1313

1314 **Figure 3.** Soil separates produced by the magnetic separator (soil 62231, 75–125 μm size
 1315 fraction), imaged (**top row**) on weighing paper and (**bottom row**) under a binocular microscope.
 1316 (**A, D**) The least magnetic particles, which fell down the chute and into the bin further from the
 1317 magnet, are visually bright and contain largely mineral fragments and no agglutinates; (**B, E**) the
 1318 moderately magnetic particles, which fell down the chute and into the bin closer to the magnet,
 1319 are intermediate in brightness and contain mineral and breccia fragments with few agglutinates;
 1320 and (**C, F**) the highly magnetic particles, which were suspended in the chute by the magnet, are
 1321 visually darkest and are rich in agglutinates.

1322

1323 **Figure 4.** Estimated agglutinate abundance vs. soil maturity for the lunar soils. All agglutinates
 1324 were magnetic–manual separated except for the manually separated 62231.52 (denoted by a
 1325 triangle marker, and likely overestimated due to substantial contamination of the agglutinate
 1326 separate with non-agglutinate particles). Note that, while the agglutinate abundance values
 1327 measured in this study are for the 125–250 μm size fraction, the soil maturity values from [Morris](#)
 1328 ([1978](#)) are for the <250 μm size fraction.

1329

1330 **Figure 5.** Microscope images of the 125–250 μm agglutinate separates from soil 62231, yielded
 1331 by (**A**) the magnetic–manual method and (**B**) the manual sorting method, showing the former’s
 1332 higher purity (fewer non-agglutinate particles). The images have been color corrected to
 1333 approximate appearance under white light. Particles we categorize as non-agglutinates are circled
 1334 in blue. Note that categorizing particles as either agglutinates or non-agglutinates is an
 1335 oversimplification, albeit a necessary one. For example, the circled “non-agglutinate” particle in
 1336 (**A**) is categorized as such due to its predominantly smooth texture, but it does have a small
 1337 amount of rough-textured agglutinitic material fused onto its upper side.

1338

1339 **Figure 6.** Microscopic images of 125–250 μm non-agglutinate separates from four mature soils
 1340 of different composition: (**A**) 62231 (highlands), (**B**) 14259 (non-mare), (**C**) 15041 (low-Ti
 1341 mare), and (**D**) 79221 (high-Ti mare). Here and in [Figures 7 and 8](#), each image was collected
 1342 with the same illumination conditions and is shown with the same relative contrast stretch. The
 1343 images have been color corrected to approximate appearance under white light.

1344

1345 **Figure 7.** Microscopic images of 125–250 μm agglutinate separates from four mature soils of
 1346 different composition: (**A**) 62231 (highlands), (**B**) 14259 (non-mare), (**C**) 15041 (low-Ti mare),
 1347 and (**D**) 79221 (high-Ti mare). Here and in [Figures 6 and 8](#), each image was collected with the

1348 same illumination conditions and is shown with the same relative contrast stretch. The images
 1349 have been color corrected to approximate appearance under white light.

1350

1351 **Figure 8.** Microscope images of 125–250 μm agglutinate separates (left) and non-agglutinates
 1352 (right) from Apollo 16 soils of different maturities: **(A, B)** immature soil 67461 ($I_S/\text{FeO} = 25$),
 1353 **(C, D)** submature soil 61141 ($I_S/\text{FeO} = 56$), and **(E, F)** mature soil 62231 ($I_S/\text{FeO} = 91$). Here
 1354 and in [Figures 6 and 7](#), each image was collected with the same illumination conditions and is
 1355 shown with the same relative contrast stretch. The images have been color corrected to
 1356 approximate appearance under white light.

1357

1358 **Figure 9.** Reflectance spectra of the 125–250 μm unsorted, non-agglutinate, and agglutinate
 1359 separates for the six soils. Each separate's mean spectrum (thick dark line) is the average of
 1360 multiple individual measurements (thin faint lines). The number of measurements contributing to
 1361 each mean is indicated in parentheses.

1362

1363 **Figure 10.** Same as [Figure 9](#), but showing the mean reflectance spectra normalized to their
 1364 values at 0.7 μm . The number of measurements contributing to each mean is indicated in
 1365 parentheses, but these individual measurement spectra are not shown.

1366

1367 **Figure 11. (top)** Continuum ratio vs integrated 1 μm band depth and **(bottom)** albedo vs
 1368 integrated 1 μm band depth for the mean reflectance spectra of the 125–250 μm unsorted, non-
 1369 agglutinate, and agglutinate separates. Continuum ratio is defined as the ratio of 1550 nm
 1370 reflectance to 750 nm reflectance, while albedo is measured by 1550 nm reflectance. Soil names
 1371 are indicated by the annotations: 16i (67461, immature), 16s (61141, submature), 16m (62231,
 1372 mature), 14 (14259), 15 (15041), 17 (79221). Dotted gray lines connect each soil's three
 1373 separates: agglutinates, unsorted, non-agglutinates.

1374

1375 **Figure 12.** Reflectance spectra of the 125–250 μm unsorted, non-agglutinate, and agglutinate
 1376 separates from the three Apollo 16 (highland) soils: immature soil 67461 ($I_S/\text{FeO} = 25$),
 1377 submature soil 61141 ($I_S/\text{FeO} = 56$), and mature soil 62231 ($I_S/\text{FeO} = 91$). **(left)** Mean spectra
 1378 and **(right)** the same spectra normalized to their values at 0.7 μm . The number of measurements
 1379 contributing to each mean is indicated in parentheses, but these individual measurement spectra
 1380 are not shown.

1381

1382 **Figure 13.** Reflectance spectra for two size fractions (125–250 μm and 75–125 μm) of unsorted,
 1383 non-agglutinate, and agglutinate separates from the 62231 soil. **(left)** Mean spectra and **(right)**
 1384 the same spectra normalized to their values at 0.7 μm . The number of measurements contributing
 1385 to each mean is indicated in parentheses, but these individual measurement spectra are not
 1386 shown.

1387

1388 **Figure 14.** Agglutinate abundance of soils in this study, as measured using different methods and
 1389 for different size fractions. Data points are plotted at the average value of the measured size
 1390 fraction, while the errors bars show the range of the size fraction. Note that ranges shown to
 1391 extend down to 1 μm indicate finest fractions (e.g., <10 μm , not 1–10 μm). Data source
 1392 abbreviations are as follows: LSCC = Lunar Soil Characterization Consortium ([Taylor et al.](#),

1393 2001a, 2010); B82 = Basu et al., 1982; T96 = Taylor et al., 1996; M72 = McKay et al., 1972;
1394 HM74 = Heiken and McKay, 1974; SP81 = Simon and Papike, 1981.

1395

1396 **Figure 15.** Comparison of reflectance spectra for 125–250 μm agglutinates (solid colored lines;
1397 this study) and npFe in silica gel (dashed/dotted black lines; Noble et al., 2007). Spectra are
1398 normalized to their reflectance values at 0.55 μm . Each of the four panels shows the same
1399 agglutinate spectra, but plotted alongside spectra of npFe with differing average size (8, 15, 35,
1400 40 nm). Annotations next to each npFe spectrum denote npFe abundance (as a weight
1401 percentage).

1402

Figure 1.

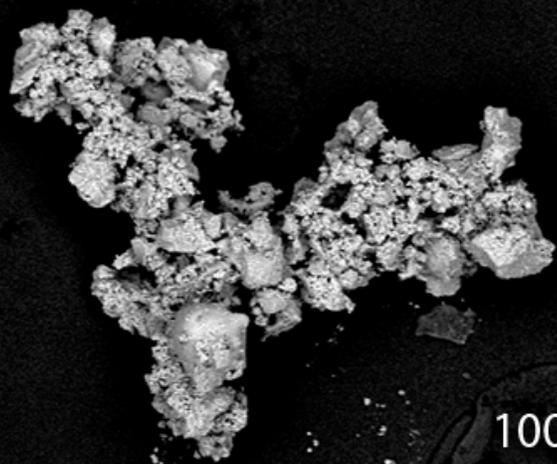
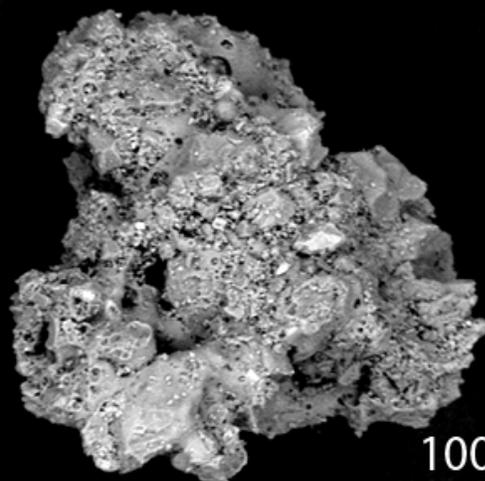
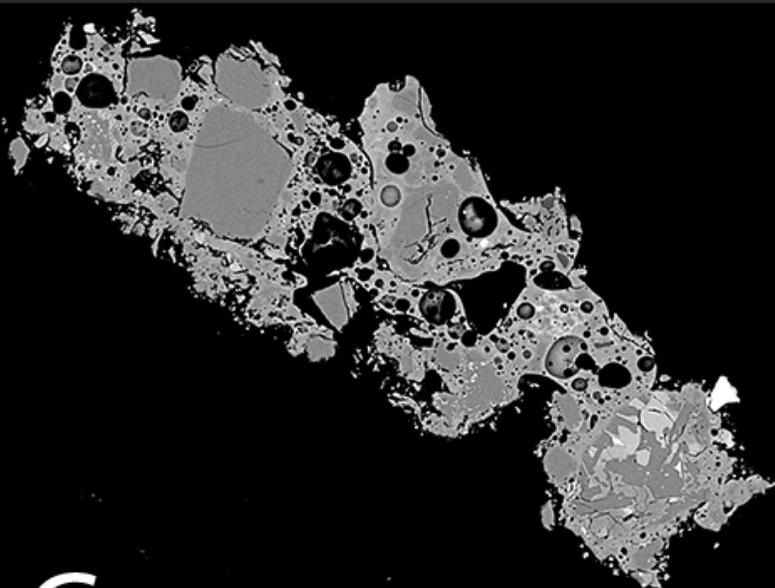
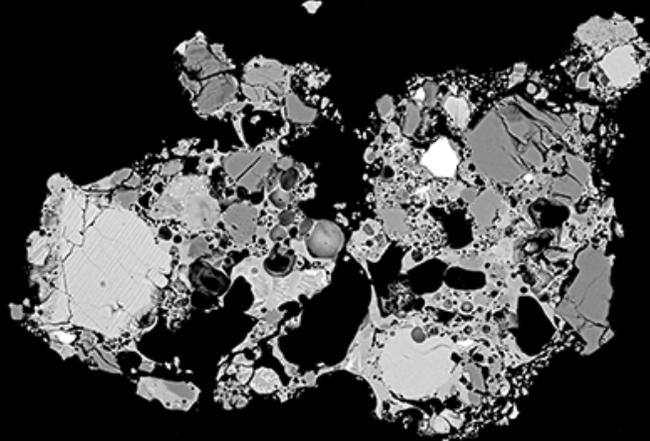
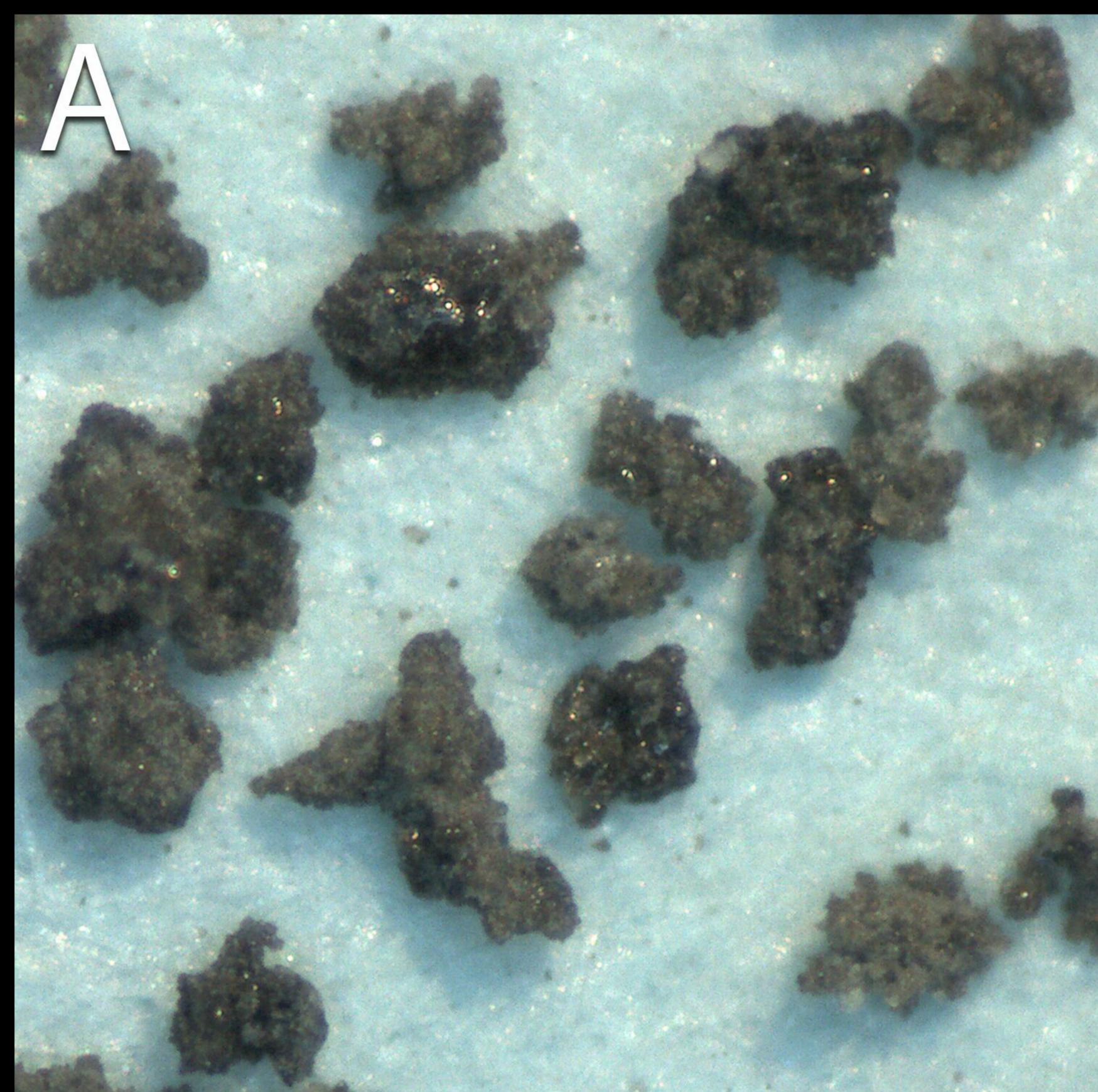
A100 μm **B**100 μm **C**100 μm **D**100 μm 

Figure 2.

A



B

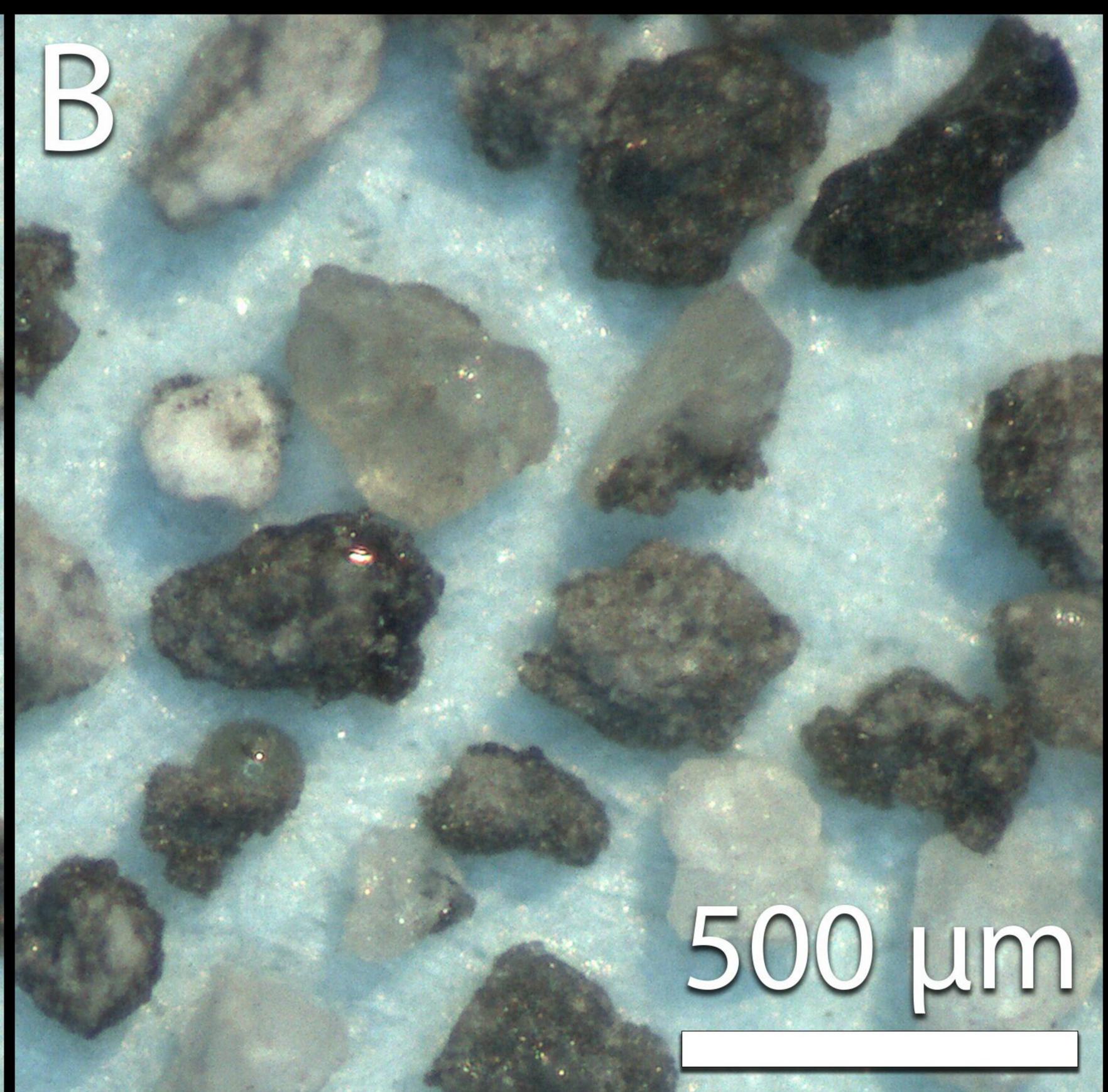
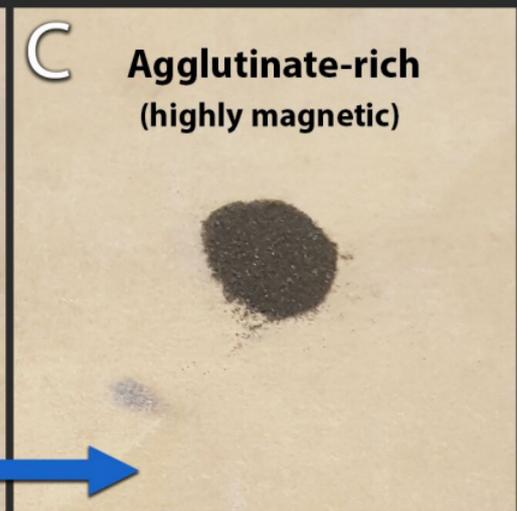
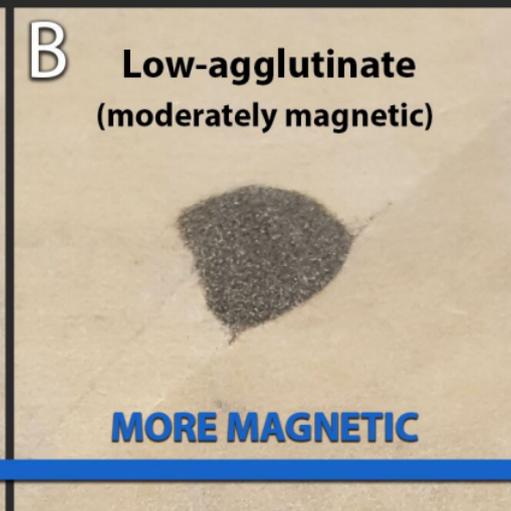
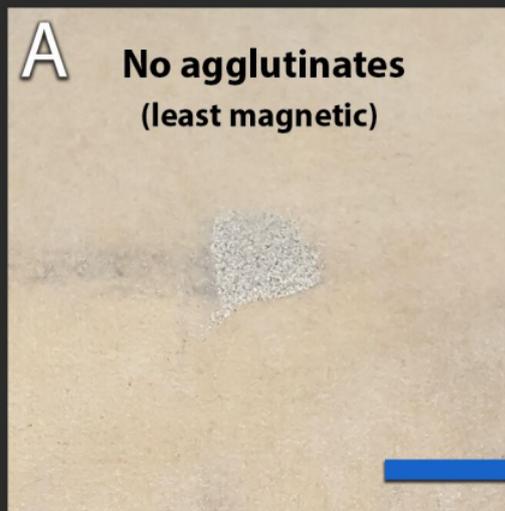


Figure 3.



MORE MAGNETIC

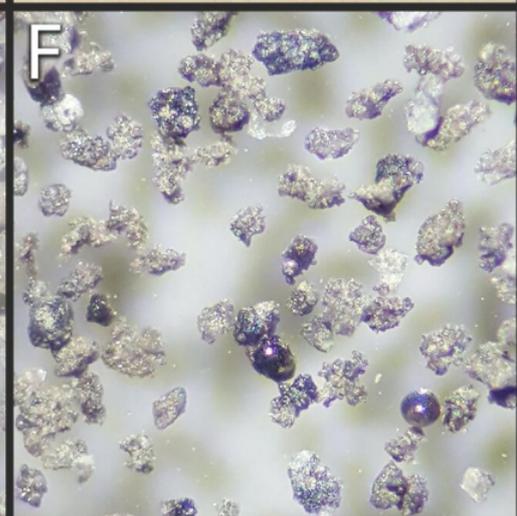
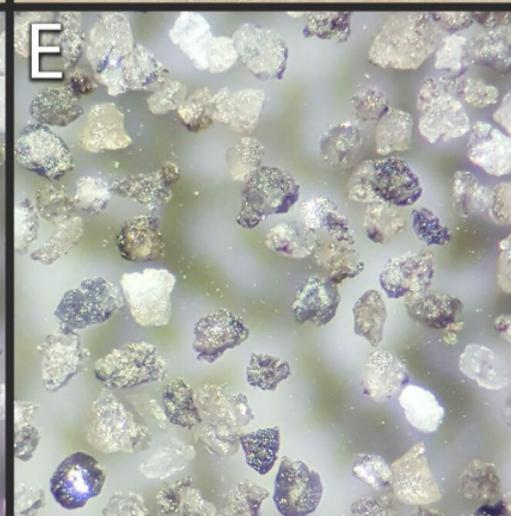
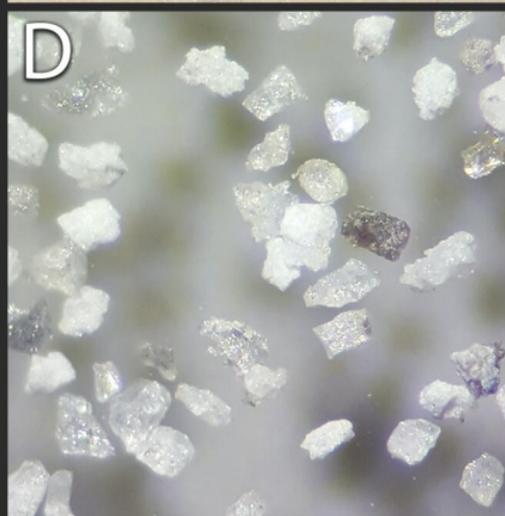
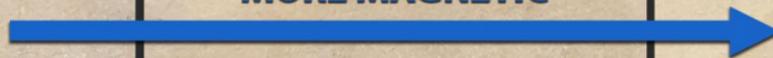
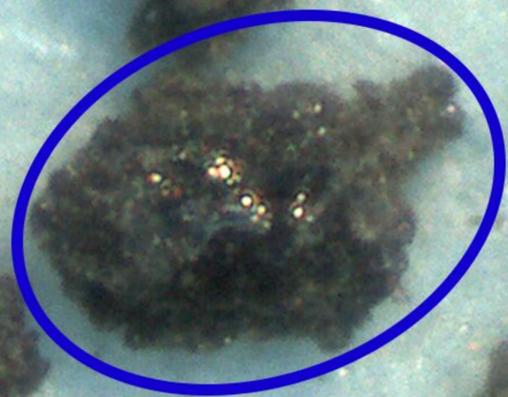
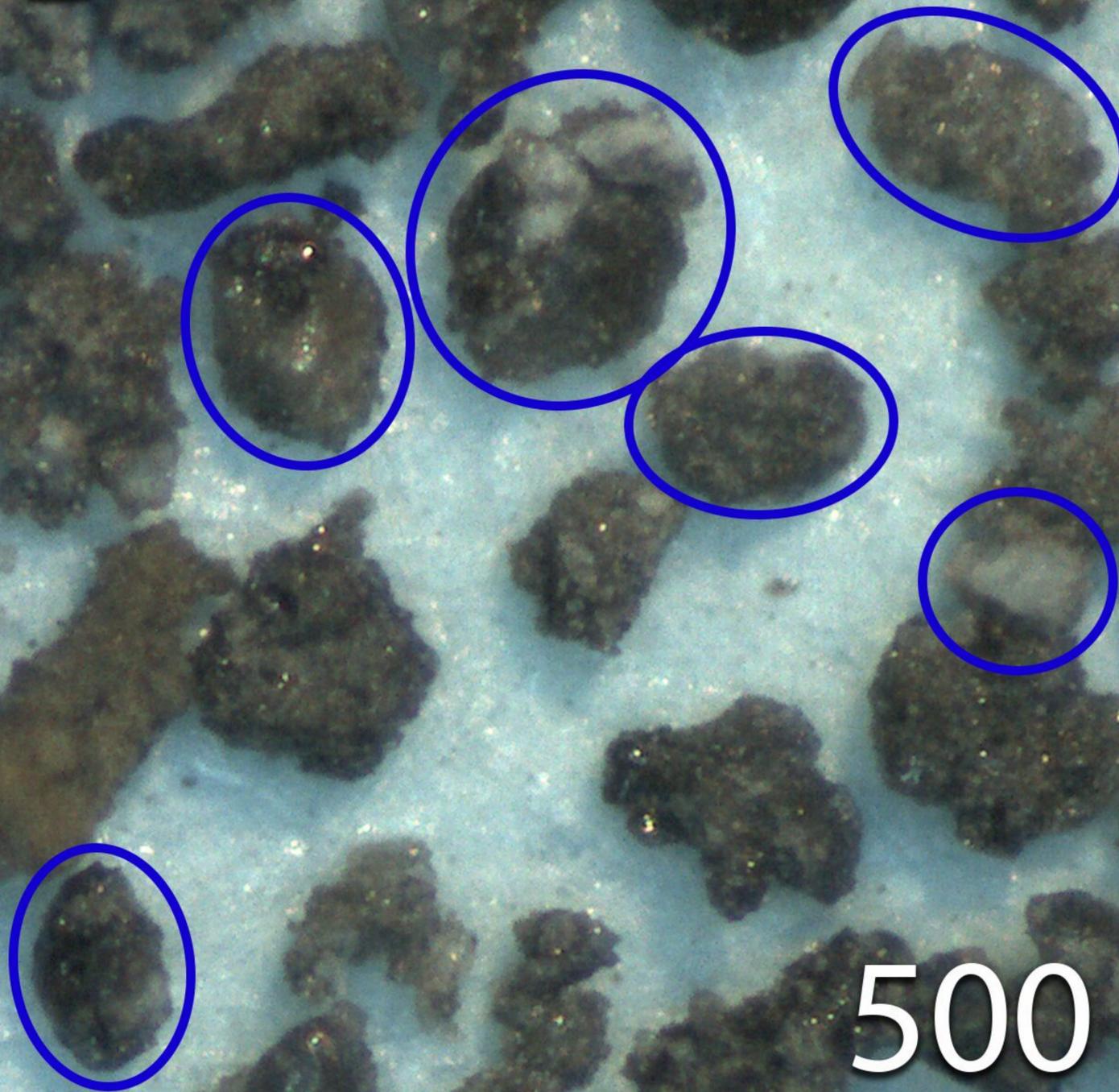


Figure 5.

A



B



500 μm



Figure 6.

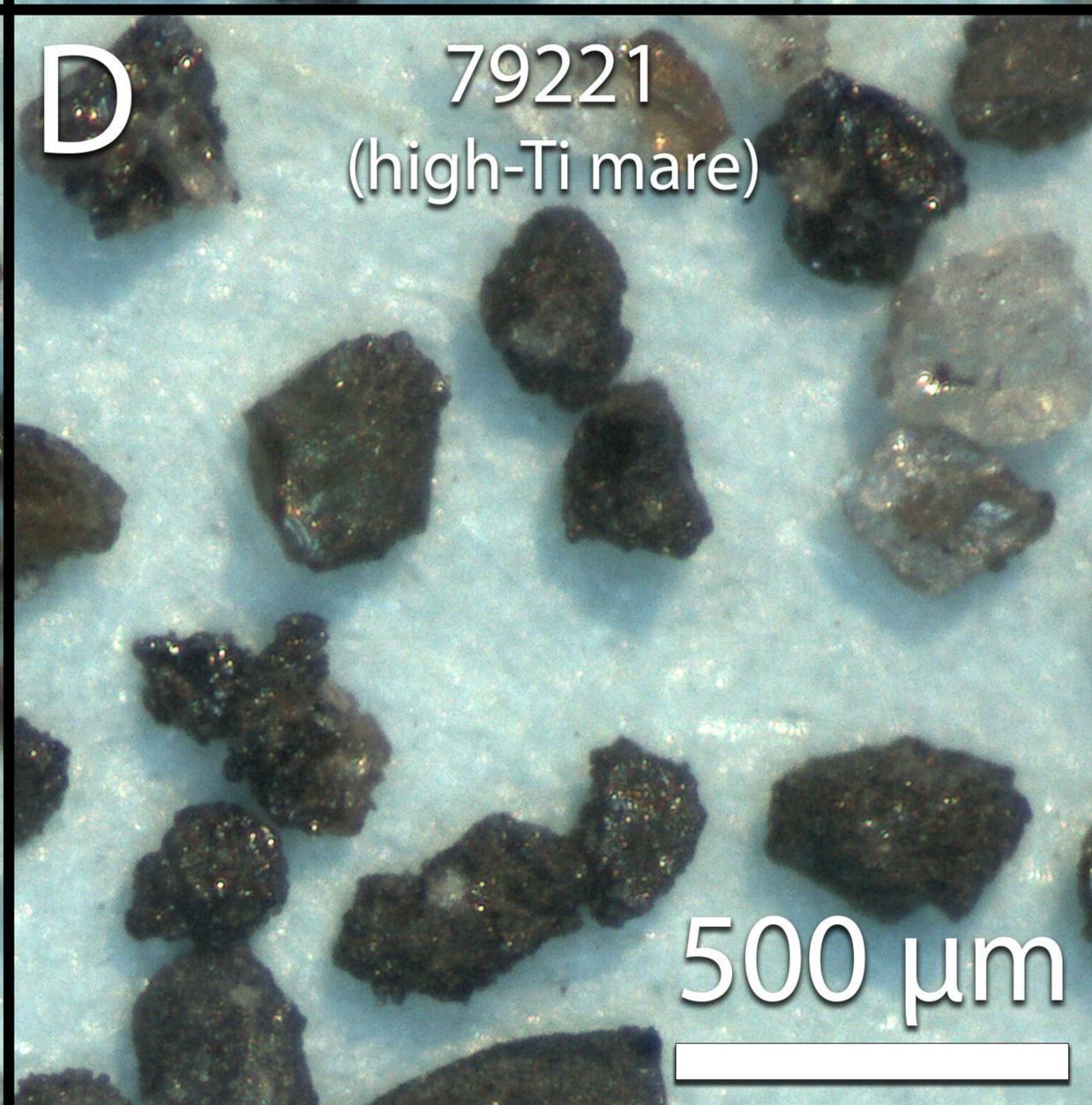
A62231
(highlands)**B**14259
(non-mare)**C**15041
(low-Ti mare)**D**79221
(high-Ti mare)500 μm

Figure 7.

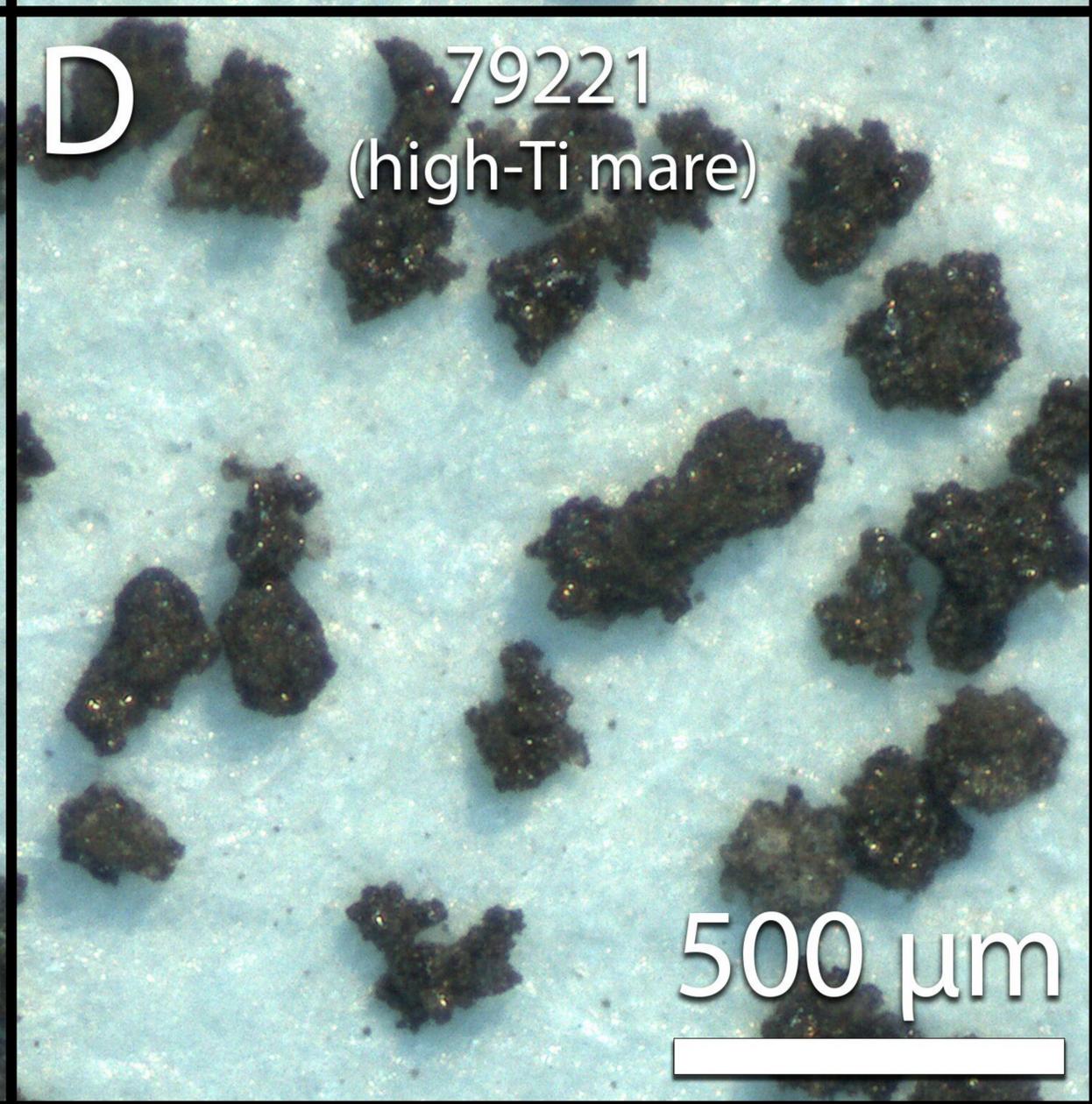
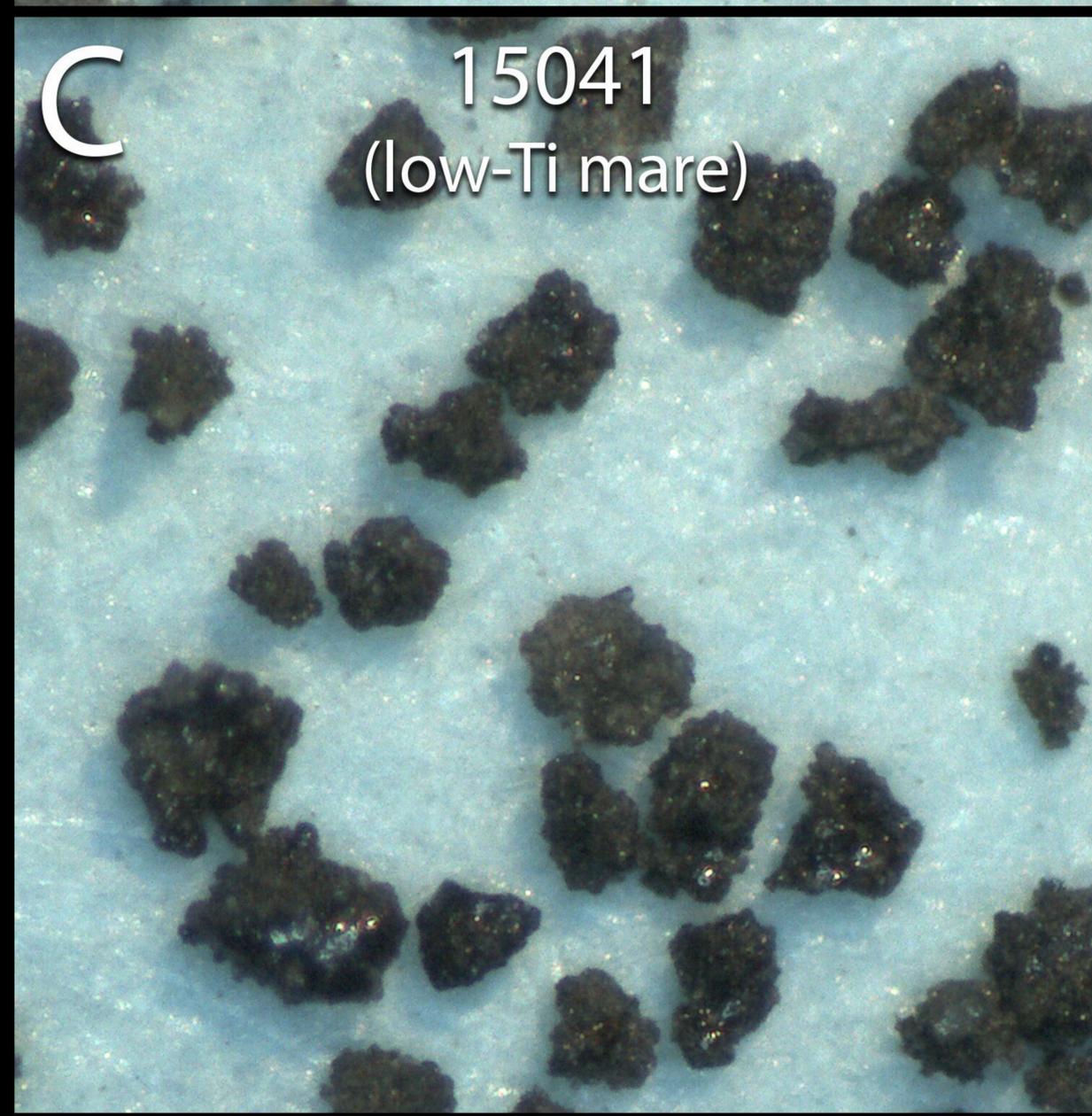
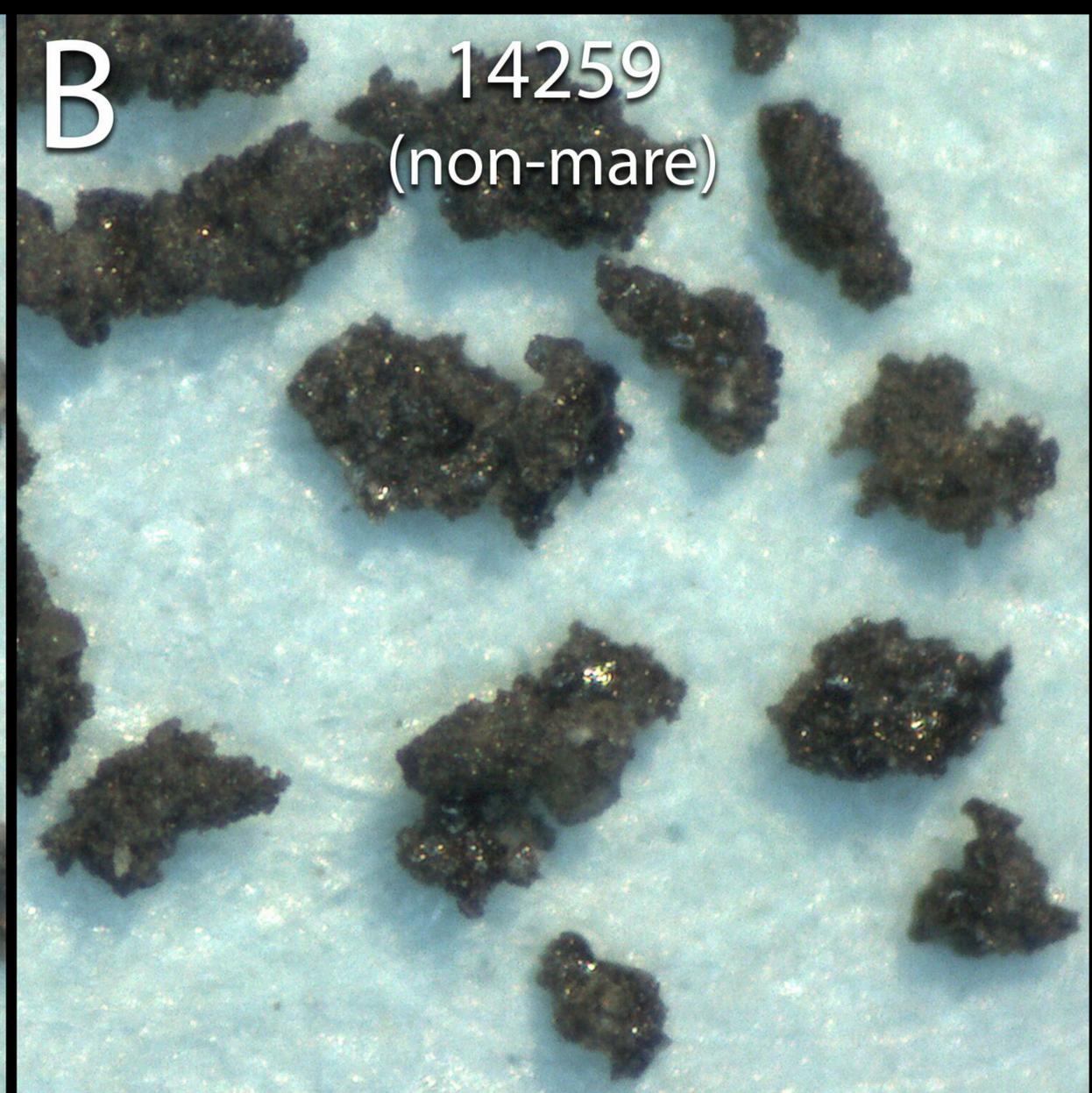
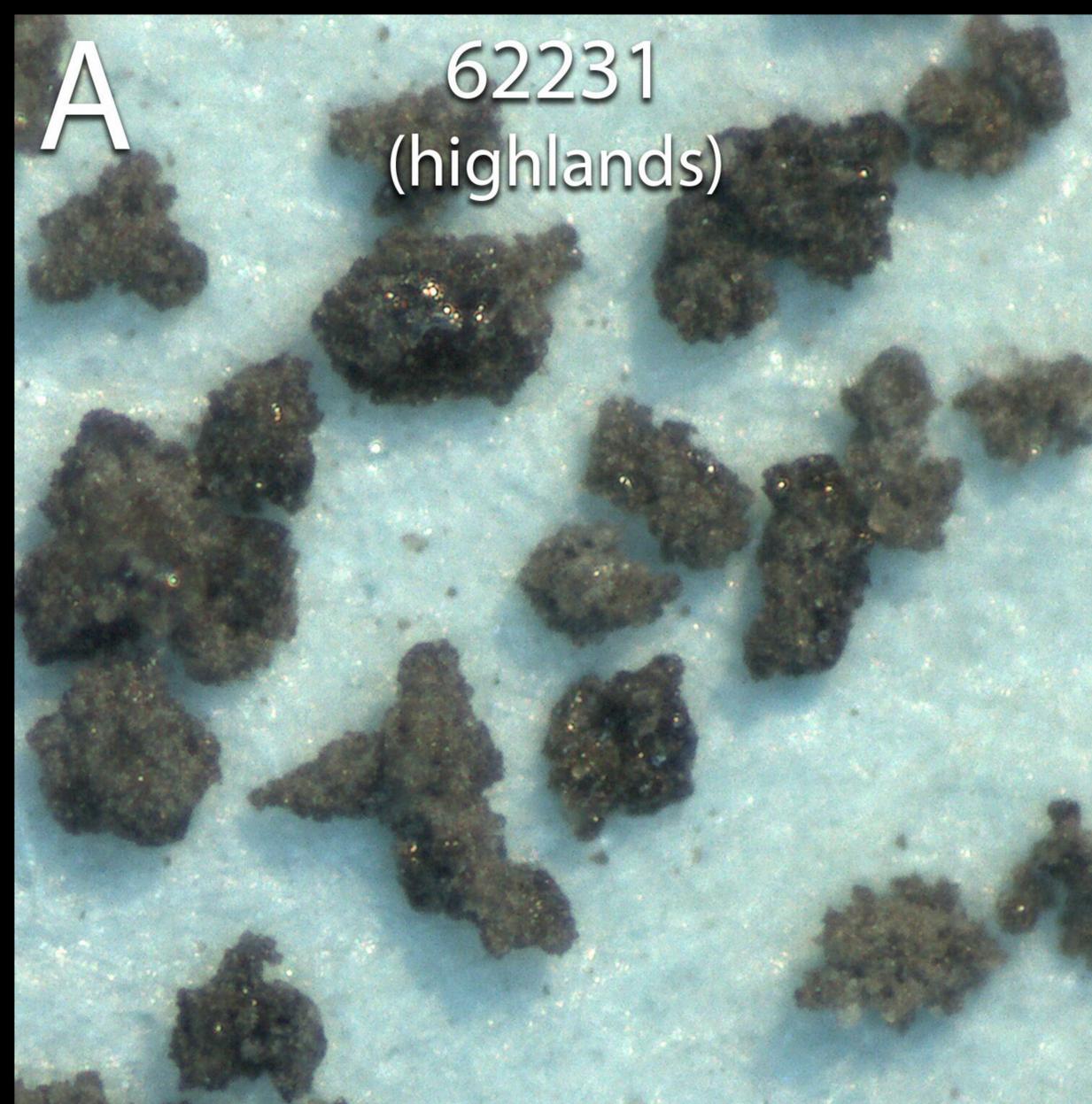
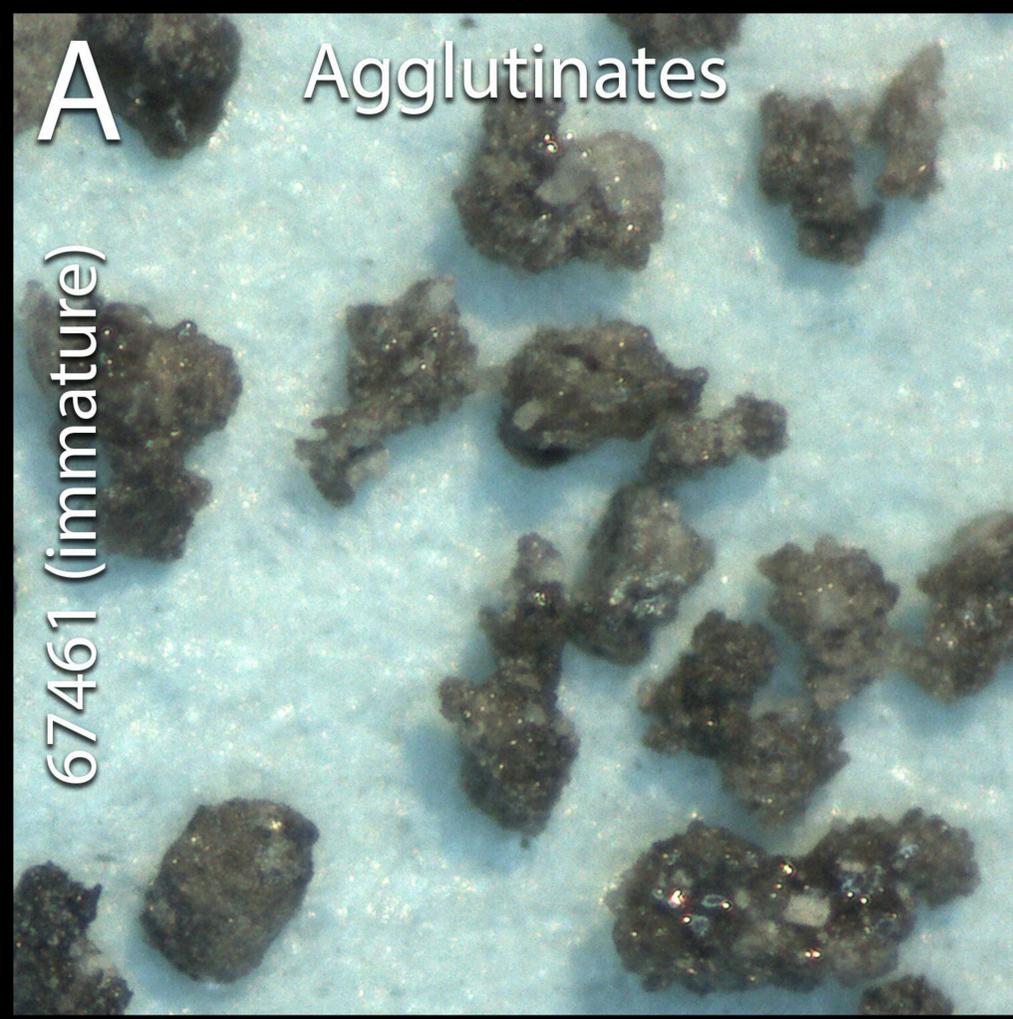


Figure 8.

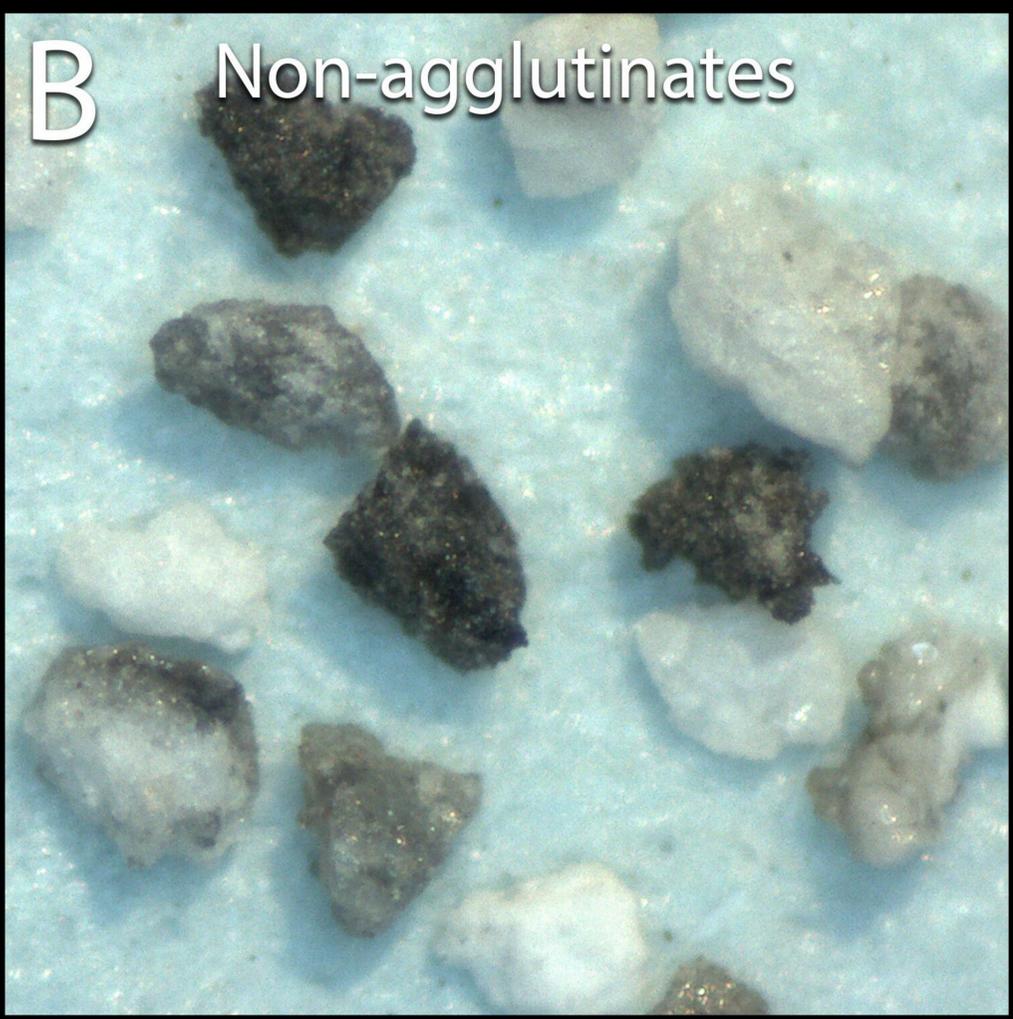
A

Agglutinates

67461 (immature)

**B**

Non-agglutinates

**C**

61141 (submature)

**D****E**

62231 (mature)

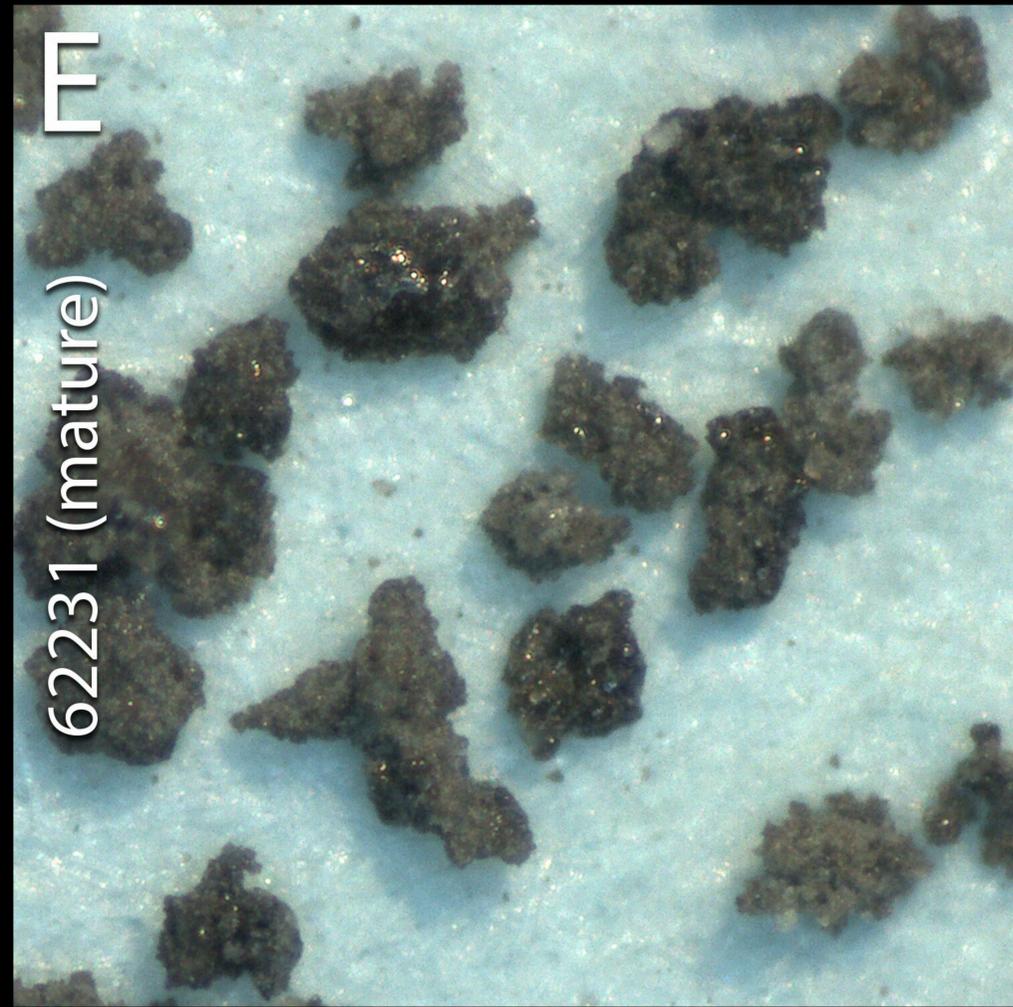
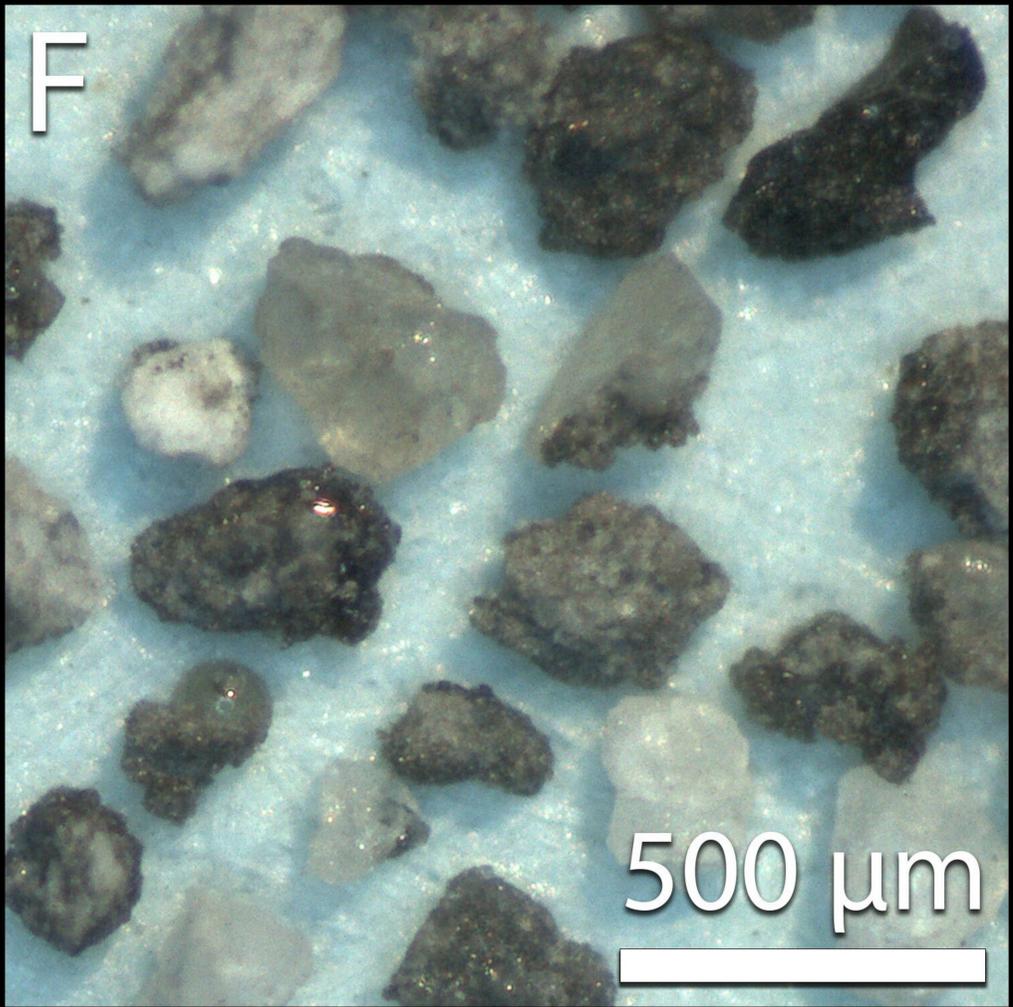
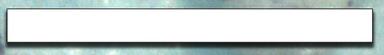
**F**500 μm 

Figure 4.

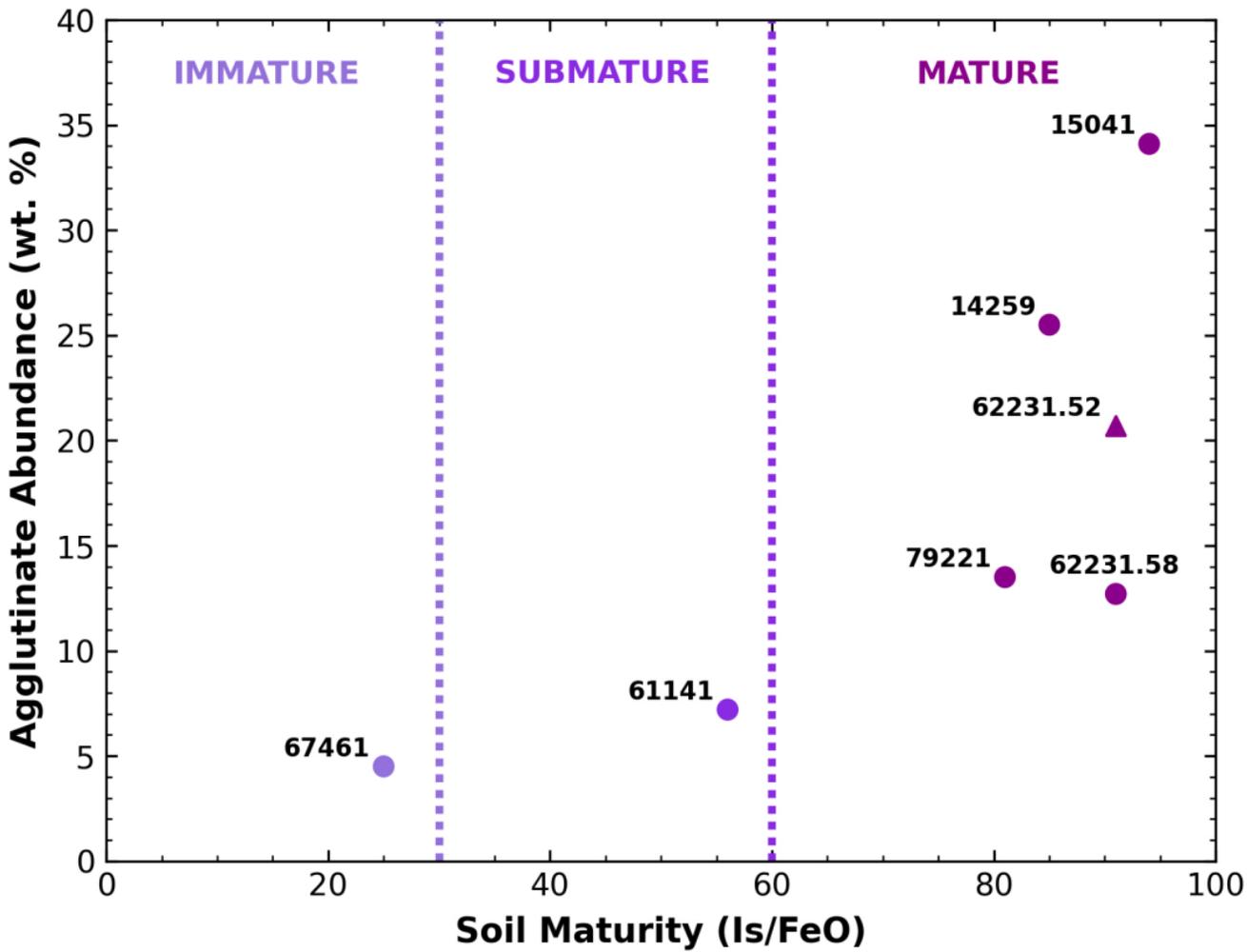


Figure 9.

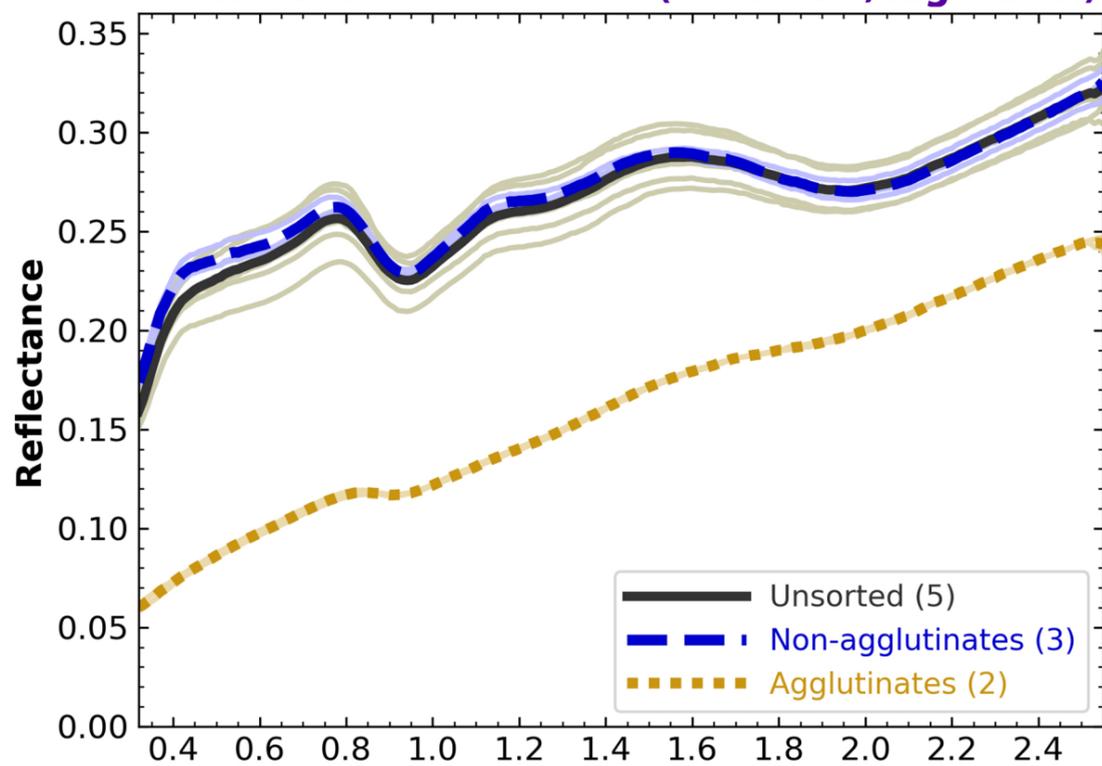
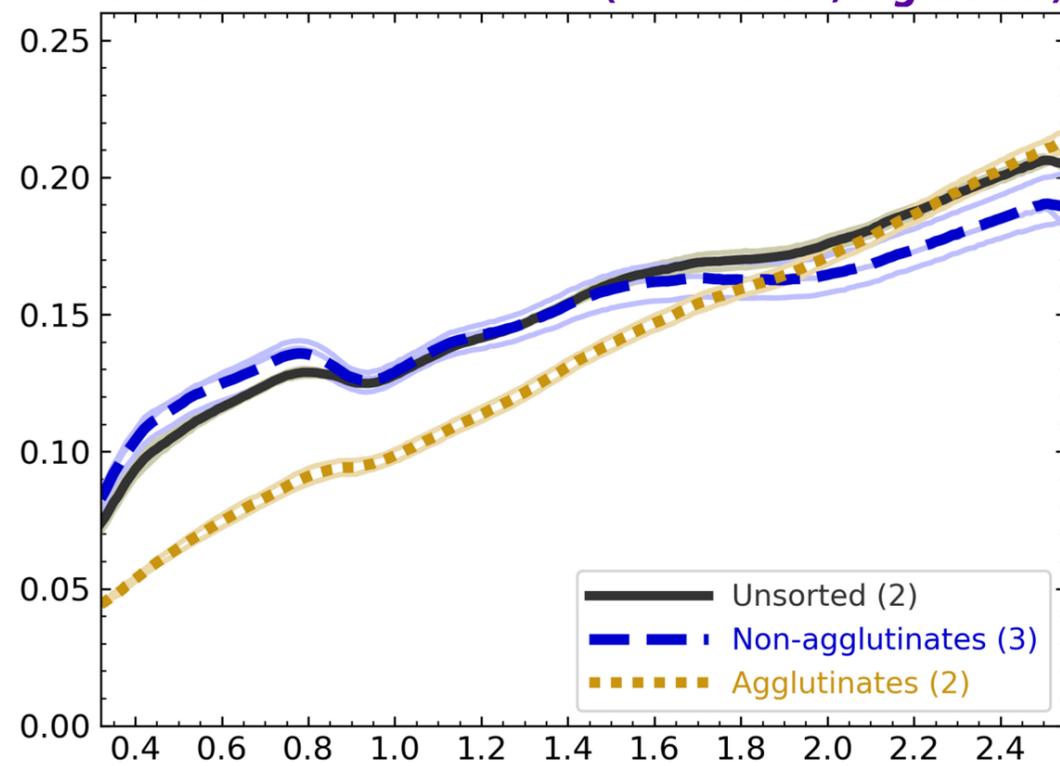
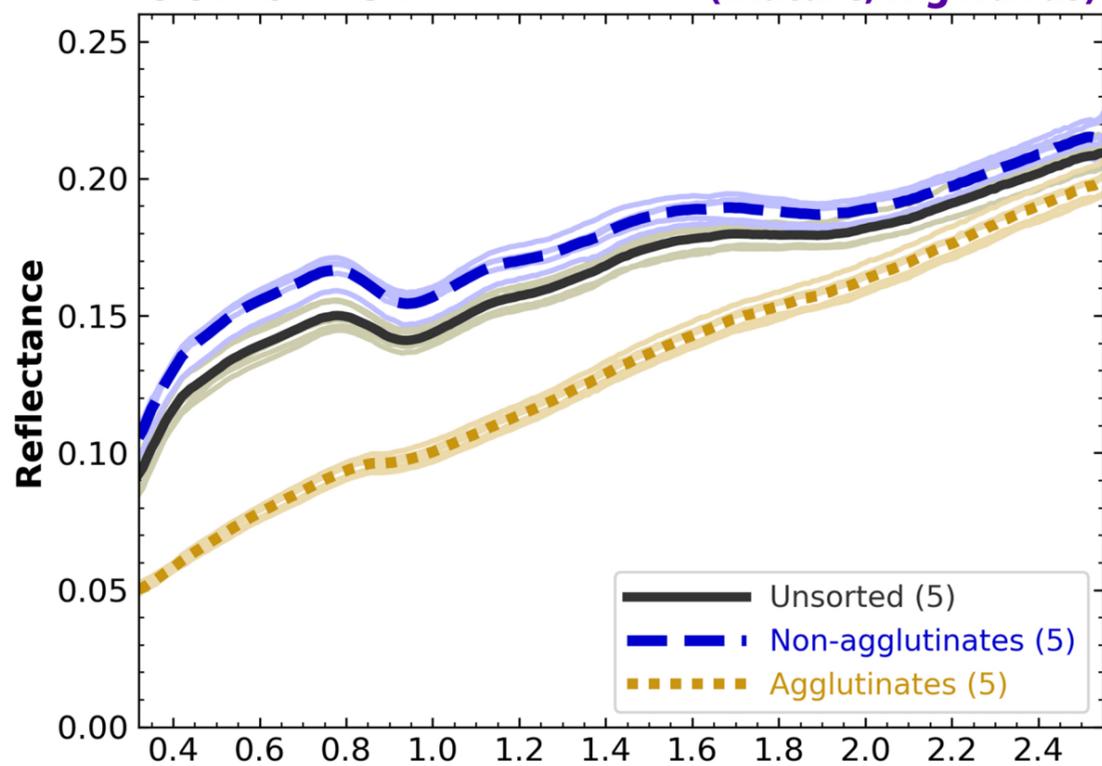
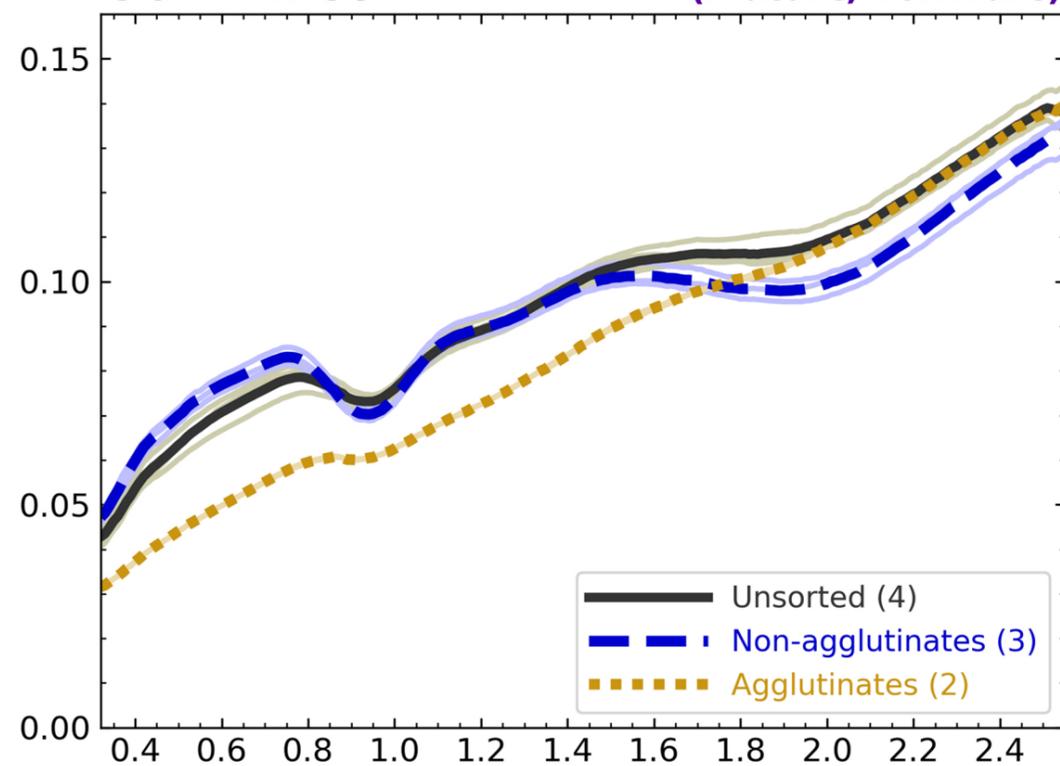
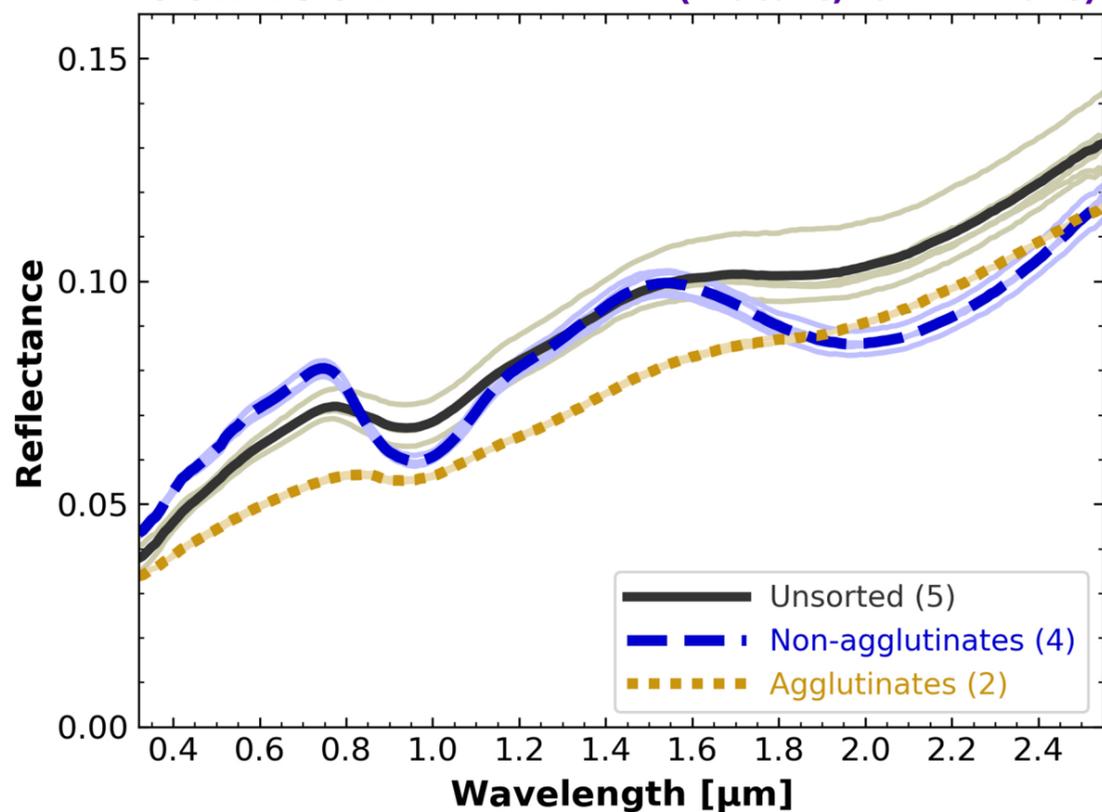
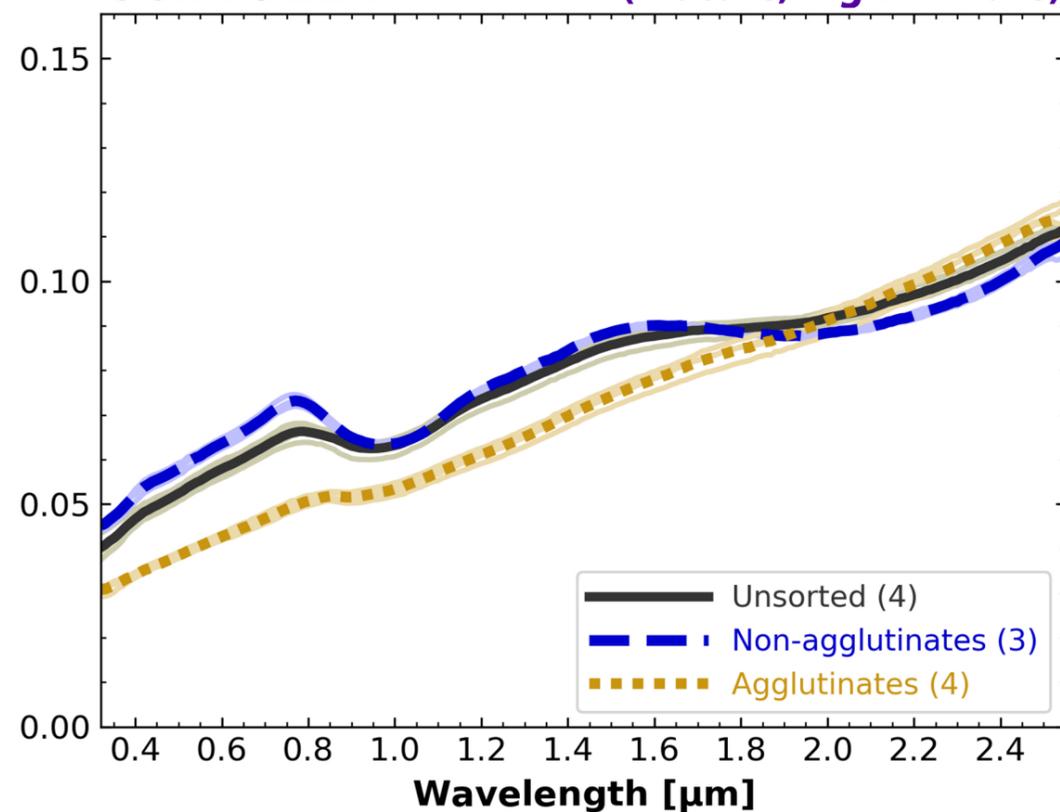
Soil 67461**(immature, highlands)****Soil 61141****(submature, highlands)****Soil 62231****(mature, highlands)****Soil 14259****(mature, nonmare)****Soil 15041****(mature, low-Ti mare)****Soil 79221****(mature, high-Ti mare)**

Figure 10.

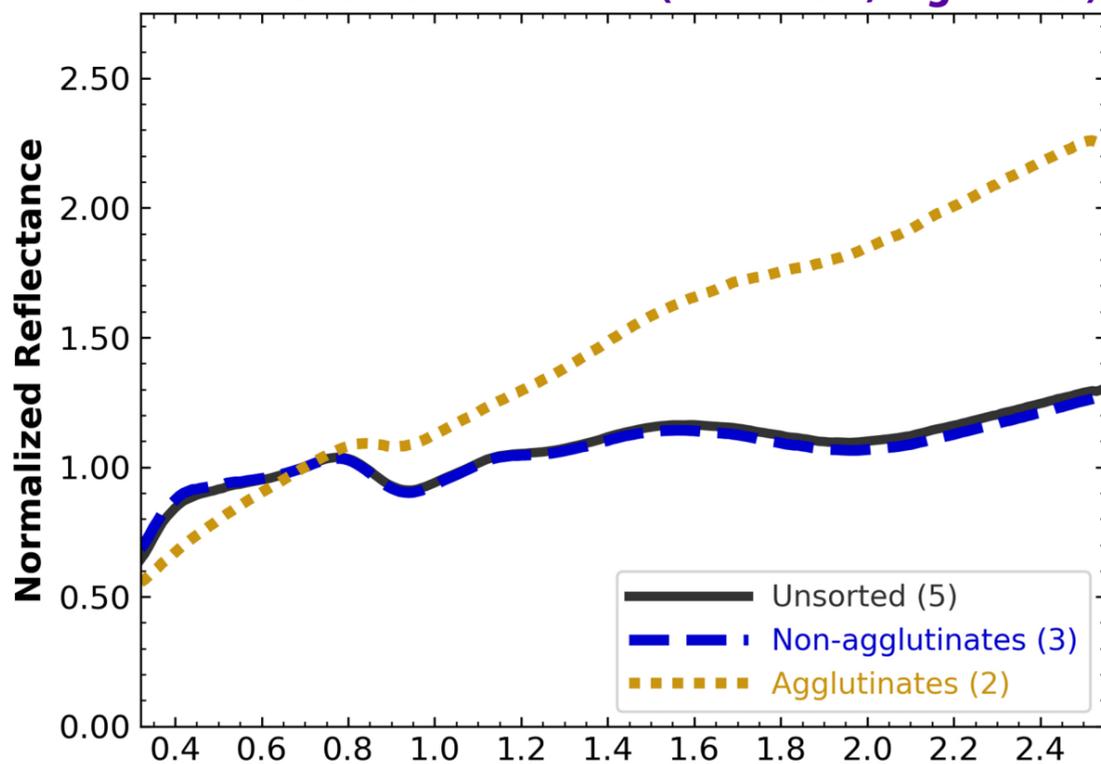
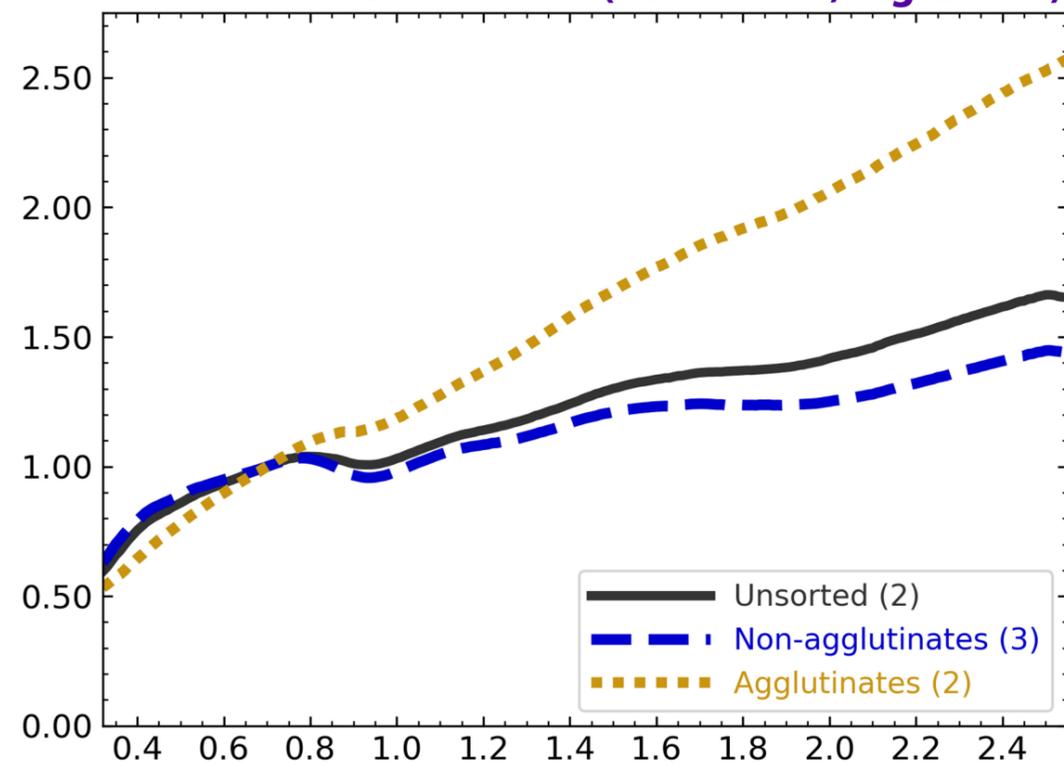
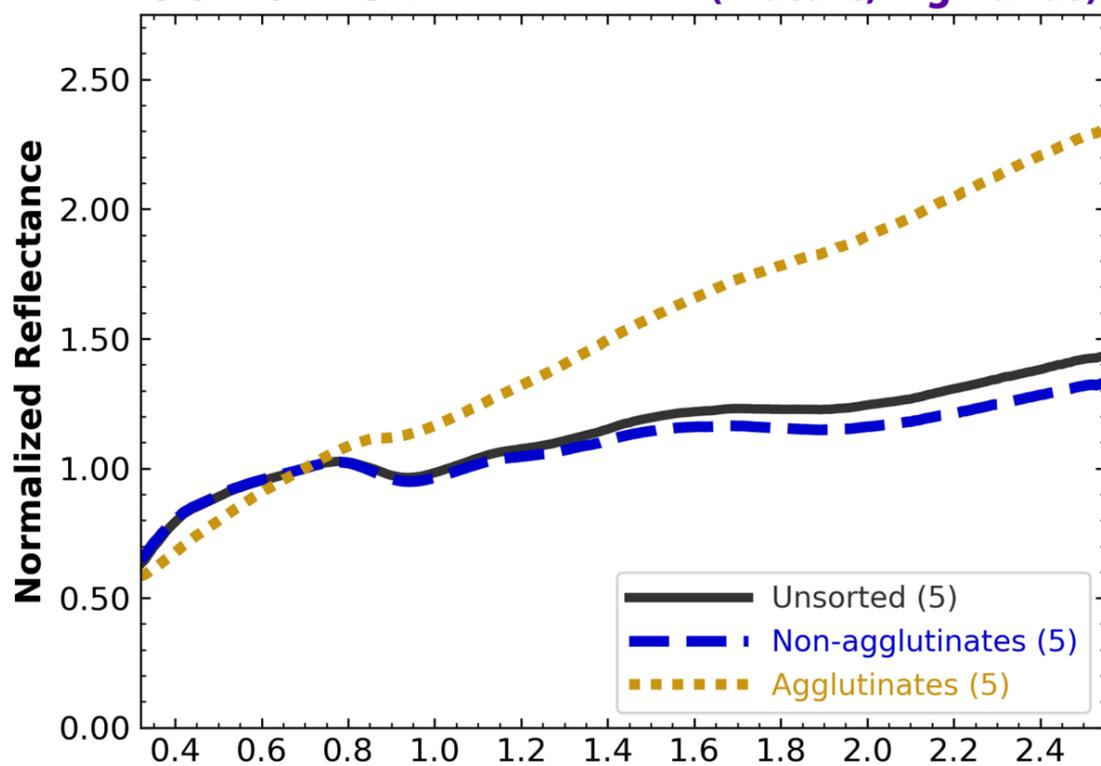
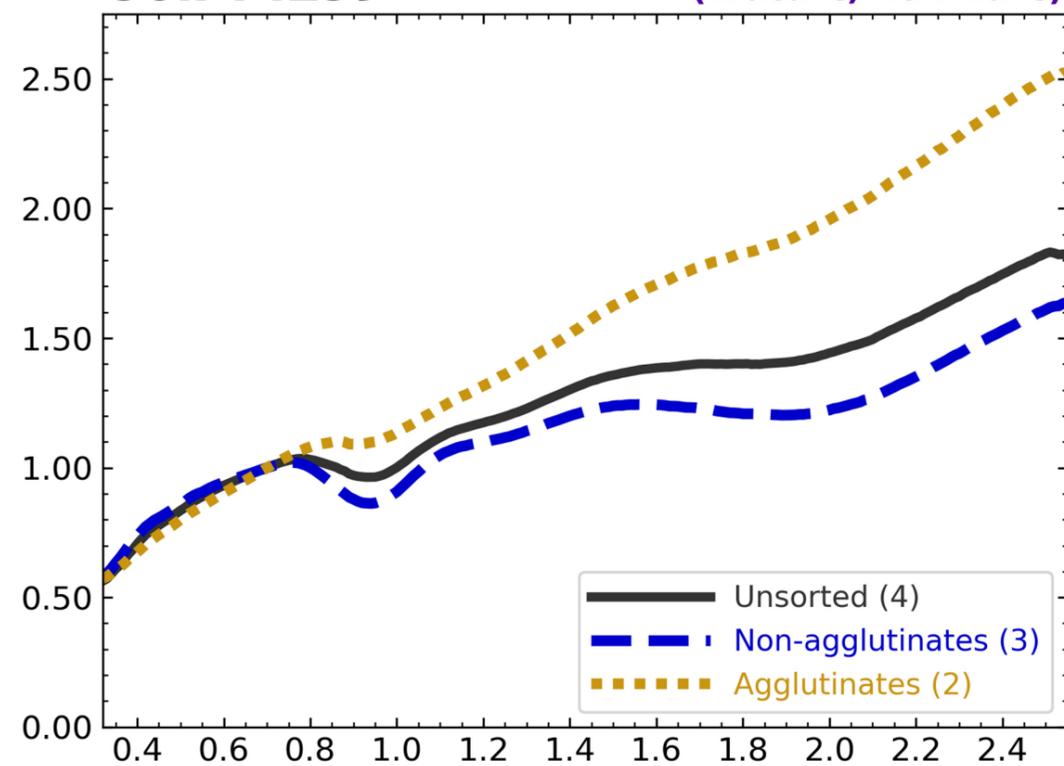
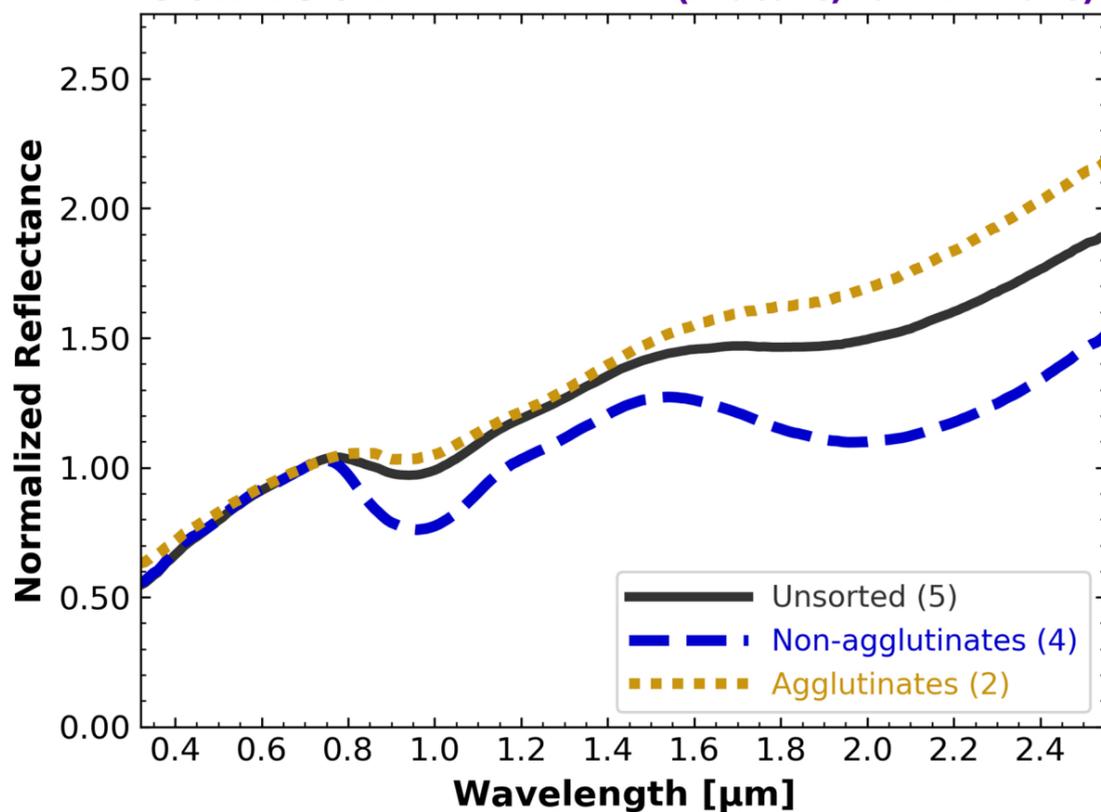
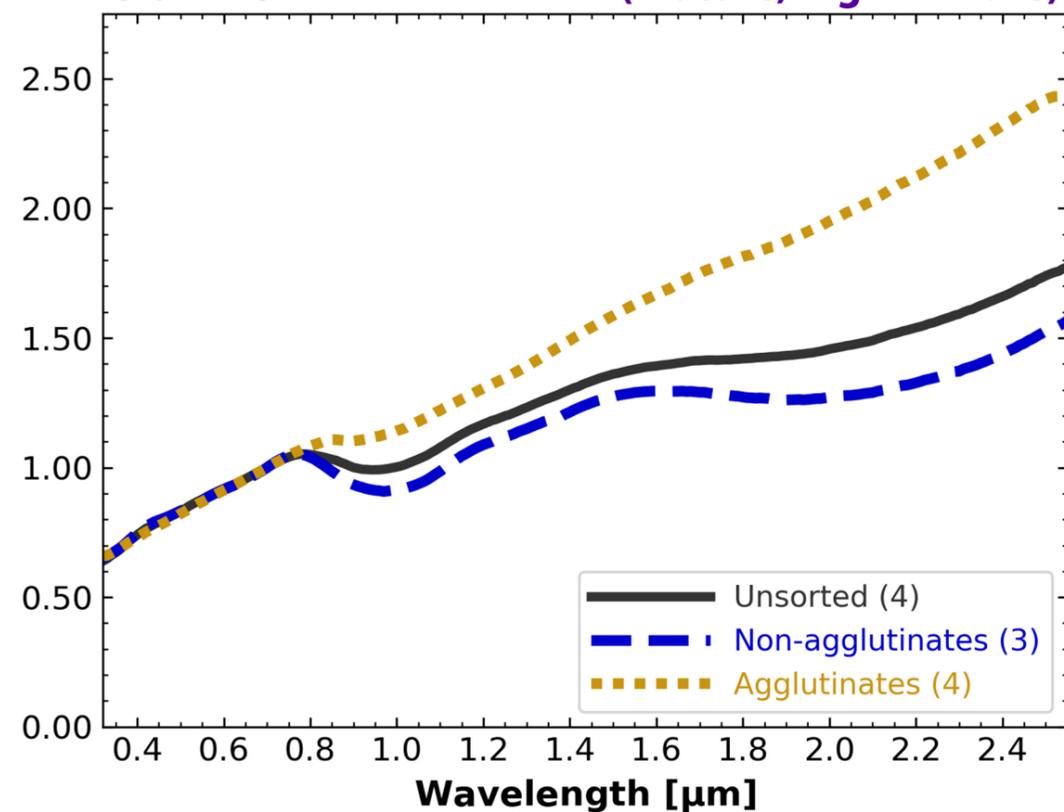
Soil 67461**(immature, highlands)****Soil 61141****(submature, highlands)****Soil 62231****(mature, highlands)****Soil 14259****(mature, nonmare)****Soil 15041****(mature, low-Ti mare)****Soil 79221****(mature, high-Ti mare)**

Figure 11.

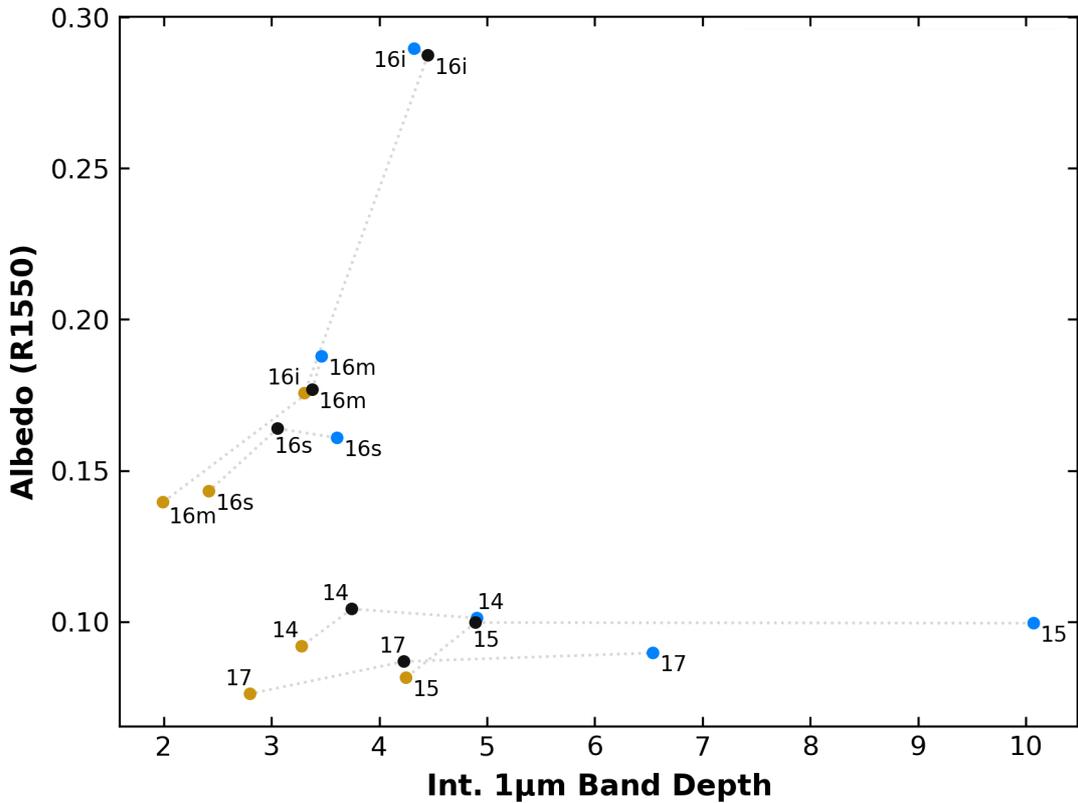
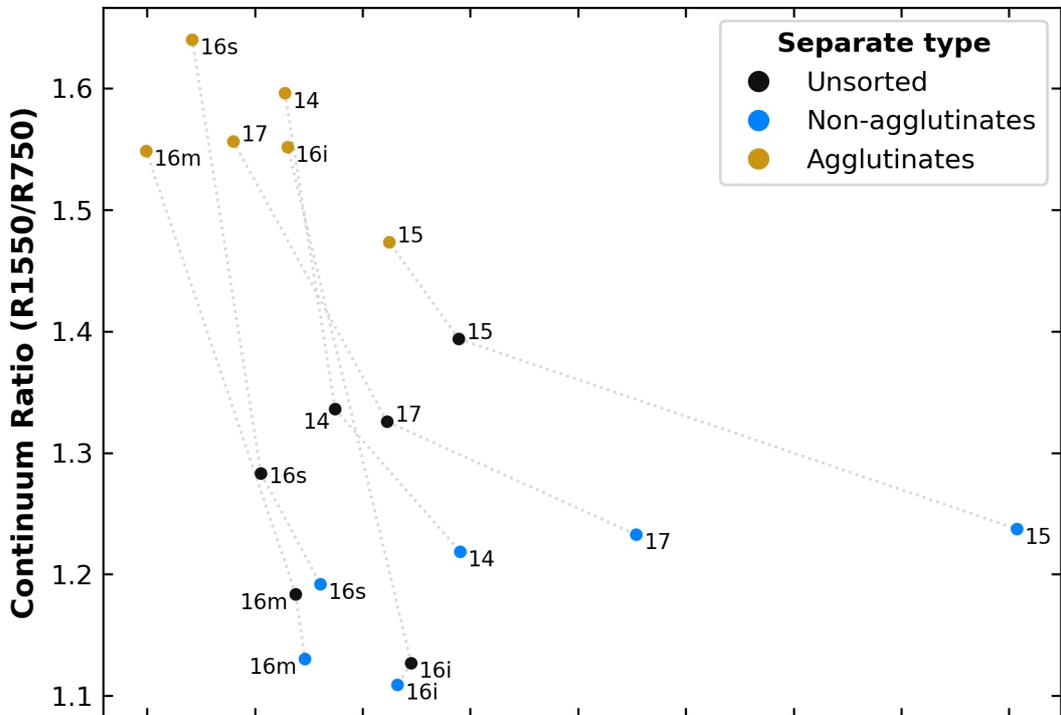


Figure 12.

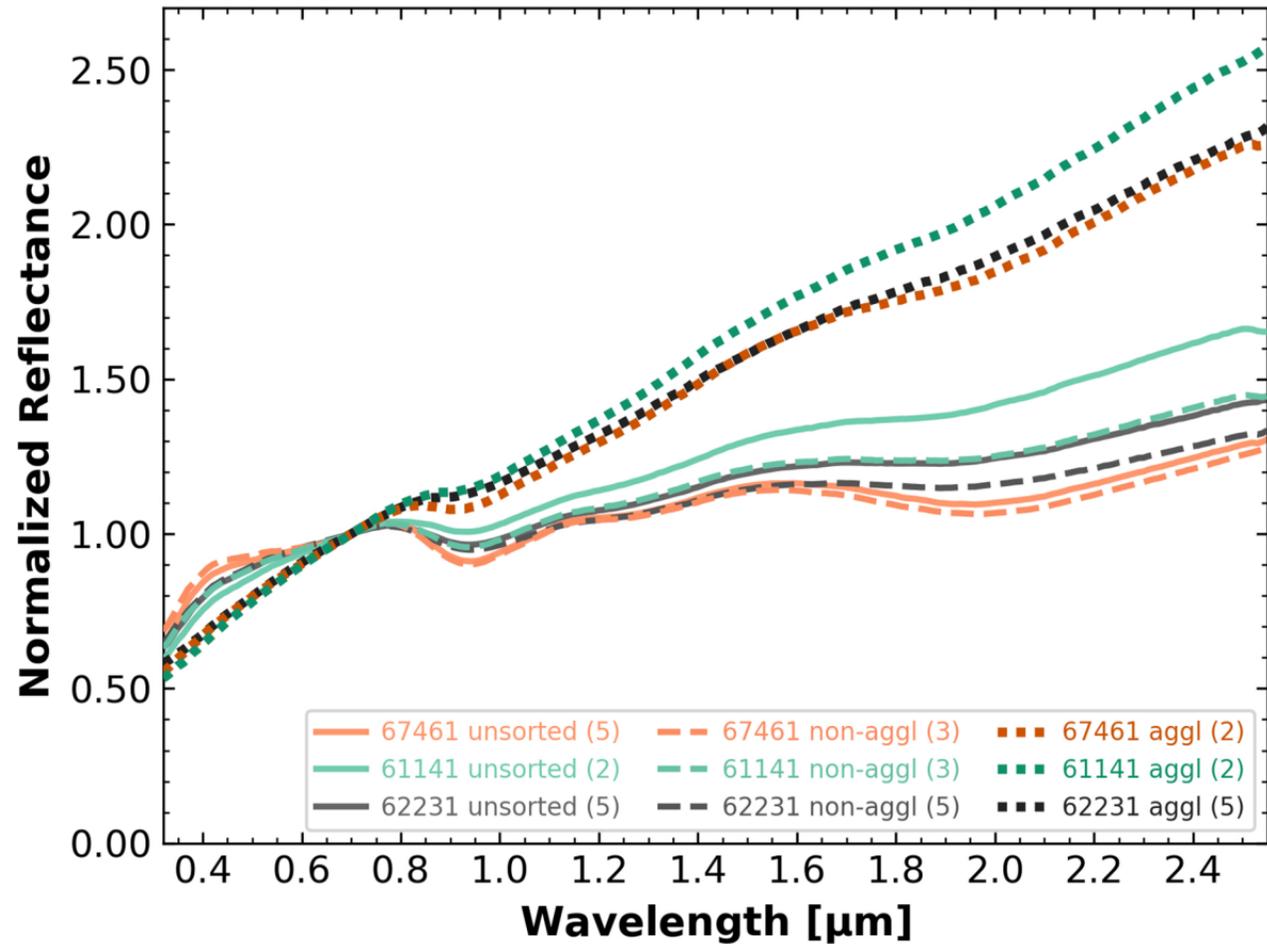
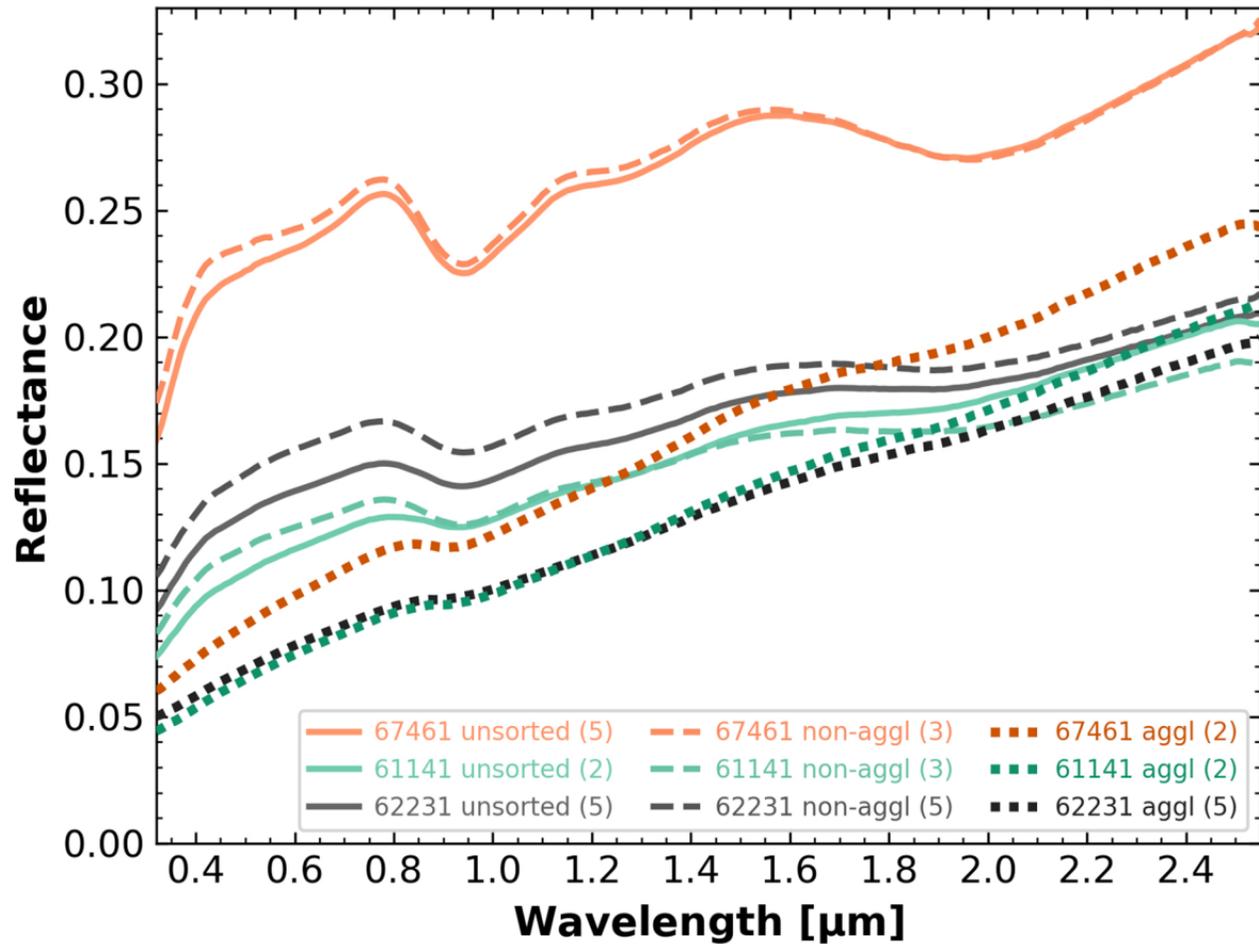


Figure 13.

Figure 14.

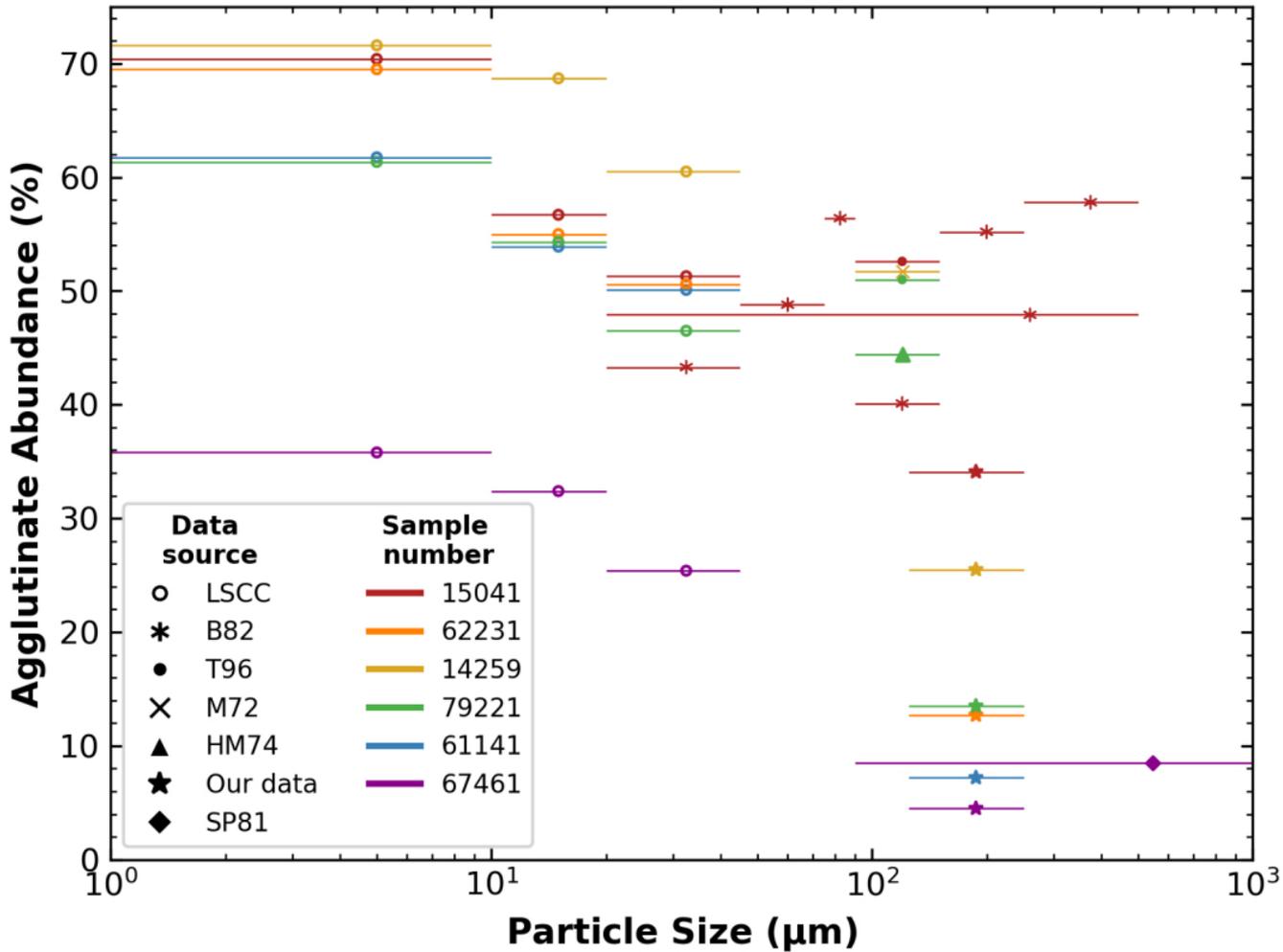
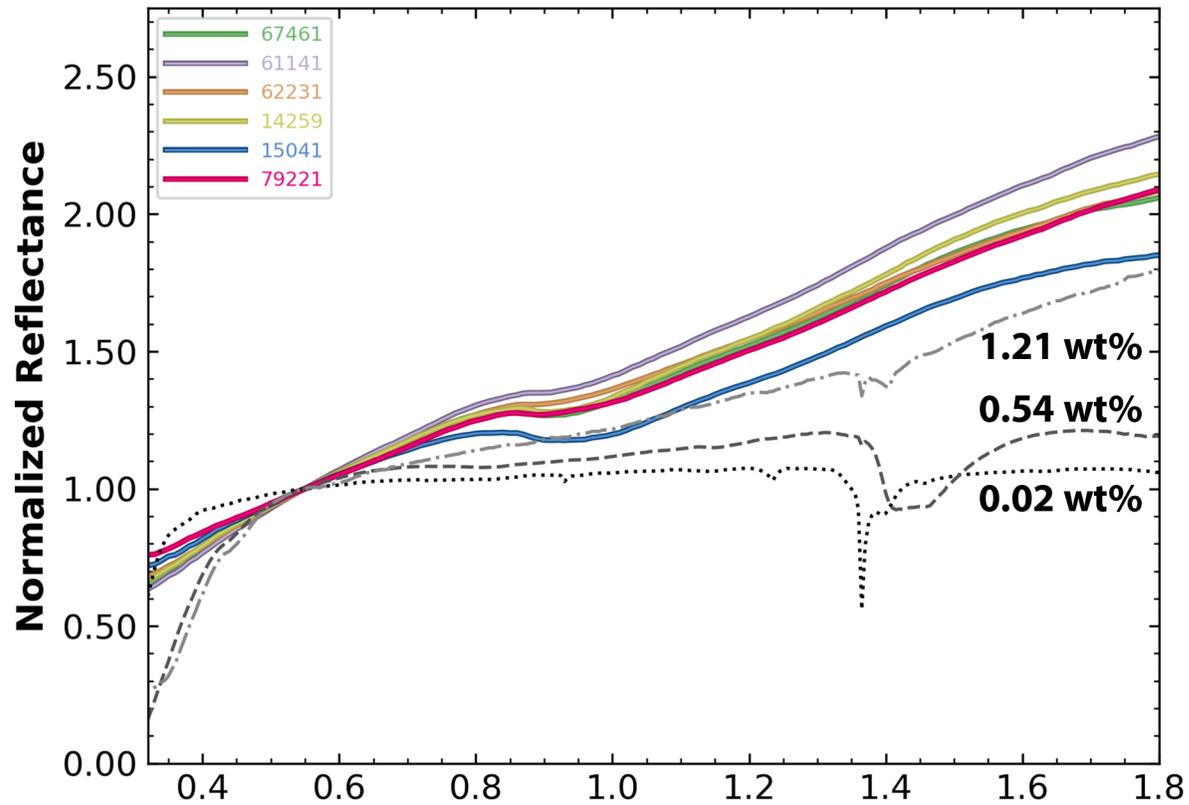
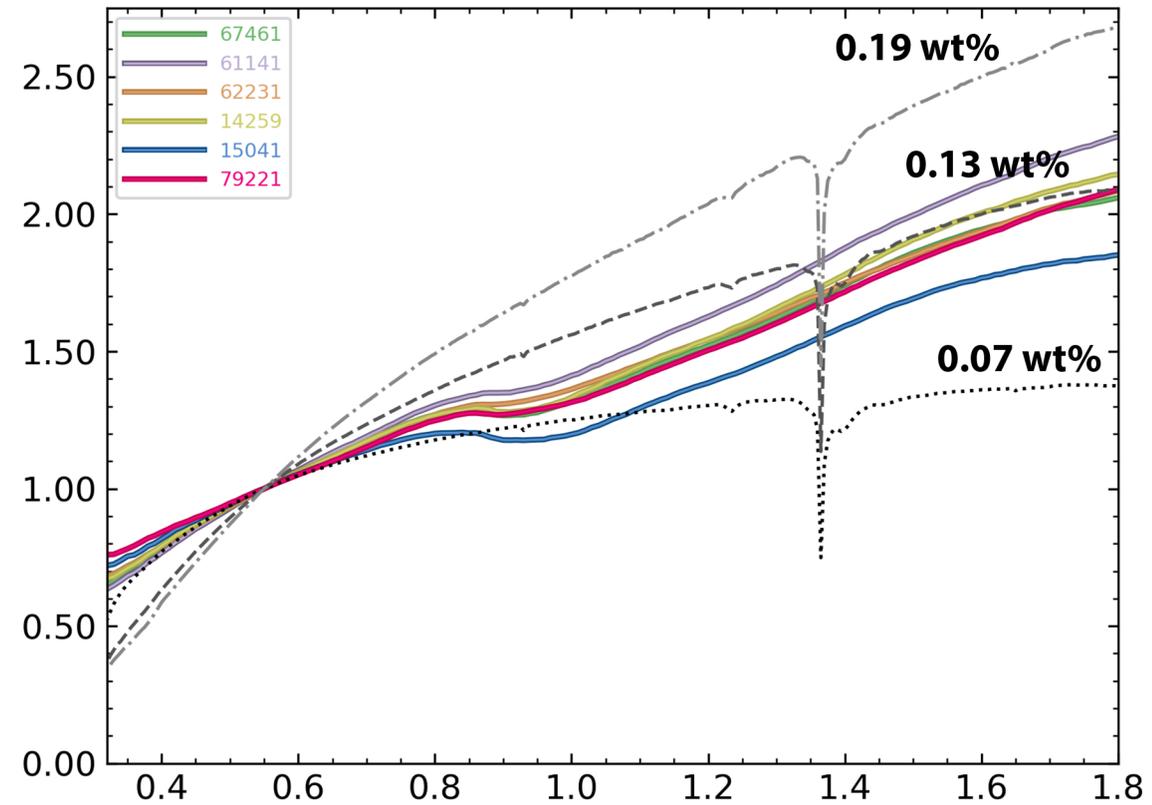


Figure 15.

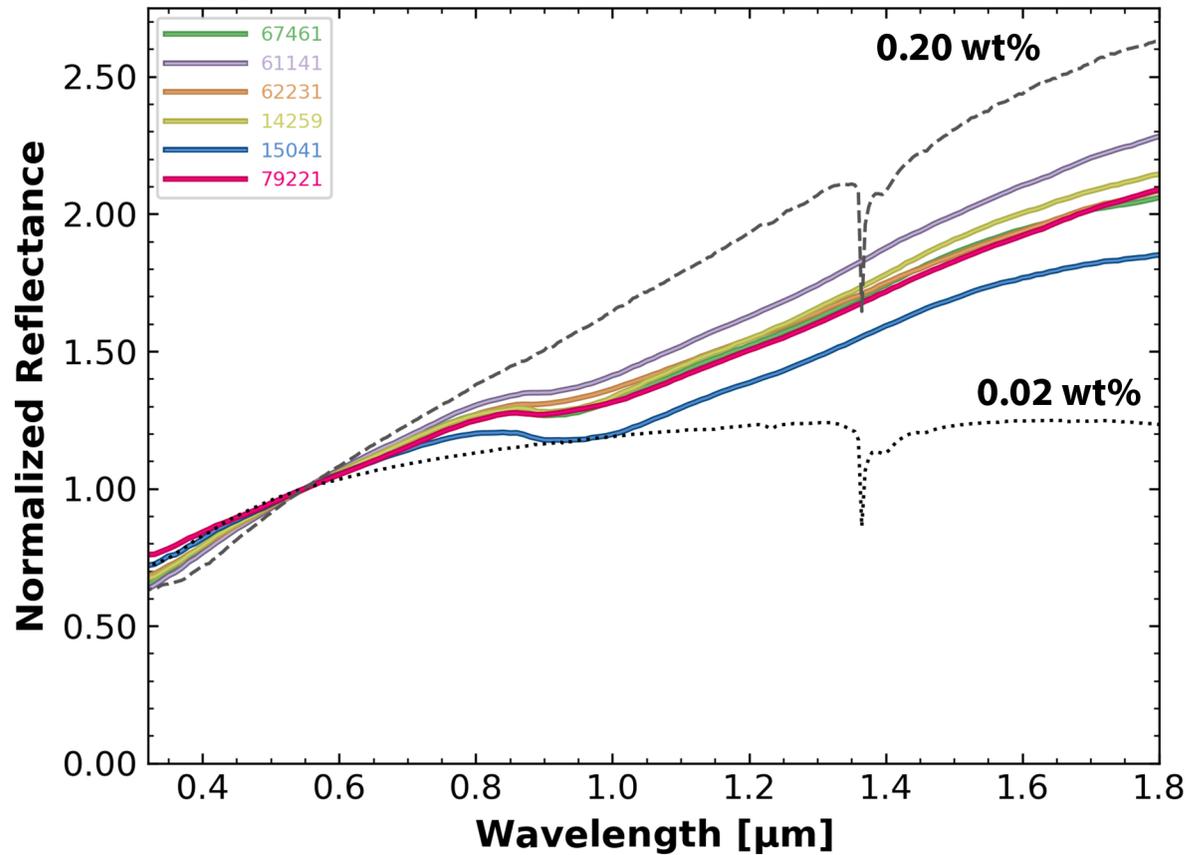
Agglutinates and ~8nm npFe



Agglutinates and ~15nm npFe



Agglutinates and ~35nm npFe



Agglutinates and ~40nm npFe

