MODAL MINERALOGY AND MATURITY ESTIMATES OF APOLLO 14, 15, AND 16 SOILS USING FTIR AND QEMSCAN TECHNIQUES. D. J. P. Martin[1], K. H. Joy[1], A. Morlok[2], H. Bagshaw[1], R. A. Wogelius[1], H. Hiesinger[2]. [1] School of Earth and Environmental Sciences, University of Manchester, Oxford Road, Manchester, M13 9PL. dayl.martin@manchester.ac.uk; [2]Institut für Planetologie, Wilhelm-Klemm Strasse 10, 48149 Münster.

Introduction: Lunar regolith samples exhibit a range of physical, chemical, and mineralogical properties including grain size distribution, grain shape, modal mineralogy, bulk chemistry, maturity (often estimated via the I_s/FeO index, where I_s = amount of nanophase iron and FeO is bulk rock iron oxide content [1], or the amount of ^{36}Ar implanted from the solar wind [2]), and spectral reflectance (or emittance) profiles [3-7]. This study has employed the use of FTIR bulk- and microspectroscopy to investigate the spectral properties of unconsolidated Apollo soils (bulk and sieved fractions) and obtain modal mineralogy and maturity estimates non-destructively. QEMSCAN (an innovative new SEM phase analysis tool [8]) analysis has been used to estimate modal mineralogy from polished grain mounts, along with finding the grain size distribution and grain shape of each sample.

Sample Preparation: 7 Apollo soils (~2 grams each of 14163, 14259, 15101, 15401, 61220, 62231, and 67481) were sieved using steel-mesh sieves and a dry-brushing technique, resulting in grain size fractions of 0-25, 25-63, 63-125, 125-250, >250 µm and a bulk soil for each sample. For each size fraction, a portion was transferred into an aluminium sample cup and the edge of an aluminium spatula was scraped across the cup to obtain a flat surface. FTIR spectroscopy was then used to analyse the surface of each size fraction of each soil. Post-sieving, a portion of 3 of the soils (14259,672, 15401,147, and 67481,96) were sent to JSC for mounting and polishing for QEMSCAN analysis.

FTIR Techniques: In Manchester, a PerkinElmer Spotlight-400 spectrometer with attached mapping unit was used to obtain mid-infrared (3-15 µm) images of the Apollo soils with a resolution of 25 µm per pixel. 32,000 pixels were collected per image. These measurements were made under ambient conditions. A series of band ratios were applied to the Reststrahlen Bands (RB; mineral sensitive region ~7-13 µm) of the images to identify each pixel as a specific mineral or phase, and the modal mineralogy estimates were derived from the proportions of each mineral or phase being identified per sample image. In Münster, a Bruker 70v Vertex FTIR instrument was used to obtain average spectra of each size fraction of the soils under both ambient and vacuum conditions. A shift in the Christiansen Feature (a reflectance minimum sensitive to bulk chemistry) was also used to estimate the maturity of each sample.

QEMSCAN Techniques: In Manchester, a FEI QUANTA 650 FEG ESEM was used to obtain quantitative information regarding the modal mineralogy, grain size distribution, and grain shape properties of the polished grain mounts. A beam strength of 10 nA and step size of 2.5 µm was used (giving an effective spot size of 2.5 µm), and data was collected using the Particle Mineralogical Analysis collection mode. This was then used to identify and analyse individual particles within the polished grain mounts. The QEMSCAN software uses a Species Identification Protocol (SIP) list containing EDS data of minerals [8]. In this study, a SIP list customized for lunar samples was used to identify each 2.5 µm pixel as a specific lunar mineral or phase, and the proportions of which were used to estimate the modal mineralogy. Grain shape was also classified using the QEMSCAN software, and grain size distribution was calculated using automated particle counting (y-axis) and a series of pre-defined size bins (x-axis).

FTIR Results: The spectral properties of the soils from the same landing site were highly similar with respect to the positions of the main reflectance bands. The exception to this was soil 15401, which is a pyroclastic glass bead-rich soil and is atypical of the Apollo 15 landing site. For each soil, the positions of the diagnostic reflectance bands were also consistent between grain sizes, though a shift to shorter wavelengths of the CF position was observed in coarser grain sizes (that could not be associated with mineralogy and, by extension, bulk chemistry) along with the appearance of a Transparency Feature (TF) in the bulk soil and 0-25 µm fraction. A general reduction in reflectance of the diagnostic reflectance bands was observed with decreasing grain size. A similar decrease in %Reflectance was also observed in more highly mature samples (for example the highly mature soil 62331 with I_s/FeO = 91 compared to the immature soil 61220 with I_s/FeO = 9.2 [8]). Modal mineralogy/phase estimates were within 2-8% of those determined by previous studies [5, 9-12]. Volcanic- and impact-derived glass was identified based on their differing CF positions (impact glass is more feldspathic so has a shorter wavelength CF position). The proportions of plagioclase and agglutinate glass were found to increase with decreasing grain size (consistent with previous studies) and bulk sample results were most similar to those of the finest grain size fraction (0-25 µm).

QEMSCAN Results: Modal mineralogy estimates from QEMSCAN analyses are also within 2-8% of the proportions quoted from previous studies, and within 5% of the FTIR estimates. However, a greater range of minerals were observed in these results compared to our FTIR method, particularly oxides and sulphides, due to such minerals being IR-inactive in the RB region.

Volcanic and impact glasses were identified based on their Mg/Al ratio (>1.1 for volcanic glass) [13]. Grain size distributions of each soil size fraction generally follow bell-curve profiles, though a large number of small particles (<5 µm) are present in each sample, skewing the results toward the finer grain size fractions. As such, for the larger grain sizes (<25 µm), data from the <5 µm fraction were discarded. Grain shapes were angular for mineral fragments and amorphous glass phases, and well-rounded for volcanic glass beads and particles <10 µm (although the latter is due to the 2.5 µm resolution not resolving the grain boundaries in enough detail for the true grain shape to be determined).

FTIR and QEMSCAN comparison: Both techniques adequately estimate the modal mineralogy (compared with each other, Figure 1, and previous studies) of each grain size fraction. However, QEMSCAN analyses are dominated by the abundant fine particles present in each section, largely because of the small number of large particles present on a single slide (this is particularly applicable to the >250 µm and bulk sample fractions). FTIR analyses are affected by a similar problem in that the fine particles (<10 µm) coat the surfaces of larger grains (<50 µm). Spectral mixing also occurs in samples <25 µm due to more than 1 grain being present in a single field of view. This causes the more reflective component (in the case of lunar samples: plagioclase) to dominate a mixed spectrum, causing a skew towards greater proportions of the mineral component. This is particularly problematic in Apollo 16 samples that are plagioclase-rich, with FTIR analyses resulting in plagioclase estimates that are 10% greater than those measured by other techniques in the 0-25 µm grain size fraction. However, for the purpose of completely non-destructive (FTIR) and largely non-mineralogy, the nature of each sample (e.g. source lithology or, in the case of Apollo 14 soils, the mixed nature of the soil) can be determined, along with identification of the variety of minerals and phases present and their approximate proportions.

Maturity estimates: CF positions of the average image spectra of samples shift to lower wavelengths with increasing grain size. Such a shift is usually caused by increased feldspathic components, but this is the opposite of what would be expected (as the feldspathic component increases with decreasing grain size). Therefore, this shift is attributed to maturity effects. Given that the finest grain size fraction has the greatest surface area, space weathering (and, therefore, maturation) has the greatest effect upon this size fraction (higher surface area for solar wind argon and nanophase iron to adhere to). As such, maturation occurs at a greater rate in the finest soil fraction, resulting in a trend of increasing maturity with decreasing grain size. In terms of spectral effects, the CF position offset between the finest (0-25 µm or Bulk fraction) and coarsest (>250 µm) fractions would be greatest in immature samples, and lowest in highly mature samples. A comparison of this CF position offset with measured I_s/FeO values of each sample display a weak logarithmic trend (R^2 = 0.54). However, FTIR results of the finest fraction of Apollo 16 samples were skewed by the high plagioclase content, resulting in reduced CF position offset. When the highly immature 61220 skewed result is removed, the logarithmic fit shows a strong correlation (R^2 = 0.91), and a linear trend shows a fair correlation (R^2 = 0.73). As such, the maturity of lunar soils can be estimated using FTIR spectroscopy, though soils require sieving prior to analysis to acquire the coarse >250 µm fraction.

Conclusions: Modal mineralogy estimates using FTIR and QEMSCAN techniques are consistent with those obtained from previous studies, with the exception of the finest fraction of some soils. The modal mineralogy proportions of soil 62231 have been estimated for the first time. Also, we highlight a non-destructive method of estimating maturity using FTIR techniques that is applicable to most samples and, potentially, could replace the destructive ferromagnetic method of estimating maturity (that is no longer used). This is important for future sample return missions to the lunar surface and to other airless bodies.

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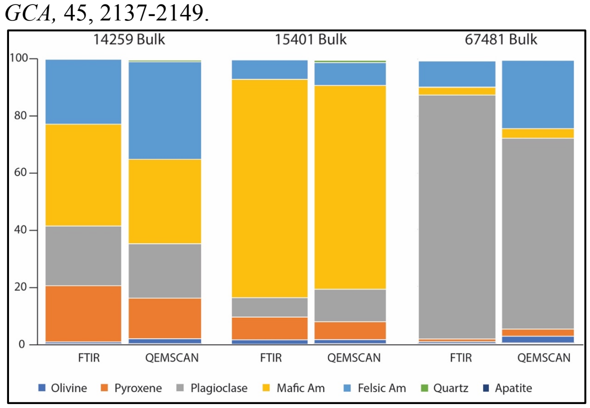

Figure 1 – A comparison of FTIR and QEMSCAN modal mineralogy estimates. Individual minerals identified through QEMSCAN were grouped for this comparison (e.g., anorthite and bytownite were combined for the plagioclase field). Am = amorphous material.