FUNDAMENTAL PROPERTIES CHARACTERISATION OF LUNAR REGOLITH SIMULANTS AT THE EUROPEAN SPACE AGENCY (ESA) SAMPLE ANALOGUE CURATION FACILITY. K. Manick[1], S-J. Gill[1], J. Najorka[1], C. L. Smith[1] and L. Duvet[2]. [1]Department of Earth Sciences, Natural History Museum, London, SW7 5BD, UK k.manick@nhm.ac.uk. [2]ESA ECSAT, Fermi Avenue, Harwell Campus, Didcot, Oxfordshire, OX11 0XD, UK.

Introduction: In this study we examine preliminary results for grain size/morphology by scanning electron microscopy (SEM) for the lunar regolith simulants EAC-1, Zybek NU-LHT-2M and USGS NU-LHT-2M, contained as voucher specimens within the ESA Exploration Sample Analogue Collection (ESA^{2}C). The grain size/morphology analysis was carried out in mid-late 2017 using the facilities at the Natural History Museum (NHM) Imaging and Analysis Centre (IAC) in London as part of activities for the ESA Sample Analogue Curation Facility [1]. Bulk mineralogy analysis by X-ray diffraction (XRD) is yet to be completed and is scheduled for late-January 2018 at the NHM.

Simulants: The EAC-1 regolith simulant is a milled basanite sourced from the Eifel region in Germany. It is a grey silty sand created by the ESA European Astronaut Centre (EAC) for their LUNA testbed facility [2]. Three batches of EAC-1 were acquired by the Curation Facility.

The Zybek NU-LHT-2M simulant was developed by Zybek Advanced Products Inc. as a Lunar highland regolith simulant [3]. The simulant includes glass, agglutinates, bricks, fiber and synthetic plagioclase melt breccias produced using an active plasma smelter. The USGS NU-LHT-2M simulant contains ~30% of an agglutinate component produced by Zybek Advanced Products Inc (USGS/NASA LHT) [4]. The majority of the material was sourced from Stillwater Mine in Nye (Montana, USA), with added minerals such as olivine and ilmenite. Visually, both Zybek and USGS appear to be light grey silty sands.

Within the Facility, the simulants are labelled as shown in Table 1.

Simulant	ESA ID	Sub-Sample ID
EAC-1 Basanite	ESA04-A	17DE01, 17DE02, 17DE03
Zybek NU-LHT-2M	ESA07-A	17US01
USGS NU-LHT-2M	ESA08-A	17US02

Table 1. Lunar regoliths simulants – vouchers specimens details.

Grain Size and Morphology Analysis: Riffle split sub-samples of each simulant were prepared as polished blocks (7 for EAC-1 and 1 each for Zybek and USGS). The sub-samples were set in Specifix resin (RI ~1.54) with hardener and then ground to expose flat and fresh mineral surfaces. It is assumed that during the polished block preparation, the grains are spread evenly in the resin. Grayscale backscattered electron (BSE) images were then obtained (Figure 1) for the polished blocks by SEM. EAC-1 was analysed using a Zeiss EVO 15LS SEM with X-ray analysis and XMAX80 silicon drift detector. Imaging was carried out at 20kV and 3nA and processed using the Oxford Instruments INCA software (pixel size 983.40 nm). Zybek and USGS were analysed using a FEI Quanta 650 FEG SEM with a BSE detector at 12kV and the images were collected using MAPS software (pixel size 366.97 nm and 351.42 nm, respectively).

The BSE images were cropped to show an area of approximately 1.01 mm width x 0.68 mm height. These images were analysed using the open source software package, ImageJ [5] and the statistical analysis was done using the Analysis ToolPak add-In in MS Excel. Using ImageJ, the BSE images were segmented and then analysed using the automated 'Analyze Particles' function.

Very small particles (with apparent area <10 μm^2) were not included in the image analysis. This is because the image resolution was not high enough for such particles to be accurately represented in the images. Also, particles at the edges of the images and holes within particles were excluded. The smallest apparent particle diameter detected in each image for all samples was either 4 μm or 5 μm.

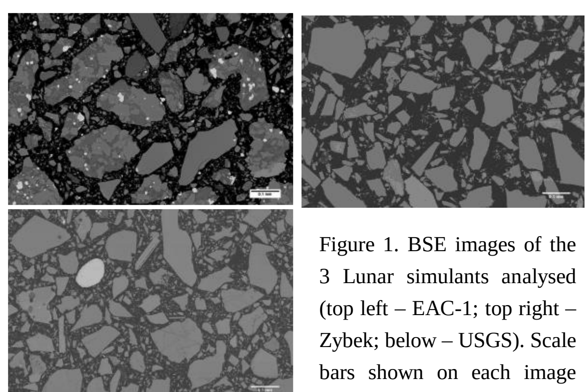

Figure 1. BSE images of the 3 Lunar simulants analysed (top left – EAC-1; top right – Zybek; below – USGS). Scale bars shown on each image represent 0.1 mm.

Bulk Mineralogy by X-Ray Diffraction: Riffle split sub-samples of each simulant were milled to a fine powder (<0.1μm) using a Retsch XRD Mill McCrone. Approximately 3g of the each powder was then mixed with ~10% of a corundum standard (Al_2O_3). The samples will be analysed at the NHM using a PANalytical X'Pert Pro diffractometer with a Cobalt X-ray source. The mineralogy was then characterised using the Rietveld method.

Grain Size and Morphology Results: Within a ~0.69 mm^2 image area, the USGS analogue contained the highest number of particles (total 1,207) suggesting that there is greater packing of the grains. By examining the number of particles within 2 micron apparent diameter ranges (Figure 2), it is shown that the USGS simulant contains a higher number of fine particles compared with the other 2 simulants, supporting the visual findings from another study [6]. This greater packing of grains (and infilling of voids by fines) may explain the higher 'cohesivity' observed on a large scale (e.g. through more stable boreholes) in the USGS simulant. Following milling however, the 'cohesivity' remains, suggesting a mineralogical/chemical control on this bulk property (also suggested in [6]).

The apparent particle aspect ratio (AR) was also analysed. The AR values were categorised according to ISO9276-6:2008: 1.0-1.2 Spherical; 1.2-2.0 Spheroidal; 2.0-5.0 Angular; 5.0-25 Rod-like. All of the samples are composed of dominantly spheroidal and angular grains with minor elongate and spherical components.

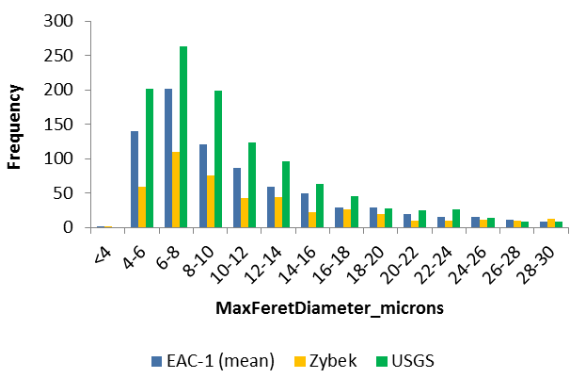

Figure 2. Particle count across 2 micron apparent particle diameter size ranges for the 3 Lunar regolith simulants.

Bulk Mineralogy Results: The quantification and identifaction of the mineral consitituents has yet to be carried out, but will be completed by 1 February 2018.

Conclusions and Further Work: The grain size and morphology workflow is designed to give a broad overview of these properties in unconsolidated soils. The polished block surface is ground and polished during preparation and the images show only 2D slices through the grains. Thus the results should be taken as an indicative, first pass overview (showing only apparent particle dimensions and shapes) and not definitive – whole, 3D grains may show a different particle diameter/morphology.

Particle size distribution (PSD) results for the 3 simulants by sieving and laser particle analysis are still pending. Results from these techniques will be used to validate the grain size (particle diameter) results determined by the SEM and image analysis techniques documented herein.

The simulants are produced from industrial processes, which don't control for grain shape. Therefore, during comminution (crushing and/or milling), the source material will break randomly along mineral grain boundaries and/or natural or induced weaknesses. The employed processes are unlikely to produce very spherical or elongate grains that are more likely to be produced from natural processes. This may explain the dominance of angular-spheroidal grain shapes in all three simulants.

Additional polished blocks for the USGS and Zybek simulants will be analysed in early 2018. The grain morphology analyses will then also be compared with the XRD results to determine the correlation between the particle shapes and the mineralogy for these three lunar simulants.

References: [1] Smith, C.L. et al. (2018) 49th LPSC, Abstract# 1623. [2] Nash et al. (2017) EPSC 2017-463, Vol. 11. [3] Zybek Advanced Products Inc. (2008) https://www3.nd.edu/~cneal/Lunar-L/Simulant-Press-release.pdf [4] Rickman et al. (2013) Manufacture of Lunar Regolith Simulants. NASA/TM-2013-217491. [5] Fiji ImageJ (v2.0.0-rc-54/1.51h) https://imagej.net/Fiji [6] Donaldson Hanna et al. (2017) 48th LPSC, Abstract# 1717.

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