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INVESTIGATION OF SURFACE PROPERTIES OF LUNAR REGOLITH
Part II

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Recently we published results of investigations of the surface properties of three lunar soil samples from the Apollo 11, 12 and 16 missions. In the present paper we summarise and supplement the results of measurements of density using a helium pycnometer, krypton and nitrogen adsorption using volumetric techniques and of water, heptane and octane adsorption using gravimetric techniques. We present additional diagrams and electron micrographs.

Keywords: *adsorption, moon, regolith, surface, water*

Introduction

Lunar soil and rock samples of the Apollo and Luna missions 1969–1972 have been already examined in detail [1–6]. Planning of manned station at the moon requires some additional information. One important question is whether there is any utilisable inventory of ice [7, 8]. Besides remainder of ice stocks delivered from asteroids, chemisorbed and vicinal water could be bound at lunar soil in regions, which are protected from solar irradiation. First results of our investigation contradict the ability of the moon regolith covering to store water [9]. In the present paper we report on additional investigations.

The Moon

Today, explaining the origin of the moon as a result of the giant impact hypothesis has been considered the most viable. It is believed that the moon formed from the ejecta resulting from a rambling collision between the early, semi-molten earth and an asteroid the size of Mars. The material ejected from this impact would have gathered in orbit around the earth and concentrated in a single sphere. This is in accordance with radiometric dating of the Moon's age as 4527±10 million years, which would imply that it was formed 30 to 50 million years after the origin of the solar system.

The lunar crust is composed of a variety of primary elements, including uranium, thorium, potassium, oxygen, silicon, magnesium, iron, titanium, calcium, aluminium and hydrogen, as determined by spectroscopy [10, 11]. Regarding its origin, the chemical and mineralogical composition of the moon is believed to be similar to that of the earth's crust. The surface of the material is, however, hardly treated by influences from outside. The moon's atmosphere is tenuous, consisting of gases produced and released from the interior, for instance radon and of solar wind.

For billions of years, comets and meteoroids continuously bombarded the Moon. Many large meteorites were able to penetrate the thin crust. Where the crust was perforated the hot lavas from the interior oozed out and spread over the surface, to cool down into the so-called maria. Thus, the very flat maria are relatively young areas on the Moon, and are very different from the heavily cratered and mountainous so-called highlands. By the impacts, rocks were shattered. The comminute material together with the refractory materials of the celestial bullets contribute to Moon's regolith covering. Lunar soil is the subcentimeter fraction of the lunar regolith. The regolith atop the crust varies from 3 to 5 m in the maria to 10 to 20 m in the highlands [12].

The samples

We investigated three soil samples from the Apollo 11, 12 and 16 missions, each of about 3 g. All samples consist of weakly coherent fines of diameter in the sub-millimeter range. Sample 12001.922 (Apollo 12, mare) and sample 64501.228 (Apollo 16,

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highland) appear light grey and include a few white particles. Sample 10084.2000 10084.2000 (Apollo 11, mare) had a dark grey colour.

Detailed studies have shown that five basic particle types make up the lunar soils: mineral fragments, pristine crystalline rock fragments, breccia fragments, glasses of various kinds, and the unique lunar structured particles called agglutinates [1]. The mineral and chemical composition of the lunar soils depends on the mission landing sites [13]. Apollo 11 and Apollo 12 landed well inside mare basalt regions, and consequently, soil samples from these sites have abundant mare-derived basaltic rock clasts and mafic minerals like olivine and pyroxene. On the other hand Apollo 16 landed in highland regions. Soils from this mission contain abundant highland-derived components, e.g. lithic fragments of anorthositic rocks, breccias and anorthitic feldspar.

The Apollo samples are handled and stored either under vacuum or in clean dry nitrogen gas at the Astromaterials and Curation Office at the NASA’s Johnson Space Center, Houston, Texas [14]. The sample vessels had been opened by us at ambient atmosphere so that adsorption of atmospheric components, as water vapour and oxygen could take place prior to the measurements.

Measurements and results

Electron microscopy was performed at the University of Münster (ICEM, Interdisciplinary Centre for Electron Microscopy and Microanalysis) (Figs 1–3). Density and sorption measurements were made at the POROTEC laboratory, Hofheim (Taunus) (Figs 4–6 and Table 1).

Scanning electron microscopy

The fine-grained textures of the three lunar soils were studied with a field-emission Scanning Electron Microscope (JEOL JSM-6300F) operating at ~5 kV.

The Apollo 16 soil sample 64501 contains abundant clastic grains, but also impact melt spherules of various sizes (Fig. 3). Fragments with internal pores like those found in the Apollo 11 and 12 soils (Figs 1 and 2) are either rare or absent. Kempa and Papike [15] found that in the particle size range of 10–90 μm sample 64501 is highly feldspathic having more than 50% plagioclase. In the Apollo 12 sample more small-grained and more molten and porous particles are found compared with the Apollo 16 soil sample (Fig. 2). Sample 12001 was previously characterized as a well-gardened soil, random mixture of debris fragments that mostly derive from nearby bedrock and whose particles largely consist of mare basalts or

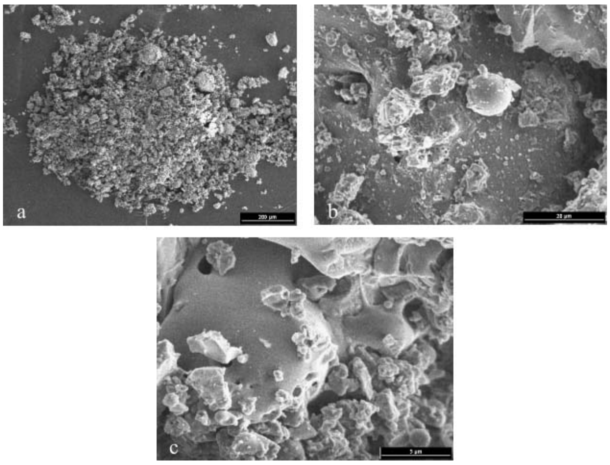

Fig. 1 (#10084-01/03/08). Electron micrographs at increasing magnification of lunar regolith sample 10084.2000 of the Apollo 11 mission from inside of a mare basalt region

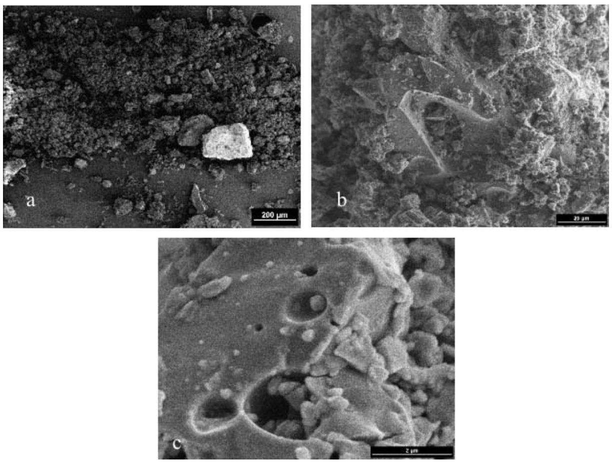

Fig. 2 (#12001-02/19/23). Electron micrographs at increasing magnification of lunar regolith sample 12001.922 of the Apollo 12 mission from inside of a mare basalt region

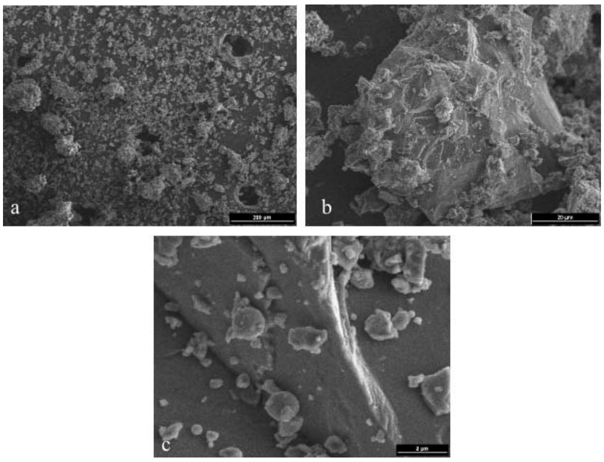

Fig. 3 (#64501-02/03/24). Electron micrographs at increasing magnification of lunar regolith sample 64501.228 of the Apollo 16 mission from highland regions

degradation products thereof [16]. The Apollo 11 soil sample (Fig. 1) has a high abundance of glassy particles or features that indicate melting (Fig. 1). It was found that the large proportion of the <10 μm material in the soil (14.2%) is consistent with its high maturity [17]. The 10084 soil is very fine-grained with a mean grain size of 51 μm and very poor uniformity. The high concentration of agglutinates (52%) was determined by Simon *et al.* [18].

Density

Helium displacement within a calibrated vessel [1, 19] at 20°C was applied using a PYCNOMATIC ATC instrument of Thermo Electron S.p.A., Milano, Italy.

The He-density values for lunar soils reported in the literature [1] range from 2.3 to >3.2 g cm^{-3} giving a mean value of 3.1 g cm^{-3}. Results of our measurements listed in Table 1 are the mean values of repeated measurements. Our results are well within the range of the literature values.

Specific surface area – krypton adsorption

Prior to the adsorption measurements the samples had been degassed at 80°C for 4 h. This is far below the temperature, which occurs on the moon during its diurnal cycle. Krypton adsorption isotherms were measured stepwise at 77.1 K using a volumetric/manometric apparatus SORPTOMATIC of Thermo Electron S.p.A., Milano, Italy.

Kr adsorption had been measured at 77.1 K and all the resulting isotherms (Fig. 4) correspond to type II of the IUPAC classification [20–22]. In the range of about $0.07<p/p_0<0.23$ the specific surface area was calculated by means of the 2-parameter BET equation using a molecular area of 0.195 nm^2. BET plots show well defined straight lines intersecting the ordinate at small positive values. The resulting values of specific surface area (0.3–0.7 m^2 g^{-1}) are within the region of values determined from N_2 isotherms by other investigators (0.2–1.5 m^2 g^{-1}).

For comparison, results from nitrogen adsorption measurements [10] are included. The much higher values are doubtful because the measurements are near the detection limit of the method. Cadenhead *et al.* [2] calculated a specific surface area of about 0.01 m^2 g^{-1} whereas nitrogen adsorption gave values of 0.1 to 1.0 m^2 g^{-1}. Differences in roughness and porosity as shown in the electron micrographs are in the micrometer range and do not contribute to the specific surface area as determined by gas adsorption. Thus, evaluations of that parameter from image analysis or particle size distribution are misleading.

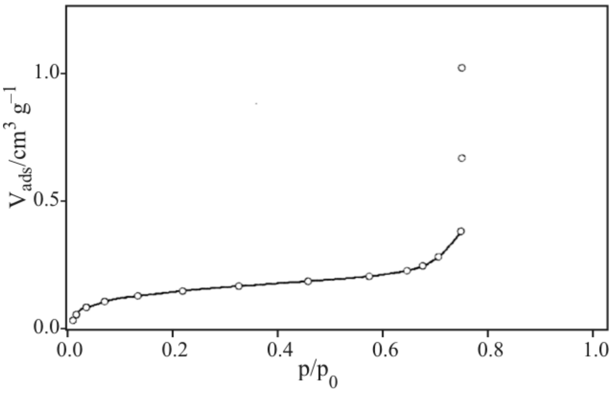

Fig. 4 Krypton adsorption isotherm at 77.1 K of lunar regolith sample 64501.228 from highland. The ordinate represents the adsorbed gas volume at S.T.P. divided by the mass of the degassed sample. The abscissa is the measured pressure above the capillary (∅10 mm) of the sample vessel related to the saturation pressure of 233 Pa. No correction was made concerning the thermal pressure difference in capillaries

Water vapour adsorption

We used a DVS 1/Advantage apparatus of Surface Measurement Systems, Ltd., Wembley, Middlesex U.K. The apparatus encompasses a Cahn microbalance with a maximum load of 1.5 g. Water vapour pressure is adjusted and varied by means of a flow of nitrogen carrier gas saturated with water vapour. The isotherms were measured stepwise at a temperature of 24.9°C slightly above ambient to avoid condensation within the apparatus. The specific surface area was calculated using a molecular area of 0.129 nm^2.

Table 1 Density and specific surface area of regolith samples

	He displacement/ g cm^{-3}	Kr/ m^2 g^{-1}	N_2/ m^2 g^{-1}	*n*-heptane/ m^2 g^{-1}	*n*-octane m^2 g^{-1}	H_2O/ m^2 g^{-1}
10084.2000 Apollo 11, Mare			2.39		0.481 0.432	0.368
12001.922 Apollo 12, Mare	3.10±0.01	0.579	1.77	0.408		0.358
64501.228 Apollo 16, Highland	2.79±0.01	0.653	2.73		0.540	0.397

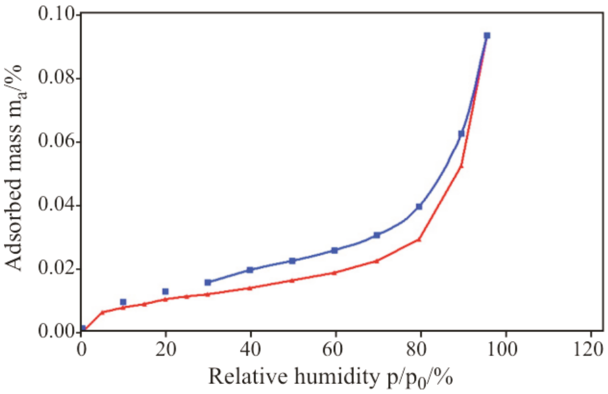

Fig. 5 Water adsorption and desorption isotherm at 24.9°C of lunar regolith sample 64501.228 from highland. ▲ – adsorption, ■ – desorption. m_a adsorbed water mass related to unit sample mass, p/p_0 water vapour pressure related to saturation pressure (relative humidity)

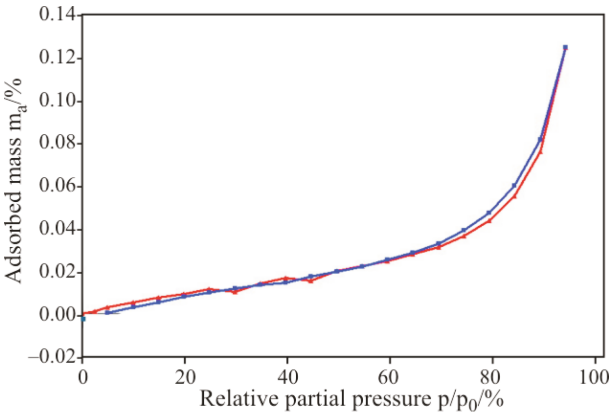

Fig. 6 Octane adsorption and desorption isotherm at 24.9°C of lunar regolith sample 64501.228 from highland. ▲ – adsorption, ■ – desorption. m_a adsorbed octane mass related to unit sample mass, p/p_0 octane partial pressure related to saturation pressure. (In Fig. 5 of the pre- ceding paper [9] the temperature is mistaken. The heptane isotherm is measured likewise at 24.9°C)

Water vapour adsorption and desorption at ambient temperature proceeds rapidly. Obviously no micropores are present which could obstruct the sorption process. The isotherm increases rather slowly from origin (Fig. 5), consistent with literature reports, which is typical for material having an intermediate character between hydrophilic and hydrophobic, and it indicates weak binding. Hysteresis loops extending over the whole region of relative pressure were observed which may be due to the fact that in many cases after changing the water vapour pressure a saturation value of the adsorbed mass was not attained. Furthermore, we also should keep in mind that these measurements are near to the detection limit of the measuring method and a rigorous interpretation of the results therefore is not possible. Indeed, investigations reported in the literature [1–5] show similar scatter of results. The specific surface area determined with water vapour adsorption (diameter 0.28 nm) is lower than that determined with krypton (diameter 0.36 nm). The reason for this is unclear and confirms uncertainties in the measurement.

n-heptane and n-octane adsorption

n-heptane and *n*-octane adsorption and desorption isotherms were measured similarly to those of water by gravimetric techniques using that DVS apparatus at 24.9°C. The specific surface area was calculated using the molecular areas of *n*-heptane and *n*-octane of 0.57 and 0.63 nm^2, respectively.

n-heptane and *n*-octane adsorption and desorption at ambient temperature proceeded rapidly, which indicates the absence of pores with entrance width of molecular size which could obstruct filling. In addition fast desorption indicates weak binding. The resulting isotherms correspond to type II of the IUPAC classification with nearly linear slopes within $0<p/p_0<0.7$ and without hysteresis between adsorption and desorption branch (Fig. 6). This suggests the absence of pores having nanometer widths. The specific surface area determined with those adsorptives is lower than that determined with krypton. This is reasonable because the molecular diameter of the *n*-hydrocarbons used are of about 0.6 nm and are larger than that of krypton (diameter 0.36 nm). They cannot penetrate into the small pores which are accessible to krypton.

Conclusions

To investigate lunar soil samples modern commercially available instruments proved to be superior in handling, sensitivity and resolution in comparison to such used in investigations reported earlier [23–25].

No significant differences in sorption properties have been detected for samples of different origin (mare and highland). Lunar regolith can hardly store water due to low specific surface area, little nano-porosity and a surface character lying between hydrophilic and hydrophobic. Structures like permafrost as observed on earth [26] and assumed on Mars [27–29] cannot be expected on the moon, on which the conditions do not allow the existence of liquid water. If remnants of ice exist as a bulk phase at the moon it may be found below the regolith covering.

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