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Higher water content observed in smaller size fraction of Chang'e-5 lunar regolith samples

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ABSTRACT

Water has been detected in lunar regolith, with multiple sources identified through the analysis of individual grains. However, the primary origin of water in the bulk lunar regolith remains uncertain. This study presents spectroscopic analyses of water content in sealed Chang'e-5 samples. These samples were sieved into various size fractions (bulk, <45 μm, and 45–355 μm) inside a glovebox filled with high-purity nitrogen. Results indicate a higher water content in the fine fractions (~87 ± 11.9 ppm) than in bulk soil (~37 ± 4.8 ppm) and coarse fractions (~11 ± 1.5 ppm). This suggests that water is predominantly concentrated in the outermost rims of the regolith grains, and thus exhibits dependence on the surface volume ratio (also known as surface correlation), indicating solar wind is a primary source of lunar surface water. Laboratory, *in-situ*, and orbital results bridge sample analysis and remote sensing, offering a cohesive understanding of lunar surface water characteristics as represented by Chang'e-5. The discovery provides statistical evidence for the origin of water in lunar soil and can be considered representative of the lunar surface conditions. The water enrichment of the finest fraction suggests the feasibility of employing size sorting of lunar soils as a potential technological approach for water resource extraction in future lunar research stations.

1. Introduction

The entire lunar surface is covered by a layer of fine-grained material called lunar regolith, which is the main focus of remote sensing exploration [1]. Water (in the forms of OH and H_2O) accumulated during the formation of lunar regolith through processes like asteroid and comet impacts [2,3], volcanic outgassing [4,5], and solar/Earth wind implantation [6–10]. The gardening process and shock heating resulting from impacts redistributed the water content within the regolith, making it challenging to determine its origins [11]. The correlation between particle size and water content helps distinguish between different water sources. For example, water implanted by solar wind shows a surface correlation, while water from the Moon itself or from comets does not [4,12]. While there have been studies on how particle size affects estimating water content in hydrated materials, the main focus has been on developing models to correct the effects of optical length on water absorption features [13]. Water on the lunar surface (OH/H_2O) has been detected using infrared data from spacecraft [12,14–16] and ground-based telescopes [17]. However, infrared remote sensing can only reveal spatial and temporal variations in water content on the lunar surface, not variations related to particle size. In addition, different methods used to correct thermal influences in lunar hyperspectral data have led to conflicting results regarding overall water content and its distribution across the lunar surface [18–20], making it harder to explore the origins of lunar water.

Examining spectral measurements from sealed lunar bulk soil samples alongside *in-situ* spectral observations of the sampling site will offer better insights into understanding water content and its origins on the lunar surface. *In-situ* measurements of reflectance spectra by the Lunar Mineralogical Spectrometer (LMS), an instrument on the Chang'e-5 lander, have revealed the presence of water in the regolith at the sampling site [21,22]. Chang'e-5 stands out as

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the only mission to retrieve lunar samples and gather *in-situ* reflectance spectra from the precise sampling location. Situated at coordinates 43.06°N and 51.92°W on the lunar surface (Fig. 1a), the sampling site for Chang'e-5 predominantly involves samples extracted a few centimeters beneath the lunar surface (Fig. 1b). This lunar sample represents the highest latitude collection compared with previous missions (i.e., Apollo and Luna). The subsequent laboratory analysis of these "fresh" samples establishes a definitive benchmark for assessing *in-situ* derived water content, thus refining our understanding of its origins. The water contents have been assessed with a very limited number of mineral grains and glass beads selected from the Chang'e-5 soil samples [7,8,23]. However, doubts arise regarding whether the analyzed regolith grains (a few tens in total) accurately represent the bulk regolith of the landing site for comparison with remote sensing observations, given the potential heterogeneity in the returned Chang'e-5 samples. Therefore, analyzing bulk samples becomes crucial for a more representative understanding. Additionally, the analysis focused only on the outermost rims of several large particles (>100 μm), overlooking the correlation between water content and particle size. Moreover, given that Chang'e-5 is the only sample returned from the mid-latitude of the Moon, it is essential to assess the water content in the bulk soil for understanding its distribution on the Moon. Thus, a thorough analysis of water in the pristine Chang'e-5 bulk soil is necessary. While reflectance measurements on pristine Apollo bulk soil samples confirmed hydration features of remote observations, determining water content and origin precisely remains challenging [24]. This study proposes a novel approach to characterizing the primary source of water in lunar regolith by comparing water content between fine and coarse Chang'e-5 lunar soil samples while considering the effects of particle size.

2. Materials and methods

1.5 g of the Chang'e-5 soil samples scooped from the lunar surface were utilized for the spectral experiment. These samples consist of lunar soil particles smaller than 1 mm [25], and have never been exposed to the atmosphere (Fig. 1c). The samples were transferred from the Ground Application System to the Institute of Geology and Geophysics, Chinese Academy of Sciences (CAS), using a sealed box filled with nitrogen to prevent contamination of atmospheric water. To avoid atmospheric water contamination, the container of the soil samples was opened, and their reflectance spectra were collected in a glovebox filled with high-purity N_2 with <0.1 ppm water, which is much lower than the detection limit of the spectrometer (Fig. S1 online). The soil was manually sieved into three fractions at <45 μm, 45–355 μm, and >355 μm within the glovebox (Fig. S2 online), with the >355 μm size fraction accounting for 4 wt.%. Reflectance spectra of the bulk soil, <45 μm size fraction, and 45–355 μm size fraction were measured. Two/three spots were measured for each of the three samples, and the averaged spectrum was used. The entire measurement process was conducted without thermal effects. The spectrometer used is a spare flight model of Chang'e-5 LMS, with a spectral range from 0.58 to 3.2 μm and the capability to observe an area of 8 mm in diameter [22]. Calibration was performed using a white diffusive reflectance standard and a gold panel. The measurement process occurred entirely within the glovebox (Fig. S1 online), and the analysis focused on the absorption at ~2.8 μm, a prominent feature of OH/H_2O.

The effective single-particle absorption thickness (ESPAT) at 2.8 μm, derived from the single-scattering albedo (SSA) spectra of the soil samples, served as the basis for estimating water content. Laboratory experiments have demonstrated a strong linear

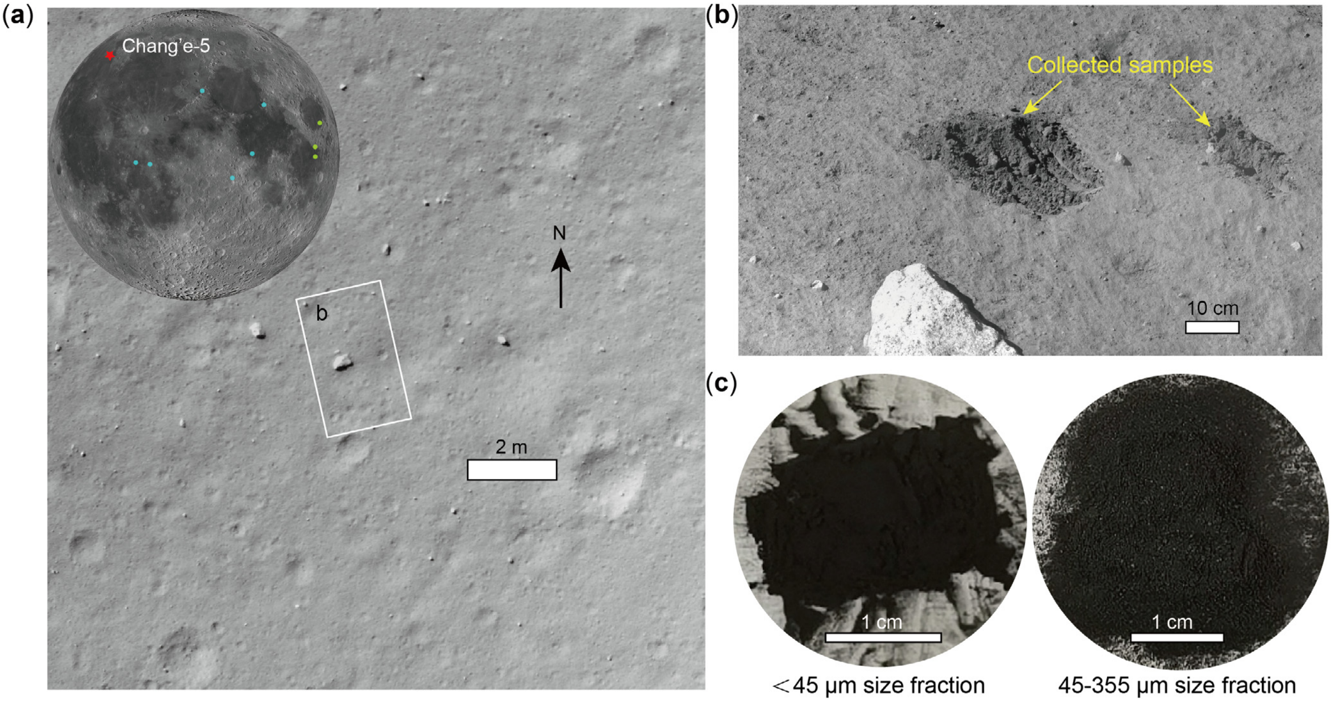

Fig. 1. The Chang'e-5 landing site and returned samples. (a) The location of the sampling site is depicted, with the background displaying an image captured by the landing camera. The inset picture shows the LROC WAC image, with all sampling sites labeled for reference. (b) The sampling area captured by the Panoramic Camera onboard the Chang'e-5 lander. (c) Pictures of the sieved soil samples, which were measured by the spectrometer, indicating statistical significance (close shots of the samples are shown in Fig. S3 online).

correlation between ESPAT at 2.8 μm and water content [20,26], with no compositional dependence [20,27]. The calculation of SSA followed the same method described in the analysis of M^3 and Chang'e-5 *in-situ* spectral data [20,21]. Continuum removed SSA spectra were obtained using a line spanning from 2.7 to 2.85 μm, as the spectral range does not extend to 3.6 μm [20,21].

To compare the compositions of samples with varying particles, backscattered electron (BSE) images were captured using a Thermo Fisher Apreo field emission scanning electron microscope equipped with a Bruker XFlash 60 energy dispersive spectroscopy (EDS) detector. The mineral distribution map was generated using Maps and Nanomin software [28]. The calculation method for mineral composition includes collecting BSE and X-ray signals at each point and comparing them with recipes from the database to automatically identify the minerals [29].

3. Results

The bulk sample and two size fractions exhibit distinct spectral characteristics near ∼3 μm, corresponding to the fundamental OH and H_2O absorptions (Fig. 2). While the bulk soil and 45–355 μm size fractions display weak 3 μm absorptions, the fine soil fraction (<45 μm) shows a more pronounced absorption depth in the raw reflectance spectra. Although the spectral shapes of the Chang'e-5 samples resemble those of Apollo samples, it is inappropriate to directly compare their features near ∼3 μm due to potential contamination of the Apollo samples by atmospheric water during measurement under ambient conditions. To facilitate comparison between the band depth of absorption features near ∼3 μm of samples with different sizes, reflectance was converted to SSA using Hapke's model. The absorption strength of the fine soil fraction (<45 μm) is approximately 1.5 times stronger than that of the bulk and coarse soils. Notably, the peak (reflectance minimum) of the OH/H_2O absorption in the Chang'e-5 samples occurred near 2.8 μm, with the wavelength calibration error of the LMS being less than 1 nm [30].

The positions of the water absorption shoulders are typically near 2.7 μm. A noticeable shift in the "downturn" position of the water absorption near 2.7 μm was observed in the *in-situ* data, approximately 50 nm towards longer wavelengths compared to the laboratory spectra of Apollo and Chang'e-5 samples (Fig. 2a). Laboratory measurements of lunar samples indicate that the "downturn" position of the water absorption near 2.7 μm is 2.6824 ± 0.0548 μm [19]. A similar shift has been observed in the Cassini flyby data of the Moon [14]. However, this shift is not observed in the M^3 and Deep Impact data [12,15]. This discrepancy cannot be attributed to differences in instrumentation because the spectrometer and samples used for Chang'e-5 *in-situ* and laboratory measurements were the same. A systematic wavelength shift may be caused by temperature variations in the spectrometer. The temperature of the spectrometer can increase from room temperature to 60 ℃ on the lunar surface. The Chang'e-5 mission employed an acousto-optic tunable filter (AOTF) made of TeO_2 as its spectroscopic device [30]. However, changes in environmental temperature can cause the diffraction wavelength of the AOTF to drift, resulting in a shift in the output of the spectrometer over time [31–33]. This disparity has little effect on water content estimation, as it only involves a small shift in absorption position.

Water content was determined from absorption features near ∼3 μm. The spectral parameter ESPAT near ∼3 μm exhibits a linear correlation with absolute water content, based on laboratory studies on lunar-relevant materials including water-bearing volcanic glasses and anorthosite [20]. The slope of the ESPAT-wt.% H_2O trend is a function of particle size due to differences in optical path length [13,20]. Smaller particles show steeper ESPAT-wt.% H_2O trends. The mean particle size of bulk soil (<1 mm) from the

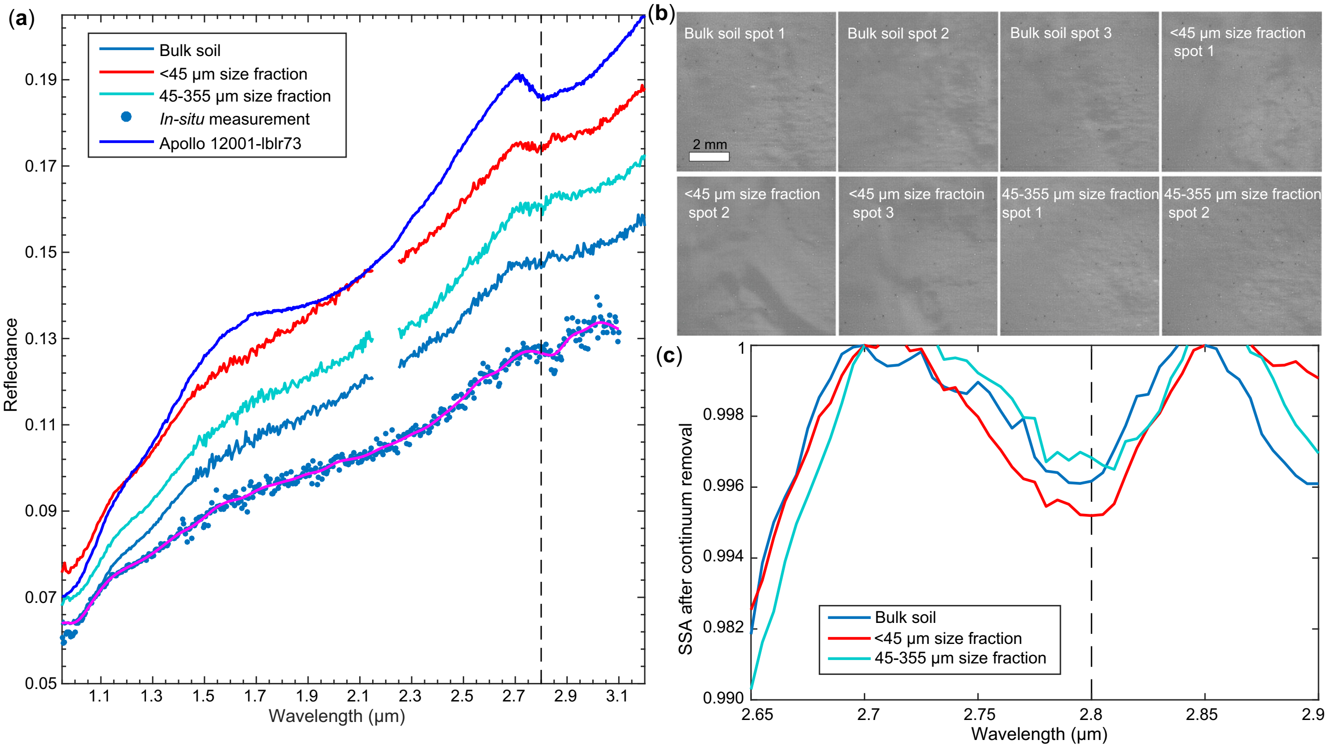

Fig. 2. Spectral measurements of Chang'e-5 soil samples in the laboratory. (a) Reflectance spectra of lunar soil samples measured using a spare flight model of the Chang'e-5 Lunar Mineralogical Spectrometer (LMS). Data between 2.15 and 2.24 μm were excluded due to a low signal-to-noise ratio caused by the connection of two detectors of the LMS. For comparison, spectra of *in-situ* measurements of lunar regolith at the Chang'e-5 sampling site and the spectrum of Apollo sample 12001 were included. (b) The images of the observed samples. (c) The single-scattering albedo spectra after continuum removal. The dashed line indicates the wavelength of 2.8 μm.

Chang'e-5 lunar samples is approximately 50 μm [25]. The Chang'e-5 sample's particle size distribution yields mean particle sizes of approximately 16 μm for the < 45 μm soil fraction and 114 μm for the 45–355 μm fraction [25]. Laboratory experiments with lunar analogs provide coefficients for calculating water content from bulk soil, <45 μm soil, and 45–355 μm soil as 9700, 18000, and 3400 [20,26], respectively. The derived water content of the fine fraction can reach 87 ppm. It should be noted that some of the finest particles (e.g., <5 μm) may be lost during the sieving process, potentially resulting in a slight underestimation of the water content of the <45 μm samples. The water contents in the bulk soil and 45–355 μm soil are approximately 37 ppm and 11 ppm, respectively. According to the size distribution of Chang'e-5 bulk soil samples [25,34], the <45 μm fraction accounted for about 40 wt.%, and the 45–355 μm fraction accounted for about 56 wt.%. Thus, the bulk soil contains an estimated 41 ppm of water after weighting, consistent with our direct spectroscopic measurements (~37 ppm), confirming the validity of the spectral analyses. The results indicate that water content in lunar regolith is strongly dependent on particle size (Fig. 3a).

4. Discussion and conclusion

4.1. Particle size-dependent water content

Variations in porosity among samples with different particle sizes may influence spectrum absorption. Different porosity values ranging from 0.1 to 0.8 were utilized in calculating the single-scattering albedo to assess the impact of porosity on the results. The investigation reveals that changes in water content induced by porosity variations (twice the standard deviations) are approximately 4.8 ppm for the bulk sample, 11.9 ppm for the <45 μm soil sample, and 1.5 ppm for the 45–355 μm soil sample (Fig. 3a). The alterations induced by the uncertain porosity, in relation to the water content, are relatively minor.

The fine fraction of Chang'e-5 samples contained more water compared with the coarse fraction, as seen in Fig. 3a. The mineral compositions of the <45 μm and 45–355 μm fractions are similar (Fig. S4 online), indicating that differences in composition did not explain the variation in water content with particle size. Instead, it seems that how water is distributed within the lunar regolith

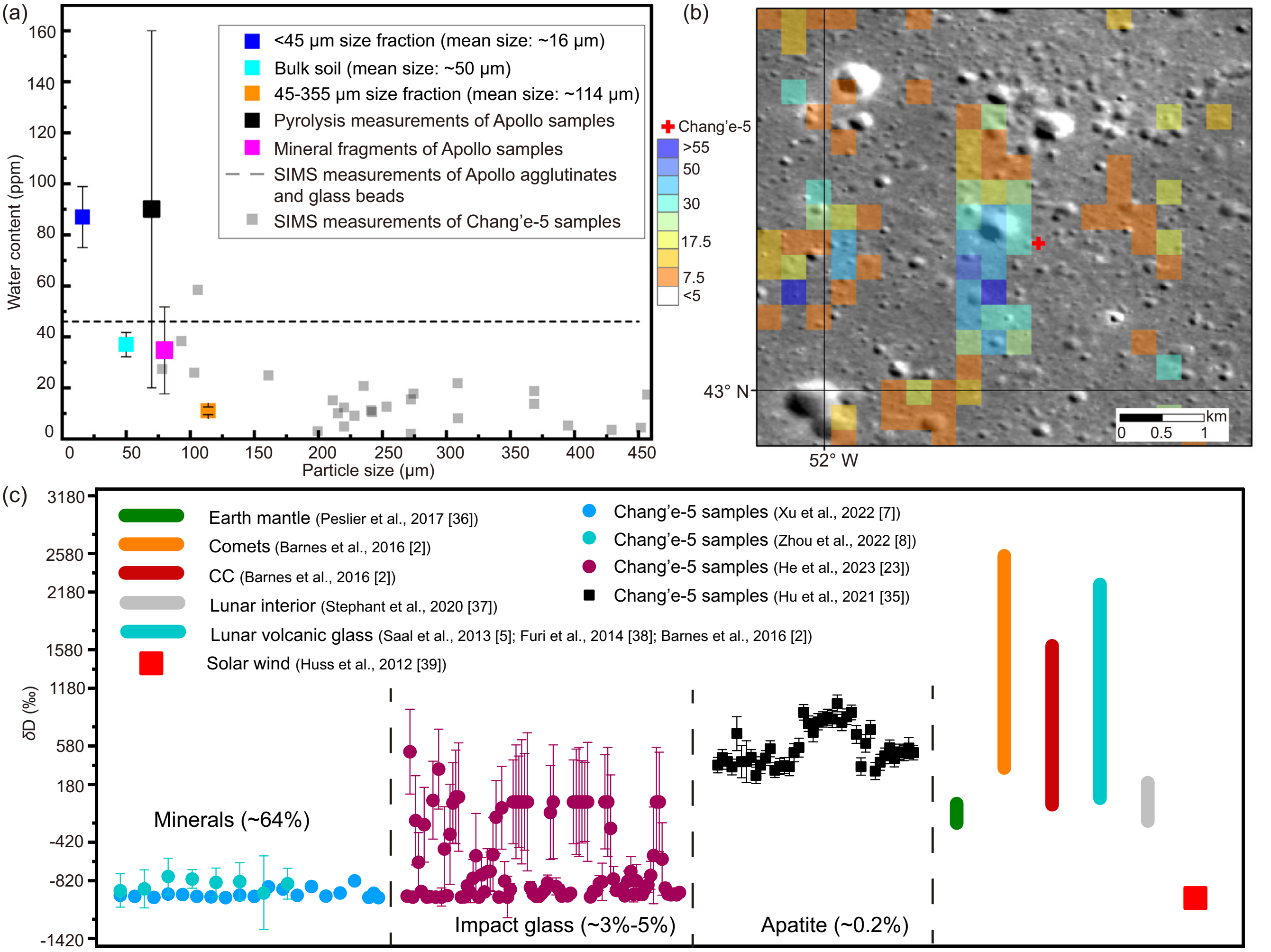

Fig. 3. Water content derived from spectra of the Chang'e-5 samples. (a) The relationship between the water content and particle size (mean size of each fraction). The dashed line and the gray square represent the water content estimated from SIMS (secondary ion mass spectrometer) measurements of Chang'e-5 soil particles [7,8] and Apollo agglutinates and glass beads [6]. (b) Water mapping at the Chang'e-5 landing area derived from the Moon Mineralogy Mapper [20]. (c) The hydrogen isotope compositions of the Chang'e-5 soil samples, indicate, upon comparison with various sources [2,7,8,23,35–39], that a majority of the particles suggest a solar wind origin.

grains provides clues about its origin. Theoretically, if the entire soil particle were saturated with water, the water absorption strength in larger particles should be stronger than in smaller particles (Fig. S5 online). However, the stronger water absorption in finer Chang'e-5 samples suggests that water content depends on the surface volume ratio, indicating a surface correlation. In the lunar regolith, solar wind hydrogen implants at consistent depths across particles of various sizes, typically reaching a peak around 30 nm and penetrating to a maximum depth of approximately 200 nm [7,8]. Beyond this depth, the presence of water is minimal, although there may be some diffusion of solar wind water into the inner regions of particles [23]. Consequently, larger particles tend to contain a lower relative proportion of water compared to smaller particles, manifesting a surface correlation phenomenon. This observation aligns with the trends observed in other solar wind implanted species found in Apollo samples [40]. Thus, the water detected in the Chang'e-5 soil samples can be primarily attributed to solar wind implantation, as supported by the hydrogen isotope compositions of the Chang'e-5 soil particles [7,8,23]. The majority of soil particles exhibit low δD values, indicative of a solar wind origin (Fig. 3c). Additionally, measurements of the Chang'e-5 mare basalts indicate a low water content with only 7 ± 3 ppm, as determined from analyses of apatites and ilmenite-hosted melt inclusions [35]. The potential contribution from Earth wind cannot be ruled out, given the Moon's exposure to Earth's magnetotail during approximately one-third of its daytime, subjecting to Earth wind [9,10]. Further investigations into Earth wind effects are warranted in the future studies.

The average water content in the outermost rims (0–100 nm) of lunar soil particles, as determined from NanoSIMS measurements, is approximately 6500 ppm, expressed as a form of H_2O [7,8]. Utilizing the size distribution data of Apollo regolith samples, an estimated 46 ppm of water content was derived for the bulk lunar regolith [7]. Nevertheless, it is important to note that the bulk soil analyzed in this study consists only of fractions smaller than 1 mm [25]. When extrapolating this water content to the bulk soil, it equates to roughly 115 ppm, accounting for the size distribution of Chang'e-5 bulk soil. This value is approximately three times higher than the content determined from reflectance measurements. This variance could stem from the specific form in which hydrogen manifests. It is deduced that approximately one-third of the hydrogen from solar wind, which was implanted into the particles, likely bonded with oxygen vacancies to form OH/H_2O compounds. These hydrogen bonds may become entrapped in the rims of regolith grains and within the impact melt found in agglutinate, assuming the influence of the impact process is disregarded. Future atomic measurements of lunar samples will provide additional insights into the nature of hydrogen species.

4.2. Implication for in-situ and orbital remote sensing spectroscopy

The absence of thermal interference in the spectra of the returned samples, analyzed under laboratory conditions, establishes a crucial baseline for *in-situ* and remote sensing observations. Accurately estimating water content on the lunar surface via remote sensing spectroscopy presents challenges due to the high surface temperature, which can obscure water-related features [19,41]. Various methods utilized to rectify thermal influences in orbital spectral observations have yielded contradictory findings regarding the overall water content, its spatial distribution across the lunar surface, and diurnal variation [18–20,42]. Although an empirical thermal correction method [19] employed to correct M^3 data [20] was tested with the Yutu-2 rover [43], the limited spectral range of the rover's spectrometer restricts its capacity to capture water-related information from the lunar surface. The water content at the Chang'e-5 landing site, derived from the M^3 spectral data, thermally corrected using the empirical method proposed by Li and Milliken [19], is about 30 ± 6 ppm (as shown in Fig. 3b). The *in-situ* spectral observations of the identical region in the returned samples significantly enhance the value of the Chang'e-5 mission. Despite the disturbance of the Chang'e-5 sampling site by the rocket plume, resulting in an uneven distribution of water over an approximately 2 m × 2 m area [21], the average water content obtained from *in-situ* spectral measurements of the regolith can effectively represent the water characteristics of the sampling site. The mean water content, based on *in-situ* measurements of regolith at seven locations within the Chang'e-5 sampling site, is approximately 32 ± 6.4 ppm [21]. The consistency between the *in-situ* measurements (approximately 2 m area) and orbital measurements (approximately 280 m per pixel) indicates that the water around the Chang'e-5 landing site is homogeneous. Therefore, the Chang'e-5 soil samples are suitable representatives for studying the water in the landing area, situated in the middle latitudes of the Moon.

Both orbital and *in-situ* spectra are susceptible to thermal radiation from the lunar surface, which can cause variations in the absorption strength near 3 μm when different thermal correction algorithms are applied. The water estimation from the spectral measurement of the Chang'e-5 bulk soil samples (~37 ppm) in the laboratory aligns with the *in-situ* and orbital remote sensing detections, indicating the effectiveness of the thermal correction model applied to the M^3 data and *in-situ* Chang'e-5 reflectance spectra [20,21]. Consequently, the results of this study support the existence of latitudinal and diurnal variations in the 3 μm absorption strength on the lunar surface, as inferred from the global mapping of water content using M^3 spectra [20]. The upcoming Chang'e-6 sample-return mission is poised to investigate the Apollo basin within the South Pole-Aitken (SPA) basin on the lunar far side [44], utilizing the same spectrometer as that was used in the Chang'e-5 mission [30]. The Chang'e-7 mission will investigate lunar surface water with high spatial resolution (tens of meters) using a Wide Band Infrared Spectrum Mineral Imaging Analyzer mounted on the orbiter [45]. These joint analyses, involving *in-situ* observations (e.g., Chang'e-5 LMS data), orbital remote sensing data (M^3), and lunar soil samples, have explicitly demonstrated the validity of the thermal correction model for remotely acquired reflectance spectra from the lunar surface and the algorithm for deriving absolute water content from the reflectance spectra near 3 μm. These models can be used to process similar spectral data that will be acquired by the upcoming Chang'e-6 and Chang'e-7 missions.

4.3. Implication for lunar resource utilization

The water content likely exceeds 37 ppm (derived from spectra of bulk soil sample) beyond the 43° latitude, i.e., the Chang'e-5 landing site. If the finer fraction than 45 μm can be separated from lunar soil *in-situ*, approximately 108 kg of water can be extracted from 1000 m^3 soil, assuming the bulk density of lunar soil is 1.24 g/cm^3 [25]. Moreover, the water extraction could be significantly more efficient if < 10 μm size separation (mean particle size is ~6 μm) can be conducted *in-situ*, yielding 840 kg from 1000 m^3 of the dust material. In the next decades, a number of countries released plans for human landings on the Moon and lunar research stations [46,47]. The lunar south pole stands out as a prime target location for landing due to the presence of water ice in the Permanently Shadowed Regions (PSRs) [48] and at the rim of the ancient and largest impact basin SPA. Despite the challenging conditions including complex topography, poor illumination, and extremely low temperatures, the current focus lies on utilizing water ice in the PSRs. The findings of this study suggest that fine particles are water-rich, indicating that size sorting of lunar regolith in the mid- to low-latitudes regions could also be a potential method for utilizing water resources in future human activities on the Moon, provided that technical challenges such as low gravity and the development of automated sieve and collection devices can be addressed. This study highlights the broader potential for utilizing lunar water resources.

Conflict of interest

The authors declare that they have no conflict of interest.

Acknowledgments

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Author contributions

Honglei Lin contributed to the conceptualization, experiments, methodology, investigation, visualization and paper writing. Rui Xu contributed to the experiments, methodology and paper review. Shuai Li contributed to methodology, investigation and paper review. Rui Chang contributed to experiments and paper review. Hejiu Hui, Yang Liu, Hengci Tian, Kai Fan, Zhiping He, and Huaiyu He contributed to paper review and editing. Wei Yang contributed to conceptualization, experiments, supervision and paper review; Yangting Lin and Yong Wei contributed to the conceptualization, supervision, paper review and editing.

Appendix A. Supplementary materials

Supplementary materials to this article can be found online at https://doi.org/10.1016/j.scib.2024.05.031.

Data availability

All data are available in the main text or the supplementary information. The reflectance spectra of the Chang'e-5 samples are provided in Table S1.

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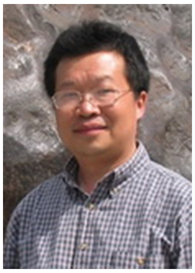

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