Research Article

Using Synchrotron-Based X-Ray Microcomputed Tomography to Characterize Water Distribution in Compacted Soils

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Water plays a vital role on the hydromechanical behavior of unsaturated soils. An important concern in unsaturated soil mechanics is to determine the distribution of water within voids and its interaction with soil grains. This paper presents some results of the spatial distribution of water in different soils using the synchrotron-based X-ray microcomputed tomography (μ -CT). Three materials (glass beads, natural sand, and clay) were first prepared at a water content of about 10%, statically compacted under vertical total stress of 500 kPa, and then scanned by synchrotron X-rays at an energy of 18 or 20 keV. The three-dimensional (3D) microstructure of the samples including air, liquid, and solid phases was reconstructed, and some new observations were obtained: (i) the iodine-based contrast medium (KI) can increase the peak greyscale value of water from 110 to 122, enhance the air-water contrast, and thus facilitate the segmentation of water phase; (ii) in the compacted glass beads and sand, water distribution is characterized using the μ -CT and image reconstruction technique. The water contents obtained by phase segmentation, i.e., 10.2% and 9.3%, are comparable with those measured by the oven-drying method, i.e., 9.7% and 9.4% for the glass beads and sand, respectively; (iii) water is preferably distributed within aggregates when it is mixed with the oven-dried particles, and an aggregate-dominated 3D structure is observed. However, it is impossible to determine the water phase for the studied material even with the resolution of 0.65 $\mu\text{m}/\text{pixel}$.

1. Introduction

In traditional soil mechanics and engineering practice, research related to the microstructure of soils was mostly limited to two-dimensional (2D) distribution of soil particles and voids generally using scanning electron microscope and mercury intrusion porosimetry [1–3]. With the development of technology, the X-ray μ -CT has recently been proved to be effective in 3D characterization of soils, and it has been used (i) to quantify the porosity or voids [4–14], (ii) to investigate the strain localization (e.g., [15–22]), (iii) to characterize the particle breakage (e.g., [15, 23–27]), (iv) to investigate the particle morphology and orientation [28–31], and (v) to estimate the particle strains and rotations [17, 25, 32–37].

The main parameters of the X-ray μ -CT tests are presented in Table 1, showing very interesting features in terms of resolution, total acquisition time, and sample size for different materials.

As liquid phase is concerned, the application of the μ -CT to characterize the water distribution was more complex and less reported [14, 44]. When a beam of X-rays penetrates a medium, its intensity will be attenuated. At a given photon energy, the degree of attenuation (e.g., the linear attenuation coefficient) is normally related to the atomic number of the medium; the larger the atomic number is, the larger the linear attenuation coefficient will be. As multiphase unsaturated soil is concerned, Figure 1(a) shows the linear attenuation coefficients of air, water, and solid (e.g., quartz)

TABLE 1: Main parameters of recent X-ray μ -CT tests on soils in the literature.

X-ray sources	Detector		Field of view (mm)	Exposure time (s)	Projection		Sample size		Reference
	Pixel size (μm)	No. of pixels			No. of projections	Total acquisition time (min)	D (mm)	H (mm)	
X-ray tube	11.2	2316 $\times$ 2316	25.9 $\times$ 25.9	2	1000	/	10	23	[8]
	75.7		77.5 $\times$ 77.5	0.5	1200	20			
	7.6	1024 $\times$ 1024	7.8 $\times$ 7.8	2.4	4800	240	35	70	[19]
	4.2		4.3 $\times$ 4.3		4800				
	9	4008 $\times$ 2672	36 $\times$ 24	0.2	1440	90	50	10	[12]
	127	1920 $\times$ 1526	244 $\times$ 194						
Synchrotron radiation	3.3	2000 $\times$ 2000	6.6 $\times$ 6.6	/	/	90	/	/	[27]
	14	2048 $\times$ 2048	28.7 $\times$ 28.7	0.08		5	10	20	
		1024 $\times$ 1024	14.3 $\times$ 14.3	0.04	1200	2.5	20	40	[38]
	14	/	/	/	/	12	11	22	[17]
	2.2	4008 $\times$ 2672	8.8 $\times$ 5.9	2	3600	120	/	/	[39]
	7.5	2680 $\times$ 1300	20.1 $\times$ 9.75	1.8–2.2	/	180–240	8	4	[40]
	4.4	/	/	/	1440	60–90	3 $\times$ 4 $\times$ 5		[41]
	0.33	/	0.8 $\times$ 0.6	0.09	1501	/	0.5	/	[42]
	0.5	/	/	0.3	1500	/	<1	<1	[43]

(a)

(b)

(c)

FIGURE 1: Linear attenuation coefficient of different phases in soil and the corresponding greyscale histogram. (a) Linear attenuation coefficients of air, water, quartz, and potassium iodide at X-ray energies from 1 to 1000 keV (data from [45] and the online FFAST database of the National Institute of Standards and Technology, USA). (b) Greyscale histogram of an undisturbed loess (from [46]). (c) Greyscale histogram of a compacted sand (from [9]).

versus the increasing X-ray energy. Due to the difference in linear attenuation coefficient of different phases, the soil grains usually seem “darker” than air in the CT projections,

whereas they feature a “lighter” color in the reconstructed CT slices. The degree of such brightness can be quantitatively reflected by the histogram of the greyscale value of the slice,

as presented in Figure 1(b) (from [46]). Based on the greyscale histogram, water is detectable and can be directly segmented using its distinctive peak greyscale value. In some cases, however, the situation may be different and complex; for example, the overlap in greyscale value between air and water phases was reported in [9], where the peaks of air and water in the greyscale histogram are completely merged, and it is impossible to simply separate the two phases (Figure 1(c)). To solve this problem, effort has been made either to enhance the air-water contrast by adding iodine-based or chlorine-based contrast medium in water (e.g., [14, 47, 48] or to directly capture water phase using neutron radiography [8, 9, 49, 50]).

In this paper, synchrotron-based X-ray μ -CT tests were performed on three statically compacted soils, e.g., glass beads, natural sand, and clay, from engineering site. Water distribution and morphology within typical compacted soils were determined and compared. The main contributions consist of the following:

- (i) Quantitatively demonstrating the effect of the contrast medium (i.e., KI in this study) on the greyscale value of the liquid phase
- (ii) Extending the idealized material (e.g., glass beads or sand in the literature) to the “real” soil (e.g., clay from the engineering site) and highlighting some difficulties in characterizing water distribution within clayey soil.

2. Materials and Methods

Three materials, namely, BioSpec glass bead, Zhuhai sand, and Guangzhou clay, were selected. BioSpec glass bead is a commercially available product from BioSpec Products Inc. (USA), with a diameter of about 0.2 mm. Zhuhai sand is a local material of Zhuhai, located alongside the South China Sea. Guangzhou clay was extracted from a slope of the Guangzhou metro line No. 21. It is a typical tropical laterite that is widely distributed in the south of China. The grain size distributions of the materials are presented in Figure 2.

The materials were first put into an oven at a temperature of 105°C for two days. Then, 500 g of the dried soil grains was carefully mixed with (i) 50 g deionized water or (ii) 60 g 20% (weight) KI solution, aiming to reach a target water content of 10%. Due to the high solubility in water and large relative contrast value [51], KI was used in this study to enhance the phase contrast. The 10% moist soils were sealed in an aluminum box for about two days to arrive at equilibrium, then taken out, and statically compacted in a 5 mm diameter stainless steel tube by applying a vertical stress of 500 kPa. After compaction, the tube was sealed with silica gel at each end and fixed on the magnetic base that can be firmly attached to the stainless-steel rotation stage (Figure 3).

X-rays were generated in the beam-line BL13W1 of Shanghai Synchrotron Radiation Facility (SSRF). Compared with the X-rays produced in X-ray tubes (e.g., for industrial and medical applications), the synchrotron-based X-rays are monochromatic (identical photon energy) and have a much higher brilliance. This can avoid beam hardening effect,

FIGURE 2: Grain size distribution of the studied materials.

reduce artefacts, and thus significantly improve the quality of the reconstructed images. The optical transform system (Optique Peter PCO2000) provides multiobjectives, with magnification ranging from 1.25 to 20 times the original size. The charge coupled device (CCD) detector (Hamamatsu ORCA-Flash4.0) has a field of view of 13.0 × 13.0 mm with 2048 × 2048 pixels, corresponding to the pixel size of 6.5 μ m in each direction. In the study, samples of BioSpec glass beads and Zhuhai sand were scanned using the 2X objective lens, whereas in the case of Guangzhou clay, samples were firstly scanned with the 2X objective lens and then with the 10X objective. The applied photon energy is 18 keV for the BioSpec glass beads and 20 keV for the rest. 1200 projections were recorded while the sample was rotated 180°. The whole scanning process took about 30 and 150 minutes for the 2X and 10X objectives, respectively (Table 2).

After CT scan, the 1200 projections (about 5 Gb) were imported into the open-source phase retrieval software PITRE [52]. PITRE provides algorithms to reconstruct the soil structures and outputs 1200 or 2000 slices (greyscale images) containing the greyscale information of each phase of the soil. 3D structure was reconstructed by stacking the slices in the commercial software Avizo Fire 8. Further analysis such as phase segmentation and volume calculation was realized based on the specific greyscale value of air, water, and soil grains in the reconstructed images (Figure 4).

3. Results and Discussion

3.1. Effect of Contrast Media on X-Ray Imaging. Figures 5(a) and 5(b) present the slice (no. 504) and greyscale histogram of the Zhuhai sand prepared with deionized water. In the slice image (enlarged view), air, water, and soil grains are qualitatively distinguishable, whereas in the corresponding histogram, the peaks of air and water phases are completely merged, the same as that reported in [9].

FIGURE 3: Experimental setup of the μ -CT at the BL13W1 SSRF.

TABLE 2: Tests performed in this study.

Materials	Sample size		CT test					Sample reconstruction
	Diameter (mm)	Photon energy (keV)	Objective lens	Pixel size (μm)	No. of pixels	Field of view (mm)	Acquisition time (min)	No. of slices
BioSpec glass beads	5	18	2X	3.25	2048×2048	6.7×6.7	30	1200
Zhuhai sand	5	20	2X	3.25	2048×2048	6.7×6.7	30	1200
Guangzhou clay	5	20	2X	3.25	2048×2048	6.7×6.7	30	1200
	1	20	10X	0.65	2048×2048	1.3×1.3	150	2000

FIGURE 4: Phase retrieval, slice creation, and 3D reconstruction of a soil sample.

When contrast medium (KI) is added, no significant improvement of the air-water contrast is observed from the slice (no. 360) in Figure 5(c). However, in the greyscale

histogram, the greyscale value of the water phase slightly increases from 110 to 122, and as a result, the peaks of air and water phases become clearer and separated (Figure 5(d)).

FIGURE 5: Slice image and greyscale histogram of the compacted Zhuhai sand. (a) Slice image and (b) histogram of the sand mixed with deionized water. (c) Slice image and (d) histogram of the sand mixed with 20% KI solution.

FIGURE 6: Continued.

(c)

(d)

FIGURE 6: 3D reconstruction and water distribution within the compacted BioSpec glass beads. (a) 3D reconstruction. (b) Enlarged view. (c) Segmented water phase. (d) Profile of water content and degree of saturation.

TABLE 3: Water content determined by different methods.

Water content (%)	Glass beads	Zhuhai sand	Guangzhou clay
Target	10	10	10
Oven-dried	9.7	9.4	9.6
Phase segmented	10.2	9.3	/

(a)

(b)

FIGURE 7: Continued.

FIGURE 7: 3D reconstruction and water distribution within the compacted Zhuhai sand. (a) 3D reconstruction. (b) Enlarged view. (c) Segmented water phase. (d) Profile of water content and degree of saturation.

The above results quantitatively show the capability of KI to enhance the air-water contrast, which contributes to (semi) automatic segmentation of water. Qualitative results on other contrast media such as CsCl were also reported (e.g., [14, 48]). In addition to the above iodine-based or chlorine-based contrast media, Van Loo et al. [51] reported a total of 50 other contrast media and their ability to enhance the X-rays imaging of water. Note that, as the addition of contrast medium changes the contact angle of water, it is necessary to consider such an effect when new contrast medium is considered. In this study, 20% KI has negligible influence on the contact angle as the capillary rise of KI solution was found to be almost the same as that of deionized water.

3.2. Water Distribution in Compacted Glass Beads and Sand.

Figure 6 shows the reconstructed 3D structure of the compacted BioSpec glass beads. In Figure 6(a), water phase was segmented and highlighted in green color based on its distinctive greyscale values ranging from 109 to 138. An enlarged view is presented in Figure 6(b) to show the detailed information in particular the water bridge between grains. The general picture of the water morphology (Figure 6(c)) reflects the complexity and heterogeneity of the structure of water within the sample. Quantitatively, the gravimetric water content (calculated with the number of pixels of air, water, and grains) varies from 8.8 to 12.7% alongside the sample height direction (Figure 6(d)). The degree of saturation deviates about $\pm 5\%$ from the mean value, 21.2%. The mean value of the water content is 10.2%, which is a bit larger than the target water content and that measured by oven-drying (Table 3). The main reason for such high water content probably results from the effect of adding KI on the quantification of water. In fact, to prepare the sample with a target gravimetric water content of 10%, 60 g 20% (weight) KI solution was mixed with 500 g glass

FIGURE 8: Formation of aggregates after mixing water with oven-dried clay particles.

beads. The volume of liquid phase (i.e., KI solution) is about 27.65% of that of the soil grains (glass beads) by considering the densities of 20% KI solution and glass beads to be 1.15 and 2.65 g/cm³, respectively. Assuming another sample of gravimetric water content of 10% by mixing 50 g deionized water with 500 g soil grains, the ratio of the water volume to grain volume is 26.5%, showing that adding the contrast medium into soil, as presented in this study, will 1.15% overestimate the quantity of segmented liquid phase.

Figure 7 presents the 3D structure of the Zhuhai sand. In the compacted sample (Figure 7(b)), water is preferably distributed within aggregates that mainly consist of small

FIGURE 9: Aggregates-dominated structure of the compacted Guangzhou clay scanned with two different objective lens. (a) 3D reconstruction with the 2X objective lens (3.25 m/pixel). (b) Slice image with the 2X objective lens. (c) 3D reconstruction with the 10X objective lens (0.65 m/pixel). (d) Slice image with the 10X objective lens.

particles with diameters ranging from several to several dozens of micrometers. In addition, water bridges with several dozens of micrometers in thickness are also observed between sand grains. As concerns the general structure of water (Figure 7(c)), the radius of the water bridge is larger than that of the BioSpec glass beads because of the larger grain size for the Zhuhai sand. Water distribution is presented in Figure 7(d), with gravimetric water contents varying from 6.6 to 13.9% and degrees of saturation from 10.0 to 19.9%. The mean value of the water content, 9.3%, is slightly smaller than the oven-drying value, i.e., 9.4% (Table 3).

From the above, it can be concluded that the synchrotron-based μ -CT is able to well characterize the 3D structure of granular materials such as glass beads and sand.

At the difference of the scanning electron microscope and Mercury intrusion porosimetry that require a delicate freeze drying step, moist samples can be directly scanned and water distribution can be determined both qualitatively and quantitatively. However, to ensure a satisfactory quality of the reconstructed image, any micromovement of soil grains and water phase should be avoided as much as possible during the rotation of the sample.

3.3. Water Distribution in the Compacted Clay. When preparing the Guangzhou clay sample, aggregates of several millimeters formed while water was added and mixed with the oven-dried clay particles (Figure 8). Water content of

aggregates was determined, and its value (11.7%) is 1.8% larger than the average value of the whole sample. 3D structure of the compacted sample, as presented in Figure 9(a), shows an aggregate-dominated skeleton structure. In the slice (Figure 9(b)), aggregates are clearly distinguishable due to the different greyscale values that result from the relatively larger water content. Under the effect of compaction effort, the size of aggregates decreases to several hundreds of micrometers in diameter. With a larger magnification, the micron-scale structure of the compacted sample is presented in Figures 9(c) and 9(d). The shape of a single aggregate is outlined by the dashed line in YZ and XY planes. It is found that the air-pores within aggregates are compressed to less than 10 micrometers. In these figures, however, it is still impossible to clearly observe the thin water films or water bridges within and outside aggregates.

A possible explanation of the above results is that the initial water content of 10% is too small for the clayey material to form water bridges that can be detected with a resolution of $0.65\ \mu\text{m}$. In fact, water absorption ability of the clays is much higher than that of granular material like glass or sand. The target water content of 10% during sample preparation should be mostly absorbed by voids between the platelets (i.e., within soil aggregates), and there should be very limited water remaining in the voids between the soil aggregates. To clearly detect the water phase within the soil aggregates, a higher resolution (e.g., in the scale of nanometer) is required.

4. Conclusions

Synchrotron-based μ -CT tests were performed in order to determine the water distribution in different statically compacted materials. The results show the following:

- (i) The addition of 20% in weight of iodine-based contrast medium (i.e., KI) can increase the grey-scale value of the liquid from 110 to 122, enhance the air-water contrast and, as a result, contribute to (semi)automatic segmentation of water phase.
- (ii) In granular materials such as glass beads and sand, water distribution and water morphology are well determined, showing good capability of the μ -CT to investigate the 3D structure of these materials.
- (iii) In the case of the clayey soil, aggregates formed when water was mixed with the oven-dried clay particles and the water content within aggregates is larger than the average value of the whole sample; an aggregate-dominated 3D structure is observed for the compacted Guangzhou clay; in this study, it is impossible to clearly observe the thin water films within aggregates with the resolution of $0.65\ \mu\text{m}/\text{pixel}$ even though the micron-scale structure of soil grains and voids are distinguishable.

Data Availability

The micro-CT data used to support the findings of this study have been deposited in Professor Lian-Sheng Tang's repository.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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