HIGH RESOLUTION IMAGING OF POROUS GEOMATERIALS AND GEOLOGICAL CORES WITH MICRO COMPUTED X-RAY TOMOGRAPHY

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ABSTRACT

High resolution X-ray tomography results of dry and partially water saturated silica sand specimens compacted to target density are presented. The concept of multi-resolution/FOV is demonstrated by using two different magnifications (1X and 10X) on the same specimen. This resulted in high contrast of the water phase compared to the silica and air phases without using a contrast agent, thus highly desirable for studying samples from the environment. Direct Numerical Simulations (DNS) using the tomography data was used to predict capillary pressure versus saturation/suction curve using full morphology method. An estimation of the capillary pressure of the partially saturated compacted sand specimen used in the tomography measurements from DNS is also included.

INTRODUCTION

Geo-materials (sand, silt, clays, and rock cores) are natural materials with complex properties resulting from three-phases composed of solid (silica), gas (air), and liquid (water and/or oil) phases. The solid phase is composed of discrete particles (sand or silt grains or clay platelets), and the interactions between solid particles affect the deformation behavior of the soil mass. The geometric arrangement of the three phases can significantly affect the total porosity, pore size distribution, fluid transport, and reservoir/storage capacity of target liquids and gases. The distribution of the liquid phase in partially saturated geo-materials also strongly affects its shear strength and permeability/hydraulic conductivity. Understanding fluid transport properties in geological samples is of increasing importance in recent years for applications such as carbon sequestration, understanding Fracking effects on ground water, transport of contaminants through ground water, and for optimizing the hydraulic fracturing schemes for oil recovery.

X-ray micro computed tomography techniques have previously been explored for imaging geo-materials [1, 2]. However, the low attenuation of water relative to the rock's silica phase typically produces low contrast in which water and air are difficult to distinguish using conventional X-ray imaging. It is necessary to add a contrast agent to the water in order to distinguish water phase more precisely [3]. However, the addition of contrast agent will affect the contact behavior of the liquid and solid phases, thus affecting the measured transport properties not representative of in situ conditions. Recently, the first two authors of this paper have performed a dual modality (neutron and X-ray) imaging to overcome the limitation of visualizing low contrast phase of water in compacted silica sand specimen using X-rays [4]. The microstructures of geo-materials obtained from X-ray tomography have also been used to perform fluid flow simulation [5, 6]. X-ray tomography can provide actual microstructure of the pore geometry, and pore scale simulation can thus be performed using such data sets.

In this paper, we demonstrate the results of using a novel laboratory X-ray microscopy platform, which uses a unique high dynamic range detector system, capable of imaging unstained water, air, silica sand in a custom specimen holder specifically designed to compact the granular material at target bulk density to mimic natural system. Very detailed quantitative information on the three phases was obtained from X-ray tomography in three dimensions. Direct numerical simulations (DNS) using actual microstructure obtained from reconstructed tomography data was used to generate capillary pressure – saturation curves. High resolution three-phase tomography imaging data can be used to predict fluid flow properties of partially saturated system. This approach shows tremendous potential to offer new insight on developing next generation predictive property based geo-materials and advanced material characterization tools for applications such as carbon sequestration, and understanding the deformation behavior of granular materials, heat transfer through porous systems, and related fluid flow behavior.

PROCEDURE

Specimen Description

A compacted dry Ottawa sand (20/40 Oil Frac, 99.98% silica phase) and a partially saturated specimens were prepared under controlled conditions using a custom designed mold suitable for tomography and known compaction effort. The compaction mold had inner dimension of approximately 10.21 mm dia. × 10.21 mm height as shown in Figure 1(a). Dry or partially saturated sand (12% gravimetric water content) are compacted in the mold using three compaction layers. The dry or wet sand (mixed to 12% water content) was placed in the compaction mold to about 1/3 height and compacted by dropping a tamping rod 25 times. The process is repeated for the next two layers. The average grains size of the sand grain is approximately 700 μm.

X-ray Tomography

Xradia microXCT-400 high resolution X-ray microscope system was used to image the specimens. The system consists of a microfocus X-ray source (150kV), multiple imaging optics to switch between different resolution modes and a CCD camera (2048 × 2048). Variable sizes of lens can be selected to change the size of field of view (FOV) and spatial resolution. The system can image specimen size from 100 mm to 50 μm and spatial resolution from 50 μm to 500 nm. In this research, the 1X and 10X magnification lens were used to change the size of FOV. For the wet sand specimen, 10X magnification imaging was performed at top, middle and bottom of the specimen corresponding to top, middle and bottom compaction layers as shown in Figure1b. For the dry sand specimen, only the top area was selected form 10X magnification imaging. Imaging parameters are shown in Table 1. A good contrast of silica, air and water phase is obtained in partially saturated sand specimen. Due to a stable microfocus X-ray source and novel imaging optics, high resolution and high contrast imaging was achieved. The data was reconstructed with filtered backprojection algorithm. The reconstructed slices were first smoothened by median 3D filter, and then non-local mean filter was applied. The data was thresholded for phase quantification. The image processing was performed with Avizo (version 7.0).

RESULTS

Multi-resolution Imaging of Identical Specimen

Example tomography slices of partially saturated sand specimen are shown in Figure 1c and 1d for 1X and 10X magnification respectively. Approximately the same region inside of the red circle shown in Figure 1c is magnified in Figure 1d with much higher resolution. The water (dark gray), sand (light gray) and air (black) phases are shown very clearly with a good contrast without the use of a contrast agent. Three dimensional images of dry and wet specimen at 10X magnification are shown in Figure 2. The porosity and saturation values of the 1X magnification data obtained from image analysis are shown in Table 2. The 1X magnification image resolution (22 μm/voxel) was high enough to quantify sand grain phase with much higher size (~700 μm) relatively well, but it was not high enough to quantify the water phase below the resolution limit accurately. Some error is expected with the saturation value of the wet sand due to partial volume effects.

Direct Numerical Simulation

Full morphology model [7, 8] is used to simulate the quasi-static drainage simulation of liquid in the porous media. The stationary distribution of wetting phase (WP) and non-wetting phase (NWP) for an arbitrary capillary pressure (p_c) can be determined. The simulations presented here run the full morphology method as implemented in the

GeoDict code (www.geodict.com). Pore size is the main factor determining the drainage at a given capillary pressure. Morphological opening is used to determine the pore size distribution as shown in Eq. (1) where X represents the pore space and B is the structuring element. O_B represents the morphologically opened pores space, and it also means the pore space where the structuring element fits in.

$$O_B(X) = \bigcup \{B \mid B \subseteq X\} \tag{1}$$

A spherical structuring element with radius r is used, and a spherical interface is assumed between WP and NWP. It is assumed that one end of the dataset is connected to the WP reservoir and the other end is connected to the NWP reservoir. The pore space is assumed to be fully saturated with the WP, and the capillary pressure is zero. The pore space is eroded by spheres with r starting from the smallest radius. Based on the radius r, the capillary pressure is computed based on Young – Laplace equation as shown in Eq. (2) where θ is the contact angle between WP and solid, and γ is the surface tension between NWP and WP.

$$p_c = \frac{2\gamma \cos\theta}{r} \tag{2}$$

The pore space is filled with NWP if the erosion of the pore space has a continuous connection to the NWP reservoir. The leftover eroded set is dilated to complete the opening process. The volume fractions of NWP and WP are determined for the given capillary pressure. The process is repeated for the next structuring element with larger radius r.

An example capillary pressure – saturation curve for drainage is generated based on the microstructure of partially saturated sand (1X magnification) by using the full morphology method as shown in Figure 3. Fully wetting condition was assumed, and contact angle of water on silica was chosen as 0°. The surface tension of water was assumed to be 72.75 mN/m. Example simulated two phase distributions obtained from the full morphology simulation are shown in a, b, c of Figure 3 and compared to the actual experimental result shown in Figure 3d. Based on the simulated capillary pressure – saturation curve, the capillary pressure of the given partially saturated sand specimen is estimated as 1.35 kPa.

CONCLUSION

Multi-resolution and high contrast X-ray tomography imaging of dry and partially saturated geomaterial were presented. High contrast of water phase was obtained without using a contrast agent. Multi-resolution capability provides option to zoom into more details at the region of interest without moving the specimen or experimental equipment.

Direct numerical simulation was applied on the actual microstructure obtained from X-ray tomography, and a capillary pressure – saturation curve was obtained. Predictive measurement of capillary pressure of the partially saturated sand specimen was obtained based on the simulated capillary pressure – saturation curve.

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Table 1: High resolution X-ray imaging parameters

Magnification	Voltage (kV)	Current (W)	FOV (mm)	Voxel size (µm)	Number of projection	Exposure Time (s)
1X	70	10	14	22	1500	10
10X	80	10	2.05	2.04	3000	15

Table 2: Porosity and saturation values obtained from image analysis of 1X magnification data

	Dry 1X	Wet 1X
Porosity (%)	36.1	32.9
Saturation (%)	0	39.0

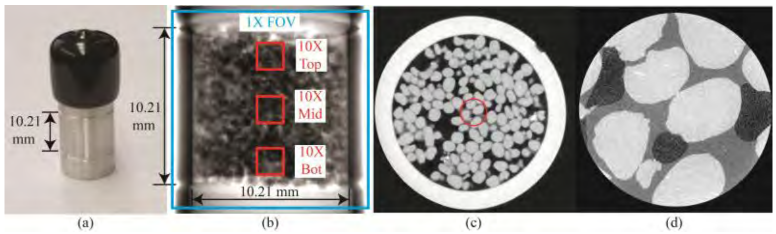

Figure 1: Picture of the partially saturated sand specimen (a), radiography of partially saturated sand specimen showing 1X and 10X magnification FOV size and location (b), tomography slice of partially saturated sand specimen at 1X magnification (c), and tomography slice of partially saturated sand specimen at 10X magnification (d)

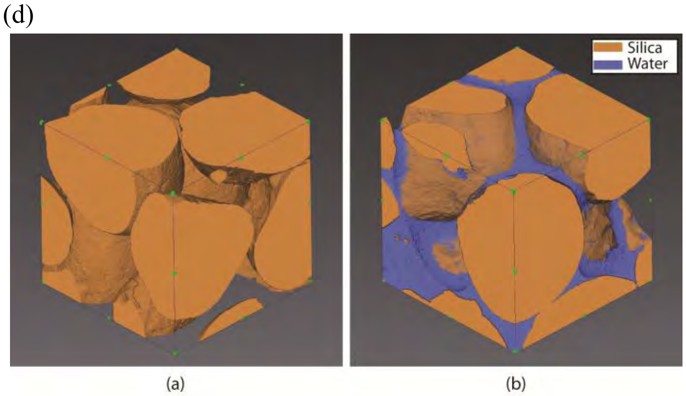

Figure 2: Three dimensional visualization (1mm^3) of dry (a) and partially saturated (b) sand specimen at 10X magnification

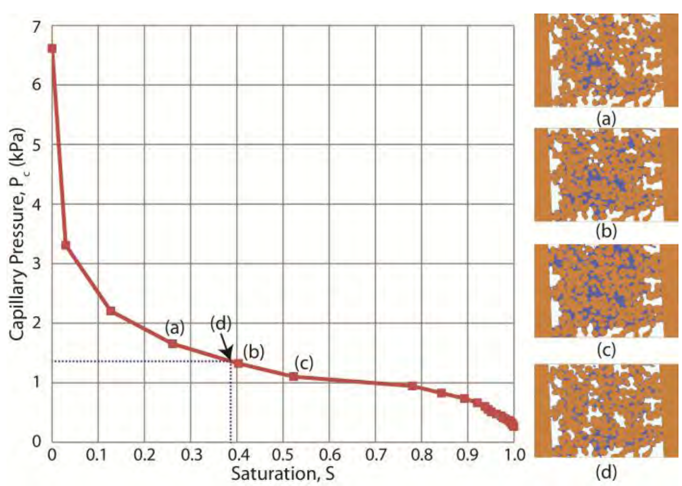

Figure 3: Simulated capillary pressure saturation curve of drainage and example two phase distributions from simulation (a, b, c) and experiment (d)