Experimental report

29/03/2022

Proposal: **1-05-53**

Council: 10/2020

Title: Combined X-ray- and neutron-tomography imaging of capillary collapse in unsaturated granular soils

Research area: Engineering

This proposal is a new proposal

Main proposer: **Nicole HUSENER**

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Local contacts: Lukas HELFEN
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Samples: Water
Glass Beads (Soda Lime)
model opencast mine dump soil (sand+lignite)
opencast mine dump soil without organics
opencast mine dump soil without fines

Instrument	Requested days	Allocated days	From	To
NEXT	3	3	26/03/2021	29/03/2021

Abstract:

Our proposal focuses on the investigation of microscopic deformations of the grain structure caused by irrigation and imbibition as well as the associated dissolution of the capillary bridges in unsaturated granular soils, known as the capillary collapse phenomenon. In order to investigate the involved microscopic processes, an opencast mine dump soil and, for comparison purposes, glass spheres with a similar grain size distribution are examined using combined X-ray- and neutron-tomography. Of particular interest is the investigation of the role of various influencing factors such as the direction of irrigation or the contained organic matter in the form of lignite. The combination of X-ray- and neutron-tomography techniques will provide experimental data with a high temporal and spatial resolution during an incremental irrigation experiment. We expect to obtain high-quality 4D-images of changes in the appearance of the water phase and the coupled particle movements as a result of the individual irrigation steps. Further image analysis is supposed to provide insights into the hydro-mechanical processes during wetting and the collapse of the grain skeleton.

Report for experiment 1-05-53: „Combined X-ray- and neutron-tomography imaging of capillary collapse in unsaturated granular soils "

Main proposer: Nicole Hüsener, Co-proposer: Marius Milatz

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Experimental date: 26/03/2021 to 29/03/2021

1 Material and methods

1.1 Experimental set-up

In experiment 1-05-53 (Hüsener et al., 2021) the capillary collapse behaviour of a sandy soil and a glass bead packing was investigated on the grain scale by means of combined neutron- and X-ray tomography using the NeXT-research facility at Institut Laue-Langevin. Due to the Corona pandemic and the resulting restrictions and precautionary measures, it was not possible to carry out the tests on-site and they were therefore controlled remotely. Originally, the investigation of several sample variations of an opencast mine dump soil as well as a glass bead packing with a grain size distribution similar to the one of the opencast mine dump soil was planned in order to be able to investigate the influence of the contained lignite on the collapse process. Unfortunately, the transport company lost the parcel with the soil samples and the experimental setup, which meant that the equipment did not arrive at the ILL in time, so other solutions had to be found spontaneously. Instead of the miniaturised setup in which a syringe pump is driven by a stepper motor and controlled with the help of a Raspberry Pi, the Nemesys pressure controller available at the ILL was used for the controlled irrigation. Using previously sent construction plans, specimen holders made of Teflon ($d_{specimen}$ = 12 mm) were produced at ILL analogous to the original experimental set-up. Teflon offers the advantage that it is less affected by the neutrons. The Laboratoire 3SR of the Université Grenoble Alpes provided Hostun sand and glass beads for the experiments. The Hostun sand had already been investigated by Bruchon et al. (2013) with regard to the capillary collapse problem and was, therefore, a good alternative, even though the influence of lignite particles on capillary collapse could not be investigated under these circumstances.

1.2 Testing procedure

A total of 5 different experiments were carried out during the allotted beamtime, 4 on Hostun sand, 1 on glass beads. A major challenge was the bubble-free connection of the saturated pressure controller to the specimen holder to ensure the controlled irrigation of the sample. In the first experiment, air remained in the channel system, so that collapse occurred by evaporation instead of wetting, as no injected water made its way into the specimen. In all other tests, the water supply succeeded, even though the combination of a highly capillary-active filter stone and the water-repellent specimen holder made of Teflon sometimes led to undesired boundary effects such as the rupture of the hydraulic contact within the specimen holder below the specimen. The quantities of sand or glass beads and water needed to prepare the specimens were first weighed in the chemistry laboratory and then thoroughly mixed together. Then the specimens were installed loosely in the specimen holder inside the scan chamber. To reduce evaporation during the experiments, the specimen holder was covered with a cap from the second experiment onwards. Before starting the wetting and after each irrigation step, the initial/current condition was captured with a combined neutron and X-ray tomography scan (duration of ca. 40 min). The changes during the injection of water were documented using 2D neutron or X-ray films. The number of irrigation steps per experiment was adapted to the presence of the local contacts at the ILL and therefore varied. Using the described experimental set-up, a voxel size of ca. 14.2 µm (FOV 1024x1024x928 px) could be achieved in the neutron tomographies and about 22.2 µm in the X-ray tomographies (experiments 1-3 FOV 640x640x2096 px, experiments 4,5 FOV 720x720x1120 px). An overview of the performed experiments can be found in table 1.

Table 1: Overview of the performed experiments

	Experiment 1	Experiment 2	Experiment 3	Experiment 4	Experiment 5
Material examined	Hostun sand	Hostun sand	Hostun sand	Glass beads	Hostun sand
Mass solid material		1,5847 g	1,592 g	1,603 g	1,5991 g
Mass water		0,1147 g	0,116 g	0,11 g	0,1168 g
No. of wetting steps	0	6	11	8	13
Target water vol. added per step	-	70 µl	35 µl	35 µl	35 µl
Additional remarks	water supply did not work because of entrapped air → specimen collapse due to evaporation	Leakage of the specimen holder observed during the experiment	Capillary bridge / hydraulic contact within the specimen holder ruptured temporarily	major collapse during the second irrigation step	Capillary bridge / hydraulic contact within the specimen holder ruptured temporarily

2 Results

2.1 Combined X-ray and neutron tomography data (reconstructed raw data)

In the 5 experiments, a lot of neutron and X-ray data on capillary collapse could be collected. The X-ray data were generally of very good quality. Only the data of scan 8 from experiment 3 could not be reconstructed and the images of scan 03 of experiment 2 are unfortunately blurred in the upper part of the sample, which could indicate movements within the specimen during the scan. The acquisition and reconstruction of the neutron data turned out to be more complicated and prone to problems. In the neutron data of experiment 2 and 5 various progressively increasing artefacts appeared, which could presumably be the result of a decreasing transmission. Despite extensive efforts, these could unfortunately not be completely eliminated in all scans and slices. Nevertheless, a partial evaluation of these data seems possibly conceivable with suitable image preprocessing. The glass beads used in experiment 4 were also not ideal, as some glass beads probably contained boron and thus appear similarly bright as the water phase. Nevertheless, the combined evaluation of the CT and neutron data can provide valuable insights, as the water phase can be visualised very well and distinguished from the other phases with the help of the neutron images, especially at low degrees of saturation at the beginning of the experiments or during the first failed experiment (collapse due to evaporation), regardless of the higher noise and lower sharpness. As an example, some obtained data from experiment 3 are shown in Figure 1.

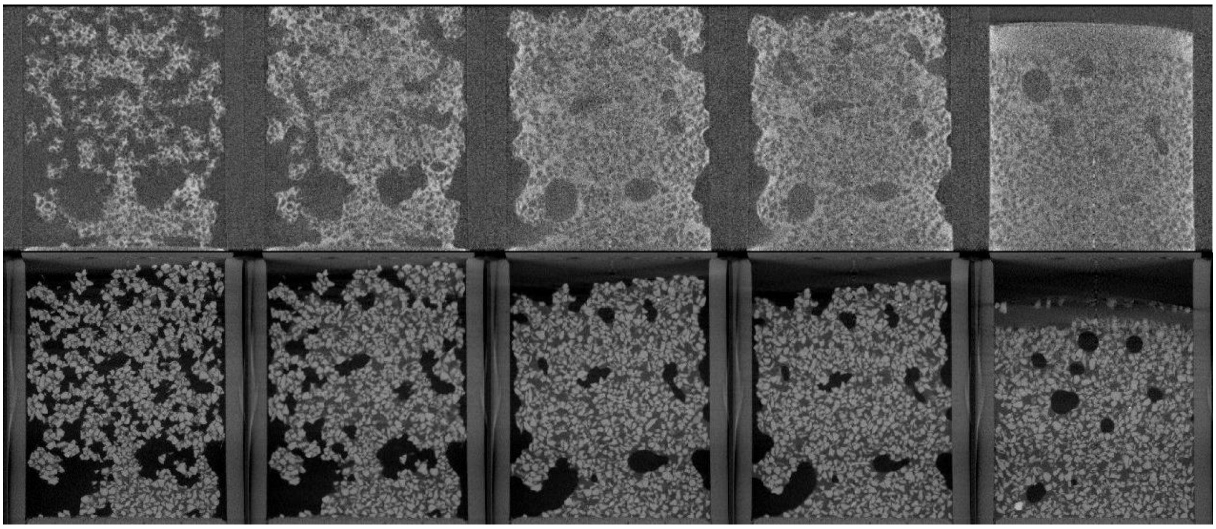

Figure 1: Central vertical slices obtained from neutron tomography (top) and X-ray tomography (bottom) during stepwise wetting through the specimen bottom (from left to right: image data of scans 0, 2, 4, 6 and 9)

While the neutron images show the rise of the liquid phase and the spread of the added water volumes within the specimen, the resulting settlements are particularly visible in the X-ray images. The individual experiments showed that in most cases the collapse does not occur abruptly, but gradually, so that the capillary collapse results as a sum of smaller micro-collapses triggered by the addition of small amounts of water. Thus, there seems to be an approximately linear correlation between the amount of water added and the induced collapse settlements, so that tomographic images before and after the irrigation steps are well suited to draw conclusions about the corresponding microscopic changes, provided that the irrigation steps are chosen small enough. However, it is noticeable that large macropores remain in the specimen, even though the addition of water was mathematically sufficient for full saturation. This effect was also observed in the other experiments and could possibly explain why a re-saturation after previous drying leads to further collapse settlements to a lesser extent. Further investigations should therefore be carried out on this issue.

2.2 First insights into the results from the evaluation of the processed data

Figure 2 shows the decrease in sample height and the evolution of porosity and degree of saturation for the processed image data of scans 0, 2, 4, 6 and 9 of experiment 3 (cf. raw data in Fig. 1). It can be seen that vertical differences in porosity and degree of saturation progressively become balanced with increasing number of irrigation steps. The data obtained will be used to analyse a variety of different parameters, such as the local degree of saturation, the contact angles, the void ratio, the collapse settlements, but also the morphological development of the phases and interfaces. In the case of small, not too chaotic particle movements due to the triggered micro-collapses, it is also planned to visualise and evaluate the grain kinematics with discrete image correlation methods.

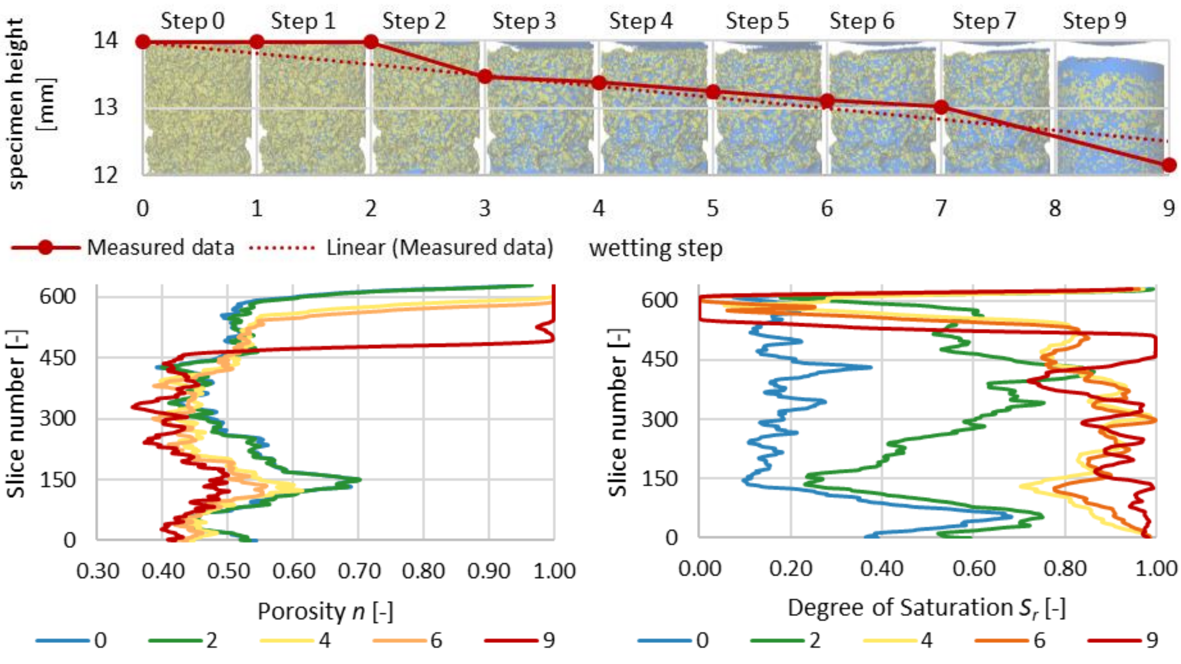

Figure 2: Development of the specimen height (top; Sand = yellow, water = blue, air = transparent) as well as the vertical porosity (bottom left) and the degree of saturation (bottom right) during the step-wise sample irrigation in experiment 3

References

Bruchon, J.-F., J.-M. Pereira, M. Vandamme, N. Lenoir, P. Delage und M. Bornert (2013). “Full 3D investigation and characterisation of capillary collapse of a loose unsaturated sand using X-ray CT”. In: Granular Matter 15. DOI: 10.1007/s10035-013-0452-6.

Hüsener, N., L. Helfen, M. Milatz und A. Tengattini (2021). Combined X-ray- and neutron-tomography imaging of capillary collapse in unsaturated granular soils. Institut Laue-Langevin (ILL). DOI: 10.5291/ILL-DATA.1-05-53.