Microscopic investigation of the hydro-mechanical behavior of unsaturated granular media with X-ray CT

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ABSTRACT

In the last years, X-ray computed tomography (CT) has been found to be a valuable tool to visualize and analyze structures in granular media from a microscopic point of view. With a sufficient resolution (voxel size) and a sequence of images over time, CT-images also allow to visualize processes in unsaturated soils, where microscopic features, such as the phase distribution (water-air-solid), the distribution of pores or air clusters as well as the interfaces between the phases, e. g. the interfacial area between the non-wetting and the wetting phase, can be studied. In the experimental program presented here, CT-imaging is applied to obtain 3D-data sets of two different soils in the unsaturated state. The hydro-mechanical behavior of free-standing unsaturated soil columns, only kept together by capillary cohesion, is investigated in two test series.

In the first test series, soil columns, made of a coarse to medium coarse sand and of a packing of glass beads, are submitted to evaporation, while their macroscopic degree of saturation is monitored by weighing over time. The evaporation process is virtually stopped at selected time points by sealing the specimen from the outer atmosphere. Then, the specimens are placed in a micro CT-scanner to investigate the unsaturated state changed by evaporation from a microscopic point of view. The distribution of pore water, the change in interfacial area as well as the shape of capillary bridges can be analyzed with focus on the loss of capillary cohesion within the specimen on its way to collapse due to drying.

In the second test series, a recently developed miniature compression device is used to perform uniaxial compression tests on free-standing cylindrical unsaturated sand and glass bead specimens. The compression device is also placed in a CT-scanner to obtain 3D-images for different compression stages, i. e. for different axial strain.

The measured CT-data of the evaporation and compression tests are finally analyzed with the focus on differences between the hydraulic and mechanical behavior of irregular shaped sand packings and ideal glass bead packings. Furthermore, the microscopic and macroscopic behavior during a change in degree of saturation by evaporation and during mechanical compression can be compared.

Keywords: Unsaturated granular soils, X-ray computed tomography, capillary cohesion, evaporation, shear strength

1 INTRODUCTION

Computed tomography (CT) is a radiographic imaging technique that allows obtaining 3D-images of matter. Originally invented for medical purposes, CT has nowadays also been discovered as a valuable tool in materials science and engineering. From the point of view of Geosciences, CT-imaging has been applied to obtain insight into the microstructure of rock and soils, even allowing to separate and analyse the different phases in unsaturated soil (Wildenschild et al., 2002), as well as to observe microscopic processes (Viggiani et al., 2015), which is also the main topic of this paper.

1.1 Imaging of capillary processes in unsaturated granular media using X-ray computed tomography

The different attenuation of X-rays passing through matter of different densities is used to distinguish material phases in granular media. The gray values of the obtained 2D-images (slices) that form a 3D-image data set, when stacked, allow to separate different phases, which is called segmentation based on a gray value threshold. In a natural unsaturated soil, frequently only three phases are present and are formed by the solid phase (soil particles), the gaseous phase (air) and the liquid phase (water). Due to the density difference,

\[ \frac{\rho_{\text{sand}}}{\rho_{\text{water}}/\rho_{\text{air}}} \approx 2.65/1.00/0.00125 \text{ (g/cm}^3\text{)}, \]

the three phases can be well distinguished, when the image
quality is good enough, i.e., when the signal to noise ratio is high enough, and the resolution, expressed in terms of voxel size, is sufficient to resolve phase boundaries and interfaces.

Different researchers have applied X-ray computed tomography in the past to investigate microscopic effects in unsaturated porous media. Frequently, laboratory X-ray sources using X-ray tubes are used. In other cases, the more powerful synchrotron radiation with very brilliant photon fluxes, created in particle accelerators, can be used in tomography experiments (Wildenschild et al., 2002; Wildenschild et al., 2005). A coarse differentiation of the investigations on unsaturated porous media conducted so far is given by hydraulic and mechanical or hydro-mechanical studies.

In the hydraulic studies, mainly the water retention behavior and its microscopic representation by multiphase flow processes, is of interest (e.g. Wildenschild et al., 2002; Higo et al., 2015; Khaddour, 2015). In the mechanical or hydro-mechanical studies, computed tomography is applied to investigate grain scale processes during saturated loading of unsaturated porous media. Similar to the hydraulic investigations, literature with focus on imaging methods applied to hydro-mechanical testing of unsaturated soils is still quite rare. Different authors have already applied triaxial testing to investigate the microscopic effects within unsaturated soils during shearing (e.g. Higo et al., 2013; Khaddour, 2015), or have investigated capillary collapse upon wetting of sand using X-ray computed tomography (Bruchon et al., 2013).

1.2 Aims of research

The experiments presented in this contribution are supposed to give insight into the hydro-mechanical behavior of unsaturated granular media by different CT-experiments. In traditional unsaturated soil mechanics, the hydro-mechanical state is described from a macroscopic point of view, ignoring the real origins of macroscopic behavior as well as possible inhomogeneity of soil properties. With the help of the results that can be derived from CT-scan data,

- the microscopic hydraulic response upon drying of granular media, and
- the microscopic hydro-mechanical behavior of unsaturated granular media causing a loss of capillary cohesion due to evaporation, and
- its microscopic hydro-mechanical response upon shearing at low stress state

are supposed to be investigated and compared to the observed macroscopic hydraulic and mechanical response throughout the experiments. Thus, the experiments are supposed

- to investigate the water distribution upon drying,
- to experience the microscopic origins of macroscopic capillary cohesion, and furthermore,
- to observe the change of water distribution and capillary bridges due to a deformation of the grain skeleton in uniaxial compression tests.

2 EXPERIMENTAL PROGRAM

In order to shed light on the hydro-mechanical behavior of unsaturated granular media, different laboratory and CT-experiments have been planned. In this contribution, we focus on evaporation tests and uniaxial compression tests under lab- and CT-conditions.

2.1 Tested material

Two different granular materials have been selected for the experiments: a coarse to medium coarse sand, “Hamburger Sand”, that is used as a model sand at Hamburg University of Technology (TUHH), and a mixture of soda-lime glass beads (SiLi beads, type S, manufactured by Sigmund Lindner GmbH, Germany) with a grain size distribution that has been adapted to that of Hamburger Sand. Here, the experimental results for irregular shaped natural sand grains are supposed to be compared to the results for ideal spherical particles. The packing of glass beads is produced by mixing several grain size fractions. To approximate the grain size distribution of Hamburger Sand, 14.02 % of glass beads with 0.25 mm ≤ d ≤ 0.5 mm, 51.98 % of beads with 0.5 mm < d ≤ 0.75 mm, 32.77 % of beads with 0.75 mm < d ≤ 1.0 mm, and 1.23 % of beads with 1.0 mm < d ≤ 1.3 mm are mixed. The grain size distribution curves of both granular materials are illustrated in figure 1 together with photos of the particles.

![Fig. 1. Grain size distribution curves of Hamburger Sand and glass beads together with photographs of the tested material.](image-url)

The basic soil properties of Hamburger Sand and the packing of glass beads are summarized in table 1.

For the evaluation of capillary effects, the water retention curve (WRC) of both materials has been measured with the HYPROP evaporation test (Peters and Durner, 2008), manufactured by UMS GmbH/METER Group (UMS, 2018). This
The experimental procedure has been selected, because the drying process comes close to the expected conditions in the laboratory evaporation tests. In the HYPROP test set-up, a cylindrical, initially water-saturated soil specimen is submitted to free evaporation at the top, while the change in gravimetric water content is measured by continuously weighing the whole set-up. Matric suction is measured over time in two heights with embedded tensiometers. From the measured continuous data, a primary drainage path of the water retention curve is obtained. Results for Hamburger Sand and the packing of glass beads, as well as fitted curves using the van Genuchten model (van Genuchten, 1980), are illustrated in figure 2. The model parameters are summarized in table 2.

Table 1. Basic soil properties of the investigated materials.

<table>
<thead>
<tr>
<th>Material</th>
<th>( \rho_s ) (g/cm³)</th>
<th>( e_{min} )</th>
<th>( e_{max} )</th>
<th>( d_{10} ) (mm)</th>
<th>( d_{50} ) (mm)</th>
<th>( d_{max} ) (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hamburger Sand</td>
<td>2.64</td>
<td>0.52</td>
<td>0.805</td>
<td>0.45</td>
<td>0.68</td>
<td>2.0</td>
</tr>
<tr>
<td>Glass beads</td>
<td>2.50</td>
<td>0.555</td>
<td>0.679</td>
<td>0.45</td>
<td>0.68</td>
<td>1.3</td>
</tr>
</tbody>
</table>

\( \rho_s \): grain density, \( e_{min}/e_{max} \): minimum/maximum void ratio, \( d_{10} \): grain diameter at 10\% passing, \( d_{50} \): grain diameter at 50\% passing, \( d_{max} \): maximum grain diameter.

Fig. 2. Water retention curves of Hamburger Sand and glass beads (primary drainage), measured in a HYPROP-evaporation test, together with fitted curves using the van Genuchten model.

Table 2. Parameters of the van Genuchten model.

<table>
<thead>
<tr>
<th>Material</th>
<th>( \alpha ) (1/kPa)</th>
<th>( n ) (-)</th>
<th>( S_{w0} ) (-)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hamburger Sand</td>
<td>0.847</td>
<td>7.3</td>
<td>0.050</td>
</tr>
<tr>
<td>Glass beads</td>
<td>1.053</td>
<td>7.7</td>
<td>0.027</td>
</tr>
</tbody>
</table>

\( \alpha, n, (m = 1–1/n) \): Fitting parameters (van Genuchten, 1980), \( S_{w0} \): Residual degree of saturation.

The water retention curves show a low air entry value (AEV) below 1 kPa for both investigated materials, typical of coarse grained granular assemblies. The capillary effects are therefore expected to be very low compared to other granular materials with finer grains. Despite very similar grain size distribution curves, Hamburger Sand shows higher matric suction compared to the glass bead packing.

### 2.2 Specimen preparation

In all tests presented in this contribution, cylindrical specimens of unsaturated granular material, only kept together by the capillary stress state, are investigated. In order to find an optimum between recorded amount of CT-data, scan-time and representativeness of specimen size with a criterion of at least 20 grains over the specimen diameter, a special specimen preparation technique, illustrated in figure 3, was developed that allows producing small-sized specimens with diameters of only 12 to 20 mm. For specimen preparation, the dry mass of either sand or glass beads, needed for a desired initial dry density \( \rho_d \) or void ratio \( e_0 \) is mixed with the calculated mass of de-ionized water \( m_w \) corresponding to a desired initial gravimetric water content \( w \) or initial degree of saturation \( S_w \). After thorough mixing in a ceramic bowl, the wet material is completely filled into an acrylic tube with a movable piston-like bottom. The inner tube-diameter corresponds to the desired specimen diameter \( d \). The wet material is inserted in layers which are compacted and then roughened before the next layer is inserted. The roughening of layer-surfaces has proven to be important for creating non-layered homogeneous specimens. The whole material is compacted into the tube and finally pushed out slowly in vertical direction with the help of the movable bottom. This procedure allows producing circular sand or glass bead specimens in a repeatable way. The specimens are standing on a base plate that allows to bring them into position for the individual experiments presented here.

Fig. 3. Preparation of an unsaturated free-standing sand specimen with a diameter and height of 12 mm, left: compacted specimen in acrylic tube on movable base plate and piston, right: final specimen.

### 2.3 Evaporation tests on unsaturated granular media

In order to investigate the link between macroscopic and microscopic drying behavior, the distribution of pore water within granular media as well as the mechanical strength due to capillary cohesion, evaporation tests have been designed and performed.
under lab conditions and in combination with CT-imaging.

**Laboratory evaporation tests**

In the lab tests, free-standing columns of Hamburger Sand and glass beads with a diameter \( d = 12 \text{ mm} \) and height \( h = 12 \text{ mm} \) are submitted to free evaporation at the specimen sides and at the specimen top. After specimen preparation according to figure 3, the specimens are placed on a laboratory balance (EW-N precision balance, manufactured by Kern & Sohn GmbH, Germany) to measure the amount of pore water loss by evaporation over time. From the measured data, the macroscopic degree of saturation can be monitored with a resolution of ca. \( \pm 0.02 \). This results in some inaccuracy, especially for lower degrees of saturation to be investigated. In order to monitor the specimen collapse due to loss of capillary cohesion, two experimental ideas are applied: First, a series of time-lapse photos is taken to monitor the specimen state over time. Additionally, a steel ball is placed on top of the specimen to act as a trigger signal, when first signs of specimen collapse by disintegrating grain skeleton occur. With the help of a specially designed test set-up, depicted in figure 4, the steel ball can roll off the specimen base plate upon a collapse event and leave the balance weighing plate on an inclined plane, thus creating a jump in the measured weight data over time. This data jump can be used to identify the exact time point of specimen collapse due to loss of capillary cohesion and can be compared to the photo series of the same test, showing the specimen’s exterior shape.

The photo series in figure 4 shows a gradual collapse process of the sand specimen upon drying. The collapse starts with exterior grains falling off, leaving an inner core of apparently wetter granular material that remains stable for several minutes, until finally a cone of dry sand with a characteristic slope angle is reached. The same macroscopic behavior could also be observed for similar glass bead specimens.

**CT-imaging of evaporation**

The microscopic interior processes upon drying by evaporation, leading to the observed macroscopic behavior, are supposed to be investigated by CT-imaging. Similar evaporation tests, however without a steel ball placed on the specimen top, are therefore started with parallel CT-scans at different temporal states of evaporation in order to have a look at the microscopic hydro-mechanical behavior within the drying material. For this purpose, free-standing specimens with different initial degrees of saturation are prepared, as described above. The specimens are placed on circular patches of sand paper that is glued to a base plate with two-sided adhesive tape, to ensure a good contact during scanning with incremental specimen-rotation. With this approach, moving artifacts, leading to noisy CT-images, especially at the top of free-standing specimen, can be minimized. During the CT-scans, the specimens are enclosed in a specimen holder to reduce evaporation.

![Principle of laboratory evaporation tests](image)

After an initial CT-scan, a sequence of further scans is started after successive evaporation intervals, in which the specimen is submitted to free evaporation by opening the specimen holder. The loss of pore water by evaporation is always monitored gravimetrically using the same balance as in the laboratory evaporation tests.

The CT-images are taken at TUHH with a desktop micro CT scanner \( \mu \text{CT} 35 \), manufactured by Scanco Medical AG, Switzerland. The \( \mu \text{CT} 35 \) is a clinical desktop CT with a cone beam that allows scanning specimens with a diameter of 75.8 mm and a height of 140 mm in a closed leaden chamber, accessed by a small hatch. The scan parameters applied in the imaging of the evaporation experiments on unsaturated sand and glass bead packings are summarized in table 3.

**Table 3.** Scan parameters of the micro CT scanner applied to imaging of the evaporation tests.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>X-ray tube voltage</td>
<td>70 kVp</td>
</tr>
<tr>
<td>X-ray tube current</td>
<td>113 ( \mu \text{A} )</td>
</tr>
<tr>
<td>Integration time</td>
<td>800 ms</td>
</tr>
<tr>
<td>Projections/180° revolution</td>
<td>1000</td>
</tr>
<tr>
<td>Isotropic voxel size</td>
<td>10 ( \mu \text{m} )</td>
</tr>
<tr>
<td>Binning</td>
<td>1</td>
</tr>
<tr>
<td>Averaging</td>
<td>2</td>
</tr>
<tr>
<td>Acquisition time (1 scan)</td>
<td>Approx. 6.2 h</td>
</tr>
<tr>
<td>Acquisition mode</td>
<td>Incremental rotation</td>
</tr>
</tbody>
</table>

Due to the small available detector screen, the full specimen has to be imaged in several image stacks,
leading to acquisition times of ca. 6.2 h. Although the specimens are sealed in a specimen holder during the scan, water evaporation cannot be entirely avoided during this long scan time. Therefore, the water contents and degrees of saturation assigned to a certain scan represent values averaged from the water content at the beginning and at the end of a CT-scan.

### 2.4 Uniaxial compression tests on unsaturated granular media

Focusing on microscopic effects in unsaturated granular media during shearing, a miniature uniaxial compression apparatus has been designed, that allows to shear small specimens of unsaturated sand or glass beads in uniaxial compression while being placed in a CT-scanner. As outlined by Khaddour (2015), who conducted triaxial tests and analyzed the microscopic triaxial responses of unsaturated sand, using a microfocus X-ray CT at the University of Grenoble, France, the impact of capillary effects on the shear strength of unsaturated granular soils is only measurable for very low confining pressures. Therefore, uniaxial compression tests on unsaturated specimens of granular media, only kept together by capillary cohesion, have been selected as a means to investigate the influence of very low matric suction on the shear strength. The microscopical changes in the three-phase system of grains, pore air and pore water can be investigated with CT-scans after different shear steps.

The new testing apparatus and its control device, the UNSAT-Pi, for the first time described in Milatz (2019), are depicted in figure 5.

![Photograph of the compression apparatus with its components and dimensions](image)

Fig. 5. Photograph of the compression apparatus with its components and dimensions (left) and the UNSAT-Pi single-board computer for test control (right).

The test set-up consists of a stepper motor, manufactured by Changzhou Fulling Motor Co., Ltd., China, and a miniature load cell of type KM10z, manufactured by ME Meßsysteme GmbH, Germany, with a low measurement range of only ±25 N and an accuracy class of ±1 %. The load cell is embedded in the base pedestal that can be fixed on the rotation stage of a CT-scanner. The stepper motor as well as the load cell are connected to the UNSAT-Pi, a microcontroller- and datalogger-system based on a Raspberry Pi 3 B (Milatz, 2019). This single-board computer controls the stepper motor that drives the axial loading plate and takes readings of the load cell during compression. The axial displacement of the loading plate is calculated from the stepper motor position using a system calibration and is also recorded with the UNSAT-Pi. The control program is coded using the Python programming language.

### Uniaxial compression tests under lab conditions

In this contribution, we firstly focus on uniaxial compression tests performed under lab conditions, i.e. without CT-imaging, in order to investigate the influence of different degrees of saturation on uniaxial compression strength and capillary cohesion (Milatz, 2019). In these tests, monotonic loading paths are applied by prescribing vertical displacements of the loading plate of up to 6 mm with a loading rate of approximately 0.48 mm/min. Additionally, the specimen shape is observed by a time-lapse series of photographs. This testing procedure allows to record paths of axial stress σ versus axial strain ε for different initial macroscopic degrees of saturation. The capillary cohesion cₖ is finally derived from the uniaxial compression strength σ_max using the effective friction angle from drained triaxial tests φ', following an approach by Grabe and Schwarz (2004) and Milatz (2016). Capillary cohesion can be derived by bringing the shear line with slope tan φ' in contact with Mohr’s circle for the stress state at failure obtained from a uniaxial compression test as illustrated in figure 6. Capillary cohesion can then be derived as the shear strength for zero axial stress or calculated according to equation 1 as the difference of the shear stresses τ₁ and τ₂ illustrated in figure 6.

![Stress conditions at failure in uniaxial compression](image)

Fig. 6. Determination of capillary cohesion cₖ from the results of uniaxial compression tests, left: Mohr’s circle at failure, right: Stress conditions at failure for soil element and shear strength components to calculate cₖ according to equation 1.

\[
cₖ = 0.5 \sigma_{max} \cos \phi' - (1 - \sin \phi') \tan \phi' \tag{1}
\]

This approach follows the assumption, that the effective friction angle φ', derived from drained triaxial tests for
the same materials, does not represent a function of saturation or suction (Fredlund et al., 1978). Furthermore, the stress state in the specimens at failure is assumed only to be due to capillary effects, i.e., a stress contribution due to gravity is neglected.

For the evaluation of \( c_e \) presented here, the effective friction angles from drained triaxial tests \( \phi' = 33.9^\circ \) for Hamburger Sand at \( e_0 = 0.65 \), and \( \phi' = 22.3^\circ \) for glass beads at \( e_0 = 0.6 \) are applied.

The capillary cohesion represents a macrosopic variable, reflecting the contribution of the unsaturated effective stress state to shear strength. The contribution of suction and degree of saturation to effective stress is typically modelled according to the traditional approach by Bishop (1959), or by the more recent suction stress concept (Lu and Likos, 2006; Lu et al., 2010).

For the theoretical evaluation of capillary cohesion, in this contribution, the effective stress is modelled following the Bishop-stress approach according to equation 2, with \( \sigma' = s \chi \) being a function of matric suction \( s \) and the Bishop parameter \( \chi \) which is assumed to be equal to \( S_e \). The net normal stress term, consisting of the difference of total stress and air pressure is already neglected in equation 2. Other approaches, such as the suction stress concept (Lu and Likos, 2006, Lu et al., 2010), assume \( \chi \) to be equal to \( S_e \), with \( S_e \) being the effective degree of saturation.

\[
\sigma' = s \chi \quad \text{with} \quad \chi = S_e \tag{2}
\]

With equation 2, capillary cohesion \( c_e \) can be calculated according to equation 3, using the effective friction angle \( \phi' \). This approach allows to derive a theoretical \( c_e \)-curve from the water retention curve which will be compared to experimental results of \( c_e \) in this paper.

\[
c_e = s \chi \tan \phi' = s S_e \tan \phi' \tag{3}
\]

2.5 Evaluation and analysis of CT-data
All reconstructed CT-images are obtained in a 16 bit .tiff-file format and are further processed with the software Avizo 9.5 (Thermo Fisher Scientific). The evaluation procedure, further explained in the following, consists mainly of the segmentation, i.e. the detection and separation of different phases in the grayscale images, and further image analysis steps.

The stack of .tiff-images is completely read into the memory of the evaluation pc (Windows 7 Professional, with Intel® Core™ i5-6500 CPU@3.20 GHz, 64 GB RAM, nvidia Quadro 2000D GPU), then a 3D median filter is applied to remove noise. In the segmentation step, the 2D-histogram segmentation algorithm based on the publication by Jones et al. (2007) is applied for the segmentation of grains, air and water. The algorithm contains different steps that allow for a semi-automatic segmentation of grayscale images containing multiple phases. In a first step, seed-voxels are classified by a gradient magnitude versus image intensity histogram. In a second step, these seed-voxels are expanded by a marker-seeded watershed transform, until all voxels are segmented. A benefit of this segmentation approach is its capability of reducing the partial volume effect (Wildenschild et al., 2005), leading to ill-segmented pixels at the interface of different phases. In the case of unsaturated sand and glass bead packings, normal threshold-based segmentation techniques have been found to lead to visible partial volume effect, with non-physical water hulls covering the grain surfaces with a thickness of only one pixel. The described procedure leads to a segmented label field that is further processed in Avizo.

The further data analysis consists of a visualization of each segmented phase in 2D-slices and 3D-volume rendering as well as different material statistics steps, which allow counting the voxels of each individual specimens, especially the glass beads. Third, the experimental facility in Grenoble allowed a very flexible set-up of specimens and the testing device due to an accessible scanning chamber in contrast to the limited space within the desktop micro CT-scanner for imaging of the evaporation tests. The scan parameters, applied in CT-imaging of the multistep uniaxial compression tests conducted at the University of Grenoble are summarized in table 4.

Table 4. Scan parameters of the CT-scanner applied to imaging of the multistep uniaxial compression tests.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>X-ray tube voltage</td>
<td>120 kVp</td>
</tr>
<tr>
<td>X-ray tube current</td>
<td>82 µA</td>
</tr>
<tr>
<td>Projections/360° revolution</td>
<td>1200</td>
</tr>
<tr>
<td>Isotropic voxel size</td>
<td>11 µm</td>
</tr>
<tr>
<td>Binning</td>
<td>1</td>
</tr>
<tr>
<td>Averaging</td>
<td>4</td>
</tr>
<tr>
<td>Acquisition time (1 scan)</td>
<td>Approx. 0.6 h</td>
</tr>
<tr>
<td>Acquisition mode</td>
<td>Continuous rotation</td>
</tr>
</tbody>
</table>

Multistep (incremental) uniaxial compression tests on unsaturated Hamburger Sand and glass beads with parallel CT-imaging have been conducted. In contrast to the evaporation tests shown before, the uniaxial compression tests are conducted within the CT-imaging set-up at Laboratoire 3SR at the University of Grenoble (Viggiani et al., 2015). The CT-scanner in Grenoble has been found to be a good choice for the multistep uniaxial compression tests, because of different factors: first, short image acquisition times of only 36 minutes per scan could be achieved, which allowed to reduce the effect of evaporation, which is undesired in this kind of experiment. Second, a continuous image acquisition could be applied, in which the rotation stage is rotating without abrupt increments, thus not creating peaks of rotational acceleration acting on the grains and the pore water in the unsaturated granular specimens (Khaddour, 2015). This has also turned out to be beneficial with respect to the fragile unsaturated
phase contained in a pre-selected subvolume. The subvolumes are supposed to be large enough to give representative results, because all properties always need a representative elementary volume (REV). For this purpose, REV-analyses can be performed, see Khaddour (2015). The voxel-count of each phase in a subvolume of representative size can then be used to calculate local microscopic properties, such as the void ratio \( e \) and the degree of saturation \( S_d \). In this contribution, soil and mixture properties calculated from a local subvolume of voxels, are referred to as “microscopic”, whereas variables, calculated from macroscopic properties, i. e. the dry mass of sand or glass beads \( m_d \), water mass inside the specimen \( m_w \) and the specimen volume \( V \), are referred to as “macroscopic” variables.

Further analysis in Avizo allows detecting the non-wetting-wetting fluid interfacial area between the segmented air and water phase, referred to as \( a^{nw} \). This variable is equivalent to the surface area of the menisci (contractile skin) and is believed to represent an important factor for effective stress in unsaturated soils (Nikooee et al., 2013) and a possible source of hysteresis in the water retention behavior (Hassanizadeh and Gray, 1993; Culligan et al., 2004). For the calculation of \( a^{nw} \), a small subvolume of 300x300x300 pixels (px) is cut out of the combined data set of air and water voxels. From this data set, the interfacial area is generated by approximating the air-water phase intersections with small triangles that allow the calculation of \( a^{nw} \).

3 EXPERIMENTAL RESULTS

3.1 Monitoring of capillary cohesion loss due to evaporation on the macro- and microscale

Macroscopic results (laboratory tests)
The laboratory evaporation tests allow to investigate the time point of collapse as well as the collapse process itself by evaluation of the photo series, see figure 4. Furthermore, macroscopic soil properties, such as the degree of saturation \( S_d \) at collapse, can be determined within the limits of available resolution of the weighing method. The development of macroscopic \( S_d \) over evaporation time \( t \) is illustrated in figure 7 for specimens of Hamburger Sand and glass beads with different initial degrees of saturation \( S_{d0} \). For both granular materials, the evaporation seems to work quasi linearly over time, from which a homogeneous evaporation over the whole specimen could be derived. All specimens remain stable up to very low degrees of saturation. These correspond to the pendular state in the water retention curves, compare figure 2.

Two out of three curves from tests on glass beads show a lower evaporation rate compared to the tests on Hamburger Sand, corresponding to longer evaporation times till collapse. The reason for this might be the active contact area between air and water. As the outside specimen surfaces are the same for sand and glass bead specimen, the key to different evaporation times might be the air-water interfacial area \( a^{nw} \) that will be evaluated later.

The evaluation of developing matric suction and capillary cohesion is based on the assumption that a primary drainage path is followed during evaporation, starting from the initial hydraulic state after specimen preparation. Under this assumption, ignoring hysteresis and the true initial state, that might rather correspond to a wetting path due to specimen preparation by mixing sand and water, a suction-path can be calculated from degree of saturation using the inverted van Genuchten model with parameters presented in table 2. In a next step, a path for the development of \( c_c \) during the evaporation tests can be calculated with equation 3, allowing to estimate \( c_c \) at collapse of the tested material, see figure 8.

Due to the nonlinear relationship in equation 3, also nonlinear curves of theoretical (calculated) \( c_c \) versus evaporation time are obtained. It can be noticed that collapse takes place for values of \( c_c < 0.1 \) kPa for glass beads and \( c_c < 0.15 \) kPa for Hamburger Sand. Although these results lie quite close to each other, they reflect the more pronounced capillary effects in the sand compared to the glass beads, which can already be derived from the WRCs in figure 2. In order to improve the presented experiment in the future, suction should be measured during evaporation to account for the true hydraulic path and a possible hydraulic hysteresis, which is not possible with the present testing method.
Fig. 8. Curves of theoretical (calculated) macroscopic capillary cohesion $c_c$ versus evaporation time $t$ until collapse (marked by a black cross) from laboratory evaporation tests on specimens of Hamburg Sand (top) and of glass bead packings (bottom).

**Microscopic results (CT)**

In order to better understand the microscopic processes during drying by evaporation and the parallel loss of capillary cohesion, two CT-scan series on Hamburg Sand and glass bead specimens on a drying path are discussed. Figures 9 and 10 show the segmented CT-data sets of Hamburg Sand and glass bead specimens for different macroscopic degrees of saturation reached from an initial state by evaporation. The bottom parts (sand paper glued to a base plate) are not shown for all 3D-data sets.

Especially the 3D-reconstructions show the specimen integrity throughout the CT-scans. For the investigated macroscopic degrees of saturation, calculated from the weighed water mass inside the specimen throughout the test, no collapse occurred, despite the incremental rotation of the specimens in the CT-scanner. However, the glass bead specimen collapsed during the attempt of a fourth CT-scan, not documented here, while for Hamburg Sand, two more scans were possible.

The water distribution in the middle slices in figures 9 and 10 shows that water evaporation starts with the emptying of larger pores. This is due to higher capillary pressure and therefore stronger water retention in smaller pores compared to larger ones. On the other hand, the inner evaporation by water vapor transport in pore air is also more pronounced at the larger air-water interfaces within larger pores.

An evaporation front occurs from the outside specimen boundaries but can also be noticed around larger pores in the middle of the specimen, leading to an apparently rather homogeneous distribution of water clusters over the specimen cross sectional area, which would be in good agreement with the linear evaporation curves from the laboratory evaporation tests, see figure 7. However, the rather homogeneous water distribution could also be a result of the small specimen sizes investigated here.

An interesting approach towards understanding of the macroscopic capillary cohesion is the analysis of the microscopic interfacial area $a_{nw}$, which has been calculated for a cubic subvolume of 300 px (3 mm) edge length, extracted from the 3D-data sets for different macroscopic degrees of saturation, see figure 11.

The interfacial area, numerically approximated by triangular facets, appears to be more structured in the packings of regular glass beads compared to a more irregular distribution within the sand. Due to evaporation, the initially large and rather connected regions of $a_{nw}$ get more and more dispersed over the considered subvolume and also isolated. The evolution of $a_{nw}$ plotted versus macroscopic degree of saturation, i. e., following a decrease of $S_r$ through evaporation, is shown in figure 12 for sand and glass beads.

Despite the limited and apparently scattering data, a reduction of $a_{nw}$ towards zero with decreasing macroscopic $S_r$ can be noticed. A similar trend has also been observed by Culligan et al. (2004) in unsaturated flow experiments on glass beads and by Khaddour (2015) in water retention tests on a sand. The values of $a_{nw}$ are generally smaller for glass beads compared to...
sand, which could explain the slower evaporation in the case of the glass beads but also their lower shear strength due to capillary cohesion. More data and further evaluation are needed to further clarify these first interpretations.

3.2 Uniaxial compression tests to investigate capillary cohesion on the macro- and microscale
Uniaxial stress conditions allow evaluating the magnitude of capillarity contribution to overall shear strength due to the low stress level. In this paper, first results of uniaxial compression tests will be presented from the macroscopic and microscopic point of view.

Macroscopic results (laboratory tests)
With the help of the developed testing apparatus for uniaxial compression tests on unsaturated granular materials, presented in detail in Milatz (2019), the influence of different macroscopic degrees of saturation on capillary cohesion can be investigated. For this purpose, specimens of Hamburg Sand and glass beads with h/d = 20 mm/20 mm are monotonously sheared and capillary cohesion is evaluated according to the approach shown in section 2.4. From the stress-strain behavior of Hamburg Sand and glass beads, shown in figure 13, an increase of uniaxial compression strength $\sigma_{\text{max}}$ with increasing $S_r$ can be noticed.
The measured shear strength of Hamburger Sand is generally higher compared to the glass bead packing.

In figure 14, the macroscopic capillary cohesion for both tested materials, calculated from the experimental data with equation 1, is compared to theoretical curves of \( c_c \) versus \( S_n \), calculated with equation 3 from the experimental WRCs shown in figure 2.

![Fig. 14. Capillary cohesion \( c_c \) derived from uniaxial compression tests on Hamburger Sand and glass beads for different macroscopic degrees of saturation \( S_n \) compared to theoretical curves calculated from WRC-data.](image)

In the case of glass beads, the theoretical capillary cohesion derived from the experimental WRC-data is in very good agreement with the data points obtained from uniaxial compression tests. For the sand, higher capillary cohesion values are obtained from uniaxial compression tests compared to the theoretical curve.

**Microscopic results (CT)**

With the focus on the hydro-mechanical behavior on the grain scale, two multistep uniaxial compression test series on specimens of Hamburger Sand and glass beads with \( h = d = 12 \) mm and at a similar initial degree of saturation are imaged by CT. Starting from a CT-scan of the initial state after specimen preparation (\( \varepsilon = 0 \)), the specimens are incrementally sheared by 0.3 mm and then again scanned with scan parameters summarized in table 4, leading to a total of seven CT-scans per test series. The segmented images, obtained from the image-analysis steps described before, are shown in figures 15 and 16 on the next page, starting with a 3D-visualization of grains and water, followed by corresponding central vertical slices and vertical slices of the water phase only for four out of seven specimen states at strain increments of \( \Delta \varepsilon \approx 0.05 \).

The specimen properties shown for every load step represent macroscopic variables, calculated from the known mass of grains and water in the specimen (under consideration of evaporative water losses) and from the current and updated specimen volume. The specimen volume is calculated from a 3D-voxel count (interior void space plus grain volume), determined by a successive dilation and erosion approach, presented in Khaddour (2015). This approach allows considering the specimen volume change during shearing, normally not recorded in uniaxial compression tests.

The voxel size of 11 \( \mu m \) allows to track the water phase during shearing. Although there is still some undesired evaporation of pore water, monitored by weighing the specimen at the beginning and end of the CT-scan series, the microscopic change of water bridges due to shearing can be monitored. For the different shear steps, a rupture and coalescence of capillary bridges around moving grains can be noticed in both tested granular materials. Due to a dilatant behavior with increasing specimen volume, the degree of saturation is gradually reduced upon shearing, which would theoretically lead to an increase in matric suction. At the same time, a stretching of capillary bridges can be noticed.

Similar to the evaporation tests, the interfacial area \( a_{\text{nw}} \) is evaluated and visualized for a central cubic subvolume with edge length of 300 px (3.3 mm), shown in figure 17 for all shear steps. A change in the shapes of the individual interfaces can be noticed with increasing strain \( \varepsilon \). This qualitative observation corresponds to an increase of \( a_{\text{nw}} \) and a final decrease at higher axial strains, quantitatively shown in figure 18.

![Fig. 17. Development of interfacial area \( a_{\text{nw}} \) during different shear steps in uniaxial compression tests on Hamburger Sand (top) and glass beads (bottom) for a central cubic subvolume with an edge length of 300 px (3.3 mm).](image)

![Fig. 18. Development of interfacial area \( a_{\text{nw}} \) with axial strain \( \varepsilon \) in uniaxial compression tests on Hamburger Sand and glass beads.](image)
Fig. 15. Segmented CT-data set of a multistep uniaxial compression test on a Hamburger Sand specimen \((h = d = 12 \text{ mm})\) with macroscopic specimen data: 3D-reconstructions of grains and water (top), central vertical slices of grains and water (middle) and central vertical slices of water phase (bottom) for each shear step.

Fig. 16. Segmented CT-data set of a multistep uniaxial compression test on a glass bead specimen \((h = d = 12 \text{ mm})\) with macroscopic specimen data: 3D-reconstructions of grains and water (top), central vertical slices of grains and water (middle) and central vertical slices of water phase (bottom) for each shear step.
The observed behavior of \( a^{\text{nw}} \) probably reflects the change of capillary bridges due to shearing, by reordering, merging, stretching and also rupturing.

4 CONCLUSIONS

The hydro-mechanical coupling in unsaturated soils still bears many not entirely answered research questions, such as the microscopic origins of hydraulic hysteresis and effective stress. However, it is believed that CT-imaging represents the key to a better understanding of these hydro-mechanical phenomena.

In this contribution, different experiments for the investigation of the hydro-mechanical behavior of unsaturated granular media have been presented and evaluated from a macroscopic and microscopic point of view. With the help of CT-imaging, interesting insights into the microscopic processes during drying by evaporation and during shearing of the grain skeleton in uniaxial compression could be obtained. The results show that the macroscopic hydro-mechanical behavior roots in microscopic changes within the three-phase material, such as the change of capillary bridges and interfacial area due to drying, but also the reordering and rupture of water clusters and capillary bridges due to a mechanical loading and grain movement.

In the future, further material analyses will be run on the available data sets to give more quantitative results, e.g. results of the distribution of microscopic soil properties, the evolution of interfaces, 3D-particle movements and the number and size distribution of water clusters. Furthermore, the gathered 3D-data can be used for the validation and improvement of discrete numerical schemes, such as the Discrete Element Method with Bonded Particle Models, allowing to simulate discrete capillary bridges.

REFERENCES