Visualization and Quantification of the Penetration Behavior of Bentonite Suspensions into the Pore Network of non-cohesive Media by using µ-CT Imaging

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Abstract

Bentonite suspensions are an essential tool for different construction techniques in horizontal and vertical drilling, in diaphragm and bored pile walls as well as in pipe jacking and tunneling. One of the main tasks of the suspension is to prevent the surrounding ground from collapsing during the excavation process of trenches, drill holes or tunnels. In order to maintain the soil stability close to the excavation, the bentonite suspension has to counteract against the earth and water pressure. Therefore, the pressure acting in the suspension has to counter the groundwater pressure and to be transferred into an effective stress to support the soil skeleton.

The creation of a pressure transfer mechanism can be achieved in two ways. A direct relation exists between the mechanism of the pressure transfer and the penetration behavior of the bentonite suspension in the subsoil. The relation of the size of the bentonite particles in the suspension and the size of the pores in soft soil is decisive. In addition, the yield strength of the bentonite suspension is a determining factor.

Concerning the penetration behavior two theoretical models exist actually: formation of a filter cake and entire penetration into the pore space. If the pore space is smaller than the size of the bentonite particles, a filtration process takes place. Here, the bentonite particles agglomerate gradually at the entrance of the pore space and create a thin nearly impermeable layer. This membrane is named filter cake. If the pore space is larger than the size of the bentonite particles, the suspension penetrates into the subsoil up to a certain depth.

These models have a more theoretical character due to missing visual evidence concerning the interaction of the bentonite suspension in the pore space. Here, the micro CT technique delivers a valuable contribution to this research.

Keywords: µ-CT, non-cohesive porous media, bentonite suspensions, non-Newtonian fluid, penetration depth, filter cake
1 Introduction

Bentonite suspensions are an essential tool for different construction techniques in horizontal and vertical drilling, in diaphragm and bored pile walls as well as in pipe jacking and tunneling. One of the main tasks of the suspension is to prevent the surrounding ground from collapsing during the excavation process of trenches, drill holes or tunnels. In order to maintain the soil stability close to the excavation process stagnates in a certain depth, when the suspension penetrates completely into the pore space of the ground up to a certain depth. In case the pore size of the soil exceeds the size of the suspended bentonite particles [Walz, 2001]. Here, the bentonite particles are filtered at the entrance of the pore space and the remaining filtrate water drains through the soil. By gradual agglomeration of more bentonite particles, a thin, impermeable membrane is build (Figure 1 (left)). Here, the suspension pressure is transferred through the membrane area in terms of effective stress to the soil skeleton.

Currently, the dominant theory in tunneling practice is adopted from diaphragm wall technology [Müller-Kirchenbauer, 1977] and summarized in DIN 4126 (2004). According to German Standard [DIN 4126, 2004] the formation of a pressure transfer mechanism in terms of effective stress can be achieved in two ways: (a) with a thin and flexible membrane or (b) with a limited penetration zone in the soil.

The membrane, named filter cake (a), develops when the pore size of the soil is smaller than the size of the suspended bentonite particles [Walz, 2001]. Here, the bentonite particles are filtered at the entrance of the pore space and the remaining filtrate water drains through the soil. By gradual agglomeration of more bentonite particles, a thin, impermeable membrane is build (Figure 1 (left)). Here, the suspension pressure is transferred through the membrane area in terms of effective stress to the soil skeleton.

In case the pore size of the soil exceeds the size of the suspended bentonite particles, the suspension penetrates completely into the pore space of the ground up to a certain depth [Walz, 2001]. Due to the yield point of the bentonite suspension, shear stresses are transferred along the surface of the soil particles within the penetration zone (b). The penetration process stagnates in a certain depth, when the suspension pressure, transfer of shear stress and groundwater pressure are balanced (Figure 2 (left)).

Mueller-Kirchenbauer 1977] describes the direct relation between the mechanism of the pressure transfer and the penetration behavior of the bentonite suspension in the subsoil in reference to the pore size in detail.

**Figure 1:** Theoretical principle of support pressure transfer in the soil due to formation of a filter cake [Zizka & Thewes, 2015] (left) and experimental result of filter cake on macroscale [Imerys, 1998] (right).
Figure 2: Theoretical principle of support pressure transfer in the soil due to formation of a penetration zone [Zizka & Thewes, 2016] (left) and experimental result of a penetration zone on macroscale [Imerys, 1998] (right).

Based on long-term practical experience, the theoretical principles are widely accepted [Anagnostou & Kovári 1994, Krause 1987, Boere 2001, Bezuijen 2001, Zizka & Thewes 2015] and in addition proven by several experimental expertise on the macroscale [IBECO, Min et.al. 2013, Heinz 2006, Arwanita 2009] (Figure 1 (right), Figure 2 (right)).

In this study the penetration process is visualized and analyzed on the microscale using µ-CT for the first time. The analysis provides detailed information concerning the interaction between the bentonite suspension and the non-cohesive media within the pore space. Furthermore, the µ-CT imaging provides the determination of parameters e.g. porosity, pore size, pore size distribution and particle size distribution. By analyzing the contact angle between the fluid and the glass beads, the bentonite suspension is identified as a “wetting fluid” with a contact angle <90°. Both principles – filter cake and penetration – are identified and analyzed using µ-CT imaging so that these phenomena are validated on the microscale.

As an important result, the single phenomena of the filtration process of the bentonite suspension can be demonstrated in detail. Beside the “standard” identification of solid particles (glass beads), air and the penetrated bentonite suspension, the filtered bentonite particles in the filter cake and the filtrated suspension water are detected.

2 Materials & Methods

2.1 Materials

Bentonite is a natural clay. Main component of bentonite is the plate like clay mineral Montmorillonite. A single Montmorillonite crystal consists of 15-20 elementary layers. Between these elementary layers different cations (e.g. Na⁺, Ca²⁺, Mg²⁺) are adsorbed. In case of Na⁺ ions the bentonite is called Sodium bentonite, in case of Ca²⁺ ions it is a Calcium bentonite.

Preparation of a bentonite suspension consists of three steps: The powdery bentonite is suspended in water (1) and dispersed by introducing high shear forces (2). The shear forces separate the single crystal layer mechanically and distribute them homogeneously in the suspension. Due to an additional swelling process (3), water molecules are embedded between the elementary layers of the Montmorillonite crystal. These water molecules are adsorbed at the cations and at the surface of the single layers as well. Hence, the distance...
between the layers increases and the volume of the dispersed/suspended solids changes. This break-up of the layer corpuses is essential for the rheological properties of the bentonite suspension to develop. The required swelling time of different bentonites varies between 4 - 16 hours. Afterwards the particle size of the suspended Na- and Ca-bentonite particles can be determined (Appendix 1). In the experimental study bentonite suspension with varying solid contents were employed: Ca-bentonite in 25 % by weight, Na-bentonite in 8 % and 13 % by weight.

Glass beads with particle size of 2 mm and 600 µm and a mean density of 2600 kg/m³ were used to ensure the reproducibility of the performed combinations of bentonite suspensions and non-cohesive media. The surface structure of the glass beads was determined using SEM (Figure 3). Here, small parts of unevenness were detected.

**Figure 3**: Image of the surface condition of the 2 mm glass beads using Scanning Electron Microscopy (SEM)

The penetration tests were conducted in test tubes made of acrylic glass (Ø 37 mm and length 160 mm) and silica glass (Ø 21 mm and length 200 mm) in order to provide the most suitable material for the µ-CT scans. The label of each sample describes the container type, the type and concentration of bentonite suspension and the size of the glass beads. Table 1 shows the combinations for scanning with µ-CT, which provide the performance of a filter cake or the penetration process.

**Table 1**: Combinations of test tube material, bentonite suspensions and glass beads size for CT scans

<table>
<thead>
<tr>
<th>Material of test tube</th>
<th>Bentonite type + solid content</th>
<th>Size of glass beads</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample 1</td>
<td>silica glass Calcium 25 %</td>
<td>2 mm</td>
</tr>
<tr>
<td>Sample 2</td>
<td>silica glass Calcium 25 %</td>
<td>600 µm</td>
</tr>
<tr>
<td>Sample 3</td>
<td>silica glass Calcium 25 %</td>
<td>2 mm + 600 µm</td>
</tr>
<tr>
<td>Sample 4</td>
<td>silica glass Sodium 8 %</td>
<td>600 µm</td>
</tr>
<tr>
<td>Sample 5</td>
<td>acrylic glass Calcium 25 %</td>
<td>2 mm</td>
</tr>
<tr>
<td>Sample 6</td>
<td>silica glass Sodium 13%</td>
<td>2 mm + 600 µm</td>
</tr>
</tbody>
</table>
2.2 Preliminary laboratory experiments

In preparation of the µ-CT scans, fundamental tests were performed in the bentonite laboratory at Ruhr-University Bochum to identify and determine the influence of different parameters on the penetration behavior of the bentonite suspensions into glass beads. Furthermore, the penetration depth of the suspension has to be limited to some extent in order to ensure a high quality of the µ-CT scans due to smaller areas of interest. This is a challenging task because bentonite suspensions are non-Newtonian fluids with a yield point and exhibit thixotropic behavior [Luckham & Rossi 1999, Maxey 2007, API RP 13B]

Here, the bentonite suspensions were prepared with varying solid contents of Ca\(^{2+}\) in 20 % and 25 % by weight and Na\(^{+}\) in 8 % and 13 % by weight. All suspensions are combined with the glass beads of 2 mm, 600 µm and a combination of 2 mm + 600 µm. After swelling times of 24, 48, 72, 96, 120 and 192 hours the penetration tests were performed using test tubes of acrylic glass and silica glass and the penetration depth was measured.

The suspension made of Ca 25 % penetrates into the glass beads size of 2 mm, 600 µm and the combination 2 mm + 600 µm. The highest penetration depth is reached in the coarse material of 2 mm; the penetration depths in 600 µm and the combination of 2 mm + 600 µm are comparable. Comparison of the penetration depth measured in the acrylic glass and silica glass tubes shows slight differences of the absolute values for the same swelling times (Figures 4 and 5).

The suspensions made of Ca\(^{2+}\) 20 % and Na\(^{+}\) 8 % show high values of penetration depth within glass beads of 600 µm and the combination of 2 mm + 600 µm (Figures 4 and 5). This area is too large for a µ-CT scan of high quality. Therefore, the solid content of Na\(^{+}\) suspension was increased to 13 % and the Ca 25 % suspension was chosen for further testing. Following general observations can be made:

- The penetration depth in the glass beads decreases with increasing swelling time of the bentonite suspension. Here, Ca\(^{2+}\) bentonite shows a distinct response in terms if a reduced penetration depth in comparison to Na\(^{+}\) bentonite.

- The general performance of the penetration behavior is irrespective of the material of the test tube within a swelling time up to 120 hours. Slight differences of absolute values of penetration depth are detected. Bentonite suspensions with swelling times beyond 120 hours should be refused for the µ-CT scans.

- The penetration depth of the same bentonite suspension in glass beads of 600 µm and the combination of 2 mm + 600 µm are equal. The penetration depth in glass beads of 2 mm is higher by trend.
Figure 4: Development of penetration depth (cm) of bentonite suspension in glass beads using test tubes made of acrylic glass for increasing swelling times (h)

Figure 5: Development of penetration depth (cm) of bentonite suspension in glass beads using test tubes made of quartz glass for increasing swelling times (h)

2.3 μ-CT Imaging

X-ray computed tomography was used for the 3D imaging of the samples, using a nanotom S 180 μ-CT (tube characteristics: 180 kV, 500 mA) device, of the Leibniz Institute for Applied Geophysics (Tab. 2). The nanotom is a compact CT system for pore scale imaging purposes, i.e. for high resolution imaging within the micrometer (typically 1-2 μm) to sub-micrometer range (about 700 nm for very small samples), featuring high image sharpness due to a significantly reduced penumbra effect [Brunke et al. 2008]. A comprehensive overview of μ-CT imaging and 3D image reconstruction is given by Buzug (2010). The 3D image data were processed with the AVIZO Fire software suite (Visualization Sciences Group). Due to the low image noise and due to the fact that only few phases exist for segmentation, phase segmentation processing has been performed by the fast and robust “automatic threshold selection method” described by Otsu (1979).
2.4 Image Processing

The preparation and execution of the µ-CT scans followed a standardized procedure:

1. Sample preparation;
2. Sample installation and µ-CT setup;
3. µ-CT scan;
4. 3D data reconstruction;
5. Image processing and analysis.

Here, the glass beads are filled in the test tube using a funnel equipment with a specific height to provide a constant dense packing or density. Afterwards, the bentonite suspension was added and the penetration process took place. The time period for finishing this process is short (< 2 min). The test tube is installed and the µ-CT device, i.e. detector and tube setup, are prepared individually (Table 2). All samples have been positioned such way, that the interfacial surface area between the suspension – glass bead – air filled void space has been investigated as a region of interest. After data reconstruction, the 3D data sets have been processed with the AVIZO Fire software suite. Image processing has been performed for all samples as followed:

1. Data filtering (either using a median or a non local means filter [Ohser & Schladitz, 2009]);
2. Phase segmentation (void space, suspension, matrix);
3. Pore space analysis (pore separation, pore size distribution, porosity);
4. Grain size analysis (grain separation, grain size distribution);
5. Suspension distribution analysis (spatial extent, air inclusions and contact angles).

Table 2: Parameters of detector and tube setup

<table>
<thead>
<tr>
<th>CT values</th>
<th>Sample 1</th>
<th>2 Ca 25 % silica glass</th>
<th>3 Ca 25 % acrylic glass</th>
<th>5 Na 8 % silica glass</th>
<th>4 Na 13% silica glass</th>
</tr>
</thead>
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<tr>
<td>Voltage [kV]</td>
<td>105</td>
<td>105</td>
<td>105</td>
<td>125</td>
<td>125</td>
</tr>
<tr>
<td>Current [µA]</td>
<td>75</td>
<td>75</td>
<td>75</td>
<td>95</td>
<td>95</td>
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<tr>
<td>Timing [ms]</td>
<td>750</td>
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<td>750</td>
<td>750</td>
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<td>3</td>
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<tr>
<td>Image Number [-]</td>
<td>1000</td>
<td>1000</td>
<td>1000</td>
<td>1200</td>
<td>1500</td>
</tr>
</tbody>
</table>

3 Results

3.1 Porosity and pore size distribution of non-cohesive media

The theoretical model of the penetration process of a bentonite suspension into non-cohesive media depends - amongst others - on the porosity and permeability. In a soil mechanical sense, porosity is a dimensionless number that quantifies the existing pore space in reference to the whole soil volume. Here, the pores may be completely or partly filled with air, bentonite suspension or water. In addition, permeability is a measure for the
connectivity of the single pores. A high permeability is associated to a high porosity, but from a high porosity a high permeability cannot be deduced. A high porosity may be based on a huge amount of small pores or on the contrary on a small amount of big pores [Engelhard 1951]. In general, the porosity is determined by the shape, grading and compactness of the non-cohesive media / soil particles.

Basically, porosity is identical for samples with the same volume, particle shape, particle grading and compactness/density. For determination of porosity using µ-CT imaging, three samples were chosen: Sample 1: silica glass – Ca 25 % - 2 mm, Sample 2: silica glass – Ca 25 % - 600 µm, Sample 3: silica glass – Ca 25 % - 2 mm + 60µm (Figure 6). The analyzed volume was identical; the non-local-means filter was applied. Table 3 presents information concerning the resulting porosity, pore size and quantity of pores.

Figure 6: 3D-illustrations of pore space of glass beads of Sample 1 (2 mm – left), Sample 2 (600 µm – center) and Sample 3 (2 mm + 600 µm – right).

The porosity obtained from µ-CT imaging fits well the experimental determined values. The latter were determined by comparison of the weight of materials under dry and water-saturated conditions. Here, distilled water with a density of 1000 kg/m³ was used.

Table 3: Results of analyzing porosity and pore size of glass beads 2 mm, 600 µm and combination 2 mm + 600 µm

<table>
<thead>
<tr>
<th>Sample</th>
<th>Porosity numerical</th>
<th>Diameter of pore space [mm]</th>
<th>Quantity of pores</th>
<th>Threshold value</th>
<th>Porosity experimental</th>
</tr>
</thead>
<tbody>
<tr>
<td>1: Silica glass Ca 25 % 2 mm</td>
<td>39.60 %</td>
<td>1.097 1.176 1.071</td>
<td>395</td>
<td>9703</td>
<td>41.40 %</td>
</tr>
<tr>
<td>2: Silica glass Ca 25 % 600 µm</td>
<td>39.28 %</td>
<td>1.369 0.746 0.726</td>
<td>355</td>
<td>7014</td>
<td>40.80 %</td>
</tr>
<tr>
<td>3: Silica glass Ca 25 % 2 mm + 600 µm</td>
<td>34.59 %</td>
<td>0.209 1.457 0.782</td>
<td>259</td>
<td>13701</td>
<td>34.08 %</td>
</tr>
</tbody>
</table>

Comparing the porosity values in Table 3 shows evidence for the general laws of soil mechanics: glass beads with only one particle size of 2 mm or 600 µm show nearly identical values of porosity of 39.60 % (2 mm) and 39.28 % (600 µm) due to the same volume and the same bulk density. Thereby, the 2 mm glass beads provide a smaller quantity of pores due to a higher median value of the pore size diameter, while the 600 µm glass beads provide a higher pore quantity due to smaller median value of the pore size diameter.

In contrast, the particle size of the glass beads of Sample 3 contains a ratio of 50 % / 50 % of 2 mm / 600 µm. Porosity is smaller due to the fact, that the smaller glass beads of 600 µm fit into the pore space of the bigger glass beads of 2 mm. Therefore, the porosity of this mixture reduces to 34.59 %.
The characterization of the pore space concerning pore size, porosity and thus permeability are evident for the analysis of the penetration behavior of the bentonite suspension. Therefore, the histograms of pore size distribution are transferred into a diagram showing the size of the pores in reference to the proportion of the pores for glass beads of 2 mm (Sample 1), 600 µm (Sample 2) and combination of 2 mm + 600 µm (Sample 3) in Figure 7.

![Figure 7: Pore size distribution of glass beads 2 mm (Sample 1), 600 µm (Sample 2) and combination 2 mm + 600 µm (Sample 3).](image)

**Figure 7**: Pore size distribution of glass beads 2 mm (Sample 1), 600 µm (Sample 2) and combination 2 mm + 600 µm (Sample 3).

### 3.2 Segmentation of penetration depth and filter cake

#### 3.2.1 Sample 1: Ca 25 % - silica glass – 2 mm

Sample 1 contains a viscous bentonite suspension Ca 25 %. The penetration depth was determined optically and by using imaging with application of module “Measurement”. Due to an even penetration performance, both results show the same value of 15 mm penetration depth of the suspension into the pore space of the 2 mm glass beads packing (Figure 8). The visualization of the sample shows artefacts below the penetration zone in the area of dry glass beads. From the optical point of view an explicit boundary exist between the pore space filled with bentonite suspension and the air filled pores. Due to the coarse material the penetration effect took place. Following the visualization and analysis of the 3D data, it can be demonstrated that the suspension flows as a homogenous fluid into the pores and stagnates at the depth of 15 mm.
This effect was revised by investigating the three-phase situation in a histogram (Figure 9). This Histogram represents a vertical line through the sample and shows different grey values of the detected media. Here, the glass beads have the highest density and therefore show the highest grey value. The lower the density of the medium, the lower the grey value in the histogram. Analyzing the grey value using the module “Line Probe” shows three different values of glass beads, bentonite suspension and air within the pore space (Figure 9). The suspension is identified at nearly the same representative values. This feature indicates that the concentration/solid content of the suspension remains constant within the pore space, the suspension stays as a homogenous fluid within the pores.

Sample 2 contains the same viscous bentonite suspension as Sample 1. Due to an uneven penetration process, the visible penetration depth of 5 mm (0.5 cm) is smaller than the penetration depth determined by applying the module “measurement” using imaging of 9 mm (0.9 cm). Furthermore, the suspension penetrates deeper in the middle of the test tube into
the pore space than at the visible edge (Figure 10, left). The small glass beads tend to be buoyant in the viscous fluid. Despite that, the bentonite suspension penetrates as a homogeneous fluid into the glass beads. Again, an explicit boundary exists between the pore space filled with bentonite suspension and the air filled pores.

Analyzing the grey value using the module “Line Probe” shows again three different values of glass beads, air and bentonite suspension within the pore space (Figure 10, right). The suspension remains at nearly the same representative value that means the concentration or solid content of the suspension is constant within the pore space (three phases).

![Figure 10: Sample 2, Identification of three phases: glass beads, bentonite suspension and air.](image)

### 3.2.3 Sample 3: Ca 25 % - silica glass – 2 mm + 600 µm

Sample 3 shows the penetration process of viscous bentonite suspension Ca 25 % into the combination of 2 mm + 600 µm. The uneven penetration process leads to the deviation of the visible penetration depth of 8 mm (0.8 cm) in comparison to the penetration depth determined by applying the module “measurement” between 6 mm (0.6 cm) and 8 mm (0.8 cm). The mean value of the penetration depth is comparable to the value of Sample 2 with glass beads of 600 µm. The visualization of the sample shows artifacts below the penetration zone in the area of dry glass beads (Figure 11, left). Some of the small glass beads tend to be buoyant too in the viscous fluid. In general, the bentonite suspension penetrates as a homogeneous fluid into the pore space of the glass beads. Again, an explicit boundary exists between the pore space filled with bentonite suspension and the air filled pores.

Analyzing the grey value using the module “Line Probe” shows again three different values of glass beads, air and bentonite suspension within the pore space (Figure 11, right). The suspension remains at nearly the same representative value that means the concentration or solid content of the suspension is constant within the pore space (three phases).
3.2.4 Sample 4: Na 8% - silica glass – 600 µm

In Sample 4 the Na 8% suspension penetrates into glass beads 600 µm and results in a penetration depth of 23 mm (Figure 12, left). This value is quite high in comparison to the penetration depth of 5 mm (0.5 cm) of Ca 25% in Sample 2 in the 600 µm glass beads. On the one hand, the Ca 25% suspension is the more viscous fluid due to the higher solid content. On the other hand, the determination of the bentonite particle size (Appendix 1) shows a mean value of 10 µm for Ca-particles and of 7 µm for Na-particles. Therefore, the Na bentonite suspensions are predestined to move deeper into a pore space of the same size than the Ca bentonite suspension. Furthermore, in Figure 12 (left) it is visible that a certain amount of water filters out of the bentonite suspension.

This phenomenon can be validated by analyzing the grey value using the module “Line Probe”. Here, four different values for glass beads, air, bentonite suspension and water are identified on the vertical sketch (Figure 12, right). The amount of water is very low and concentrates on a very limited area within the test tube. As it can be seen from Figure 12, the water flows at the inner tube walls. The histogram in Figure 12 shows a nearly constant value of the bentonite suspension. It appears that the concentration/solid content of the suspension does not change significantly within the pore space.
3.2.5 Sample 5: Ca 25 % - acrylic glass – 2 mm

Sample 5 shows the penetration process of viscous bentonite suspension Ca 25 % into 2 mm glass beads whereas the test tube is made of acrylic glass. The even penetration process leads to the visible penetration depth of 15 mm (1.5 cm) and to the penetration depth of 15 mm (1.5 cm) determined by applying the module “Measurement” (Figure 13). This value matches the penetration depth of Sample 1 using Ca 25 % and glass beads of 2 mm in a test tube made of silica glass (Figure 8). It provides evidence that the material of the test tube has no influence on the penetration depth.

The visualization of Sample 5 shows artifacts below the penetration zone in the area of dry glass beads (Figure 13). Some of the small glass beads tend to be buoyant too in the viscous fluid. In general, the bentonite suspension penetrates as a homogeneous fluid into the pore space of the glass beads. Again, an explicit boundary exists between the pore space filled with bentonite suspension and the air filled pores.

The analysis of the grey value using the module “Line Probe” shows an additional phase of water below the end of the penetration zone of the suspension within the pore space (Figure 13). The water does not appear at the walls of the test tubes, it is located inside the glass beads.

The µ-CT scanning is based on the detection of media with different densities. The larger the difference in density, the easier the single media can be identified. However, the density value of water 1000 kg/m³ is close to the density of the Ca 25 % suspension 1025 kg/m³. The left side of the histogram (Figure 13) shows the grey value of the bentonite suspension that is placed above the area of glass beads (sample length approx. 0 - 2500 µm). Within the pore space of the glass beads (sample length approx. 5000 – 16000 µm), the grey value of the bentonite suspension is slightly higher. In addition, the filtrated water shows a lower grey value close to the original bentonite suspension. This gives evidence that a filter process took place within the pore space. Here, the suspension water is separated in a small amount from the bentonite particles. The particles remain in the pore space, the filtrate water drains into the pore space below. The solid content/density of the bentonite suspension within the pores increases gently, in place where some suspension water is filtrated.

![Figure 13: Sample 5, identification of four phases: glass beads, bentonite suspension, water and air using Histogram.](image-url)
3.2.6 Sample 6: Na 13% - silica glass – 2 mm + 600 µm

In Sample 6 the penetration process of bentonite suspension Na 13% performs into the combination of 2 mm + 600 µm glass beads in a test tube of silica glass. Here, the infiltration leads to a filter cake performance at the entrance of the pore space of glass beads. The bentonite particles are filtered from the suspension, the bentonite particles attach gradually at the pore access and the filtrate water of the suspension drains through the glass beads (Figure 14). Therefore, the visualization of Sample 6 shows no penetration depth within the analysis using the module “measurement”.

![Figure 14: Sample 6: 3D embodiment of the area of filter cake as an assembly of solid material with a closed surface (left) and apparent accumulation of bentonite particles at the pore access (right).](image1)

The Histogram in Figure 15 shows the increase of the density of the bentonite suspension within a short area/depth followed by the detection of the filtrate water. The grey value of the bentonite suspension above the level of glass beads is shown. Within the pore space of the glass beads the grey value of the bentonite suspension is slightly higher than the filtrate water shows a lower grey value. This gives evidence that the filtration process of the bentonite particles took place at the pore access. The suspension water is separated from the bentonite particles. The particles remain in the pore space, the filtrate water drains into the pore space below. The solid content/density of the bentonite suspension within the pores increases, which is verified by increasing grey values.

![Figure 15: Sample 6, identification of four phases: glass beads, bentonite, filtrate water and air, especially the density of the bentonite suspension varies.](image2)
3.4 Contact angle of bentonite suspensions and glass beads

In general, the contact angle determined using µ-CT imaging helps to classify the manner of interaction between a fluid and the surface of a solid, e.g. type of fluid, material and surface roughness of solid. A contact angle of 90° is the limit between a wetting and non-wetting fluid. Contact angles over are typical for Mercury, contact angles less than 90° present the behavior of Water (Figure 16).

Figure 16: General classification of a non-wetting fluid (contact angle > 90°, left), partial wetting fluid (contact angle = 90°, center) and completely wetting fluid (contact angle < 90°, right).

For the bentonite suspension Ca 25 % the contact angles are determined using the module “angular measurement” for the different glass beads 2 mm (Sample 1), 600 µm (Sample 2) and 2 mm + 600 µm (Sample 3). Table 4 shows the results of contact angles. In general, the contact angle for bentonite suspension Ca 25 % is smaller than 90°. The suspension is classified as a wetting fluid.

Table 4: Results of determination of contact angles between bentonite suspension and glass beads in Sample 1 Ca 25 % 2 mm, Sample 2 Ca 25 % 600 µm and Sample 3 Ca 25 % 2 mm + 600 µm in test tubes of silica glass.
4 Discussion

In this study, two different bentonites Ca\(^{2+}\) and Na\(^{+}\) were used. Samples 1, 2, 3 and 5 contain Ca-bentonite with solid content of 25 %. In Sample 1 and 5 the bentonite suspension penetrates into glass beads 2 mm and both tests perform identical values for penetration depth of 15 mm. It can be shown, that the material of the test tube - silica glass or acrylic glass – does not influence the result of the penetration test (Figure 8 and 12).

Using the smaller glass beads of 600 µm and the combination of 2 mm + 600 µm, the penetration depth reduces considerably (Figure 10 and 11). As shown in Figure 7, the distribution of the pore size is nearly identical for glass beads of 600 µm and the combination of 2 mm + 600 µm. In general, the pore size ranges between 0.3 mm – 1.5 mm with a peak around 0.7 mm. The range of pore size for the 2 mm glass beads is between 0.6 mm – 2.1 mm with a peak around 0.9 mm. As a consequence, the same bentonite suspension Ca 25 % with a particle size of approximately 10 µm (Appendix 1) penetrates deeper into the glass beads 2 mm with larger pore size (Figure 17).

<table>
<thead>
<tr>
<th>Sample</th>
<th>Contact Angle</th>
<th>Penetration behaviour between</th>
<th>Determination using AVIZO</th>
</tr>
</thead>
<tbody>
<tr>
<td>1: Silica glass 25 % 2 mm</td>
<td>46.69°</td>
<td>1.17 mm</td>
<td><img src="image1" alt="AVIZO Image" /></td>
</tr>
<tr>
<td>2: Silica glass Ca 25 % 600 µm</td>
<td>61.25°</td>
<td>0.367 mm</td>
<td><img src="image2" alt="AVIZO Image" /></td>
</tr>
<tr>
<td>3: Silica glass Ca 25 % 2mm + 600 µm</td>
<td>61.01°</td>
<td>0.515 mm</td>
<td><img src="image3" alt="AVIZO Image" /></td>
</tr>
<tr>
<td>4: Silica glass Ca 25 %</td>
<td>29.88°</td>
<td>0.421 mm</td>
<td><img src="image4" alt="AVIZO Image" /></td>
</tr>
</tbody>
</table>
The Histogram of Samples 1, 2, and 3 identifies three different phases: glass beads, bentonite suspension and air within the pore space. Here, the bentonite suspension penetrates as a homogeneous fluid into the pores of the coarse material. Within this study, this is defined as the “standard penetration behavior” and can be linked directly to the experimentally performed penetration zones in Figure 2 (right) on the macroscale. For the first time, this penetration behavior of bentonite suspension into coarse material is visualized on microscale using μ-CT.

According to Appendix 1, Na-bentonites show a size of approximately 3 µm. Therefore, the penetration depth of Na 8 % into glass beads of 600 µm in Sample 4 reaches the largest value of the conducted test series 23 mm (see also Figure 5 and Figure 6). The larger the particles of Ca-bentonite (approximately 10 µm) the smaller the penetration depth into the same size of glass beads of Sample 2 and Sample 3.

Exceptionally, four different phases are detected within the histogram of Sample 4: glass beads, bentonite suspension, air and filtrated water at the lower area of the penetration depth. In addition, the histogram of Sample 5 identifies the same phases: glass beads, bentonite suspension, air and filtrated water. Compared to Sample 5, the area of filtrated water within Sample 4 is quite large. This may be due to the lower solid content of Na 8 % (Sample 4) compared to Ca 25 % (Sample 5).

Sample 6 using Na 13 % and glass beads of combination 2 mm + 600 µm performs uniquely the filter cake within this test series. The bentonite particles remain at the "entrance" of the pore space of the glass beads and the filtrated suspension water flows deeper into the tube. The filter cake is illustrated in the 3D embodiment of Figure 14. Furthermore, the Histogram shows a varying density of the bentonite suspension within the limited filtration area at the pore space “entrance”. For the first time, the performance of a filter cake of bentonite suspension is visualized on microscale using μ-CT.

From the gained results of the six samples, a general classification concerning the performance of a penetration zone and of a filter cake cannot be derived. Furthermore, the reasons of the filtration of water within the penetration effect as seen in Sample 4 and 5 using different types of bentonite cannot be explained satisfyingly. Here, further research work is needed.

Figure 17: Average diameter of pore space [mm] within glass beads 2 mm, 600 µm and combination 2 mm + 600 µm in reference to the determined penetration depth [cm].
5 Conclusions

The aim of this study is the visualization and analysis of the penetration behavior of bentonite suspensions in non-cohesive granular material on microscale using µ-CT scanning.

The widely accepted scenarios of filter cake formation and entire penetration of the suspension into the pore space were conducted experimentally in test tubes using different combinations of bentonite suspension and granular material (glass beads). These phenomena were scanned with high-resolution µ-CT technique. The 3D embodiment of the different samples were analyzed concerning soil mechanical aspects, e.g. particle size distribution, pore size distribution, porosity of the granular material, and concerning the interaction of the bentonite suspension within the pore space, e.g. contact angle, penetration depth and filter cake thickness.

These effects are verified by investigating the different phase situation in a histogram. The Histogram represents a vertical line through the sample and shows different grey values of the detected media. The lower the density of the medium, the lower the grey value in the histogram. Here, the histogram of each sample offers the identification of single phases: glass beads, bentonite suspension and air as well as the filter cake at the “entrance” of the pore space of the glass beads or the variation of density of a bentonite suspension filtrating within the penetration depth.

Sample 1, 2 and 3 show the penetration behavior of bentonite suspension into coarse material is visualized on microscale using µ-CT. Three phases - glass beads, bentonite suspension and air - are detected in the Histogram. An additional phase – water filtrated from the suspension - is identified in Sample 4 and 5. The performance of a filter cake of bentonite suspension is visualized in Sample 6 on microscale using µ-CT. The agglomeration of the bentonite particles at the entrance of the pore space results in the development of a filter cake. This area is identified as a distinct media. Furthermore, the histogram of Sample 6 shows a varying density of the bentonite suspension within the limited filtration area at the pore space “entrance”.

In summary, the µ-CT technique delivers a valuable contribution for the research on the interaction of bentonite suspensions penetration the pore space of non-cohesive media. This study shows the missing visual evidence concerning the theoretical interaction models of the bentonite suspension in the pore space on microscale.

Acknowledgements

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References


Appendix

Appendix 1: Distribution of particle size (µm) of Na<sup>+</sup> (blue line) and Ca<sup>2+</sup> bentonite particles within the suspension (red line) in reference to quantity (%).