



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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13 Abstract

Bentonite suspensions are an essential tool for different construction techniques in horizontal 14 15 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 16 17 collapsing during the excavation process of trenches, drill holes or tunnels. In order to 18 maintain the soil stability close to the excavation, the bentonite suspension has to counteract 19 against the earth and water pressure. Therefore, the pressure acting in the suspension has 20 to counter the groundwater pressure and to be transferred into an effective stress to support the soil skeleton. 21

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.

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38 <u>Keywords:</u> μ-CT, non-cohesive porous media, bentonite suspensions, non-Newtonian fluid,
 39 penetration depth, filter cake





40 **1** Introduction

41 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 42 43 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 44 45 maintain the soil stability close to the excavation, the bentonite suspension has to counteract 46 against the earth and water pressure. Therefore, the pressure acting in the suspension has 47 to counteract the groundwater pressure and has to be transferred into effective stress to 48 support 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)).

66 [Mueller-Kirchenbauer 1977] describes the direct relation between the mechanism of the 67 pressure transfer and the penetration behavior of the bentonite suspension in the subsoil in 68 reference to the pore size in detail.



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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]

72 (right).







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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.at. 2013, Heinz 2006, Arwanitaki 2009] (Figure 1 (right), Figure 2 (right)).

82 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 83 84 between the bentonite suspension and the non-cohesive media within the pore space. 85 Furthermore, the µ-CT imaging provides the determination of parameters e.g. porosity, pore 86 size, pore size distribution and particle size distribution. By analyzing the contact angle 87 between the fluid and the glass beads, the bentonite suspension is identified as a "wetting 88 fluid" with a contact angle <90°. Both principles - filter cake and penetration - are identified 89 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.

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95 2 Materials & Methods

96 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

118 SEM (Figure 3). Here, small parts of unevenness were detected.



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

122 The penetration tests were conducted in test tubes made of acrylic glass (Ø 37 mm and 123 length 160 mm) and silica glass (Ø 21 mm and length 200 mm) in order to provide the most 124 suitable material for the μ -CT scans. The label of each sample describes the container type, 125 the type and concentration of bentonite suspension and the size of the glass beads. Table 1 126 shows the combinations for scanning with μ -CT, which provide the performance of a filter 127 cake or the penetration process.

128	Table 1: Combinations of test tube material,	bentonite suspensions	s and glass beads size for	CT scans
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	Material of	Bentonite type +	Size of glass
	test tube	solid content	beads
Sample 1	silica glass	Calcium 25 %	2 mm
Sample 2	silica glass	Calcium 25 %	600 µm
Sample 3	silica glass	Calcium 25 %	2 mm + 600 μm
Sample 4	silica glass	Sodium 8 %	600 µm
Sample 5	acrylic glass	Calcium 25 %	2 mm
Sample 6	silica glass	Sodium 13%	2 mm + 600 μm

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131 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 extend 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.

144 The suspension made of Ca 25 % penetrates into the glass beads size of 2 mm, 600 μ m and 145 the combination 2 mm + 600 μ m. The highest penetration depth is reached in the coarse 146 material of 2 mm; the penetration depths in 600 μ m and the combination of 2 mm + 600 μ m 147 are comparable. Comparison of the penetration depth measured in the acrylic glass and 148 silica glass tubes shows slight differences of the absolute values for the same swelling times 149 (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, Ca²⁺ bentonite shows a distinct response in terms if a reduced
 penetration depth in comparison to Na⁺ bentonite.

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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.

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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.







167

- 168 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)



170

171 **Figure 5:** Development of penetration depth (cm) of bentonite suspension in glass beads using test 172 tubes made of guartz glass for increasing swelling times (h)

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174 2.3 μ-CT Imaging

X-ray computed tomography was used for the 3D imaging of the samples, using a nanotom 175 176 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, 177 178 i.e. for high resolution imaging within the micrometer (typically 1-2 µm) to sub-micrometer 179 range (about 700 nm for very small samples), featuring high image sharpness due to a 180 significantly reduced penumbra effect [Brunke et al. 2008]. A comprehensive overview of u-CT imaging and 3D image reconstruction is given by Buzug (2010). The 3D image data were 181 182 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 183 184 segmentation processing has been performed by the fast and robust "automatic threshold 185 selection method" described by Otsu (1979).

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188 2.4 Image Processing

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

- 190 1. Sample preparation;
- 191 2. Sample installation and μ-CT setup;
- 192 3. μ-CT scan;
- 193 4. 3D data reconstruction;
- 194 5. Image processing and analysis.

Here, the glass beads are filled in the test tube using a funnel equipment with a specific 195 height to provide a constant dense packing or density. Afterwards, the bentonite suspension 196 was added and the penetration process took place. The time period for finishing this process 197 198 is short (< 2 min). The test tube is installed and the μ -CT device, i.e. detector and tube setup, 199 are prepared individually (Table 2). All samples have been positioned such way, that the 200 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 201 processed with the AVIZO Fire software suite. Image processing has been performed for all 202 samples as followed: 203

- Data filtering (either using a median or a non local means filter [Ohser & Schladitz, 205
 2009]);
- 206 2. Phase segmentation (void space, suspension, matrix);
- 207 3. Pore space analysis (pore separation, pore size distribution, porosity);
- 208 4. Grain size analysis (grain separation, grain size distribution);
- 5. Suspension distribution analysis (spatial extent, air inclusions and contact angles).
- 210 **<u>Table 2:</u>** Parameters of detector and tube setup

Sample	1	2	3	5	4	6
		Ca 25 %		Ca 25 %	Na 8 %	Na 13%
		silica glas	S	acrylic glass	silica glass	silica glass
CT values	2 mm	600 µm	2 mm + 600 µm	2 mm	600 µm	2 mm + 600 μ m
Voltage [kV]	105	105	105	125	125	125
Current [µA] 75		75	75	95	95	95
Timing [ms]	750	750	750	750	750	750
Averaging [-]	3	3	3	4	4	4
Skip [-]	1	1	1	1	1	1
Image Number [-]	1000	1000	1000	1200	1500	1500

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213 **3 Results**

214 3.1 Porosity and pore size distribution of non-cohesive media

The theoretical model of the penetration process of a bentonite suspension into noncohesive 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: <u>Sample 1</u>: silica glass – Ca 25 % - 2 mm, <u>Sample 2</u>: silica glass – Ca 25 % - 600 μ m, <u>Sample 3</u>: silica glass – Ca 25 % - 2 mm + 60 μ m (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.



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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 watersaturated 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

Sample	Porosity	osity Diameter of pore space [mm]			Quantity	Threshold	Porosity	
	numerical	Min.	Max.	Average	Median	of pores	value	experimental
1: Silica glass Ca 25 % 2 mm	39.60 %	0.597	2.081	1.167	1.071	81	9703	41.40 %
2: Silica glass Ca 25 % 600 μm	39.28 %	0.377	1.369	0.746	0.726	355	7014	40.80 %
3: Silica glass Ca 25 % 2 mm + 600 μm	34.59 %	0.209	1.457	0.782	0.783	259	13701	34.08 %

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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 / 600μ m. Porosity is smaller due to the fact, that the smaller glass beads of 600μ m 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
- 254 1), 600 μm (Sample 2) and combination of 2 mm + 600 μm (Sample 3) in Figure 7.

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257 Figure 7: Pore size distribution of glass beads 2 mm (Sample 1), 600 μm (Sample 2) and combination
 258 2 mm + 600 μm (Sample 3).

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260 **3.2** Segmentation of penetration depth and filter cake

261 3.2.1 Sample 1: Ca 25 % - silica glass - 2 mm

Sample 1 contains a viscous bentonite suspension Ca 25 %. The penetration depth was 262 determined optically and by using imaging with application of module "Measurement". Due to 263 264 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 265 visualization of the sample shows artefacts below the penetration zone in the area of dry 266 267 glass beads. From the optical point of view an explicit boundary exist between the pore 268 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 269 be demonstrated that the suspension flows as a homogenous fluid into the pores and 270 271 stagnates at the depth of 15 mm.







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274 **Figure 8:** Sample 1, 3D embodiment of penetration depth.

This effect was revised by investigating the three-phase situation in a histogram (Figure 9). 275 276 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 277 278 the highest grey value. The lower the density of the medium, the lower the grey value in the 279 histogram. Analyzing the grey value using the module "Line Probe" shows three different 280 values of glass beads, bentonite suspension and air within the pore space (Figure 9). The 281 suspension is identified at nearly the same representative values. This feature indicates that 282 the concentration/solid content of the suspension remains constant within the pore space, the suspension stays as a homogenous fluid within the pores. 283



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285 **Figure 9:** Sample 1: Identification of three phases: glass beads, bentonite suspension and air.

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287 3.2.2 Sample 2: Ca 25 % - silica glass – 600 μm

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).



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301 Figure 10: Sample 2, Identification of three phases: glass beads, bentonite suspension and air.

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303 **3.2.3 Sample 3: Ca 25 % - silica glass – 2 mm + 600 μm**

304 Sample 3 shows the penetration process of viscous bentonite suspension Ca 25 % into the 305 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 306 determined by applying the module "measurement" between 6 mm (0.6 cm) and 8 mm (0.8 307 308 cm). The mean value of the penetration depth is comparable to the value of Sample 2 with 309 glass beads of 600 µm. The visualization of the sample shows artifacts below the penetration 310 zone in the area of dry glass beads (Figure 11, left). Some of the small glass beads tend to 311 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 312 between the pore space filled with bentonite suspension and the air filled pores. 313

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).







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319 Figure 11: Sample 3: Identification of 3 phases: glass beads, bentonite suspension and air.

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321 3.2.4 Sample 4: Na 8 % - silica glass - 600 µm

322 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 323 penetration depth of 5 mm (0.5 cm) of Ca 25 % in Sample 2 in the 600 µm glass beads. On 324 325 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) 326 327 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 328 329 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. 330

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.



339 Figure 12: Sample 4, identification of four phases: glass beads, bentonite suspension, water and air.





340 **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 357 358 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 359 360 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 361 space of the glass beads (sample length approx, 5000 - 16000 µm), the grev value of the 362 363 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 364 365 took place within the pore space. Here, the suspension water is separated in a small amount 366 from the bentonite particles. The particles remain in the pore space, the filtrate water drains 367 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. 368



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Figure 13: Sample 5, identification of four phases: glass beads, bentonite suspension, water and air
 using Histogram.





373 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".



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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).

384 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 385 386 bentonite suspension above the level of glass beads is shown. Within the pore space of the 387 glass beads the grey value of the bentonite suspension is slightly higher than the filtrate 388 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 389 the bentonite particles. The particles remain in the pore space, the filtrate water drains into 390 the pore space below. The solid content/density of the bentonite suspension within the pores 391 increases, which is verified by increasing grey values. 392



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Figure 15: Sample 6, identification of four phases: glass beads, bentonite, filtrate water and air,
 especially the density of the bentonite suspension varies.

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398 **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).



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405 <u>Figure 16:</u> General classification of a non-wetting fluid (contact angle > 90°, left), partial wetting fluid
 406 (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 μ m (Sample 2) and 2 mm + 600 μ m (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.

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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.

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Sample	Contact Angle	Penetration behaviour between	Determination using AVIZO
1: Silica glass 25 % 2 mm	46.69°	1.17 mm	
2: Silica glass Ca 25 % 600 μm	61.25°	0.367 mm	Angle 81.25
• •	34.76°	0.461 mm	Anglet 34.76
3: Silica glass Ca 25 % 2mm + 600 μm	61.01°	0.515 mm	Line: 515,15
•	29.88°	0.421 mm	Links 42(1 33

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430 **4 Discussion**

In this study, two different bentonites Ca²⁺ 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 436 penetration depth reduces considerably (Figure 10 and 11). As shown in Figure 7, the 437 distribution of the pore size is nearly identical for glass beads of 600 µm and the combination 438 of 2 mm + 600 µm. In general, the pore size ranges between 0.3 mm - 1.5 mm with a peak 439 440 around 0.7 mm. The range of pore size for the 2 mm glass beads is between 0.6 mm - 2.1 441 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 442 beads 2 mm with larger pore size (Figure 17). 443







444

445 Figure 17: Average diameter of pore space [mm] within glass beads 2 mm, 600 μm and combination
 446 2 mm + 600 μm in reference to the determined penetration depth [cm].

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.

454 According to Appendix 1, Na-bentonites show a size of approximately 3 μ m. Therefore, the 455 penetration depth of Na 8 % into glass beads of 600 μ m in Sample 4 reaches the largest 456 value of the conducted test series 23 mm (see also Figure 5 and Figure 6). The larger the 457 particles of Ca-bentonite (approximately 10 μ m) the smaller the penetration depth into the 458 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 μ m 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.





478 **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.

481 The widely accepted scenarios of filter cake formation and entire penetration of the 482 suspension into the pore space were conducted experimentally in test tubes using different combinations of bentonite suspension and granular material (glass beads). These 483 phenomena were scanned with high-resolution µ-CT technique. The 3D embodiment of the 484 different samples were analyzed concerning soil mechanical aspects, e.g. particle size 485 distribution, pore size distribution, porosity of the granular material, and concerning the 486 487 interaction of the bentonite suspension within the pore space, e.g. contact angle, penetration 488 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.

496 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 497 suspension and air - are detected in the Histogram. An additional phase - water filtrated from 498 the suspension - is identified in Sample 4 and 5. The performance of a filter cake of bentonite 499 suspension is visualized in Sample 6 on microscale using µ-CT. The agglomeration of the 500 501 bentonite particles at the entrance of the pore space results in the development of a filter 502 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 503 504 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.

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590 Appendix



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592 <u>Appendix 1:</u> Distribution of particle size (μ m) of Na⁺ (blue line) and Ca²⁺ bentonite particles within the 593 suspension (red line) in reference to quantity (%)