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Summary of the Doctoral thesis

Investigation of the thermo-hydro-mechanical behavior of Czech bentonite used as a model material for planning of high level nuclear waste disposal

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Abstract

The work involved in the thesis is mainly focused on the Czech bentonite which is originally from Cerny vrch deposit (north western region of the Czech Republic). The compacted bentonites are prepared from the industry provided bentonite powder with an initial water content around 10%. Dry densities from 1.27 to 2 g/cm^3 were used for laboratory testing, specifically 1.27, 1.60 and 1.90 g/cm^3 were used for water retention measurements, microstructures and fractal pore analysis. Dry densities of 1.25 to 1.95 g/cm^3 were used for mechanical tests such as one dimensional swelling strain and oedometer load-unload tests. The vapor equilibrium method was used to impose the suction on samples ranging from 3.29 MPa to 286.7 MPa. Mercury intrusion porosimetry (MIP) and environmental scanning electron microscope (ESEM) were utilized for the microstructure analysis. The water retention measurements were performed at 20, 40, 60 and 80 °C respectively, results show that the increasing of temperature can decrease the water retention capacity. The influence of compaction and suction on microstructure was compared and studied. MIP tests were performed on the samples which were equilibrated at suction of 3.29, 38 and 286.7 MPa on wetting path of both low and high dry densities. The samples equilibrated at suction of 286.7 MPa on wetting path were firstly observed in ESEM chamber with different magnification, then followed by wetting and drying path performed in the chamber with increasing and decreasing relative humidity. The changes in aggregates and macropores were recorded. The pore families (macropores and micropores) recognized by the MIP results were consistent with the ESEM observation. Moreover, the influence of dry density and suction on the microstructure was studied by fractal analysis with different methods. Fractal analysis confirmed the pore families definition by MIP pore size distribution curve. The mechanical study of compacted bentonite showed a unique relationship between (aggregate) dry density and effective stress. The proposed equations for predicting swelling pressure were developed based on the diffuse double layer (DDL) theory, which was proved to be applicable in both sodium and calcium bentonites. The prediction of swelling pressure of Czech bentonite was presented and discussed.

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1. Introduction

With the increasing utilization of nuclear energy, the wasted high level spent nuclear fuel must be properly and safely stored. The most appropriate way to handle these high level nuclear waste is the deep geological disposal, which is usually excavated in depths of 400 - 500 meters in the natural rock formations. The principle of deep geological disposal is to isolate the waste from the human being biosphere environment for more than 1 million years until its radioavtivity decayes to harmless levels. Considering the safety of the deep geological disposal, barriers are usually used to keep the system independent. Bentonite material was popularly chosen as the barriers, thanks to its low permeability, high adsorption potention and high swelling pressure. Bentonite is a clay of high montmorillonite content. The compacted bentonite is used as engineered barrier material between canister and surrounding rocks, while the bentonite pellets is planned to be used as backfill materials to fill the disposal tunnels. The initially unsaturated compacted bentonite near the rock will be hydrated by the underground water, while the bentonite near the canister will be heated by the radioactivity generated by the spent fuel. When the bentonite adsorbs water, the swelling pressure will be generated. The swelling properties are related to the type and the initial state of bentonite. Thus, understanding the thermo-hydro-mechanical behavior of bentonite is necessary for the properly choosing of barrier materials and safety operation of the nuclear waste disposal.

Over the past decades, bentonite has been an extensively studied material. It is now well accepted that the compacted bentonite has a structure with two distinct pore systems, denoted as a double structure (Gens & Alonso, 1992; Alonso et al., 1999, 2010). The double structure plays an important role in the thermal-hydro-mechanical behavior of compacted bentonite and it is explicitly considered in many bentonite constitutive models. The microstructure is investigated using mercury intrusion posorimetry (MIP) (Wang et al., 2014; Přikryl & Weishauptová, 2010; Monroy et al., 2012) and an environmental scanning electron microscopy (ESEM) method (Romero, 1999; Romero and Simms, 2008), which is supplemented by water retention measurements (Dieudonne et al., 2017; Villar, 2007).

2. Aims of the study

The work involved in this thesis is targeting at better understanding of the behavior of the Czech bentonite at various conditions. The aim of this thesis is to study the thermo-hydro-mechanical properties of Czech bentonite by laboratory tests and modeling, which are key in dealing with the backfill materials or the engineered barrier systems utilized in planned high level nuclear waste disposal galleries. The water retention properties, microstructure characteristics and mechanical behavior of the Czech bentonite are presented in this thesis. The results obtained in the thesis can further be used for bentonite material evaluation and also as input data to simulate the behavior of bentonite performed in mock-up tests by modeling.

3. Material and methods

The thesis mainly focus on the Czech bentonite extracted from the Cerny vrch deposit (north-western region of the Czech Republic) and commercially supplied in the form of powder, but B75 was specially treated by slight vitrification only. The initial water content of the bentonite powder was around 10%. The montmorillonite content was about 60%. The plastic limit, liquid limit and specific gravity of the solid are 65%, 229% and 2.87, respectively.

The vapor equilibrium method (Delage et al. 1998) was applied to control the total suction. Relative humidity in the closed desiccator was controlled by different saturated salt solutions, the values of which were dependent on temperature. In this thesis, eight different salt solutions were used. The total suction can be calculated by the Kelvin equation by knowing the value of relative humidity. The temperature was imposed at 20, 40, 60 and 80 °C respectively. The 20 °C tests were performed in the air condition room with constant temperature. Other higher temperatures tests were done in a temperature controlled box, in which the desiccators were placed in. The suction varied from 3.29 MPa to 286.7 MPa (20 °C).

Mercury intrusion porosimetry (MIP) is based on the capillary law governing non-wetting liquid penetration into small pores. The pore entrance diameter (D) can be determined from the applied mercury pressure (P) by assuming that the cylindrical pores existed in soil according to Washburn equation. The MIP tests were performed at the Department of Inorganic Technology at the University of Chemistry and Technology Prague (Apparatus Autopore IV, Micromeritics). The measurement was done in two regimes, one was the low pressure regime from 0.01 MPa to 0.2 MPa (corresponding the pore radius between 100 μm and 3 μm); another one was the high pressure regime from 0.2 MPa to 400 MPa (corresponding the pore radius between 3μm to 1.5nm). MIP tests were conducted on freeze dried samples, which can keep its original structure. In freeze drying methods, the samples were firstly immersed into the liquid nitrogen to quickly freeze to preserve its original structure, then the frozen samples were placed under deep vacuum. Finally, the samples went through sublimation in the vacuumed chamber of a freeze dryer.

The environmental scanning electron microscope (ESEM) is widely used for the observations of micro-fabric. And the ESEM has advantages with respect to traditional SEM, the former can be used for observation under different relative humidity, while the latter is only suitable for completely dry samples which only can be observed at the atmospheric pressure and laboratory temperature. The Environmental Scanning Electron Microscopy (ESEM) tests have been performed using QUANTA 650 FEG scanning electron microscope at the Institute of Scientific Instruments of the Czech Academy of Sciences, Brno, Czech Republic. Conditions of observation were kept constant throughout the experiment. The compacted bentonite samples were observed at different magnifications (from 800 to 50000 times) at different states. Especially, the bentonite samples with different initial dry densities were observed following wetting and drying path at the magnification of 2500 times. The water vapor pressure of 93 Pa (relative humidity of 10%) was determined as an optimal initial state for the experiment. Then the vapor pressure was gradually increased up to 850 Pa (relative humidity 97%). After the maximum value of the relative humidity was reached, the relative humidity was gradually decreased again down to 10%. The microphotographs were taken at each stage. The time interval between vapor pressure changes was 15 minutes.

The conventional oedometer apparatus was used for measuring the swelling deformation of Czech bentonite B75. The compacted bentonite was tested in the standard fixed stainless-steel ring, 50 mm inside diameter and 20 mm height. Silicone grease was applied to the inner wall of the stainless-steel ring to reduce friction between the specimen and the wall. Once the compacted bentonite was introduced in the stainless-steel ring, the prescribed vertical stress was applied. Then the specimen was infiltrated by distilled water. The vertical deformation and time were recorded. For saturated oedometer test, the loading and unloading test continued after the swelling deformation test. Eventually, the water content of the specimen was measured. The ASTM D2435/D2435M standard recommends the correction for oedometer apparatus compressibility. The deflection of the apparatus was measured by substituting a smooth hard steel disk for the soil specimen before the experiment for all the test conditions.

4. Results and discussion

4.1 Water retention properties

The water retention curves of three dry densities of 1.27, 1.60 and 1.90 g/cm^3 at 20 °C, 40 °C, 60 °C, 80 °C are shown in Figure 1 (a-f) respectively. It can be seen that the water content decreased with increasing temperature for the same initial dry density. This trend was more obvious at low suctions. Comparison of wetting and drying paths showed that wetting curves at high suctions (above 100 MPa) exhibited almost no temperature dependence for all dry densities. The effect of temperature was generally more clear in drying paths. The decrease of

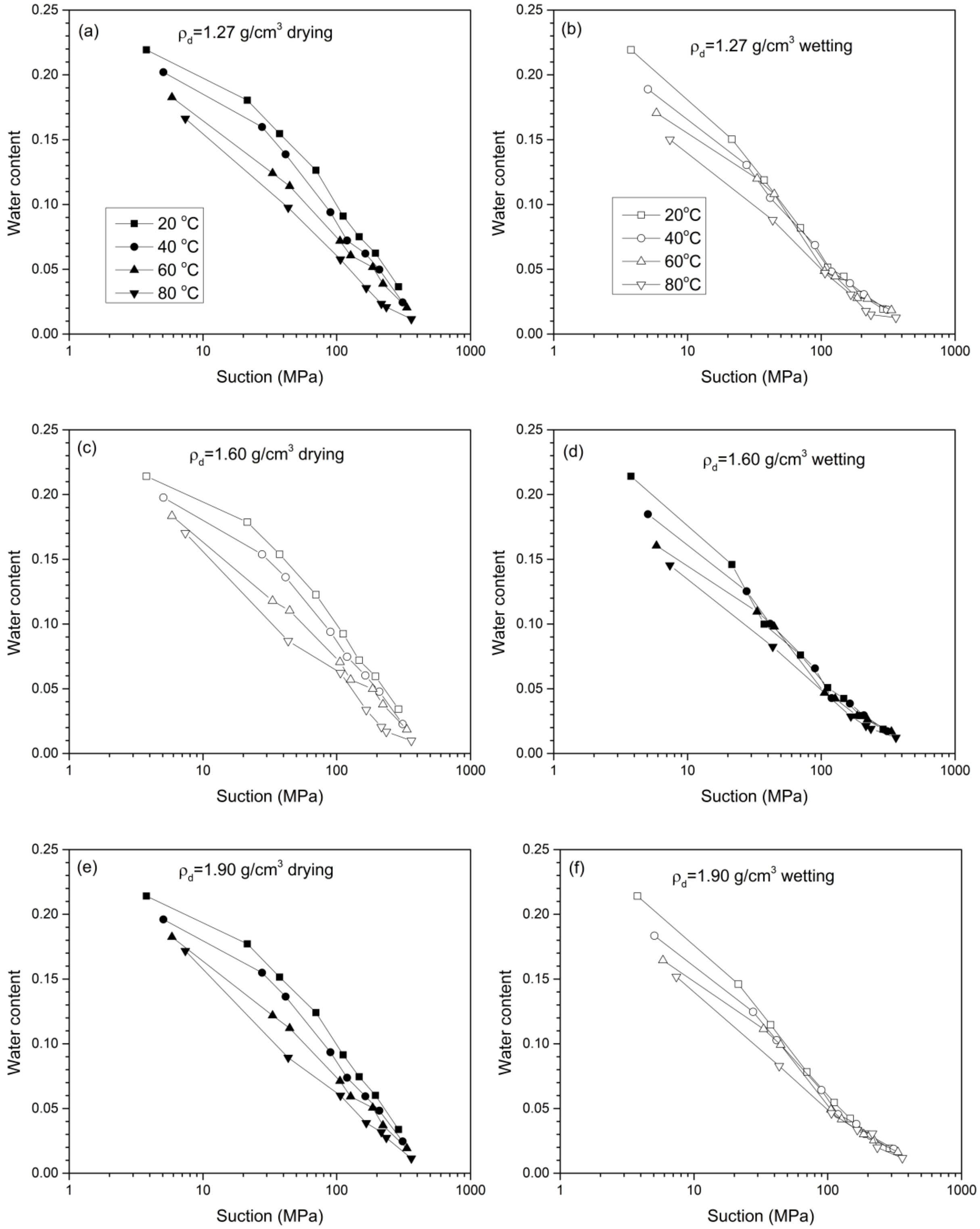

Figure 1. Water retention curves at various temperatures for dry density of 1.27 g/cm^3 ((a) - drying path; (b) – wetting path), 1.6 g/cm^3 ((c) - drying path, (d) – wetting path) and 1.9 g/cm^3 ((e) - drying path, (f) – wetting path)

water retention capacity with increasing temperature was influenced by the water surface tension, the liquid-gas interfacial tension or contact angle between water and soil. The increased temperature can also drive volumetric changes of the aggregates. These volumetric changes can include both expansion and contraction, depending on initial dry density and applied suction (Villar and Lloret, 2004). These structural changes were associated with the transfer of the intra-aggregate water into inter-aggregate pores, which would explain the lower water retention capacity at a higher temperature at certain suction (Villar and Lloret, 2004). The presented results of Czech bentonite B75 were consistent with this theory and confirmed that the temperature has an important influence on the water retention capacity.

4.2 Microstructure evaluation

The micrographs of compacted bentonite equilibrated at the suction of 286.7 MPa with an initial dry density of 1.27 g/cm^3 was presented in Figure 2. The arrangement of the aggregates may clearly be seen, along with different pore families. The aggregates were clearly visible at lower magnification (Complete photo and Zoom 1), Zoom 2 and Zoom 3 then show details of the microstructure.

Figure 3 shows the MIP results of low (1.27 g/cm^3) and high (1.90 g/cm^3) density samples at different suction levels. It is clearly identifies the effect of suction on pore size density curves. The MIP data allows us to identify the effects of suction and compaction level on the individual pore sizes. We can distinguish three pore size domains:

1) "Nanopores": smaller than 3nm. These pores can be attributed to the inter-layer of clay sheets. They can't be detected by the MIP technique and thus they are not indicated in Figure 3.

2) "Micropores": between 3nm and micro-macro transition pore radius, which is found to be suction dependent. In this paper, the upper limits of 0.07 μm, 0.15 μm and 0.3 μm were found to correspond to the suction of 286.7 MPa, 38MPa and 3.29 MPa respectively based on high density samples (in low density samples, the transition pore radius is not clear due to the dominant effect of macropores). It follows from Figure 3 that the largest micropores are influenced by suction and micropores are, in general, unaffected by compaction level.

3) "Macropores", which are larger than the micro-macro transition pore radius. Macropores up to approximately 2 μm are significantly influenced by compaction level and they are not significantly affected by suction. Contrary, macropores larger than approx. 2 μm are affected both by suction and by compaction.

Pore surface fractal dimension (D_s) was calculated based on Menger fractal dimension model. The calculated D_s value was dependent on the pore size range, the fractal dimension for dry densities of 1.27 and 1.90 g/cm^3 and all suctions were plotted in one graph shown in Figure 4. Both micropores and macropores can be clearly identified based on the fractal analysis. Inside the family of macro-pores, fine macropores and coarse macropores can be distinguished. Micropores were represented by parallel regression lines of all samples demonstrating similar values of surface fractal dimension D_s. It showed that this domain representing the pores inside the aggregates was compaction-insensitive. The size of the micro-pores domain was therefore not uniquely defined and depended on the value of suction. The macropores and micropores were defined based on the fractal analysis, which was consistent with the pore size distribution curves presented by Sun et al. (2018).

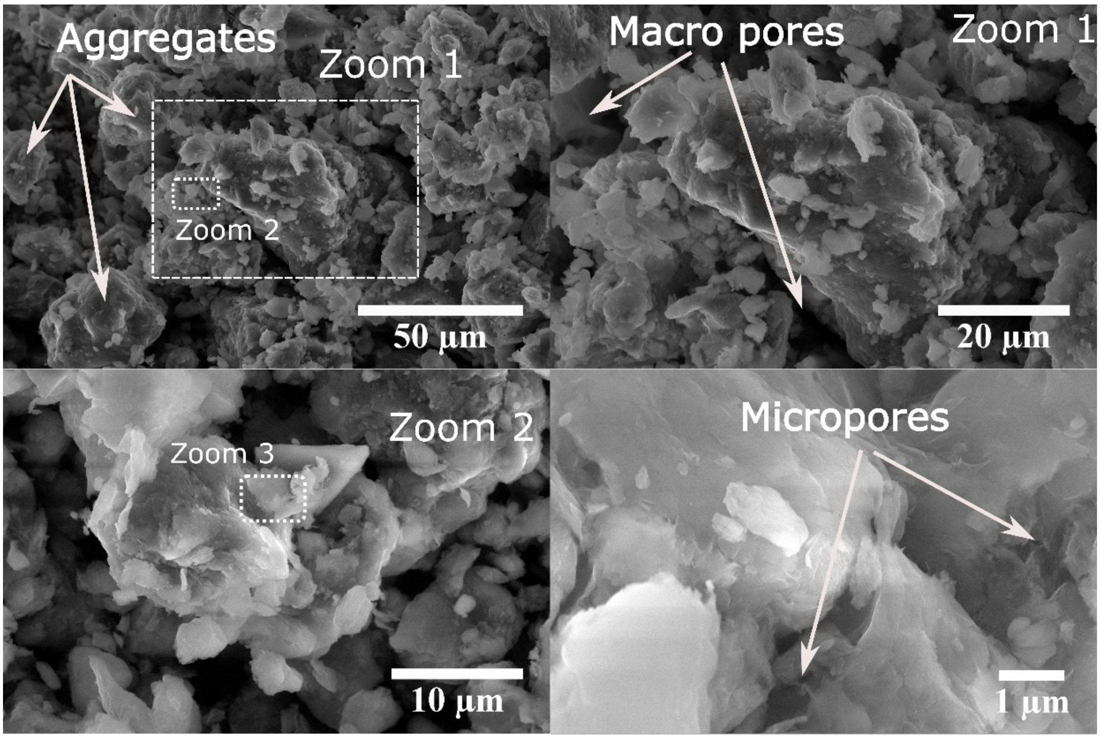

Figure 2. ESEM micrographs of compacted bentonite with a dry density of 1.27 g/cm^3 at different magnifications

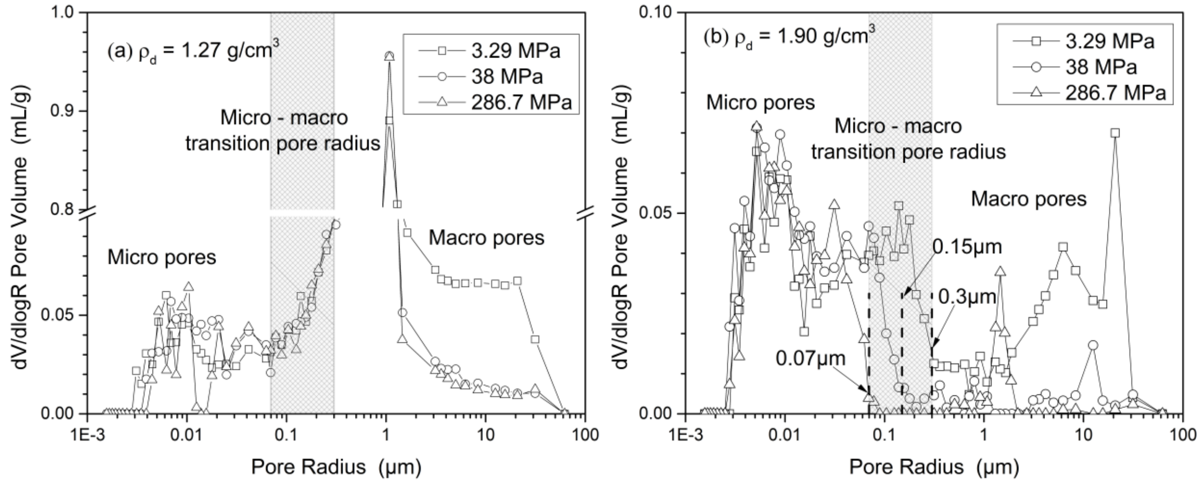

Figure 3. Pore size distribution curves – the effect of suction and indication of micro-macro transition pore radius: (a) $\rho_d = 1.27$ g/cm^3, (b) $\rho_d = 1.90$ g/cm^3

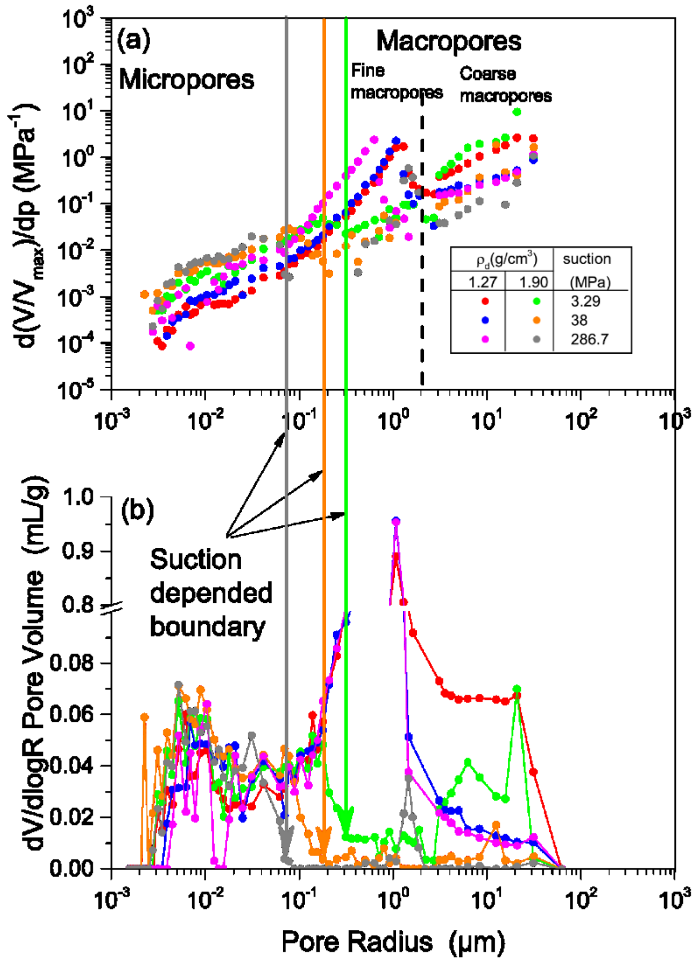

Figure 4. Fractal dimension (a) and pore size distribution (b) with pore radius under each suction level for both dry densities

4.3 Mechanical behavior

The swelling under constant load, the saturated oedometer unloading, the water retention measurement at wetting path were performed. The data of swelling at constant volume tests were obtained from Hausmannova and Vasicek (2014). Here, the dry density refered to the dry density of the aggregates, the method adopted for calculation was presented by Mašín and Khalili (2015). Fianlly, all the results are shown in Figure 5. A unique linear trend occurred between pressure and dry density, but swelling pressure tests gave higher pressures than other methods. This unique relationship showed that the bentonite deformation depended only on the effective stress, which was consistent with the Terzaghi's effective stress definition.

I summarized the following equations to calculate the swelling pressure based on the MX80 and FoCa bentonite. These equations were developed based on the diffuse double layer theory. Equations (1-2) were for sodium bentonite, while equations (3-4) were for calcium bentonite.

$$p=2n_0kT[\cosh(u_{theory}+\Delta u_{MX80})-1] \tag{1}$$

$$p=2n_0kT[\cosh((-3.462\log(Kd)+3.292)-1.729\ln(e+0.203))-1] \tag{2}$$

$$p=2n_0kT[\cosh(u_{theory}+\Delta u_{FoCa})-1] \tag{3}$$

$$p=2n_0kT[\cosh((-3.957\log(Kd)+2.869)-5.158\ln(e+0.153))-1] \tag{4}$$

Where e is the void ratio of compacted bentonite, d is the half of the distance of clay platelets (m), u is the nondimensional midplane potential, n_0 is the ionic concentration of the bulk fluid in ions/m^3, k is the Boltzmann's constant (1.38×10^{-23} J/K), K is the diffuse double layer parameter (1/m).

The swelling pressure of Czech bentonite was used to further to check the developed model. There were two types of bentonite, namely Sab65 bentonite which is a sodium bentonite, another was the B75 bentonite which is a calcium bentonite. The experimental data was obtained from Hausmannova and Vasicek (2014). Figure 6 shows the experimental data compared with the theoretical value and developed model predictions. It can be seen that the proposed model prediction is much better than the theoretical DDL values. However, the theoretical DDL values were predicted well when the dry density was lower than 1.55 g/cm^3, this phenomena was presented by Tripathy et al. (2004), Schanz and Tripathy (2009) and Sun (2018).

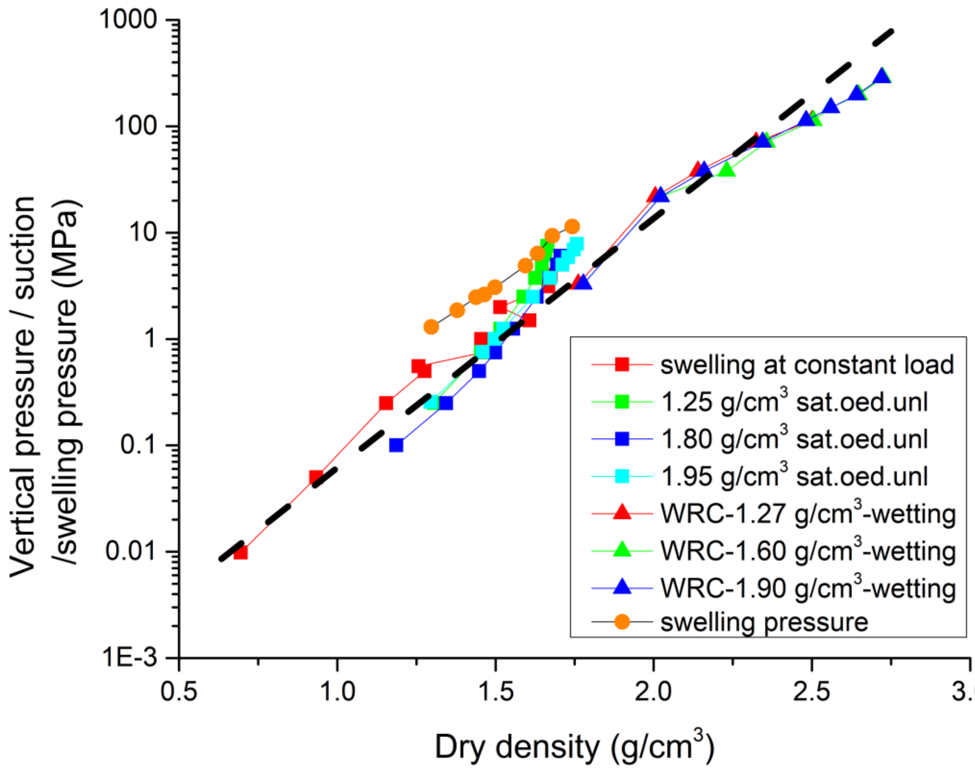

Figure 5. Swelling pressure (Hausmannova and Vasicek et al.,2014), oedometer unloading pressure, oedometer constant pressure and suction versus dry density

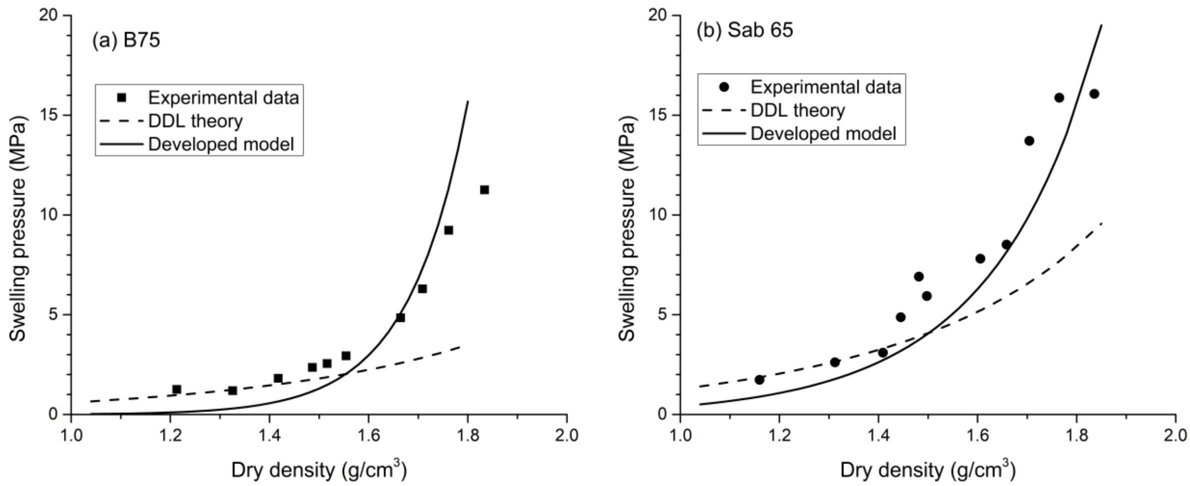

Figure 6. Theoretical DDL prediction, experimental and developed model for swelling pressure of Czech bentonite (a) B75 and (b) Sab65

5. Conclusions

In the thesis, I presented experimental and theoretical works on the water retention properties, microstructure characteristics and mechanical behavior of compacted Czech bentonite. The following conclusions can be drawn:

Water retention properties:

1) No variations of retention curves (water content vs. suction) with initial dry density was observed at 20°C and the same result was confirmed at higher temperatures. This conclusion is due to the fact that suctions adopted are quite high, where no water is between aggregates. The water retention capacity of compacted bentonite decreased with increasing temperature for all the dry densities. This trend is more obvious for drying paths. For wetting path and suctions over 100 MPa, no obvious change of the retention capacity with temperature was observed.

Microstructural analysis:

2) Apart from the inter-lamellar pores, which were not accessible to the adopted observation methods, two main pore families (macropores and micropores) could be identified. Their transition pore size was suction dependent (0.07 to 0.3 μm for suctions varying between 286.7 and 3.29 MPa). The micropores were practically insensitive to compaction and only the largest micropores were sensitive to suction. The smaller macropores were sensitive to compaction only, whereas the larger macropores were sensitive to both compaction and suction.

3) Two pore size domains (macropores and micropores) were defined based on the fractal analysis of MIP data. The macropores can be subdivided to fine macropores and coarse macropores. The zones influenced by suction and different compaction energy were identified. An estimation of pore geometry of different size domains can be made based on the value of fractal dimension D_s. Pore size domains determined from fractal analysis correlates with size domains obtained by visual evaluation of MIP distribution curves. The fractal analysis of MIP data thus proved to be a useful tool, which complements the information obtained from MIP distribution curves.

4) The fractal analysis using the box counting method on ESEM images proved high fractal characteristics of bentonite pore system. The fractal dimension decreases with increasing magnification due to more smooth and regular structures observed under high

magnifications. However, the fractal dimension increases again when approaching extremely high magnifications. The effect of freeze-drying and oven-drying was investigated, showing a higher fractal dimension of oven dried samples. This indicates a certain change of the pore structure of the samples exposed to oven drying.

Mechanical behavior:

5) The swelling-consolidation method gives a higher swelling pressure than constant volume method because of the crystalline expansion and osmotic expansion. The swelling pressure determined by constant volume method and dry density at saturation shows a good linear relationship in semi-logarithmic coordinates. This unique relationship showed that the bentonite deformation depends on the effective stress only, which is consistent with the Terzaghi's effective stress definition.

6) The newly proposed equations based on DDL theory were developed by consideration the variablity of void ratio. The swelling pressures calculated by proposed equations indicated a good agreement with the experimental swelling pressures. Moreover, this proposed model worked well for Czech Sab65 bentonite, while it was less accurate on Czech B75 bentonite at lower dry density range.

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7. Curriculum vitae

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01.10.2014 - present	*PhD candidate*, Faculty of Science, Charles University, Prague, Czechia
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01.09.2011- 01.07.2014	*Master degree* in engineering geology, Nanjing University, Nanjing, China
01.09.2007- 01.07.2011	*Bachelor degree* in engineering geology, Southwest Jiaotong University, Chengdu, China

8. Publications

Journal articles

1) **Sun, H**., Mašín, D., Najser, J., Neděla, V., & Navrátilová, E. (2019). Bentonite microstructure and saturation evolution in wetting–drying cycles evaluated using ESEM, MIP and WRC measurements. Géotechnique, 69(8), pp. 713-726, doi: doi.org/10.1680/jgeot.17.P.253

2) **Sun, H**. (2018). A new method to predict swelling pressure of compacted bentonites based on diffuse double layer theory. Geomechanics and Engineering, 16(1), 71-83. https://doi.org/10.12989/GAE.2018.16.1.071

3) **Sun, H**., Mašín, D. Najser, J., Neděla, V., & Navrátilová, E. (2019), Fractal characteristics of pore structure of compacted bentonite studied by ESEM and MIP methods, Acta Geotechnica, DOI: 10.1007/s11440-019-00857-z, (Accepted)

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2) **Sun, H**., Mašín, D., & Najser, J.(2018). Investigation of the mechanical behavior of compacted bentonite. The 7th International Conference on Unsaturated Soils Conference, Hong Kong

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