Swelling Properties and Permeability of Expandable Clays of Potential use for Nuclear Waste Disposal

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Abstract

Bentonite clay has attracted considerable attention as isolating material for safe disposal of high-level radioactive nuclear waste (HLW) on account of its low hydraulic conductivity and high swelling capacity. In the presently described study, three candidate smectitic clay materials were investigated and compared with respect to hydraulic conductivity and expandability. The clay samples were prepared from desiccated and crushed raw material placed and compacted in oedometer cells for saturation with distilled water and 3.5% $CaCl_2$ solution in separate test series to a density at fluid saturation of 1200 ~ 1900 kg/m3. The samples were tested with respect to the hydraulic conductivity and swelling capacity.

The MX-80 and Homehus clays matured as expected giving a successively monotonous increase in swelling pressure during the wetting process while GMZ clay showed two pressure peaks. The pressure development was different for low and high densities and can be explained by crystal expansion via interlamellar wetting, followed by osmotic swelling including establishment of electrical double-layers. The phenomena can alternatively be explained by the microstructural changes when clay minerals absorb solutions in the hydration phase. Differences in granule size distributions and mineral composition can explain different swelling abilities and permeabilities.

Keywords: Highly Radioactive Waste (HLW), clay, hydraulic conductivity, swelling pressure.

1 Introduction

Hydraulic conductivity, swelling and ion sorption capacity are properties that determine whether smectitic clay can be selected as isolation material for canisters with highly radioactive waste (HLW) and for use as seals in various parts of a repository for such

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waste. Compacted smectite-rich clay ("bentonite") can be an effective barrier to migration of radionuclides and for providing a ductile embedment of canisters [1]. Its constitution as raw material depends on the chemical and pressure conditions where the clay was formed. Chinese GMZ clay, which has a similar content of montmorillonite as American MX-80 clay, which is taken as reference material in the study, is considered as a possible material for preparing engineered barriers ("buffers") in Chinese HLW disposal program. Danish Holmehus clay, which is illite/smectite mixed layer but smectite-rich clay, represents another type of clay buffer that has also attracted considerable attention for isolation of HLW. In the present study, the swelling ability and hydraulic conductivity of these three clays prepared with distilled water and 3.5% $CaCl_2$ solution were determined and assessed.

Beishan, which is located in Gansu province in China, has been selected as one of several potential disposal sites. The groundwater here is rich in Na^+ and Ca^{2+} and the main chemical compounds $Cl,SO_4/Na$ and $Cl,SO_4/NaCa$ [2]. This will cause some change of the physical or chemical properties of the clay, and will thereby affect the role of buffer clay candidates to serve acceptably as buffer/backfill.

2 Material

2.1 Location and Origin

Holmehus clay beds are situated in the northern Lillebält area in Denmark. Several geologists have investigated the origin of this clay and concluded that it belongs to a Paleocene formation of Tertiary age comprising also the Æbelö and Rödbyhavn clays according to Foged& Bauman, [3, 4].

Gaomiaozibentonite (GMZ) originates from Xinghe county, Inner Mongolia Autonomous Region (113º58' to 114º06' E, 40º27' to 40º44'N), China (Figure 1.).   The total reserve is about 120 million tons of Na-bentonite. In the Gaomiaozi deposit, which represents a mining area about 72 km^2, five bentonite layers have been identified. The clay mass is homogeneous with uniform high smectite content, especially the part termed "the III layer bed" (Figure 2.).
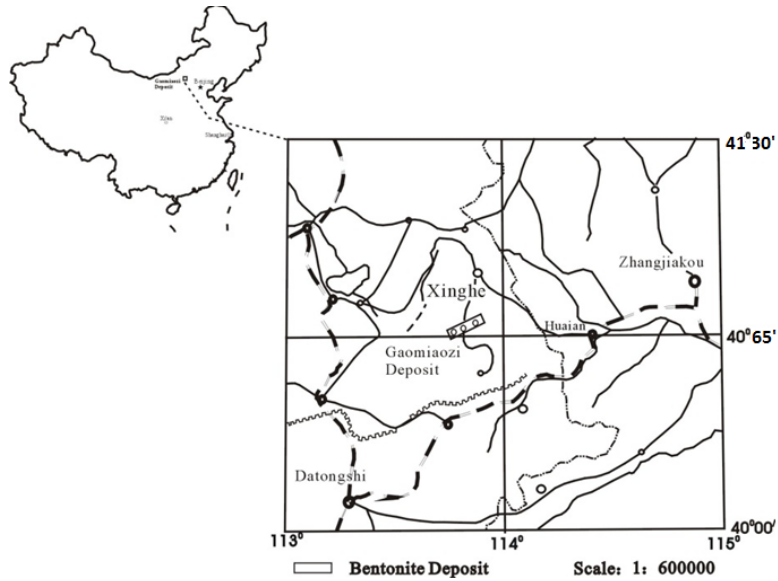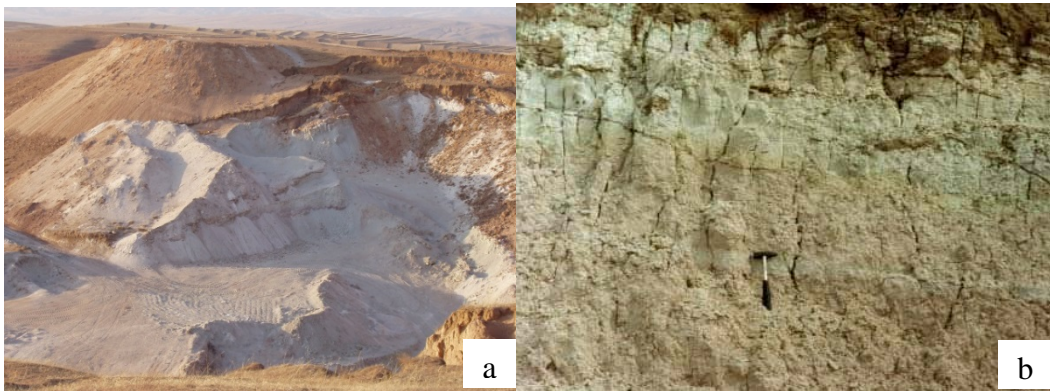
Figure 1: GMZ clay deposit location in China

Figure 2: GMZ clay beds. (a: open pits, b: the "III layer bed" outbowed.)

2.2 Basic Properties of Materials

2.2.1 Mineralogy

X-ray diffraction

Mineralogical characterization of the GMZ clay samples was made by X-ray diffraction analysis (Figure 3.) using a Rigaku D/MAX-IIIB diffractometer of East China University of Technology in China. The system uses CuKα radiation with a wavelength of 1.54Å also as secondary mono-chromator. The scanningangle, voltage and current intensity were 3-70° 2 θ, 40kV and 30 mA, respectively. Analysis of oriented samples of <2μm material in air-dry and ethylene-glycol treated forms have shown dominance of montmorillonite (75-78%) for GMZ clay, IS-ml (55%) for Holmehus clay [4] and montmorillonite (77-85%) for MX-80 clay [5]. Semiquantitative evaluations give additional essential components. (Table1.)
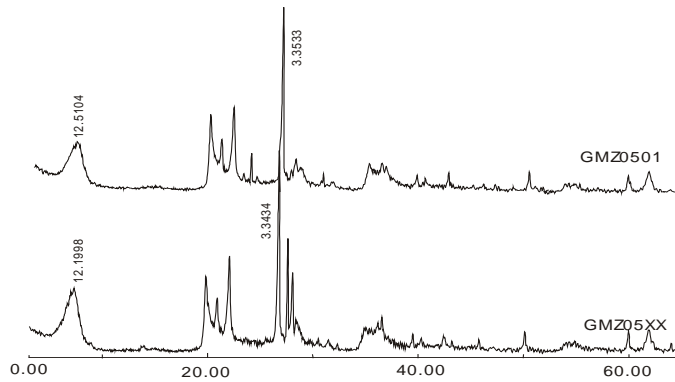
Figure 3: XRD pattern of GMZ clay.

Table 1: Mineralogical composition of virgin clay samples as determined by XRD. (Weight percentage)

Clay	Muscovite, 2M$_1$	Chlorite	Quartz	Cristobalite	Plagioclase	K-feldspar	Anatase	Gypsum	Jarosite	Pyrite	Feldspars
Holmehus	10-20	<5	10-15	-	<5	5	0.5	2	2	0.3	
MX-80 [5]	1	-	5	3	13	-	-	-	-	-	
MX-80 [6]	2	-	4	2	5	-	-	<1	-	-	5-8
MX-80 [7]	<1	-	15	-	5-8	-	-	-	-	0.3	4.3
GMZ			11.7	7.3	-						

FT-IR Analysis

FT-IR analyze of GMZ clay shows a strong infrared absorption peak at 800 cm^{-1} due to the existence of MgAl-OH bonds and hydration ion (mainly Na$^+$) that weakens H-O-HOH Mg Al bonds. The analysis verified that the clay is Na- montmorillonite clay (Figure 4.)
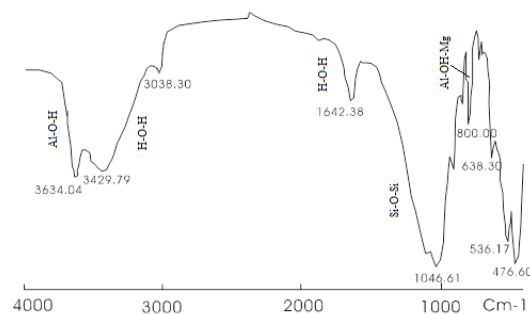

Figure 4: FT-IR of GMZ clay. (East China University of Technology)

2.2.2 Chemical composition and physical properties

The evaluated element composition of GMZ clay is compiled in Table 2, the corresponding data for Holmehus and GMZ clays being shown for comparison.

Table2: Element data of bulk clay samples. ([a,b] [4]; [c][8].)

Element	SiO_2	Al_2O_3	Fe_2O_3	MgO	CaO	Na_2O	K_2O
Holmehus, [a]	58.6	15.3	6.5	2.2	0.7	1.4	2.8
MX-80, [b]	63.6	19.8	5.0	3.2	3.1	2.8	1.0
GMZ, [c]	69.2	14.4	3.1	3.3	1.3	2.0	0.8

Essential physical properties and cation exchange capacity (CEC) of Holmehus, MX-80 and GMZ clay are shown in Table 3. The higher montmorillonite content agrees with the higher CEC (The Cu-triethylenetetramine method for Holmehus clay; the neutral NH_4Cl-ethyl alcohol method, [9]). It is also confirmed by the high Plasticity Index (I_P) of this clay. The dominance of sorbed Na explains their high liquid limits, the highest value represented by the MX-80 clay (W_L).

Table3: Cation exchange data and Atterberg consistency limits of the three clays [10], $\rho_s$is the specific gravity of soil grain.

Clay	CEC (meq/100 g)	Exchangeable cations	W_L	W_P	I_P	ρ_s
Holmehus	29.7-31.6	K, Na	234	29	205	2.58
MX-80	78.8±4.8	Na	519	35	484	2.76
GMZ	77.3	Na- Ca	313	38	275	2.66

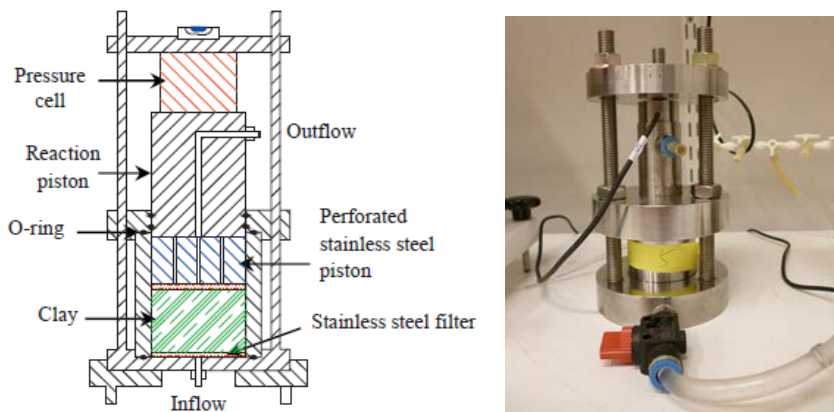## 3 Swelling Pressure and Hydraulic Conductivity Tests

Figure 5: Experimental setup for fluid saturation and determination of the hydraulic conductivity and swelling pressure [11]

Stainless steel oedometer cells were used for the experiments (Figure 5).Two sets of 2cm

high and 3cm diameter Holmehus clay blocks with saturated densities 1675, 1800, 1900 kg/m³ were compared; one saturated and permeated with distilled water and the other with 3.5% $CaCl_2$ solution. The permeation was made from the lower end of the cells. After saturating the samples, permeation started until stable flow was reached. The discharged permeant was collected in 100ml glass containers with rubber caps that had thin needlesfor keeping equal air pressure in and outside the containers. The hydraulic conductivity wasevaluated by using Darcy's law, the hydraulic gradient being maintained at 100m/m (meter water head difference over one meter flow length).Stiff pressure cells of 3-5kN load capacity were mounted on the oedometers for logging the swelling pressure evolved in the saturation phase [11]. The pressure was perfectly stable after 3 weeks using distilled water and around 2 weeks using the salt solution.

GMZ clay samples were prepared in the same way with the desired   water contents 15.1%, 19.6%, 23.9% corresponding to "wet" densities of 1200 ~ 1700 kg/m³. Figures 6-a, b, c, d give that the evolution curves of the swelling pressure of GMZ clay samples (water contents a, b=15.1%, c=19.6%, d=23.9%) for distilled water as a function of time. Figure 7 and 8 show the relationship between swelling pressure and hydraulic conductivity at saturationfor the three clays saturated with   distilled water and salt water (3.5% $CaCl_2$ solution), respectively. Examination of these graphs is described in Paragraphs4.1 respecting the swelling behavior, and 4.2 concerning the hydraulic behavior.

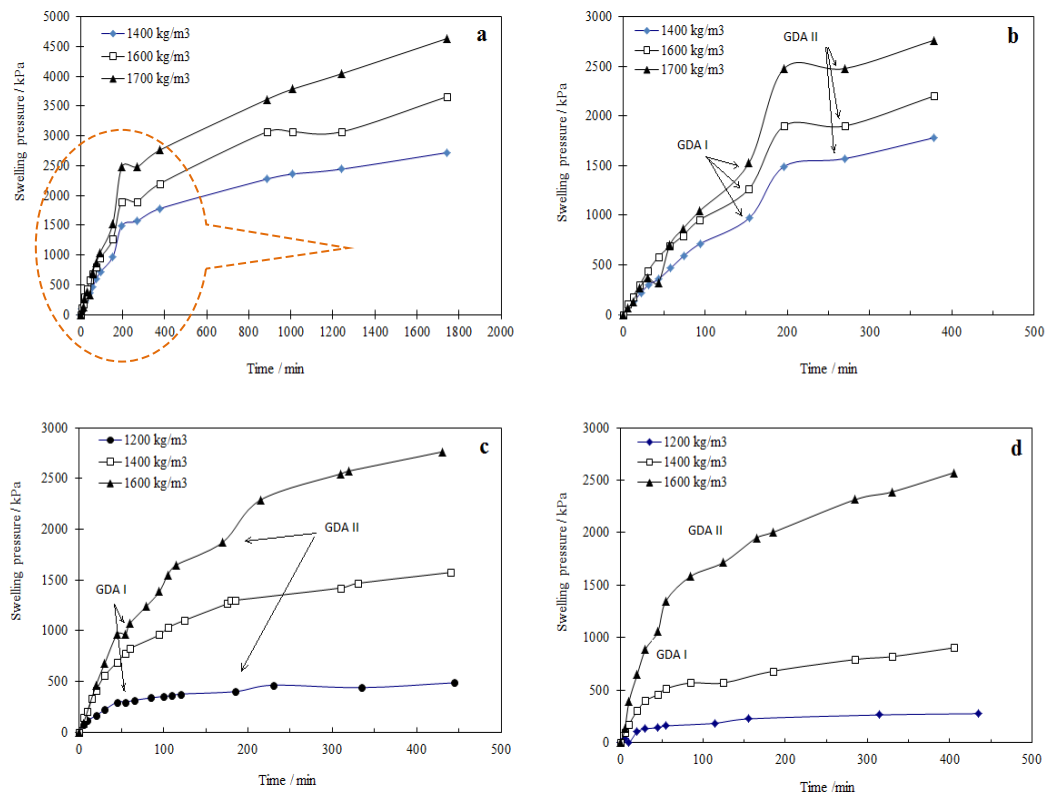Figure 6: Relationship between swelling pressure (p_s) and swelling time (T) of GMZ clay. a: completep_s -T curve when w%= 15.1, b: part p_s -T curve when w%= 15.1, c: part p_s -T curve when w%= 19.6, d: part p_s -T curve when w%= 23.9.
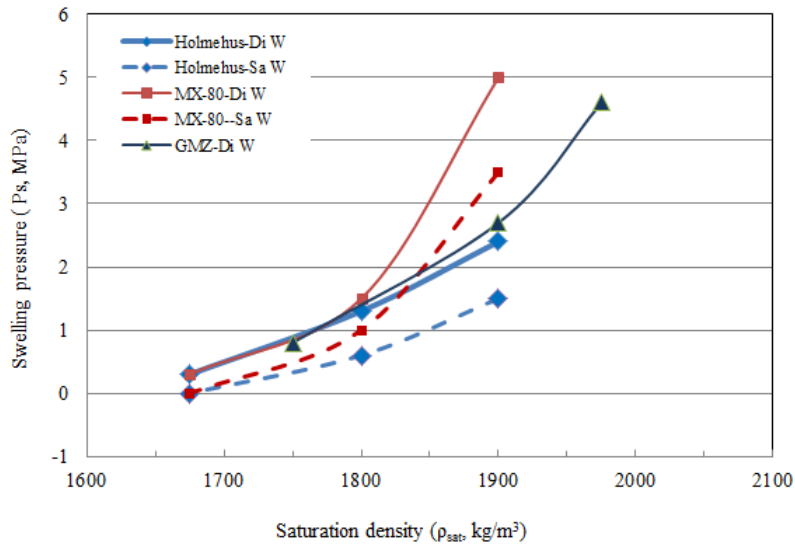
Figure 7: Relationship between swelling pressure and density at saturation. (-Di W is distilled water; -Sa W is salt water)

Figure 8: Relationship between hydraulic conductivity and density at complete saturation.

4   Discussion

4.1 Swelling Mechanism

4.1.1 Influence of accessory minerals

The MX-80 and GMZ clays have a content of montmorillonite of 77-85% and 75-78%, respectively. The accessory minerals are mainly quartz, plagioclase, feldspar, cristobalite,

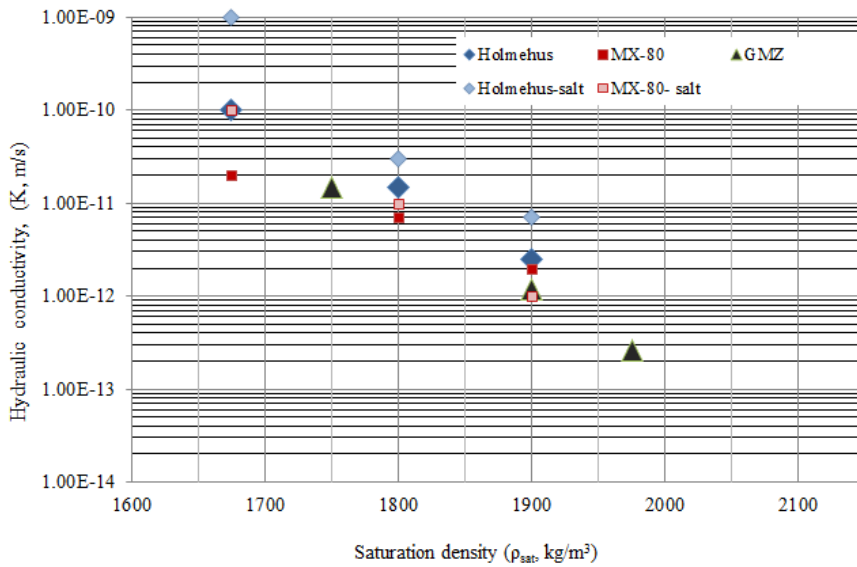gypsum, pyrite, and chlorite. They make up about 20 % in these clays and have a limited influence on the bulk physical properties except for those belonging to the fine-silt fraction [12]. Their role is negligible in the GMZ and MX-80 clays but can possibly have some impact on the behavior of Holmehus clay in which they make up some 20-30 % of the mineral phase.

4.1.2 Crystalline swelling

It is recognized that the swelling of clay on exposure to water or saline water was primarily controlled by the crystalline swelling and the interaction of diffuse double-layers [13, 14].

Crystalline swelling is related to the hydration of the exchangeable cations (K^+, Na^+, Ca^{2+}, and Mg^{2+}) between montmorillonite unit layers (lamellae). The matter may be rather complex, in fact, as pointed out by Tan [15], who conducted a test with Na-montmorillonite prepared with NH_4NO_3 solution of different concentrations and then made XRD analysis of the mixture. The results showed that the crystal lattice swelled when the concentration of solution was low, remained   unchanged when concentration was intermediate, and shrank when the concentration was high (Figure 9.). This phenomenon cannot be explained by ordinary hydration and diffuse double layer theories.

In natural saturated soil, montmorillonite crystal layers are in contact with interlamellar (interlayer) water and free porewater. Isomorphous replacement of Si^{4+} in silicon-oxygen tetrahedron can be partly replaced by Al^{3+}, Fe^{3+}, while and Al^{3+} in aluminium-oxygen octahedrons can be exchanged by Mg^{2+}, Fe^{2+}, according to classical colloidal chemistry and basic clay mineralogy. It is well known, also, that the charge deficiencies in the crystal structure are largely balanced by uptake of exchangeable cations in the interlamellar space. For montmorillonite, the ion concentration in crystal layers is about 1-7 mol associated with different numbers of water layers. Based on the physico/chemical theory, ion osmotic pressure in crystal layers at various ion concentrations is about 2.4-17.0 MPa [15].
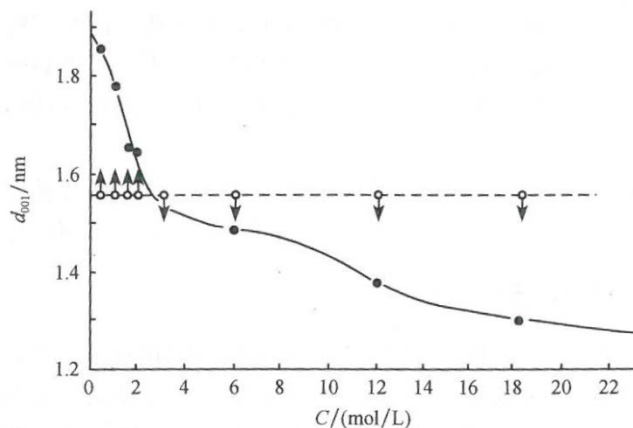

Figure 9: Relationship distance of crystal layer d_{001} of Na- montmorillontie and concentration of NH_4NO_3 [15].

For Holmehus clay, which is an illite/smectite mixed layer (IS-ml) clay, isomorphous replacement can take place of Al^{3+} in aluminium-oxygen octahedron being replaced by K^+

instead of by Mg^{2+} and Fe^{2+}. K^+ makes firm contact between the crystal layer units, and cations in the interlamellar space are thence not easily exchanged. The vacant charges for IS-ml are mainly in the tetrahedrons which are different from montmorillonite where they mainly take place in the octahedrons, making the swelling pressure of Holmehus clay lower than for MX-80 and GMZ clays. The differences depend on the proportion of smectite in Holmehus clay.

For Na-montmorillonite it is well known that when the Ca^{2+} concentration in the interlamellar space is higher than the Na^+ concentration in the free porewater, Na^+ will be gradually be replaced by Ca^{2+}. Furthermore, when the Na^+ concentration in the interlamellar space is higher, there is no ionic migration because of the higher osmotic pressure, since hydrogens will enter and reduce the high ion osmotic pressure. The process makes crystal layers enlarge and swell; the effect being called osmotic swelling potential. The ion osmotic pressure and osmotic swelling potential can also explain why the swelling pressure at saturation with distilled water is higher than for salt water (3.5% $CaCl_2$ solution).

Except for the impact of the ion osmotic swelling potential, the matrix suction potential for unsaturated clay should also be considered. The montmorillonite matric suction in Tan and Kong's research [16] could reach up to 39MPa, which is equal to the suction potential at RH (relative humidity) of about 70% (cf. [17]).

4.1.2 Role of diffuse electric double-layers

Diffuse electric double-layersare considered to be the main reason for the second expands stage. The Gouy–Chapman diffuse double layer theory has been widely used for explaining basic particle/water/cation interaction. According to this theory, the interaction force between two double layers depends on the ion concentration at the mid-plane between two adjacent parallel clay layers and is given by the osmotic pressure in that plane. Therefore, for any given separation distance between two clay layers the osmotic pressure can be determined from the theory by knowing the concentration of the ions in the central plane between the layers [18]. In general, the thicker the diffused layer, the larger is the space between particles. This has a strong impact on the size of smectite particles and thereby on the size of water-filled voids and on the bulk permeability.

The exchange capacity of cations is of fundamental importance. The order of exchange potential of different cations is $Fe^{3+}>Al^{3+}>H^+>Ba^{2+}>Ca^{2+}>Mg^{2+}>K^+>Li^+>Na^+$. For MX-80 and GMZ clay, Na^+ is the main exchangeable cation, while in Holmehus clay, K^+ was sorbed in the formation of this clay in nature, leaving only a rather small amount of exchangeable positions in the crystal aggregates. In Ca-dominated salt water, Na^+ is expelled from the montmorillonite aggregates, and K^+, Na^+ from those in illite-smectite clay. This lead to thicker electrical double-layers when MX-80 and GMZ clay are immersed in water Holmehus clay. The swelling capacity of MX-80 and GMZ clay is therefore much higher than of Holmehus clay.

Some of the graphs in Figure 6 show two maxima indicating that there are two stages in the swelling process. In the early one the swelling pressure increases rapidly and reaches a peak that may be caused by crystalline swelling. Rao and Shivananda [19] proposed that this swelling stage is reached relatively quickly and is related to the dissipation of matric suction, which drops when the degree of saturation rises. According to these investigators the suction of unsaturated specimens absorbs water to dissipate both matric suction and osmotic suction and to satisfy electrostatic force on the clay surface. Matric suction

dissipates on saturation of void spaces while osmotic suction dissipates by equalization of the dissolved salts concentration in the pore water and reservoir water. The electrostatic force reaches to a balance in the soil-water system when inter-particle repulsion force becomes equal to the force of inter-particle attraction, which arises because of electrostatic repulsion between diffuse ion layers of adjacent clay particles.

The curve for samples with 15.1% initial water content (Figure 6-a, b) indicates a first delay in pressure rise (marked GDA I). Compared with the 19.6% and 23.9% sample curves (Figure 6-c and d), the delay in curves with higher water content samples becomes weaker and finally disappeared at highest water content and lowest dry density. It is probable when the water fills the clay voids, and lightly collapses the soil skeleton takes parallel to the crystalline swelling. Furthermore, the higher water contents of samples, i.e. higher saturation degrees, relieve the extent of the collapse in this stage.

After the first peak diffuse double-layer repulsion successively dominates and shearing of big particles and particle aggregates makes them disintegrate in conjunction with a drop in swelling pressure. The large number of thin stacks of smectite lamellae, exfoliated from dense aggregates, reorganizes and forms soft gels in the voids without contributing to the macroscopic swelling pressure. For the samples with 15.1% initial water content this phenomenon may represent the second delay in pressure build-up (GDA II). For the 19.6% and 23.9% samples, we see the same effect but they are less obvious.

For very densely compacted clay samples, there is only a small amount of adsorbed water in the interlamellar space of montmorillonite and illite/smectite particles or in the contact region of adjacent particles [20]. It may, however, be sufficient for formation of local double-layers in parts of the regions [21]. The structure of expandable clays may hence rebuild and become stable, representing the aforementioned second stage. This explanation is similar to an alternative model proposed by other investigators, who claim that there is a very high contact pressure between smectite grains in compacted granular clay before they become weakened by water uptake. Until this takes place the particles are intact and exert a very high contact pressure that drops when the contact zone is wetted, by which inter particle slip takes place that brings the particles in new positions where they establish new bonds and cause successive "secondary" growth of the bulk swelling pressure [22]. The distribution of swelling pressure in confined smectite soil samples will therefore not be uniform and the swelling of individual aggregates is different [20, 23].

4.2 Hydraulic Behavior

The hydraulic conductivity depends on the degree of saturation and on the density of the clay, as well as of the strongly temperature-dependent viscosity of the porewater. When water is infiltrated into the clay, capillary forces make it migrate in the network of big pores, which are first filled with water then transported by the hydration potential of the smectite clay particles causing expansion of the particles and aggregates of particles. As mentioned, the density of a smectitic clay formed by compaction of clay granules followed by water saturation, varies throughout the clay and even for very smectite-rich clays there are a number of channels - empty or filled with soft clay gels - of varying size and interconnectivity that are responsible for the bulk hydraulic conductivity. The expansion causes initial collapse of the soil skeleton, followed by structural rebuilding and renewed swelling [17].

Figure 8 verifies that the hydraulic conductivity significantly changes with the dry density. In a microstructural perspective compaction to a high dry density implies that the cross

section of macro-pores forming channels decreases and that the interconnectivity of the latter is reduced. The electrical double-layers integrated in viscous water films that exist on all the clay surfaces exposed in the channels provide resistance to porewater flow making the conductivity extremely low for highly compacted smectite-rich clays. The role of the electrical double-layers is particularly important in this context: a high salt concentration in the porewater causes coagulation of the clay gels and soft parts of the microstructural particle network and hence widening of the voids, which results in a raised average bulk hydraulic conductivity.

These considerations are validated by comparing the results of tests with electrolyte-free and salt percolates: one finds that the value of hydraulic conductivity changes from 2.50E-12 m/s (with distilled water) to 7.00E-12 m/s (with 3.5% $CaCl_2$ solution) for Holmehus clay (saturation density 1900 kg/m^3), and from 7.00E-12 m/s to 1.00E-11 m/s for MX-80 (saturation density 1800kg/m^3).

5 Conclusion

In HLW repositories, buffer/ backfill material should have good swelling capacity, and low hydraulic conductivity. The chemical compositions in the groundwater in a deep geological repository can affect the properties of seals of compacted smectite-rich clay, like most bentonites. They are considered as potential candidate materials for buffer/backfill material. The main conclusions of the study are the following:

- The most effective barrier to flow of radioactively contaminated water migrating from a HLW repository is provided by low-permeable clays, i.e. those with high content of smectite minerals, with montmorillonite as major constituent. This makes the MX-80 and GMZ clays particularly suitable for use in practice. However, also the mixed-layer I/S Holmehus clay with significantly lower content of montmorillonite can be considered to be suitable as buffer or seals. The reason for the lower permeability of the clays with much montmorillonite is that the expandability of these particles makes the microstructural voids and channels largely filled with clay gels with considerable density when the porewater is poor in electrolytes, and that the voids are relatively well filled with (somewhat denser) clay gels also when the porewater is salt. Clays with lower montmorillonite contents have larger voids and less ability of the expandable montmorillonite to fill up voids, and they are therefore more conductive.

- Clay buffer placed to isolate canisters with HLW, and clay seals in boreholes in repository rock are preferably prepared by strong compaction of granular raw material. This gives both high density and low permeability but also the required expandability to establish tight contact with surrounding rock by exerting a sufficiently high swelling pressure on the contacting rock. As for the permeability the most smectite-rich clays MX-80 and GMZ give the highest swelling pressures, but the significantly less montmorillonitic mixed-layers Holmehus clay gives only a slightly lower pressure. This apparently strange condition can be explained by different granular size of the raw-material of clay, or by higher dispersibility of the Holmehus clay.

- The peculiar appearance of pressure peaks in the recorded development of the swelling pressure in the course of wetting in the laboratory deserves attention since it

reveals the mechanisms in the transformation of initially desiccated granulated clay confined in a closed space to the ultimately apparent homogeneous state of the clay. The phenomenon can be explained by assuming two wetting stages: a first caused by swelling of the clay aggregates giving the primary pressure peak followed by relaxation, and a second in which electrical double-layers are being developed and start exerting delayed interparticle pressure. A slightly different explanation is that an initial high contact pressure between particles in desiccated clay, representing the first pressure peak, drops when the particles soften by water uptake in their contact region, a process that is followed by shearing that causes disintegration and slip of particles to new positions where reconstruction of a homogenized particle skeleton can take place. This matter deserves more attention by considering the force-fields on the microstructral scale.

- Longevity of the smectite content in clays described in the study has not been in focus but naturally requires attention. This issue has been and is considered in the literature [17]. At present there are reasons to believe that most of the smectite component of bentonite clays, and particularly of mixed-layer S/I clays exposed to moderate temperatures (<100ºC) under the hydrothermal conditions in repositories for high-level radioactive waste, survive for many thousands of years. Their degradation appears primarily in the form of cementation by precipitated silica and iron complexes.

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