



Experimental investigation into temperature effect on hydro-mechanical behaviours of bentonite

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Abstract: The bentonite barrier of underground repositories for high-level radioactive waste will be hydrated by the groundwater while it is subjected to high temperatures due to the radioactive decay of the wastes. These changes of temperature affect the hydraulic and mechanical responses of bentonite, which has important effects on design and performance of repositories. The temperature influence on the hydro-mechanical behaviour of bentonite was studied in this paper by experiments, which were carried out with the Spanish FEBEX bentonite compacted at dry densities expected in the repository (from 1.5 to 1.8 Mg/m³). The dependence of the swelling strains of bentonite on the temperature has been measured from 30 °C to 90 °C. At high temperatures the swelling capacity of clay slightly decreases. Also, a clear decrease of swelling pressure as a function of temperature was observed for the same dry densities. Nevertheless, the deformation of bentonite is more dependent on the stress than the temperature. An increase in the permeability of water saturated bentonite with temperature has also been detected. The water retention curves of bentonite compacted at different dry densities were determined under isochoric conditions and in the range of temperatures from 20 °C to 120 °C. For a given density and water content, the suction decreases as the temperature increases at a rate, which is larger than the one predicted on the basis of water surface tension changing with temperature. Mechanisms related to the physico-chemical interactions that take place at microscopic level, in particular the transfer of interlayer water to the macropores triggered by temperature, seem to explain qualitatively the experimental observations.

Key words: bentonite; temperature; swelling; permeability; water retention capacity

1 Introduction

The research has been carried out in the context of projects concerning the engineered clay barrier of underground repositories for high-level radioactive waste (HLW). The barrier, made of compacted bentonite, a highly swelling material, will be placed between the waste canisters and the host rock, and will be saturated by the groundwater while it is subjected to high temperatures due to the radioactive decay of the wastes. Temperature changes affect important hydraulic characteristics of compacted clays including water retention and permeability, which are crucial to

predict the hydration rate of the barrier: any small variation can lead to very significant changes in saturation time. In addition, the mechanical response of the material, which has important effects on the design and performance of repositories, is also affected by temperature.

Laboratory tests may help to understand the processes that take place in the clay barrier under simple and controlled conditions and to develop the governing equations. The laboratory tests enable to isolate different processes, making their interpretation easier, and to provide fundamental data concerning the parameters used in models.

2 Material and methods

2.1 Material

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The FEBEX bentonite comes from the Cortijo de Archidona deposit (Almería, Spain) and its detailed characterisation can be found in Refs.[9–11]. The smectite content of the FEBEX bentonite is higher than 90% ($92\% \pm 3\%$), and it contains variable quantities of quartz ($2\% \pm 1\%$), plagioclase ($2\% \pm 1\%$), K-felspar, calcite and opal-CT. The cationic exchange capacity varies from 96 to 102 meq/(100 g), and the major exchangeable cations are Ca^{2+} (35–42 meq/(100 g)), Mg^{2+} (31–32 meq/(100 g)), Na^+ (24–27 meq/(100 g)) and K^+ (2–3 meq/(100 g)). The liquid limit of the bentonite is $102\% \pm 4\%$, the plastic limit is $53\% \pm 3\%$, the total specific surface area is $(725 \pm 47) \text{ m}^2/\text{g}$, and the specific gravity is 2.70 ± 0.04 . The hygroscopic water content in equilibrium with the laboratory atmosphere is $13.7\% \pm 1.3\%$. In all the laboratory tests, the grain size less than 5 mm was used.

2.2 Swelling pressure and hydraulic conductivity

The determination of swelling pressure and hydraulic conductivity as a function of temperature has been performed in high-pressure oedometer equipment (Fig.1). The clay is compacted uniaxially and statically at room temperature in the oedometer ring, which has an inner diameter of 5.0 cm, the height of the resulted specimen being 1.2 cm. Nominal dry densities of 1.5, 1.6 and 1.7 Mg/m^3 were reached by applying vertical stresses of (10.0 ± 1.3) , (16.0 ± 1.5) and $(29.1 \pm 1.0) \text{ MPa}$, respectively. The specimens thus obtained are confined between porous stainless steel sinters.

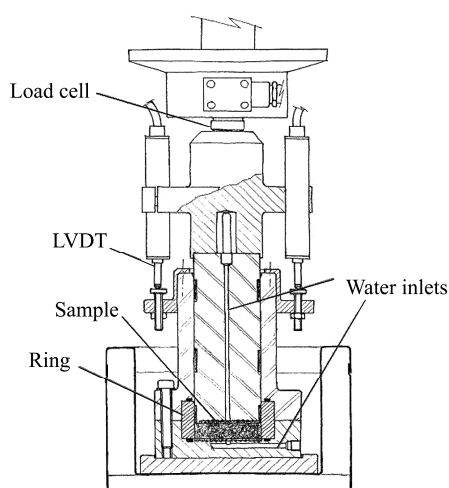


Fig.1 Schematic layout of high-pressure oedometric cell inside the thermostatic bath.

The oedometer assemblage is placed inside a silicone oil thermostatic bath. Before increasing the temperature of the bath, a small vertical load of around 0.4 MPa is applied to the sample to ensure a good contact with the

load cell installed in the loading frame, and the deformation of the sample is hindered by means of setscrews. The stabilisation of temperature is reached in less than 24 hours. At the end of this period, the equipment deformation gives rise to vertical displacements between -0.06 and 0.24 mm (corresponding to -0.5% and 2.0% of the specimen height, respectively) measured by two LVDTs placed in each oedometer assemblage. Also, the load cell records pressure increases due to the deformation of the metallic parts of the equipment during the temperature stabilisation period, ranging from 0.1 to 2.7 MPa for temperatures between $30 \text{ }^\circ\text{C}$ and $80 \text{ }^\circ\text{C}$. These have been calibrated and deducted from the initial values corresponding to the sample behaviour. After 24 hours, the sample is hydrated at constant volume through the bottom surface with deionised water injected at a pressure of either 0.6 MPa for most samples with a dry density of 1.6 Mg/m^3 or 0.01 MPa for the samples with dry densities of 1.5 and 1.7 Mg/m^3 , while the upper outlet remains open to atmosphere. At the same time, the load cell measures the swelling pressure exerted by the clay. The small vertical deformation of the specimen, due mainly to the load cell and frame deformation, is measured by LVDTs. An automatic volume change apparatus measures the water exchange of the specimen. The values of load, strain and water exchange are automatically recorded.

Once the sample is completely saturated (which is assumed by the stabilisation of water intake and swelling pressure development), the injection of water is stopped, and the pressure registered is considered with the swelling pressure value for the dry density attained. The actual density may differ slightly from the nominal one due to the small displacement allowed by the equipment (about 0.1 mm when the vertical stress is 1 MPa, and 0.4 mm when the vertical stress is 7 MPa, i.e. between 1% and 3% of the sample height, respectively).

Afterwards, hydraulic conductivity is determined in the same equipment and on the same samples, which are kept at constant volume. To perform this determination, the water pressure at the bottom of the samples is increased, while a backpressure of 0.6 MPa is applied on top, resulting in hydraulic gradients between 800 and 9 600. At least two different hydraulic gradients have been applied for each sample. The water outflow is measured by a volume change apparatus, and the hydraulic conductivity is calculated according to Darcy's law.

2.3 Swelling capacity

The influence of temperature on the swelling capacity of clay was checked by swelling under vertical load tests. They are performed in oedometers whose cell is placed in a thermostatic bath with controlled temperature (Fig.2). Granulated bentonite with its hygroscopic water content (about 14%) is compacted inside the cell ring at room temperature using static uniaxial compaction. The specimens, 5.0 cm in diameter and 1.2 cm in height, were obtained using the same method and pressures described above.

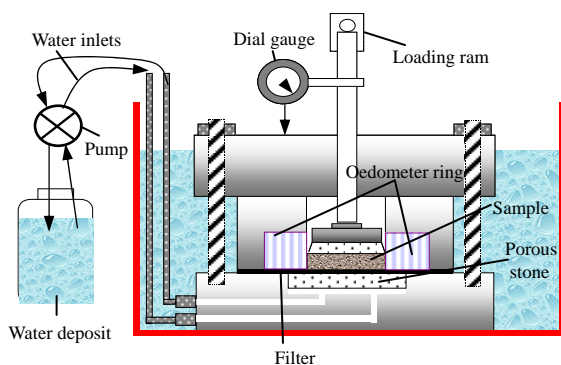


Fig.2 Schematic representation of the oedometer cell for tests at high temperature.

Once in the oedometer, the stabilisation of temperature is reached in less than 24 hours. After having reached the stabilisation of the target temperature in the oedometer, vertical pressures of 0.1, 0.5, 1.5 and 3.0 MPa are applied to the samples. Afterwards, they are put in contact with deionised water at atmospheric pressure from the bottom porous plate immediately. The swelling strain experienced by the specimens upon saturation is recorded as a function of time until stabilisation. On completion of the tests, the water content of the specimens is determined and full saturation is verified. The tests were performed at temperatures ranging from 30 °C to 90 °C.

2.4 Water retention capacity

To determine the water retention curve of the compacted bentonite at constant volume, two methods and different theoretical principles were expounded.

The cell method is carried out in special cells designed to avoid the swelling of clay in wetting paths [6, 10]. The cells consist of a corrosion-resistant stainless steel cylindrical body with two perforated covers joined by bolts (Fig.3). Granulated clay is compacted directly inside the cell ring at room temperature using static uniaxial compaction. Porous stones are

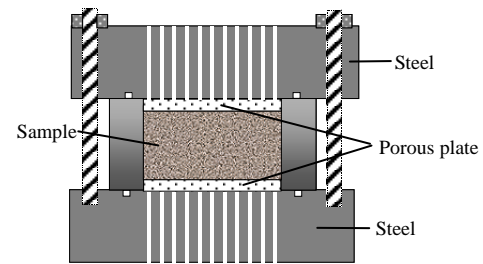


Fig.3 Schematic layout of the non-deformable cell for the determination of the water retention curve.

placed between the specimen and the cell covers at top and bottom. The height of the specimen is 1.20 cm and its cross-sectional area is 11.34 cm². The cells are placed in desiccators with a H₂SO₄ or NaCl solution. There are temperature-dependent experimental relations between the concentration of solution and its water activity (a_w). The calculation of suction on the basis of relative humidity ($RH = 100a_w$) is accomplished through Kelvin's equation. In the cell method, the suction is, therefore, imposed through the control of relative humidity. The perforated covers allow the exchange of water in the vapour phase between the clay and the atmosphere of the desiccators. Once the water content of the clay is stable (after several months, checked by periodic weighting), the solution in the desiccators is changed to apply different suctions. To determine the curve at different temperatures, the desiccators are placed inside ovens. At the end of the tests the final density of the solution (related to its concentration) is checked with pycnometers, and the final water content of the specimens is measured by oven drying.

The sensor/cell method consists in the compaction of a bentonite block with the clay previously mixed with the desired quantity of deionised water and the measurement of its relative humidity by means of a capacitive sensor while the bentonite is kept inside a hermetic cell made of stainless steel [12, 13]. To convert the values of relative humidity to suction, Kelvin's law is used. Either the clay with its hygroscopic water content mixed with deionised water, or the slightly dried one at temperatures below 50 °C, was used to obtain water contents between 7% and 22%. The block is introduced in the cell, the dimensions of which are equal to the internal volume of the cell, 7 cm in diameter and 10 cm in height. A hole is drilled in the central upper part of the block to insert the sensor, and the cell is closed (Fig.4). The external wall of the cell is covered with a siliconerubber laminated heater that fixes the tem-

perature all over the cell. After measuring the suction corresponding to the laboratory temperature, the temperature of the external heating mat was increased up to 120 °C at intervals of 20 °C. Each target temperature was kept for about two days, although the relative humidity equilibrium was reached very quickly (in a few hours). Afterwards, the temperature was decreased according to the same pattern. This allows, in a single test, the determination of the suction change with temperature for given density and water content. At the end of the test, the block is extracted and its water content and dry density are checked.



Fig.4 Appearance of the cell with the compacted bentonite inside, drilled to allow the entrance of the sensor.

The drawback of the cell method is the duration of the tests, because the time to reach equilibrium for each suction is very long. This is why the sensor/cell method was fine-tuned. The results obtained with both methods are largely consistent, although the sensor/cell method is unsuitable for the very low and very high suctions [14].

3 Results

3.1 Swelling pressure

The equilibrium swelling pressure values obtained in the high-pressure oedometers are plotted in Fig.5, where the dispersion of data can be mostly attributed to the variations in dry density, as the swelling pressure value is very sensitive to small density changes. The error bars shown in the figure were obtained from values measured in tests performed at laboratory temperature. A decrease of swelling pressure as a function of temperature is observed. Figure 6 shows the evolution of swelling pressure in the tests performed with the bentonite compacted at nominal dry density 1.5 Mg/m³. The swelling pressure does not develop in a

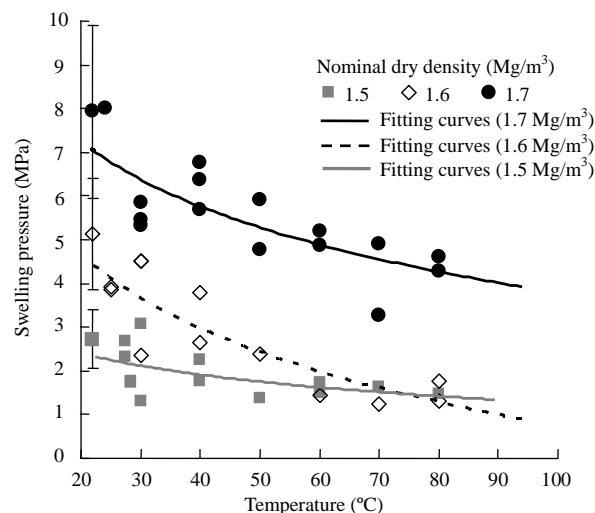


Fig.5 Swelling pressure as a function of temperature for saturated compacted samples.

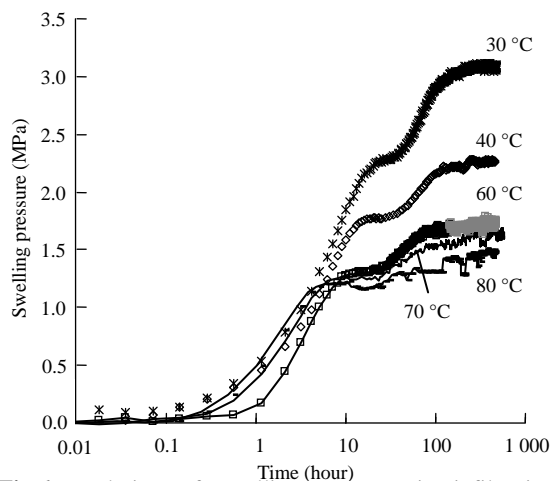


Fig.6 Evolution of swelling pressure in infiltration tests performed at different temperatures in samples compacted at nominal dry density 1.5 Mg/m³.

uniform way: after a sharp initial increase, there is a period during which the swelling pressure increases more slowly, even decreases in the case of densities 1.5 and 1.6 Mg/m³. This behaviour is clearer in samples of lower dry density whose swelling pressure is lower, but it takes place for all temperatures tested.

3.2 Hydraulic conductivity

The hydraulic conductivity was measured in the samples described above, kept in the same equipments, once they were fully saturated. For that, various hydraulic gradients were applied between top and bottom of the specimens. Lower hydraulic gradients were needed to obtain measurable flow as the temperature was higher. Below certain hydraulic gradients, no flow was obtained. These threshold values depend on the temperature and dry density of bentonite.

The permeability results are plotted in Fig.7. The permeability tends to increase with the temperature, as expected from the decrease in water kinematic viscosity (the continuous lines follow the measured trend). In the same figure, the change of permeability expected as a consequence of changes in water properties with the temperature (viscosity and density decrease) has been indicated with dotted lines. For the nominal dry density 1.6 Mg/m³, the measured increase in permeability with temperature seems smaller than that expected according to the changes in water viscosity. However, for the other two densities, the behaviour is not so clear, especially for the high temperatures, the permeabilities measured are higher than that expected on the basis of the changes in water viscosity.

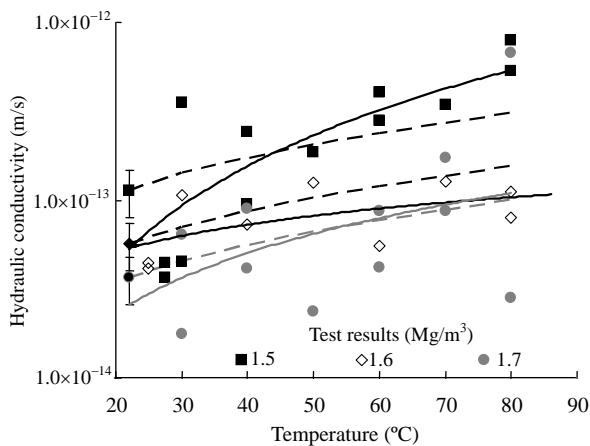


Fig.7 Hydraulic conductivity vs. temperature for saturated samples compacted to different nominal dry densities.

3.3 Swelling capacity

Swelling under vertical loading tests were performed on samples with initial dry densities 1.5, 1.6 and 1.7 Mg/m³ which were tested at temperatures between 20 °C and 90 °C and vertical loads between 0.1 and 3.0 MPa. The final strains obtained in all the tests were plotted in Fig.8. A single empirical equation takes into account the effect of vertical load σ (MPa), temperature T (°C) and dry density ρ_d (Mg/m³) on the final vertical strain ε (%) of the bentonite upon saturation with deionised water for the ranges of temperature and load. It can be expressed as

$$\varepsilon = 0.04T + 7.76 \ln \sigma - 52.90\rho_d + 68.60 \quad (1)$$

The tests show that the effect of temperature on the swelling capacity is smaller than that of the vertical load applied during hydration or that of initial dry density. The effect of temperature on the decrease in swelling capacity is also rather independent of the dry density of bentonite or the vertical load applied.

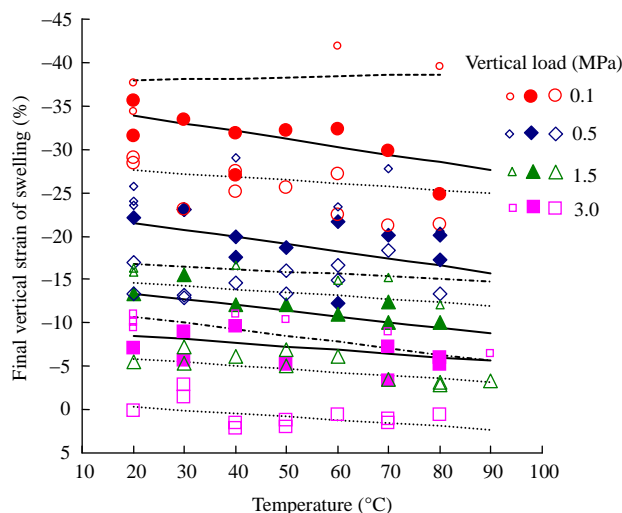


Fig.8 Final vertical strain of swelling under load tests performed on samples compacted to nominal dry densities 1.5 Mg/m³ (big open symbols and dotted lines), 1.6 Mg/m³ (big filled symbols and continuous lines) and 1.7 Mg/m³ (small open symbols and discontinuous lines, respectively).

3.4 Water retention capacity

The effect of dry density on water retention capacity of the FEBEX bentonite was checked using the two methods described above. It was observed that there is a suction threshold value, above which, for a given water content, the suction of the higher density samples is slightly higher, and below which the trend clearly inverts. This threshold value is between 10 and 20 MPa for the range of temperatures studied [15, 16].

Tests with different densities have been also performed at different temperatures using both methods. Some of the results obtained are plotted in Fig.9. The water retention capacity of the samples decreases with temperature [10, 17] and the effect of temperature on the water retention capacity is higher for the high dry density; as for dry density 1.5 Mg/m³, the water retention capacity only decreases clearly for the highest temperatures (above 70 °C).

Taking into account all the results obtained, the following modified van Genuchten expression for the water retention curve has been fitted:

$$w = [37.5n^{0.32}e^{-0.001(T-20)}] \cdot \left\{ 1 + \left[\frac{s}{21.9e^{-8.2(n-0.4)}e^{-0.001(T-20)}} \right]^{1-0.295} \right\}^{-0.295} \quad (2)$$

where w is the water content (%), s is the suction (MPa), n is the porosity, and T is the temperature (°C). The difference between measured values and the ones estimated using this equation is smaller than 1.2% in terms of water content, with a trend to be lower for a higher density.

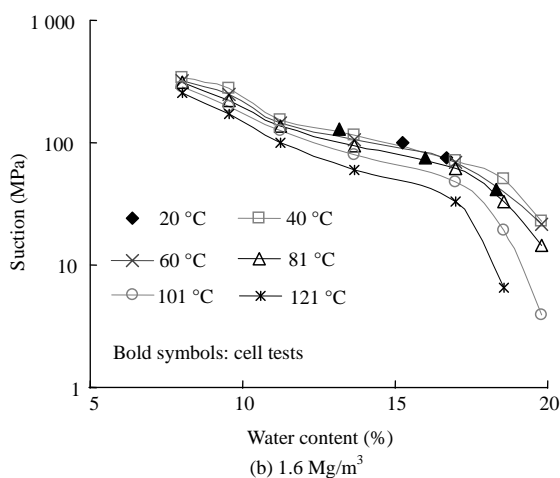
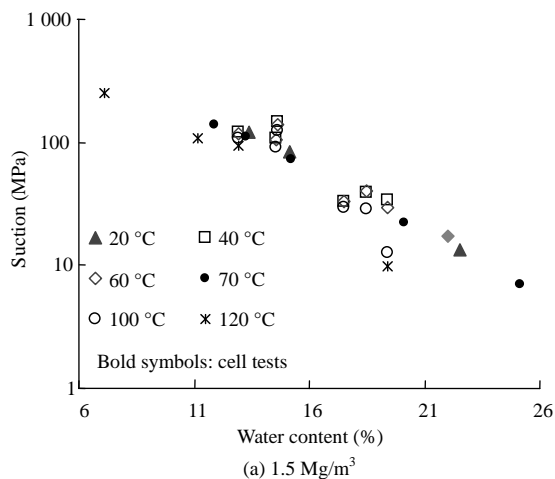


Fig.9 Retention curves obtained for samples compacted to different dry densities and temperatures.

Figure 10 shows the suction evolution of samples with temperature tested with the sensor/cell method. The samples have different water contents and are compacted

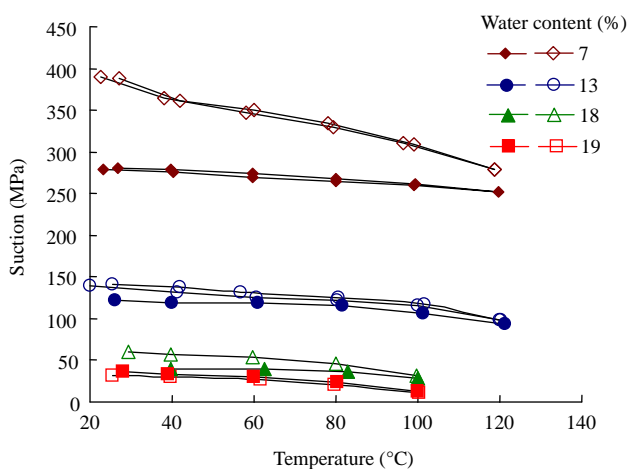


Fig.10 Evolution of suction with temperature (heating-cooling paths) for samples with different water contents compacted at dry densities 1.75 Mg/m^3 (open symbols) and 1.5 Mg/m^3 (filled symbols).

at two dry densities. The decrease in suction with temperature is significant, especially when the temperature is above $60 \text{ }^\circ\text{C}$ and the water content is low. There is barely any hysteresis between the initial heating and the subsequent cooling. The influence of temperature on the water retention capacity seems to be higher than that of density for suctions below $150\text{--}200 \text{ MPa}$. Anyway, the smaller slope of the curves for the dry density 1.5 Mg/m^3 would indicate a smaller effect of temperature on the water retention capacity for the low-density samples.

4 Discussions

Laboratory tests were performed to check the effect of temperature in the range from $20 \text{ }^\circ\text{C}$ to $120 \text{ }^\circ\text{C}$ on some hydro-mechanical properties of the bentonite compacted at dry densities between 1.5 and 1.75 Mg/m^3 .

The results of the swelling under loading tests show that, despite the slight decrease in swelling capacity with temperature observed, the effect of temperature on the swelling capacity of the bentonite is smaller than that of the vertical load applied during hydration, or that of initial dry density. However, the results obtained in the swelling pressure tests (Fig.5) show that the effect of temperature is higher for the higher density. All the dry densities tested, including the lowest one, suffer a clear decrease of swelling pressure for high temperature, which suggests that the effect of temperature is more important as the vertical load is higher, since the load applied in swelling pressure tests is higher than that applied in swelling under load tests. Nevertheless, the extrapolation of the empirical correlations between swelling pressure and temperature towards higher temperatures indicates that the swelling pressures higher than 1 MPa would develop even for temperature of $100 \text{ }^\circ\text{C}$ for the three densities tested.

The decrease in swelling pressure and swelling capacity of the FEBEX bentonite with temperature has been explained as a consequence of the transfer of microstructural (interlayer) water to the macrostructure triggered by temperature [6, 17]. The swelling of a predominantly divalent bentonite (as it is the FEBEX one) is mainly caused by the microstructural water placed in the interlayer. When it is transferred to the macrostructure, it no longer causes swelling. This process would be more significant in high-density

samples, in which the interlayer water predominates initially over the “free” macroscopic water [18], and this is why the effect of temperature is clearer in swelling pressure tests, in which the density remains high and constant during the saturation process, whereas in the swelling under loading tests, the density usually decreases with saturation.

No conclusive results were obtained with respect to the influence of temperature on permeability. As expected, permeability increases with temperature, but it seems that this increase cannot be attributed solely to the water viscosity changes. The same processes responsible for the swelling decrease are probably involved in the additional increase of permeability.

Although it is generally acknowledged that suction in clayey soils is not exclusively a capillary process, the Laplace equation, which relates the capillary pressure and the pore size distribution, is a first approximation to explain the water retention processes in soils. Thus, for the prediction of the effect of temperature on the water retention capacity, the change of surface tension of water with temperature is usually included in this equation. However, the observed evolution of suction with temperature cannot be explained on the basis of the change in surface tension of water with temperature [12]. In Fig.11 the measured evolution of suction for samples with nominal dry density 1.60 Mg/m^3 tested with the sensor/cell method has been plotted under heating. The suction values were calculated by taking into account the change in the water surface tension when the temperature increases from $20 \text{ }^\circ\text{C}$ to $120 \text{ }^\circ\text{C}$. The slopes of the lines relating the suction to the temperature have been calculated and are indicated in the figure. There is a discrepancy between the lines corresponding to measured values and those corresponding to computed values, the latter displaying smaller slopes. This difference between measured and computed values is more significant for temperatures above $60 \text{ }^\circ\text{C}$, and is probably due to the fact that capillarity is not the main mechanism of water retention in bentonite. Instead, physico-chemical interactions between the clay particles and the water tightly attached to them are responsible for the soil retention capacity, especially in the high suction range. In this low water content region, changes in the interaction mechanisms between the clay and water are considered the main temperature effects on water retention capacity [5, 6, 12].

As mentioned above, an increase in temperature produces a transfer of water from the interlayer region

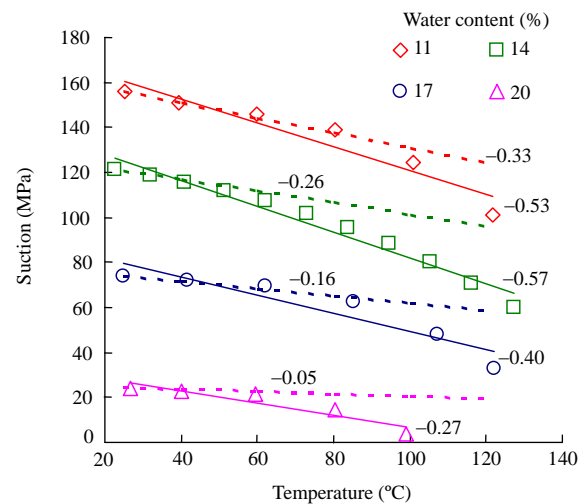


Fig.11 Change of suction for the FEBEX bentonite measured with the sensor/cell method (points and continuous lines) and computed by the change of water surface tension (dashed lines).

to the pores between the clay aggregates (macropores) [19, 20]. Since the density of the interlayer tightly-bound water in smectites is higher than 1 Mg/m^3 [9, 21, 22], the volume occupied by the interlayer water transferred to the macropores will be higher and the degree of saturation of the sample will increase, provoking a suction decrease when the temperature is increased [6]. It is known that the proportion of water in the interlayer of the smectite increases with the density of the bentonite [18]. This would explain the larger effect of temperature on the water retention capacity of high-density samples.

5 Conclusions

Laboratory tests about the effect of temperature on some hydro-mechanical properties of the compacted FEBEX bentonite were reported.

The dependence of the swelling strains of the bentonite compacted to dry densities of 1.5, 1.6 and 1.7 Mg/m^3 on temperature at the interval from $20 \text{ }^\circ\text{C}$ to $90 \text{ }^\circ\text{C}$ was measured. At high temperatures, the swelling capacity of the clay slightly decreases. On the other hand, a clear decrease in swelling pressure as a function of temperature was observed for the same dry density. Nevertheless, the deformation of the bentonite is more dependent on the stress than temperature. An increase with temperature in the water saturated permeability of the FEBEX bentonite compacted to these dry densities was also detected.

The effect of temperature on the suction of bentonite has been determined for dry densities 1.5, 1.6 and 1.75

Mg/m³ and water contents between 7% and 22% in a range of temperature between 20 °C and 120 °C. The water retention capacity of the bentonite decreases clearly with temperature, especially when it is above 60 °C and the density of the bentonite is high, although the effect of the dry density on the water retention capacity seems lower than that of temperature, except for high suctions. This decrease cannot be explained on the basis of the changes of water surface tension with temperature. Instead, mechanisms related to the physico-chemical interactions that take place at microscopic level, in particular the transfer of interlayer water to the macropores triggered by temperature, seem to explain qualitatively the experimental observations.

In spite of these observations, the FEBEX bentonite remains suitable as a sealing material in HLW repositories (from the hydro-mechanical point of view) for temperatures up to 80 °C, as it keeps low permeability, self-healing ability and water retention capacity. Not enough data are still available for higher temperatures, although the extrapolation of results points out the preservation of properties for at least up to 100 °C.

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