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Bentonite strain due to cyclic suction changes

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ABSTRACT: A series of suction-controlled oedometer tests were performed on a highly compacted bentonite following drying/wetting and wetting/drying paths from the initial hygroscopic conditions (about 120 MPa) under low vertical load. The increase of suction to values of close to 500 MPa induced practically no reduction in volume. During the subsequent hydration, the swelling strains occurring between 500 and 300 MPa were very small, which could be due to plastification of the material on drying, this displacing the elastic range to the interval between those suctions. Afterwards, when suction decreased to the initial value, the initial void ratio was practically recovered and below this value, the reduction in suction did cause a significant increase in volume. In fact, drying did not modify the swelling capacity of the sample during subsequent hydration, on the contrary, the deformations observed during wetting were larger in the samples previously dried. In wetting/drying paths the swelling deformation occurred on suction decrease was irreversible and mainly caused by the increase in the microstructural void ratio.

1 INTRODUCTION

The system of barriers in a deep geological repository for high-level radioactive waste (HLW) aims to prevent the possible leakage paths for radionuclides to the environment. The sealing materials will be in contact with the waste containers and their basic functions are to prevent or limit the entry of water to the wastes, provide mechanical protection for the canisters and contribute to radionuclide retention (Chapman & McCombie 2003).

Bentonite has been widely considered as a potential sealing material, since it provides very low permeability, high exchange capacity, sufficient thermal conductivity, mechanical resistance to withstand the weight of the canister, plastic behaviour, swelling potential, good compressibility, low shrinkage in response to drying, and physical and chemical stability. When used as a sealing material, the bentonite – that in most repository concepts will be installed compacted and in an unsaturated state – will be hydrated with the water coming from the host rock at the same time that will be subjected to a thermal gradient because of the high temperatures generated by the wastes on their radioactive decay. In the Spanish HLW repository concept, the temperatures on the surface of the canisters can be of 100°C (ENRESA 1995) and temperatures as high as 150°C are considered in other repository concepts (Johnson et al.

2002). These high temperatures can cause an intense drying of the sealing material, as has already been observed in many large-scale *in situ* tests (Gaus et al. 2014). As an example, Figure 1 shows the relative humidity measured over time by two sensors placed close to a heater simulating the waste canister in the FEBEX large-scale *in situ* test (ENRESA 2006). The relative humidity reached values as low as 7% at locations where the temperature was close to 90°C, corresponding to a suction of about 445 MPa. According to the water retention curve of the material (the FEBEX bentonite), this suction would correspond to a water content of 4% at room temperature (Figure 2) but taking into account that the water retention capacity of bentonite decreases with temperature (Villar & Gómez-Espina 2008; Jacinto et al. 2011), the water content close to the heater during the first stages of the repository would be even lower. Afterwards, the relative humidity would recover, as can also be seen in Figure 1. In fact, it is expected that eventually the bentonite closest to the canister will be also saturated, and in this way, it will have been subjected to a drying/wetting process.

One of the concerns about the behaviour of the bentonite barrier is the preservation of its good mechanical properties after being intensely dried in the conditions of the repository. Even if the sealing capacity is well preserved, as some studies have demonstrated, the impact on its volumetric behav-

ior has to be well known in order to predict its long-term evolution. In the context of an investigation of the near field for a repository of high-level radioactive waste, the FEBEX Project, a set of suction-controlled oedometer tests were performed on a compacted highly-expansive bentonite to give a better understanding of the thermo-hydro-mechanical behaviour of unsaturated, expansive clays and to achieve an experimental corroboration of the constitutive models for these clays. In particular, this work focuses on the volumetric behaviour of bentonite when it is subjected to wetting/drying or drying/wetting processes. The latter are particularly relevant for the study of the behaviour of the bentonite close to the waste canister.

Although the volumetric behaviour of unsaturated bentonite has been studied for decades, there are very few experimental results referring to wetting/drying cycles (Lloret et al. 2003; Alonso et al. 2005; Nowamooz & Masrouri 2008), because of the long-term periods needed for equilibrium in expansive materials. This paper intends to contribute filling this gap.

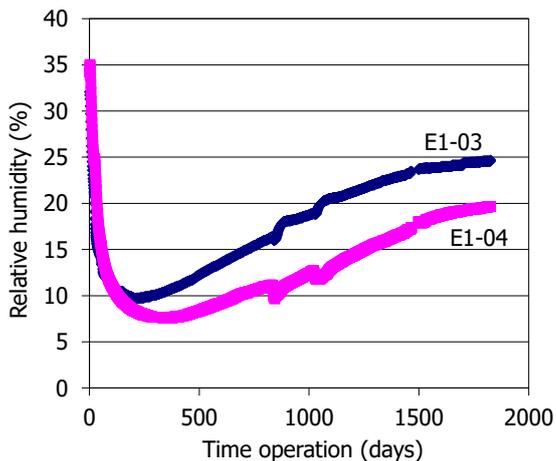


Figure 1. Evolution of the relative humidity of the bentonite recorded by two sensors at 3.5 cm from the heater in the FEBEX in situ test (modified from Villar et al. 2012).

2 MATERIALS AND METHODS

2.1 Material

The FEBEX bentonite was extracted from the Cortijo de Archidona deposit in SE Spain (ENRESA 2006). The montmorillonite content of the FEBEX bentonite is $92 \pm 3\%$ and its cation exchange capacity is 102 ± 4 meq/100g, the main exchangeable cations being calcium, magnesium and sodium. The liquid limit of the bentonite is $102 \pm 4\%$, the plastic limit $53 \pm 3\%$, the density of the solid particles 2.70 ± 0.04 g/cm³, and $67 \pm 3\%$ of particles are smaller than 2 μ m. The hygroscopic water content in equilibrium with the laboratory atmosphere (total suction about 100 MPa) is $13.7 \pm 1.3\%$.

The water retention curve of the bentonite compacted with its hygroscopic water content at an initial nominal dry density of 1.7 g/cm³ was determined at 20°C using the vapour transfer technique and following a drying/wetting path (Figure 2). Although it is well known that the retention capacity of the bentonite depends on its density and consequently on the volume restriction (if the sample is allowed to swell or its volume is confined), it has been checked that for the FEBEX bentonite the difference between the two procedures is very significant only for suctions below 10 MPa (Villar 2002). However, in drying paths from the hygroscopic water content the results obtained under free or isochoric conditions are equivalent, because the sample volume barely changes.

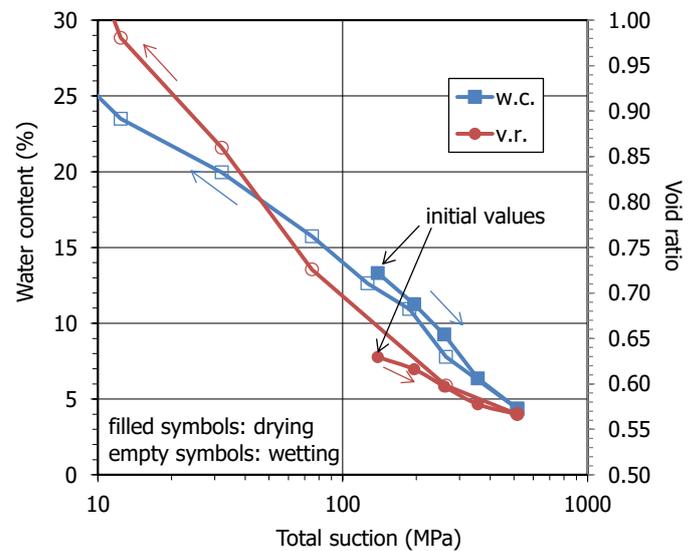


Figure 2. Water retention curve of the FEBEX bentonite obtained in a drying/wetting path at 20°C under no volume restriction, with indication of the changes in void ratio (Villar 2002).

2.2 Methodology

Suction-controlled oedometer tests were performed in oedometer apparatus equipped with cells specially designed to apply the desired suctions (Figure 3; Esteban 1990; Villar 2002). Suction was controlled in the oedometer cells by the vapour transfer technique, specifically through the control of the relative humidity by means of sulphuric acid solutions. The relative humidity is correlated to suction through the psychrometric (Kelvin's law). Suctions from 13 to 500 MPa can be obtained with sulphuric acid solutions.

The granulated bentonite with its hygroscopic water content was directly compacted in the cell ring applying static uniaxial pressures around 30 MPa to obtain nominal dry densities of 1.70 g/cm³. These initial conditions were selected to reproduce those of the blocks manufactured to construct the engineered barrier of the FEBEX in situ test (ENRESA 2006).

The initial height of the samples was 1.20 cm, and their diameter was 3.80 or 4.95 cm. The tests were performed at 20°C and consisted of a series of successive suction stages applied under a load vertical load.

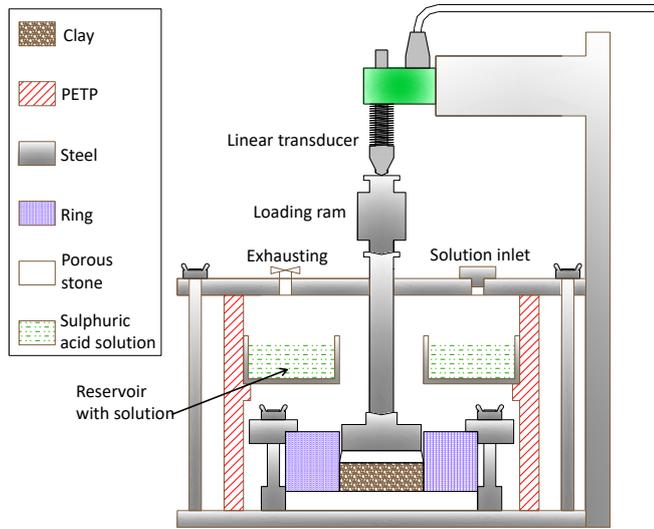


Figure 3. Schematic representation of a suction-controlled oedometer cell.

3 RESULTS

Table 1 shows the initial characteristics of the samples tested and the suction paths followed during the tests. Some of them were first presented in Villar (2002) and discussed in Lloret et al. (2003) but have been included again for completion and comparison.

Table 1. Characteristics of the oedometric tests performed (constant vertical load 0.1 MPa, except test S10: $\sigma_v=0.2$ MPa).

Test	Initial conditions		Suction range (MPa)			Duration (days)
	ρ_d (g/cm ³)	w (%)	Wetting	Drying	Wetting	
S4 ^{a, b}	1.71	12.9	119-4			118
S9	1.70	13.8	122-1	1-97		3833
S5 ^{a, b}	1.72	13.2		138-520	520-0	266
S6 ^a	1.71	12.8		118-523	523-1	659
S8	1.70	14.1		99-550	550-13	587
S10	1.73	12.1		131-471	471-1	1018 ^c

^a Villar 2002; ^b Lloret et al. 2003, ^c The last suction step (1 MPa) took 473 days

The duration of most of the suction steps was fixed to 20-30 days, except for test S9, in which every suction step was prolonged until complete stabilisation of the vertical strain, which caused the total duration of the test to be of 3833 days. The results of the initial wetting path of this test are plotted in Figure 4

along with those of test S4, which followed a similar path but with shorter duration for each suction step. Although there were certain differences in the initial kinetics of both tests, the strains reached after a given time at a particular suction were not very different (the maximum differences were below 2%), which gives confidence in the results. Nevertheless, it is clear that, particularly for the lowest suctions, the stabilisation of the vertical strain took much longer than the 20-30 days set for test S4.

Figure 5 shows the evolution of void ratio in the tests that followed drying/wetting paths under a load of 0.1 MPa (0.2 MPa in test S10), and

Figure 6 is an enlargement for the suctions above the initial value. The increase of suction to values of close to 500 MPa implied practically no reduction in volume, although the void ratio decreased with increasing suction in accordance with a logarithmic relation whose slope was almost independent from initial dry density (Villar 2002). This minor variation in volume with drying from 120 MPa would indicate that this value of suction was probably above the air entry value. The suction from which the loss of volume of water due to drying is compensated by the entry of a similar volume of air, with which the loss of water does not translate into loss of total porosity. Values of 100 MPa were proposed for the air entry value in montmorillonite (Tessier et al. 1992). Also, this would mean that the water content of the bentonite was lower than its shrinkage limit. Furthermore, for suction in excess of 120 MPa, external load did not lead to any important consolidation of the samples, whose compressibility became very low (Villar 2002; Lloret et al. 2003).

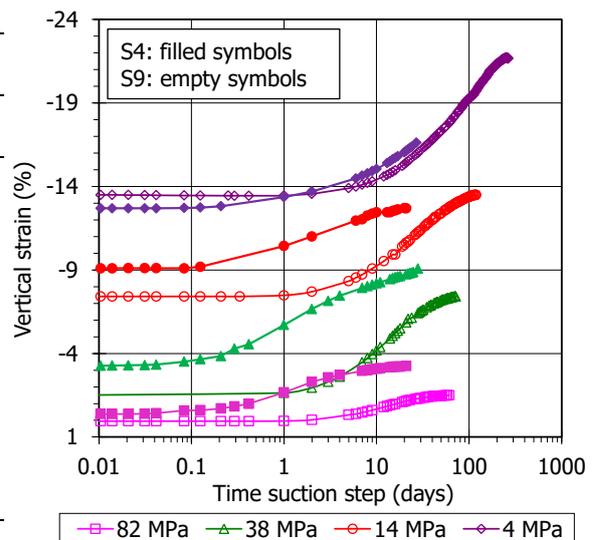


Figure 4: Evolution of vertical strain in wetting paths of two oedometric tests performed under vertical load 0.1 MPa (the total suction applied in each step is indicated in the legend).

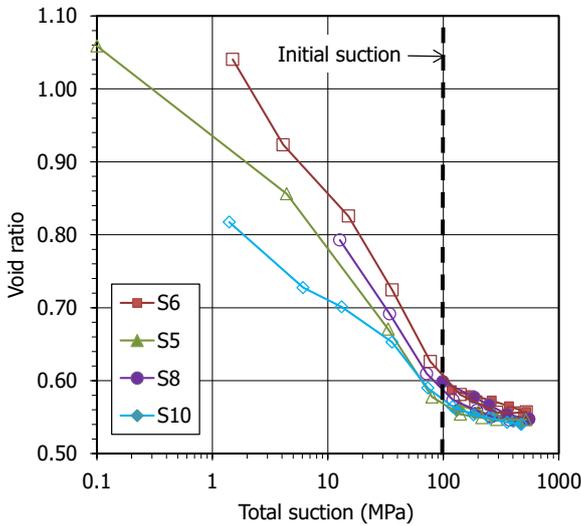


Figure 5. Evolution of void ratio during drying/wetting under low vertical load in different tests.

During the subsequent hydration, the swelling strains occurring between 500 and 300 MPa were very small, which could be because of the plastification of the material on drying, this displacing the elastic range to the interval between those suctions. The elastic range would now be between approximately 500 and 300 MPa, whereas prior to drying it would be below the initial value of 130 MPa. Below these values, the reduction in suction did cause a significant increase in volume, with a very sharp change in slope when suction decreased to below the initial value (Figure 5).

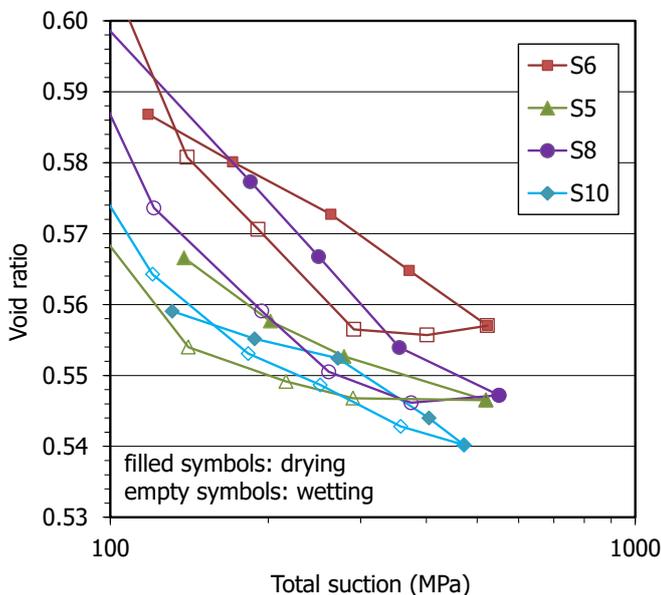


Figure 6. Evolution of void ratio during drying and wetting above 100 MPa under a vertical load of 0.1 MPa in different tests.

Figure 7 represents the void ratios on the hydration path below 100 MPa for the tests performed under vertical load 0.1 MPa. In tests S5, S6 and S8 hydration occurred following intense drying to suctions above 520 MPa, which did not occur in the case of tests S4 and S9. The samples in tests S4 and S6 had the same initial dry density (1.71 g/cm^3), but follow-

ing hydration to a suction of 4 MPa the dry density was lower in test S6 than in the case of test S4, despite the fact that the path in test S6 included a particularly intense previous process of drying, probably to water content values around 4% according to the water retention curves (Figure 2). In these two tests each suction step was kept for 20-30 days, hence, the equilibrium time was the same for the two of them. On the contrary, the equilibrium times were much longer in test S9 (complete stabilisation of strain was attained in every suction step), and despite this difference, the void ratios for the hydration path below 100 MPa were not different to those after intense drying for the potentially non-stabilised test S6. This higher swelling in samples that had undergone previous drying was observed also on the retention curves determined under free volume conditions (Villar 2002). The models of microstructural behaviour show that the swelling experienced by a soil on decreasing suction under a given load is higher the higher the suction at the beginning of hydration, and that the relation between the percentage of strain and the decrease in suction increases on reaching lower values of suction (Gens & Alonso 1992; Sánchez et al. 2005), both of which aspects have been confirmed in these tests. After completing the hydration path, drying back to the initial suction was carried out in test S9 (Figure 9). The results show that in wetting/drying the swelling deformation occurred on suction decrease was irreversible, and the initial void ratio could not be recovered upon drying. Gens & Alonso (1992) stated that, in expansive soils, the swelling of the microstructure when wetting the samples would cause irreversible macrostructural rearrangements, and that this interaction would be stronger when the applied stresses were low, which is the case in these tests.

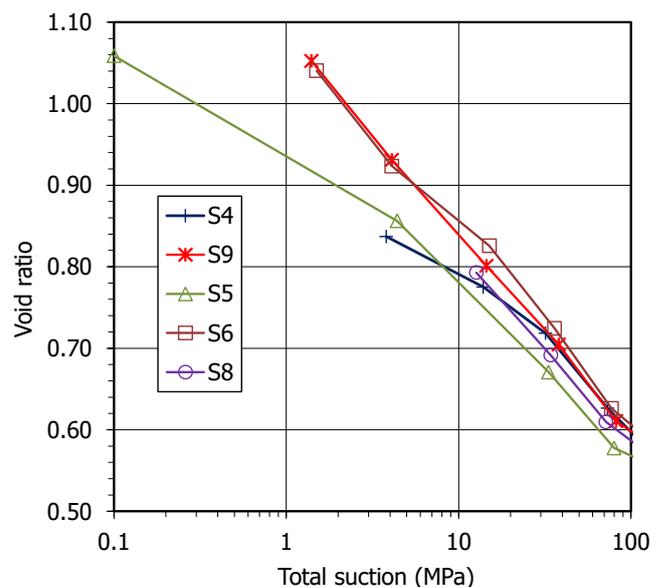


Figure 7. Evolution of void ratio during wetting above 100 MPa for different tests (tests S5, S6 and S8 experienced previously drying).

The pore size distribution of the sample from test S9 was determined by mercury intrusion porosimetry (MIP) and the results obtained are plotted in Figure 9, along with the results for a sample of FEBEX bentonite compacted with its hygroscopic water content at a nominal dry density of 1.70 g/cm^3 , which is considered representative of the initial conditions of all the oedometric tests performed. In the two cases the sample was previously lyophilised. For the range of pores explored by MIP, the two samples presented two major pore families, one of them in the range of the macro-pores, with a pore mode around $20 \mu\text{m}$, and another one in the range of the mesopores, with a pore mode around 7 nm . However, the pore size distribution, particularly concerning the pores smaller than 6 nm that cannot be intruded by mercury, was greatly modified during the test, as it can be observed in Figure 10, where the results have been plotted in terms of intruded void ratio. The percentage of macro-pores decreased (the macrostructural void ratio decreased from 0.32 to 0.28 , despite the overall increase in void ratio) and the percentage of pores smaller than 50 nm increased from 54 to 68% (the void ratio of pores smaller than 50 nm increased from 0.38 to 0.61). Hence, the volume increase brought about by hydration under a low vertical load would have been accomplished through a significant increase in the volume of smaller pores (micropores). Sánchez et al. (2012) modelled the irreversibility of the bentonite strains occurred upon wetting under a low vertical load by considering that the expansion of the aggregates leads to an invasion of the macro-pores with a consequent reduction of the macrostructural void ratio, which has been confirmed by the MIP curves presented here.

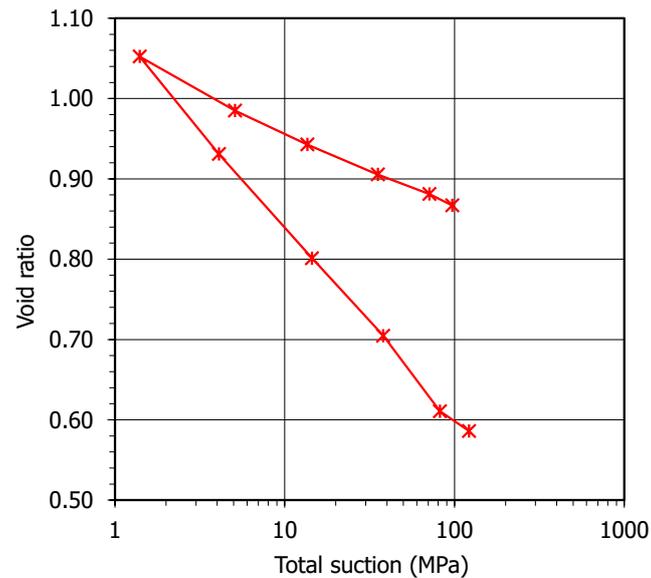


Figure 8. Evolution of void ratio during wetting/drying under vertical load 0.1 MPa in test S9.

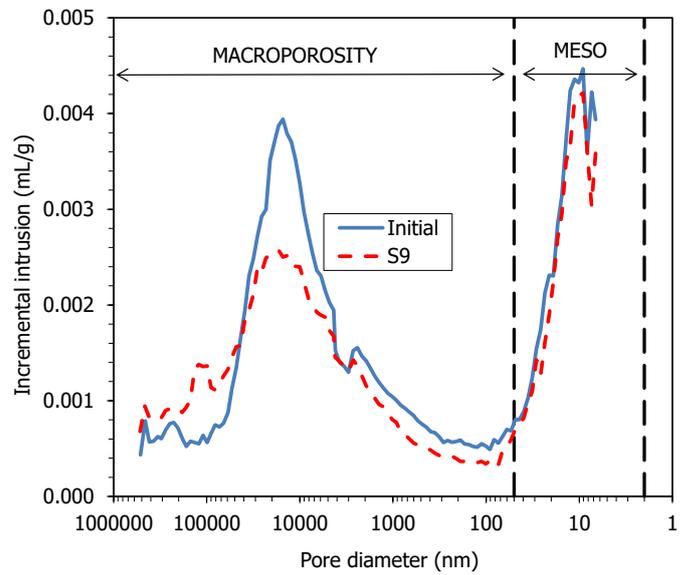


Figure 9. Mercury intrusion porosimetry curves determined at the end of test S9 and in a sample representative of the initial conditions.

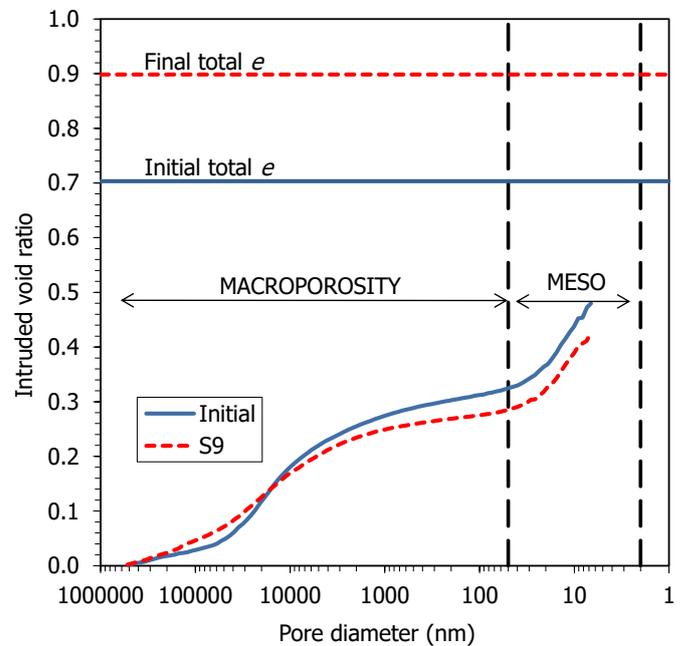


Figure 10: Total and intruded void ratio as determined by MIP at the end of test S9 and in a sample representative of the initial conditions.

Also, the water content at the end of the drying path was higher than the initial one for the same suction (17.1 vs. 13.8%). Accordingly, the interlayer distance as determined by X-ray diffraction increased during the test from the 1.48 nm typical of the FEBEX bentonite at hygroscopic suction conditions, to 1.53 nm .

4 CONCLUSIONS

Samples of highly-compacted FEBEX bentonite were submitted to wetting/drying and drying/wetting paths in suction-controlled oedometers under low

vertical loads. These tests have revealed the following features of behaviour:

- The bentonite showed a stiff behaviour when suction increased from hygroscopic conditions to values of close to 500 MPa, corresponding to water contents around 4%.
- During the subsequent hydration, the swelling strains occurring between 500 and 300 MPa were very small, which could be due to plastification of the material on drying, this displacing the elastic range to the interval between those suctions.
- The decrease of suction below the initial value of 130 MPa did cause a significant increase in volume. In fact, drying did not modify the swelling capacity of the sample during subsequent hydration, on the contrary, the deformations observed during wetting seemed to be larger in the samples previously dried.
- In wetting/drying paths the swelling deformation occurred on suction decrease under low vertical stress was irreversible and mainly caused by the increase in the volume of pores smaller than 6 nm and the increase in the water content of the interlayer.
- During wetting, the time for stabilization of the deformation under a low vertical load and for suctions below 14 MPa was extremely long, despite the small size of the samples. This limits the validity of this kind of results obtained under conventional testing times.

5 ACKNOWLEDGEMENTS

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