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M. V. Villar
F. J. Romero

GOBIERNO DE ESPAÑA
MINISTERIO DE ECONOMÍA Y COMPETITIVIDAD
Ciemat
Centro de Investigaciones Energéticas, Medioambientales y Tecnológicas

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SAMPLING

Water Retention Curves of Opalinus Clay

Villar, M. V.; Romero, F. J.
33 pp. 7 ref. 25 figs. 21 tables

Abstract:
The water retention curve of Opalinus clay samples was determined under different conditions: total and matric suction, stress or no-stress conditions, wetting and drying paths. Through the fitting of these results to the van Genuchten expression the P parameter, related to the air entry value (AEV), was obtained. The AEV is the suction value above which air is able to enter the pores of the sample, and consequently, above which 2-phase flow can take place in the soil pore structure. The samples used in this research came from two different boreholes, BHT-1 and BHG-D1, but the behaviour of them did not depend on their location, what was probably due to the fact that both were drilled in the shaly facies of the Opalinus clay.
There was not a distinct difference between the results obtained under total or matric suctions. In the drying paths, both the water contents and the degrees of saturation tended to be higher when total suction was applied, however the reverse trend was observed for the water contents reached in wetting paths. As well, no clear difference was observed in the water retention curves obtained in oedometers under matric and total suctions, what points to the osmotic component of suction in Opalinus clay not being significant. Overall, the water contents were lower and the degrees of saturation higher when suction was applied under vertical stress, what would indicate that the water retention capacity was lower under 8 MPa vertical stress than under free volume conditions. This vertical stress value is slightly higher than the maximum in situ stress. Also, the samples showed hysteresis according to the expected behaviour, i.e. the water contents for a given suction were higher during a drying path than during a wetting path. The P values obtained were between 6 and 34 MPa, and tended to be higher for the samples tested under stress, in drying paths and when total suction was used. The air entry value calculated from the mercury intrusion porosimetry tests was 28 MPa.

Curvas de Retención de Agua de la Argilita Opalinus

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33 pp. 7 ref. 25 figs. 21 tablas

Resumen:
Se ha determinado la curva de retención de la arcilla Opalinus bajo diferentes condiciones: succión total y matricial, condiciones de tensión y no tensión, trayectorias de mojado y secado. Al ajustar estos resultados a las expresión de van Genuchten se obtiene el parámetro P, que está relacionado con el valor de entrada de aire (VEA), que es la succión por encima de la cual el aire puede entrar en los poros de la muestra y por tanto, a partir de la que se puede producir flujo bifásico en la estructura porosa del suelo. Las muestras utilizadas provenían de dos sondeos, BHT-1 y BHG-D1, pero el comportamiento de unas y otras es similar, probablemente debido a que ambos sondeos se perforaron en la facies arcillosa de la formación.
No se observó diferencia clara entre los resultados obtenidos bajo succión total y matricial. En las trayectorias de secado tanto las humedades como los grados de saturación tendieron a ser mayores cuando se aplicó succión total, pero se observó la tendencia contraria para las humedades en trayectorias de mojado. Así mismo, no se observaron diferencias claras en las curvas de retención obtenidas en edómetro bajo succión total o matricial, lo que parece indicar que el componente osmótico de la succión de la Opalinus no es significativo. En general las humedades fueron menores y los grados de saturación mayores cuando se aplicó la succión bajo tensión vertical, lo que indicaría que la capacidad de retención fue menor bajo tensión vertical de 8 MPa que en condiciones de volumen libre. Este valor de tensión vertical es algo mayor que la máxima tensión medida in situ. Los resultados mostraron histéresis de acuerdo a lo esperado: las humedades para una succión dada fueron mayores en las trayectorias de secado que en las de mojado. El valor obtenido para el parámetro P fue de entre 6 y 34 MPa, con tendencia a ser mayor para las muestras ensayadas bajo tensión vertical, en las trayectorias de secado y cuando se usó succión total. El VEA calculado a partir de la porosimetría por intrusión de mercurio fue de 28 MPa.

Contents

1 Introduction

The multiple barrier concept is the cornerstone of all proposed schemes for underground disposal of high-level radioactive wastes. The concept invokes a series of barriers, both engineered and natural, between the waste and the surface. Understanding gas generation and migration is vital in the quantitative assessment of repositories and was the focus of the research in the integrated, multi-disciplinary project FORGE. The FORGE project was a pan-European project with links to international radioactive waste management organisations, regulators and academia, specifically designed to tackle the key research issues associated with the generation and movement of repository gasses in bentonite buffers, plastic clays, indurated mudrocks and crystalline formations. These issues were addressed through a series of laboratory and field-scale experiments. One of the materials studied in the project was the Opalinus claystone in Switzerland, which is considered as potential host rock in the Swiss radioactive waste disposal concept.

This report includes part of the work carried out by CIEMAT in FORGE WP5.1 "Gas transport laboratory experiments". One of the tasks undertaken by CIEMAT in this work package was the determination of parameters of two-phase flow –which is one of the possible gas migration mechanisms– in Opalinus clay. With this aim the water retention curves (WRC) of Opalinus clay samples were determined under high confinement –to simulate the *in situ* conditions– and under free volume conditions for comparison, since this gas transport mechanism is sensitive to the stress state, in particular to porosity changes. The fitting parameter *P* of the WRCs is usually related to the air-entry value, which is key to evaluate the two-phase flow.

To determine the WRC of the undisturbed material under vertical stress, suction-controlled oedometers –in which the sample was laterally confined and placed under constant vertical stress and suction– were used. The vertical stress applied during the tests was 8 MPa, the maximum that could be reached in the equipments, and the suction applied was either matric or total. The measurements of the stress state at Mont Terri indicate that σ_1 is 6-7 MPa (Corkum & Martin 2007), *i.e*. slightly lower than the vertical pressure applied in the oedometers. To apply matric suctions up to 14 MPa the axis translation technique was used. To apply total suctions higher than 3 MPa the relative humidity of the atmosphere in which the sample was placed was controlled by means of sulphuric acid solutions of different concentrations. The water retention curves were also determined with the same suction control techniques in non confined samples, so that to evaluate the effect of the mechanical stress on the water retention capacity and in particular, on the air entry value.

2 Material

The material used in the tests came from two different boreholes drilled in the Opalinus Clay Mesozoic formation in the Mont Terri Underground Research Laboratory in the Folded Jura mountains (http://www.mont-terri.ch). This formation is a mainly marly claystone with differing proportions of sand and carbonates around 180 million years old (Aalenian). At the rock laboratory, the Opalinus Clay has a layer thickness of around 140 m.

From a mineralogical point of view the Opalinus Clay consists of 40-80% clay minerals (including mixed layers of swelling illite and smectite), 10-40% quartz, 5-40% calcite and smaller proportions of siderite, pyrite and organic carbon. The dry density range is between 2.20 and

2.41 g/cm^3, the water content between 5.0 and 8.9% and the hydraulic conductivity between $2\cdot10^{-14}$ and $1\cdot10^{-12}$ m/s.

Of the three facies of Opalinus Clay that can be distinguished, the materials used in this investigation belong to the shaly one, which is a homogeneous, barely visible laminated claystone with low sand content. They are undisturbed cores of two different boreholes:

- BHT-1, from the Hydrogen Transfer (HT) test run by ANDRA. Core samples from metres 7.35 to 14.73 were sent to CIEMAT by ANDRA on May 2009 and they were kept since then vacuum packed at 4°C. These samples were mainly used for mineralogical and geochemical determinations in the context of the HT Project (Fernández *et al*. 2011). Their water content and dry density were between 6.6 and 8.0% and around 2.26 g/cm^3, respectively. The samples used for the water retention curve determinations belong to the depth 14.30-14.73 and 12.42-12.96, and grain densities of 2.70 and 2.72 g/cm^3, respectively, were determined.
- BHG-D1, from the Reactive Gas Transport in Opalinus Clay (HG-D) test run by NAGRA. A core sample kept in resin from metres 11.8 to 12.5 was sent to CIEMAT by NAGRA on March 2010 specifically for the tests to be performed in the context of the FORGE project. It was kept since then at 20°C. The water content and dry density determined in some samples from this core were 7.1% and 2.28 g/cm^3, respectively, and its measured grain density was 2.70 g/cm^3.

Additionally, the total suction of a core from borehole BDR-1 was measured at laboratory temperature (21°C) with two capacitive sensors inserted in a suitable perforated hole. The equilibrium value was found to be 31.3±0.1 MPa for a dry density of 2.33 g/cm^3 and water content of 6.4% (determined in samples drilled from the core). The measured grain density for this sample was 2.71 g/cm^3.

A sample from the BHG-D1 core of dry density 2.4 g/cm^3 and water content 3.4% (slightly air-dried) was lyophilised and analysed by mercury intrusion porosimetry (MIP). The mercury intrusion method allows access only to the macroporosity (pores smaller than 600 µm) and to part of the mesopores (pores of diameter larger than 0.006 µm). An estimation of the percentage of pores actually intruded can be made by comparing the actual void ratio of the samples (computed from their dry density and density of solid particles) and the apparent void ratio calculated from mercury intrusion. Thus, the pore size distribution curves obtained by MIP were corrected to take into account the percentage of pores not intruded and are shown in Figure 1 for two subsamples. Most of the pore sizes were comprised in the range 2-50 nm, *i.e*. in the mesopore range, with a dominant pore mode of 11 nm. The air entry value corresponding to this dominant pore mode calculated from the Laplace's equation is 27.8 MPa.

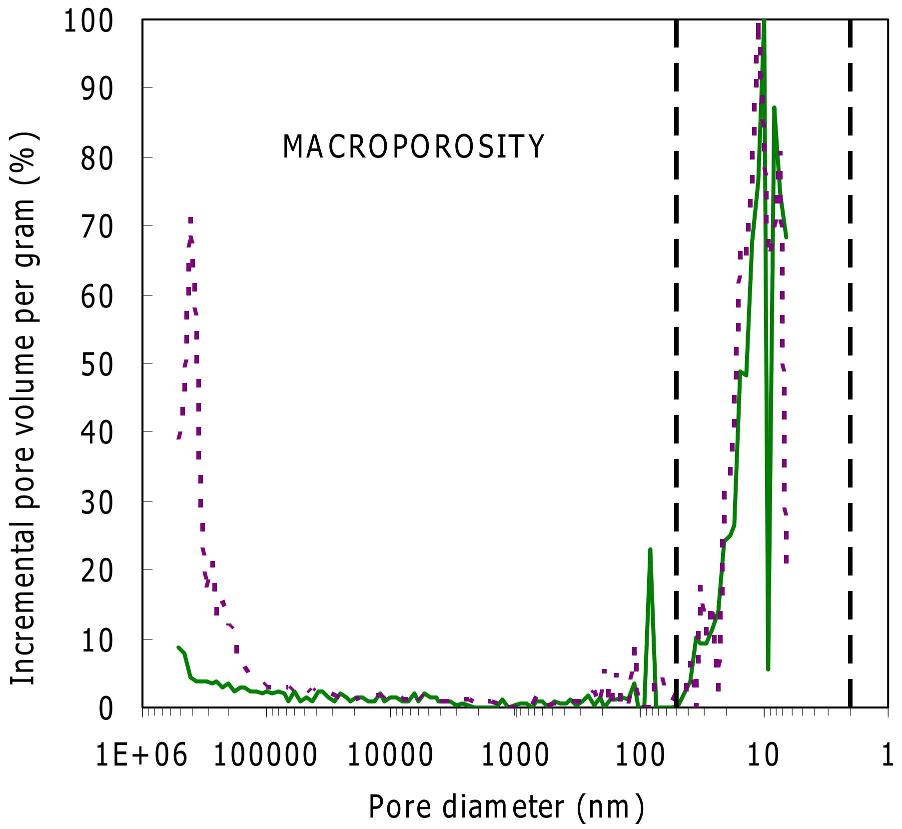

Figure 1: Mercury intrusion porosimetry curves for two samples taken from BHG-D1

3 Methodology

The techniques applied for the determination of the water retention curve (WRC) allow the study of the unsaturated behaviour of the material by imposing suction instead of measuring it, that is to say, by subjecting the sample to a given and known suction that conditions its water content, while the other variables (stresses, strains) are modified or measured (Villar 2002). Two different techniques were used to impose suction: axis translation (matric suction, in membrane cells) and the imposition of relative humidity (total suction, in cells with deposit for solution). The axis translation technique was applied both in oedometers –for determination of the WRC under constant stress– and in membrane cells, in which the sample experienced no mechanical stress imposition. The control of relative humidity was applied both in oedometers –for determination of the WRC under constant stress– and in desiccators, in which the sample experienced no mechanical stress imposition.

The characteristics of the tests performed in oedometers and the suction paths followed in each of them are shown in Table I. The same information is given in Table II for the tests performed in desiccators and in Table III for the tests performed in membrane cells. In the case of the tests in oedometers the initial dry densities were systematically lower than the dry density measured in other samples of the same boreholes. This is due to the fact that the samples trimmed were not exactly cylindrical and they did not fit precisely the ring contour at the beginning of the tests.

Table I: Initial characteristics of the samples used in the tests in oedometers and suction paths followed

Reference	Sample	ρ_d (g/cm^3)	*w* (%)	S_r (%)	Suction path (MPa)
MATRIC SUCTION					
EDN1_F1	BHG-D1	2.18	7.9	87	0-1-2-4
EDN3_F1	BHT-1 14.30-14.73	1.94	8.3	56	0-0.3-1-2-4-8-10-12
EDN5_F1	BHG-D1	2.14	7.5	76	14-11-8-5
TOTAL SUCTION					
EDS2_F1	BHG-D1	2.09	8.3	77	3-2
EDS2_F2	BHG-D1	2.21	7.2	87	4-7-11
EDS2_F3	BHG-D1	2.21	5.6	68	19-7
EDS3_F1	BHT-1 14.30-14.73	2.11	6.5	62	2-6-11-15-25-39-37-57
EDS3_F2	BHG-D1	2.14	7.3	75	70
EDS5_F1	BHG-D1	2.09			12-11-6-3
EDS5_F2	BHG-D1	2.19	6.6	78	11-19-23
Average		2.13±0.08	7.2±1.4	74±16	

Table II: Initial characteristics of the samples used in the tests in desiccators and suction paths followed

Reference	Depth (m)	ρ_d (g/cm^3)	*w* (%)	S_r (%)	Suction path (MPa)
WETTING					
BHT1-1	14.30-14.73	2.20	7.6	91	12-10-7-4
BHT1-2	14.30-14.73	2.22	6.8	85	
BHT1-3	14.30-14.73	2.21	6.6	80	
BHT1-4	12.42-12.96	2.20	6.5	74	
BHT1-9	12.42-12.96	2.18			
BHT1-10	12.42-12.96	2.20			
DRYING					
BHT1-5	14.30-14.73	2.20	8.1	96	2-7-10-13-19-35
BHT1-6	14.30-14.73	2.14	7.8	81	
BHT1-7	14.30-14.73	2.17	7.2	80	
BHT1-8	12.42-12.96	2.18	6.4	69	
BHT1-11	12.42-12.96	2.18	6.3	70	
BHT1-12	12.42-12.96	2.14	6.1	61	
Average		2.19±0.03	6.9±0.7	79±11	

Table III: Initial characteristics of the samples used in the tests in membrane cells and suction paths followed

Reference	ρ_d (g/cm^3)	w (%)	S_r (%)	Suction path (MPa)
BHT-1-1	2.21	6.3	74	0.2-0.5-1-2
BHT-1-2	2.22	7.2	87	14-12-10-8-6-4-2
BHT-1-3	2.20	7.0	81	14-12-10-8-6-4-2-1
BHG-D1-1	2.12	8.7	86	14-12-10
BHG-D1-2	2.21	7.0	85	0.5
BHG-D1-3	2.22	4.4	55	0.8
BHG-D1-4	2.21	7.9	96	8
BHG-D1-5	2.20	6.5	77	2
BHG-D1-6	2.16	6.9	75	4-8-12
BHG-D1-7	2.20	7.1	85	6
BHG-D1-8	2.15	7.2	76	14-10-6
BHG-D1-9	2.26	6.7	94	1-2-4
BHG-D1-10	2.23	6.5	83	5-3
BHG-D1-11	2.20	7.0	83	3-5
BHG-D1-12	2.25	5.2	71	0.5-1-1.5
BHG-D1-13	2.23	6.7	85	12-10
BHG-D1-14	2.22	6.2	78	10-8
BHG-D1-15	2.19	7.3	83	10
BHG-D1-16	2.12	6.6	65	0.5
BHG-D1-17	2.24	6.1	80	2
BHG-D1-18	2.20	6.9	82	3
BHG-D1-19	2.26	6.3	87	6
BHG-D1-20	2.26	5.8	80	11
BHG-D1-21	2.23	6.8	88	13
Average	2.21±0.04	6.7±0.8	81±9	

3.1 SUCTION CONTROL TECHNIQUES

3.1.1 Matric suction

Matric suction was applied to the samples through the axis translation technique. The principle of axis translation is the modification of the suction of a sample by increasing the pressure of the gaseous phase in its pores. The sample is placed in a cell in contact with water at atmospheric pressure through a membrane permeable to water but not to gas. These regenerated cellulose membranes have a pore diameter of 2.4 nm. The pressure in the cell is increased by injecting gas at the desired pressure, this increasing the air pressure in the pores of the sample. This new situation forces the sample to exchange water through the membrane until equilibrium is reached once again. Given that the changes in capillary (matric) suction are caused by the difference between the pressure of the air in the pores (u_a) and the pressure of the water (u_w), when air pressure is applied to the sample an increase in u_a is induced, while u_w remains the same as atmospheric pressure. In this way, capillary suction varies by the same amount as gas pressure. The membrane allows ions to pass through, as a result of which osmotic suction is not controlled by this method.

The oedometer membrane cells used are manufactured in stainless steel and consists of a base, cover and central body (Figure 2). The oedometer ring is housed in the body of the cell. The upper part of the cell has a central orifice for passage of the loading ram. This rests on the load distributing piston, which has a porous stone attached to it at its lower end, which remains directly in contact with the sample. The cover of the cell also has a gas inlet with a manometer for values of up to 16 MPa. Externally, a linear potentiometric transducer, with a level of accuracy of thousands of a millimetre, coupled to the loading ram and connected to a data acquisition system, rests on the cell cover so that to measure the vertical deformation of the sample. The base of the cell has an embedded porous stone, below which there are two inlet and outlet orifices, connected to a deposit with water at atmospheric pressure. The semipermeable, regenerated cellulose membrane is placed over this stone, with the sample resting directly on it. A peristaltic pump, installed between the deposit and the cell inlets, facilitates the removal of the gas that could diffuse through the membrane. Given the mechanical limitations of the cell, it is possible only to apply matric suctions of less than 14 MPa. Industrial nitrogen was used as the gas, deionised water for saturation and the tests were performed at 20°C.

The cells used for the determination of the WRC under no mechanical stress are essentially like the one just described except for there is no loading ram or ring to confine the sample (Figure 3).

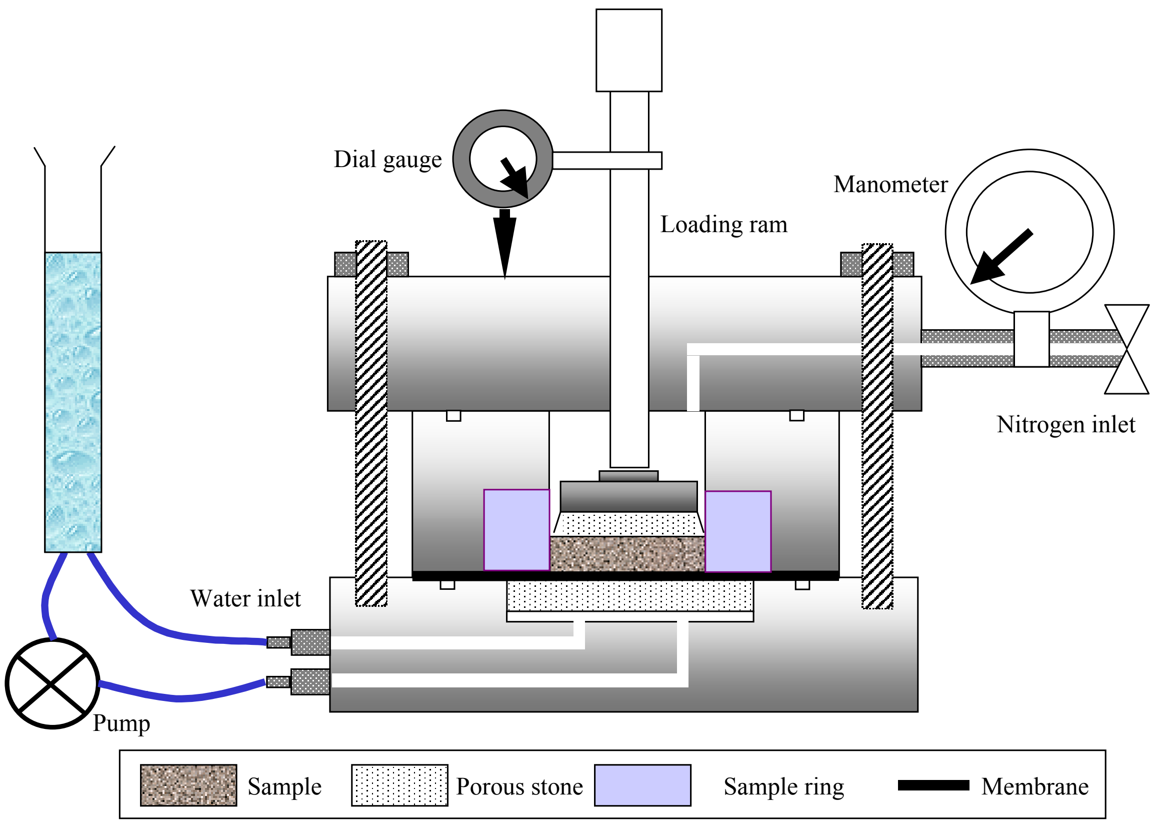

Figure 2: Schematic cross section of an oedometric membrane cell (Villar 2002)

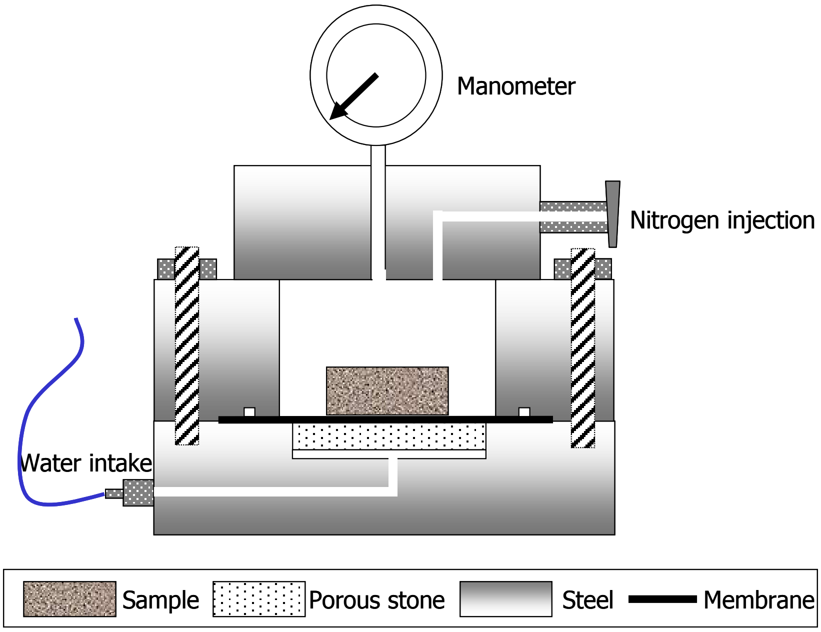

Figure 3: Schematic cross section of a membrane cell (Villar 2002)

3.1.2 Total suction

The method of imposing relative humidity (RH) is based on the fact that this conditions the pressure of the water and gas in the pores (u_w and u_a). This humidity may be imposed by means of solutions of sulphuric acid (although any other solution of known water activity may be used). The sample exchanges water with the atmosphere until thermodynamic equilibrium is reached with the vapour pressure of the solution, as a result of which total suction is modified. The suction in the pores of the sample (*s*, in MPa) is related to the activity of the water in the solution (a_w=RH/100) by means of Kelvin's law:

$$s = -10^{-6} \frac{R \times T}{V_w} \ln\left(\frac{\text{HR}}{100}\right) \qquad [1]$$

where *R* is the universal constant of the gases (8.3143 J/mol·K), *T* is absolute temperature and V_w is the molar volume of the water ($1.80\cdot10^{-5}$ m^3/mol).

The relation between the activity of the solution and the percentage in weight of sulphuric acid used to prepare it is reflected in experimental tables. The transfer of water between the clay and the atmosphere may cause the density of the solution to vary, as a result of which this should be checked prior to and following stabilisation, this being accomplished by means of pycnometers. There is an experimental relation between the specific gravity of the solution and the percentage in weight of the sulphuric acid in the solution (which in turn depends on activity), which is temperature-dependent.

Total suctions of between 3 and 500 MPa may be obtained using this method. In view of the influence of temperature on the activity of the solutions, this is kept constant at 20°C throughout the entire tests.

The oedometric equipment used includes modified oedometric cells in which suction may be applied. The cell with a deposit for solutions consists of a base and cover of high corrosion-resistant stainless steel (AISI 316L) and a cylindrical body of transparent material with an internal border on which rests a ring-shaped glass deposit (Figure 4). A porous stone rests on the base of the cell, over which is the oedometer ring with the sample and finally, the upper porous stone attached to the piston. This assembly is attached to the base of the cell by means of a steel flange. The upper cover has an orifice for the loading ram, a perforation for insertion of the sulphuric acid and an inlet for the creation of the vacuum. Externally, a linear potentiometric transducer, accurate to thousands of a millimetre and connected to a data acquisition system, rests on the cell cover, coupled to the loading ram, to measure the vertical deformation of the sample.

This technique is also applied in desiccators to determine the WRC under no mechanical stress (Figure 8).

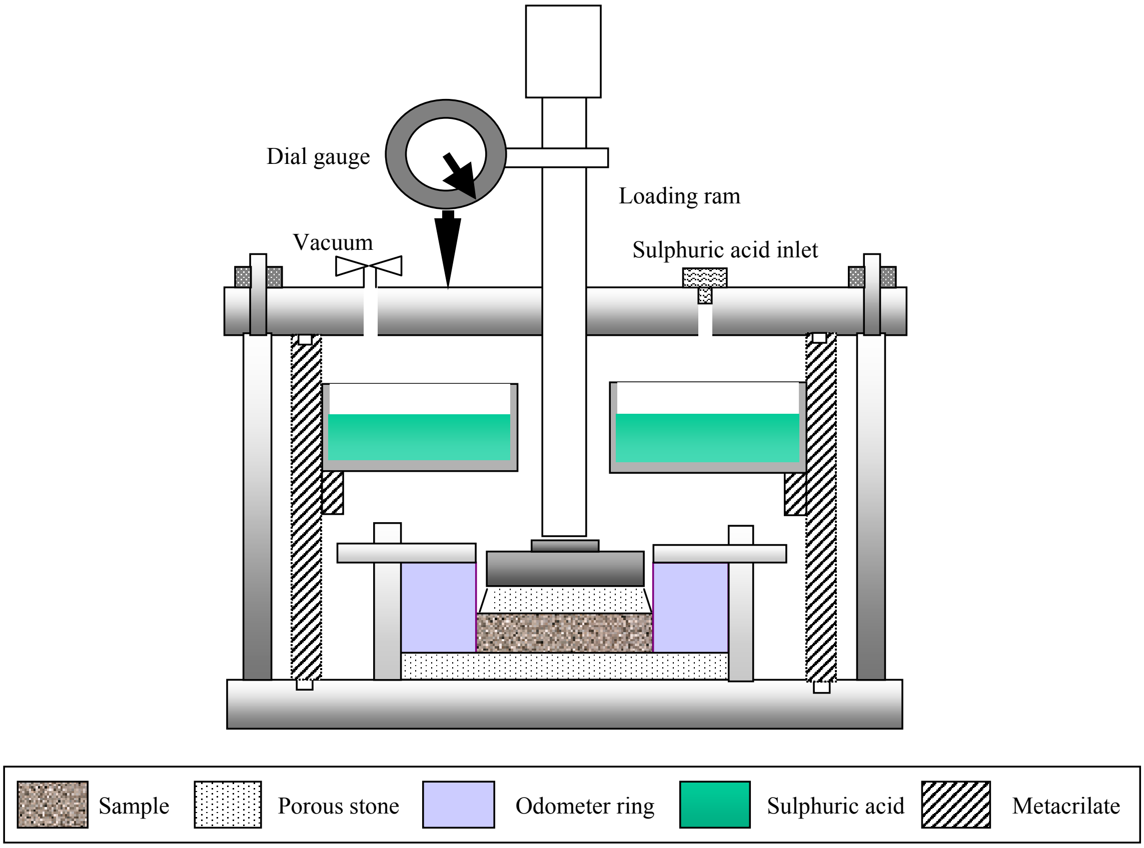

Figure 4: Schematic cross section of an oedometric cell with deposit for solutions (Villar 2002)

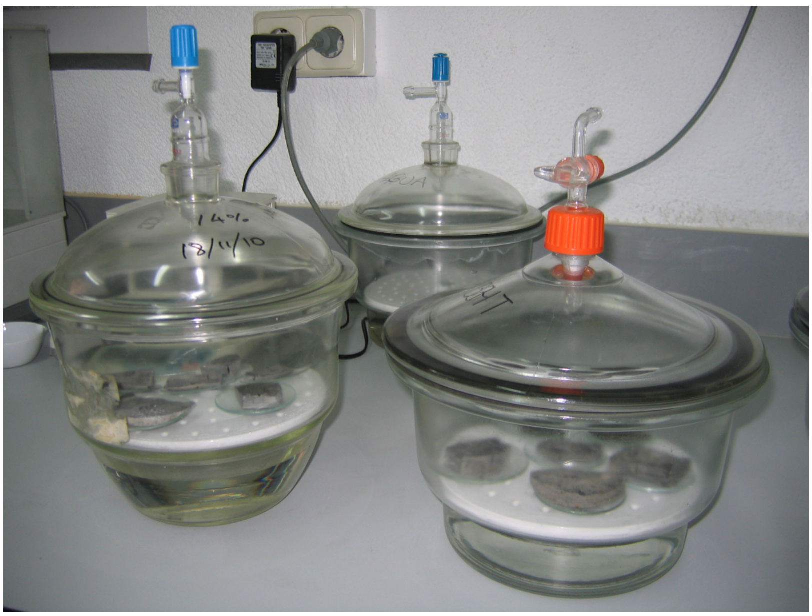

Figure 5: Desiccators for control of the relative humidity

3.2 EXPERIMENTAL PROCEDURE

3.2.1 Water retention curves under vertical stress

The undisturbed clay was trimmed in cylindrical specimens to fit the oedometer ring, *i.e*. 1.2-2.0 cm in length and 11.40 cm^2 in cross section. For that, an approximately 2-cm thick slice of the boreholes was cut with a band saw and a cylindrical specimen was obtained from it by trimming (Figure 6). This was a particularly difficult task due to the bad consistency of the material, which easily crumbled and exfoliated. In some cases the voids between the sample and the ring were filled up with crumbled adjacent material and slightly pressed. In spite of it, the initial dry density of the samples was lower than the borehole density (2.13±0.08 g/cm^3, see Table I).

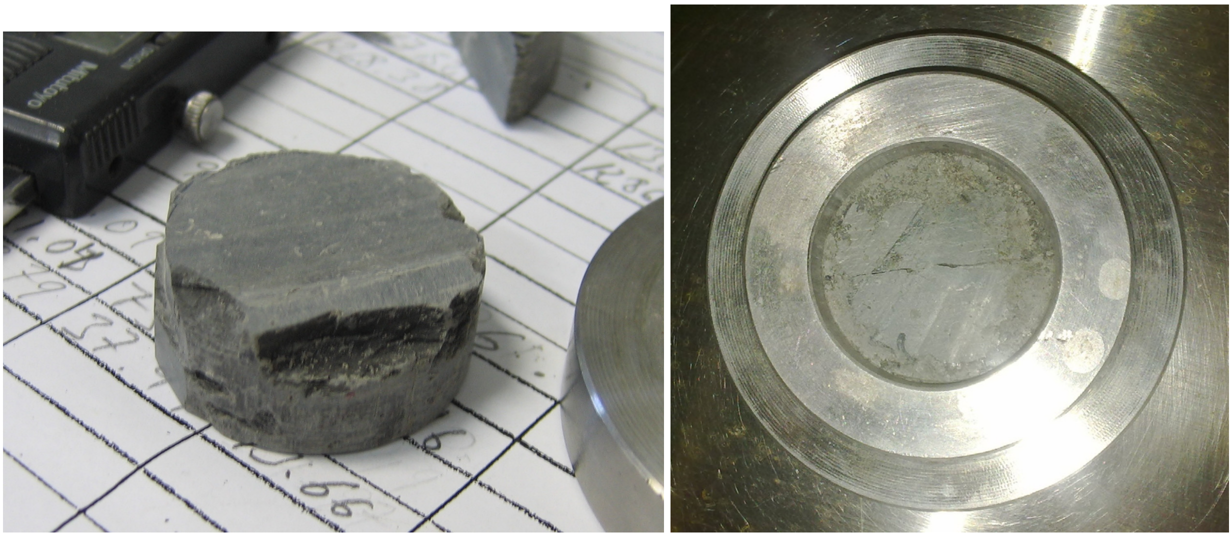

Figure 6: Appearance of samples prepared for the oedometer tests (left: EDS5_F1, right: EDS5_F2)

Once the oedometer ring with the sample was placed in the oedometer cell and load frame, the desired suction and vertical stress were applied simultaneously and the vertical strain recorded. Once equilibrium was reached for a given suction and stress (from previous experience it was considered that equilibrium was reached after two months or when there was no vertical strain change), the cell was released, the ring and the sample were weighed together (since the sample could not be removed from the ring without undergoing major disturbance) and the specimen height was indirectly measured. Afterwards, the oedometer ring was immediately assembled in the cell, which was placed in the oedometer frame, and the vertical stress was applied, along with the new suction.

At the end of the tests, the samples were extracted from the ring, measured, weighed and dried in the oven at 110°C for 48 h to determine their final water content.

Drying or wetting paths were followed, according to the equipment limitations. In the tests performed in oedometers with suction control by nitrogen pressure, the maximum applicable suction is 14 MPa, which was close to the *in situ* value of the Opalinus clay as determined in a previous research in samples from the VE test (Villar et al. 2009). In oedometers with suction controlled by solutions, the minimum suction applicable was 2 MPa.

In one of the tests performed in membrane cells (EDN3_F1) it was tried to measure the water exchange by using a piston pump to apply a water pressure of 200 kPa (instead of it being atmospheric). The piston pump was capable of applying a fixed pressure and measure small fluid volume variations. However, due to the quantity of gas that diffused through the membrane and entered the pump, the accurate measurement of water exchange failed and the pump was removed after the fourth suction step.

3.2.2 Water retention curves under no vertical stress

The WRC under no mechanical stress were determined both in membrane cells (axis translation technique, matric suction) and in desiccators (control of relative humidity, total suction). The samples used were obtained by cutting slices from the boreholes and trimming from them samples. In some cases these samples were the remainings of trimming circular-shaped samples for the tests in oedometers.

The samples were initially weighed and measured and subjected to the corresponding suction. Usually, the actual dry density and water content of fragments adjacent to those used in the determinations were initially determined. The samples' weight was periodically checked and the suction was kept in the same value until no weight variation was observed. Some samples were just subjected to one suction value, and after equilibrium they were measured (and sometimes also immersed in mercury to determine their volume) and dried in the oven at 110°C for 48 h to determine their water content. Other samples were submitted to a suction path, *i.e*. once equilibrium was reached for a given suction the sample was weighed and measured (and sometimes also immersed in mercury) and a new suction value was imposed. Drying in the oven did not take place until the end of the suction path.

These samples had not regular shapes (Figure 7, Figure 8), and their volume after hydraulic equilibrium was measured in some cases by immersing them in mercury, and in others by just measuring a couple of dimensions. The first method had the drawback that some mercury could get trapped inside the sample and affect the subsequent weight measurements, for which reason it was not applied when the samples were very exfoliated. The second method gave only an indication of the density change, which was considered proportional to the dimensions' change.

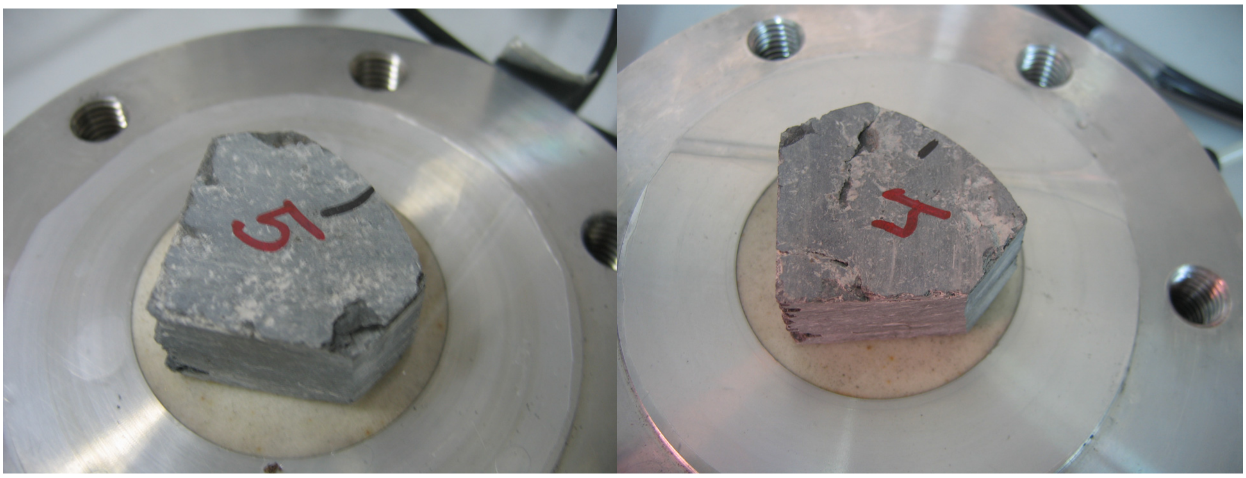

Figure 7: Samples used for the determination of the WRC in membrane cells

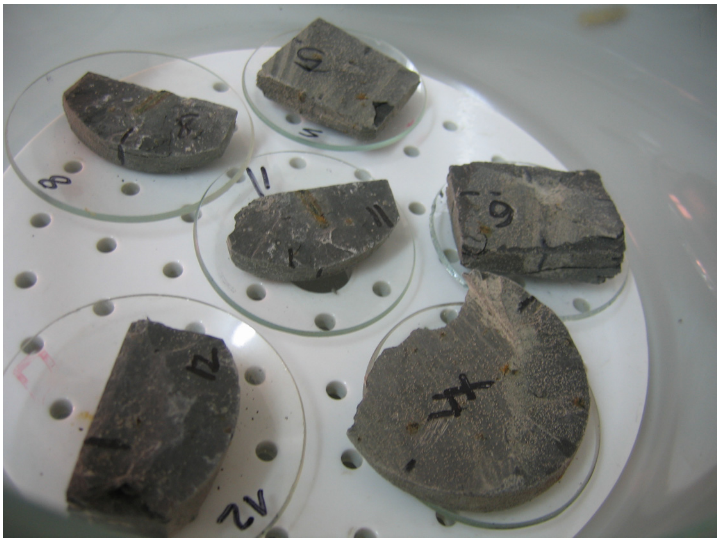

Figure 8: Samples for determination of the WRC in desiccators

4 Results

4.1 WATER RETENTION CURVES UNDER VERTICAL STRESS

The samples analysed had, once in the oedometer ring, and average dry density of 2.13±0.08 g/cm^3 and an initial water content of 7.2±1.4%. As explained above, the initial dry density was lower than the value corresponding to the borehole due to the fact that the shape of the samples was not perfectly regular and they did not fit entirely the oedometer ring at the beginning of the tests. Overall, the initial dry density of the samples clearly increased after the first suction step, due to the consolidation caused by the mechanical stress. In the subsequent steps, the dry density barely changed.

The results obtained in the suction-controlled oedometers can be found in Table V to Table XIV (Appendix 1) and they are shown in Figure 9 in terms of water content vs. suction changes and in Figure 10 in terms of degree of saturation. To analyse these results the following has to be taken into account:

- The paths followed were either of drying or of wetting, what could influence the equilibrium water content due to hysteresis, drying paths usually giving higher water contents.
- The two different methodologies applied for the control of suction applied total suction in one case (deposit cells, tests EDS) and matric suction in the other (membrane cells, tests EDN), what could give rise to different water contents if the osmotic component of suction were significant.
- To determine the degree of saturation for each suction step, the sample dimensions measurements taken upon relieving the vertical pressure and removing the oedometer ring from the cell were considered. Certain expansion of the sample could be expected after removal of the vertical stress, and thus the degrees of saturation could be underestimated with respect to those the samples actually had when they were submitted to the 8 MPa vertical stress in the oedometer.
- The samples used came from two different boreholes, BHT-1 (tests EDN3_F1 and EDS3_F1) and BHG-D1 (rest of the tests). As well as in the tests performed under free volume conditions (see below), no evidence of a different behaviour between both kinds of samples

was discernible though. However, the behaviour of the sample in test EDN3_F1 could be affected by the fact that it was initially flooded under suction 0 and that during the first steps a water pressure of 200 kPa was applied.

Overall, the water content and degree of saturation for a given suction was higher in drying than in wetting paths, what is the expected behaviour. However, there is not a clear difference between the results obtained under matric suction (tests EDN) or under total suction (tests EDS), for which reason no conclusion about the osmotic component of suction in Opalinus clay can be drawn.

Figure 11 and Figure 12 show the final appearance of samples from some of the tests performed in oedometers. The bedding planes were no longer visible, particularly in the samples coming from tests performed in membrane cells oedometers, in which the final values of water content were higher and the total duration longer (Figure 12).

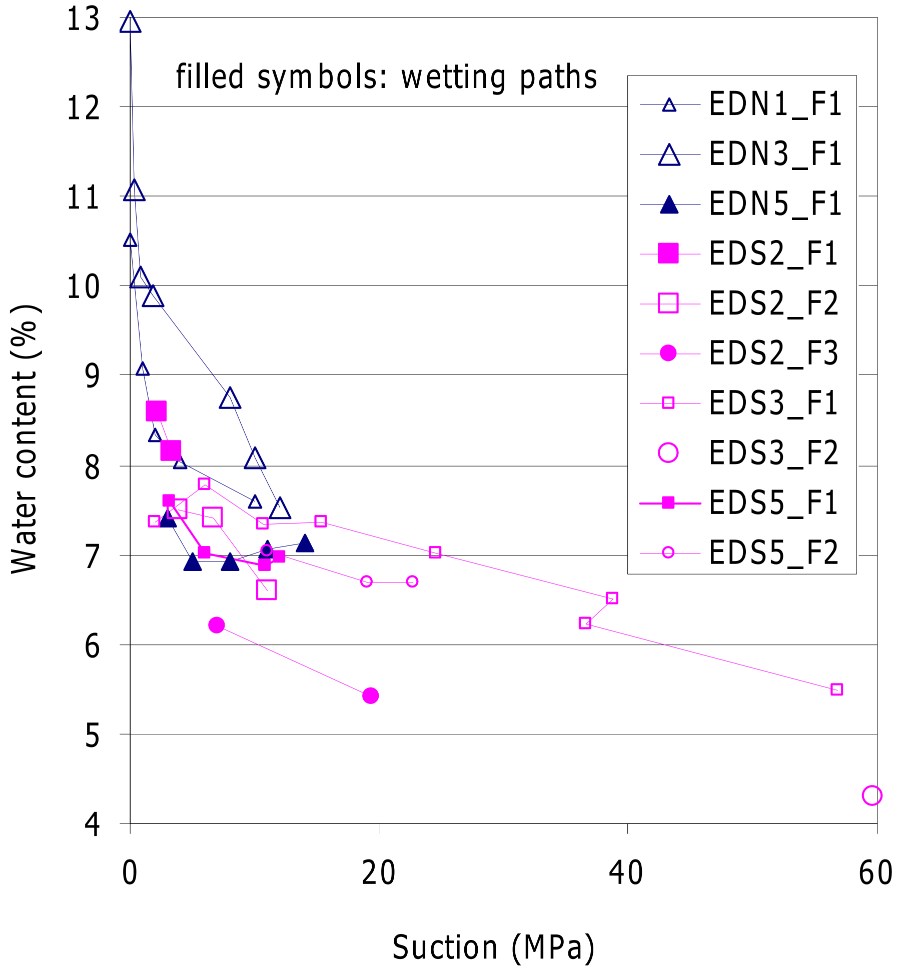

Figure 9: Results obtained in suction-controlled oeometers under vertical stress 8 MPa. Tests EDN: matric suction, tests EDS: total suction (detailed values in Table V to Table XIV)

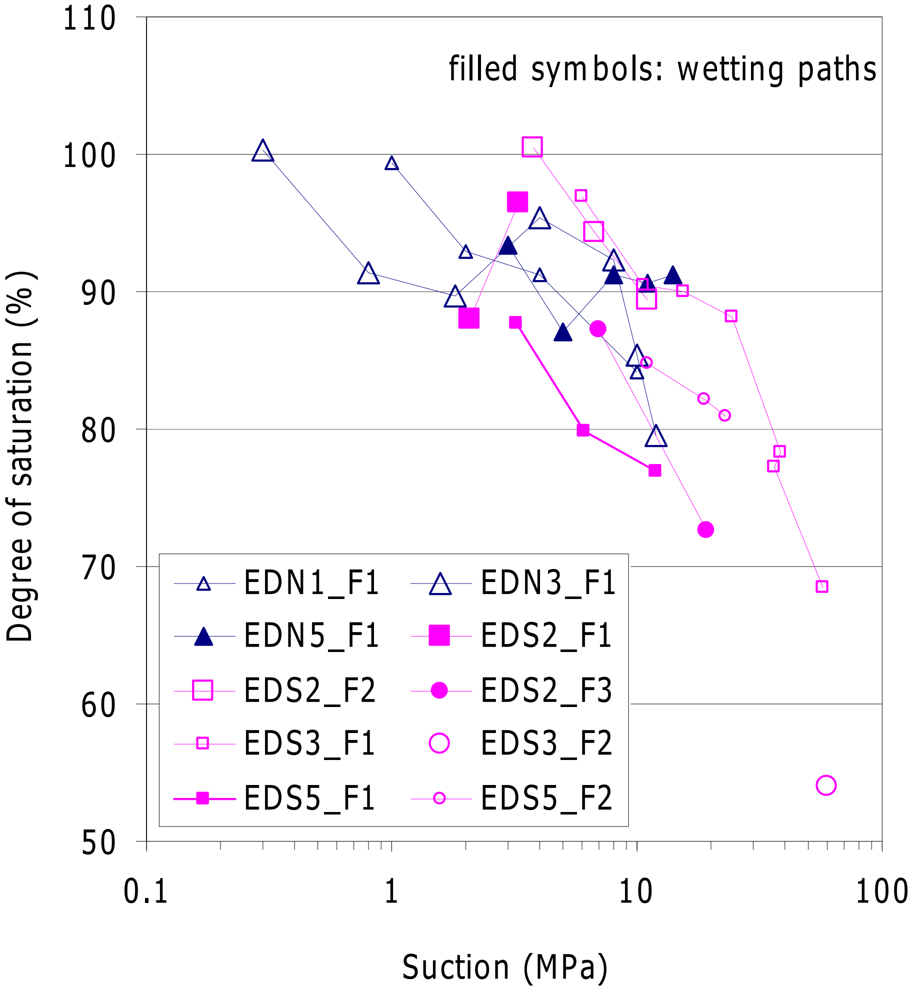

Figure 10: Results obtained in suction-controlled oeometers under vertical stress 8 MPa. Tests EDN: matric suction, tests EDS: total suction (detailed values in Table V to Table XIV)

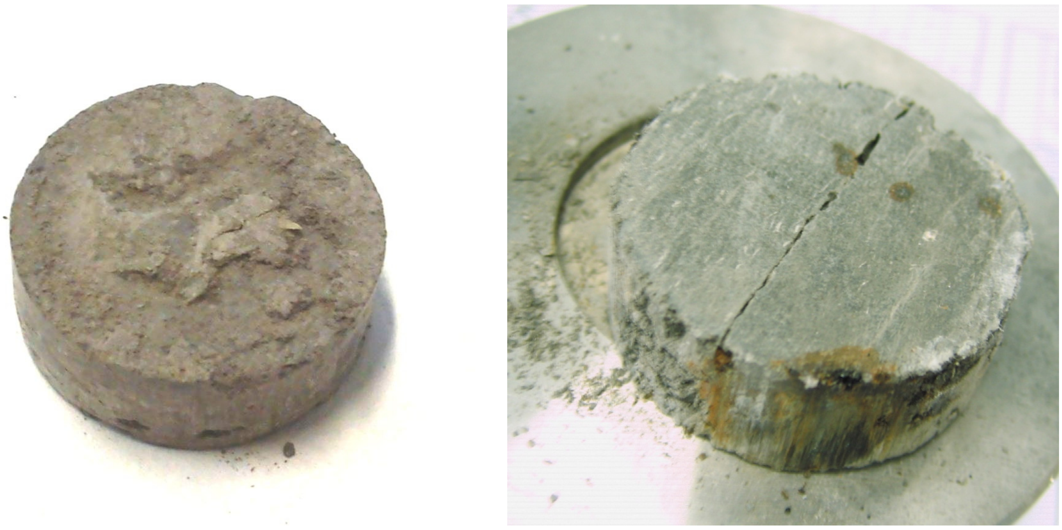

Figure 11: Final appearance of samples from test EDS2_F3 (left) and EDS5_F2 (right)

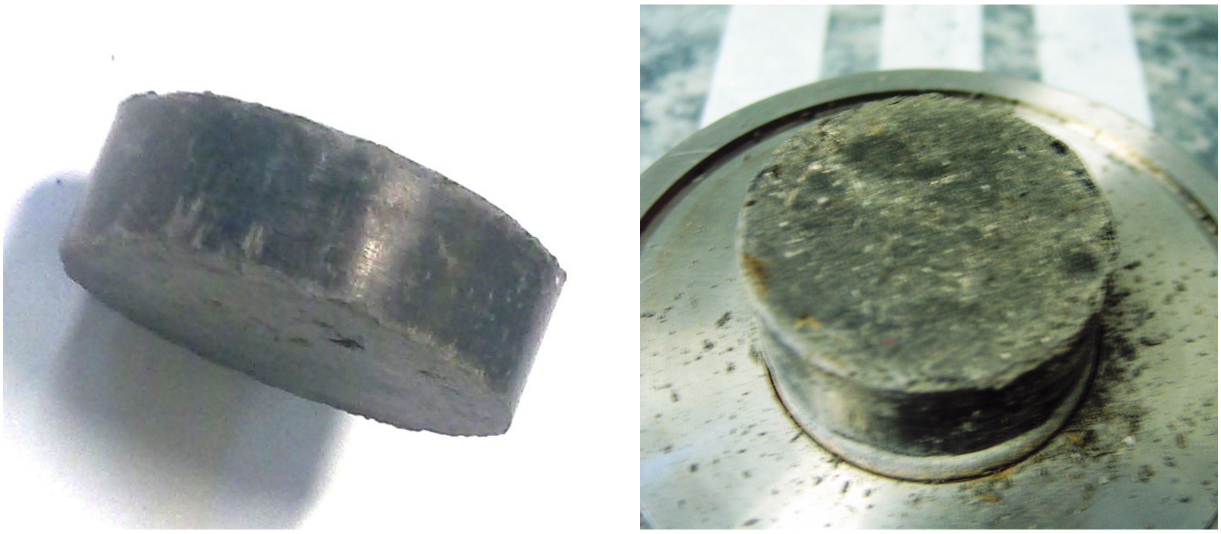

Figure 12: Final appearance of samples from test EDN3_F1 (left) and EDN1_F1 (right)

4.2 WATER RETENTION CURVES UNDER NO MECHANICAL STRESS

In order to analyse the effect of stress on the water retention capacity and the air entry value, the retention curve of the Opalinus clay was also determined in similar samples under no mechanical stress conditions, both in desiccators applying total suction and in membrane cells applying matric suction. The same concerns expressed in the previous section with respect to the interpretation of these results have to be taken into account. In addition, from borehole BHT-1 samples from two different depths (expressed in metres in the legend of the Figures) were used.

4.2.1 Water retention curves obtained in desiccators

Twelve samples from borehole BHT1 were tested in desiccators, six of them taken at depth 14.30-14.73 m (average initial ρ_d 2.19±0.03 g/cm^3 and *w* 7.4±0.6%) and six others at depth 12.42-12.96 m (average initial ρ_d 2.18±0.03 g/cm^3 and *w* 6.3±0.1%). Wetting paths from 12 to 4 MPa and drying paths from 2 to 35 MPa were followed. The detailed results are shown in Table XV and Table XVI (Appendix 1) and are plotted in Figure 13.

The results obtained in samples coming from the same depth were averaged and are plotted in Figure 14 in terms of water content and degree of saturation. The water contents and degrees of saturation for a given suction were higher for the drying paths in both kinds of samples. This can also be observed in Figure 15, where the results obtained for each sample are plotted grouped according to the path followed. In fact, for the drying path no difference was found between the samples coming from various depths, and the variation observed in the wetting path is not considered caused by the different provenance of the samples.

The control of the dry density evolution of the samples during the tests was performed by measuring the samples once equilibrium for a given suction was reached or by determining their volume by immersing them in mercury. Although the latter method gave a more precise result, it was only used in some of the steps because, due to the foliation of the samples, mercury got trapped in the porosity and this jeopardized the water content computation by weight change. On the other hand, the dimensional control of the sample gave only an approximate value for the dry density. Nevertheless the approximate results obtained showed that the dry density increased slightly in the first suction step and remained approximately constant during the rest of the tests.

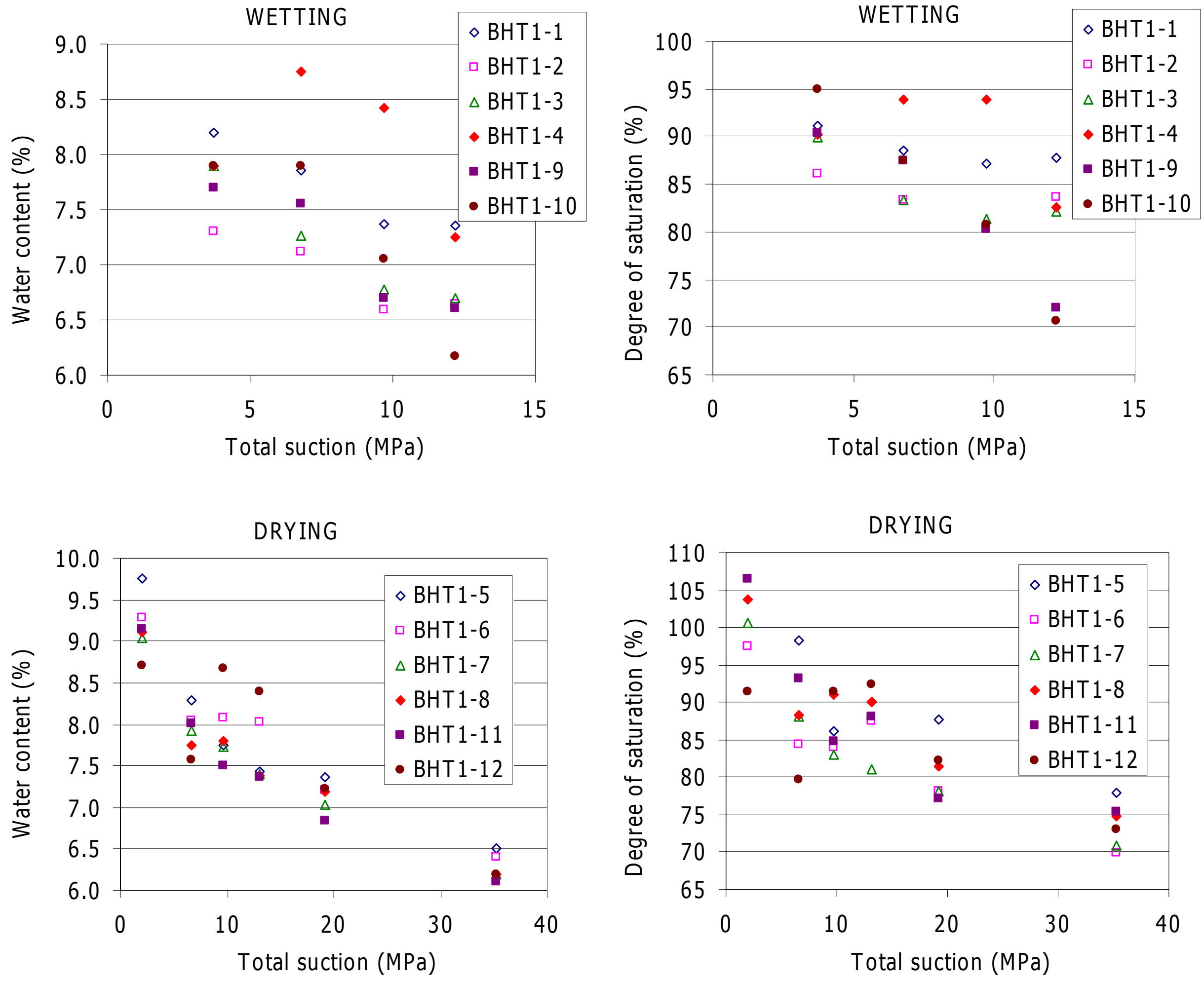

Figure 13: Water retention curves of samples from borehole BHT1 tested in desiccators following wetting and drying paths (samples 1 to 3 and 5 to 6 from m 14.30-14.73, samples 4 and 8 to 12 from m 12.42-12.96) (detailed values in Table XV and Table XVI)

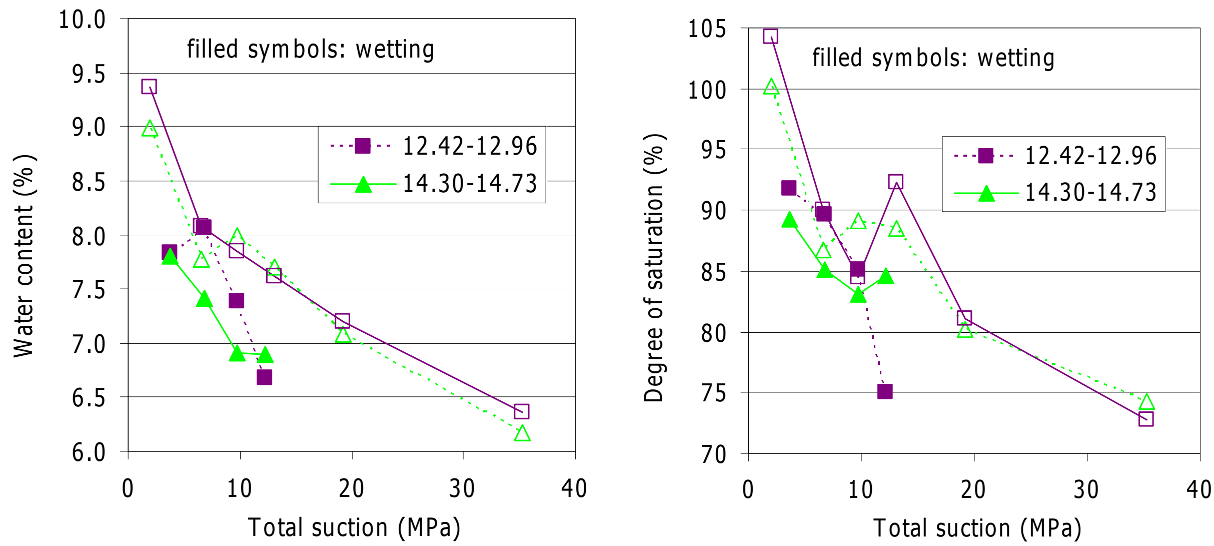

Figure 14: Water retention curves of samples from borehole BHT1 tested in desiccators following wetting and drying paths (each point is the average of 3 determinations)

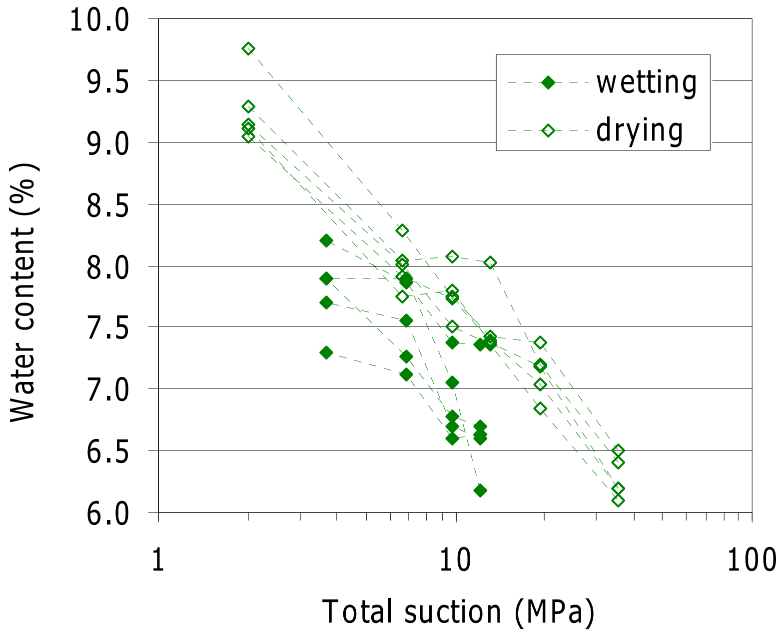

Figure 15: Water retention curves of samples from borehole BHT1 tested in desiccators following wetting and drying paths

4.2.2 Water retention curves obtained in membrane cells

The samples for determining the water retention curve in membrane cells, *i.e*. applying matric suction, were taken from boreholes BHT-1 (m 14.30-14.73) and BHG-D1. Some of the samples were subjected to different suctions following wetting or drying paths (results in Table XVII to Table XX in Appendix 1), whereas others were submitted just to a single suction value (Table XXI in Appendix 1). In both cases, upon equilibrium for a given suction step and in order to estimate the dry density change, the dimensions of the samples were measured and/or the volume of the samples was determined by immersion in mercury. In any case, the dry density values given in the Tables can only be considered as indicative, as well as the degree of saturation values (see section 3.2.2).

The results obtained are plotted in Figure 16 in terms of water content. The behaviour of the samples from the two boreholes is similar, and consequently they have not been in what follows treated separately. The results of all the determinations in membrane cells are plotted in terms of degree of saturation in Figure 17. The increase of degree of saturation with suction decrease was not as noticeable as the increase of water content, due to the fact that the samples experienced a decrease in dry density linked to the water content increase, because of the broadening of the foliation. The repercussion of the kind of path (wetting or drying) on the retention capacity was not clear.

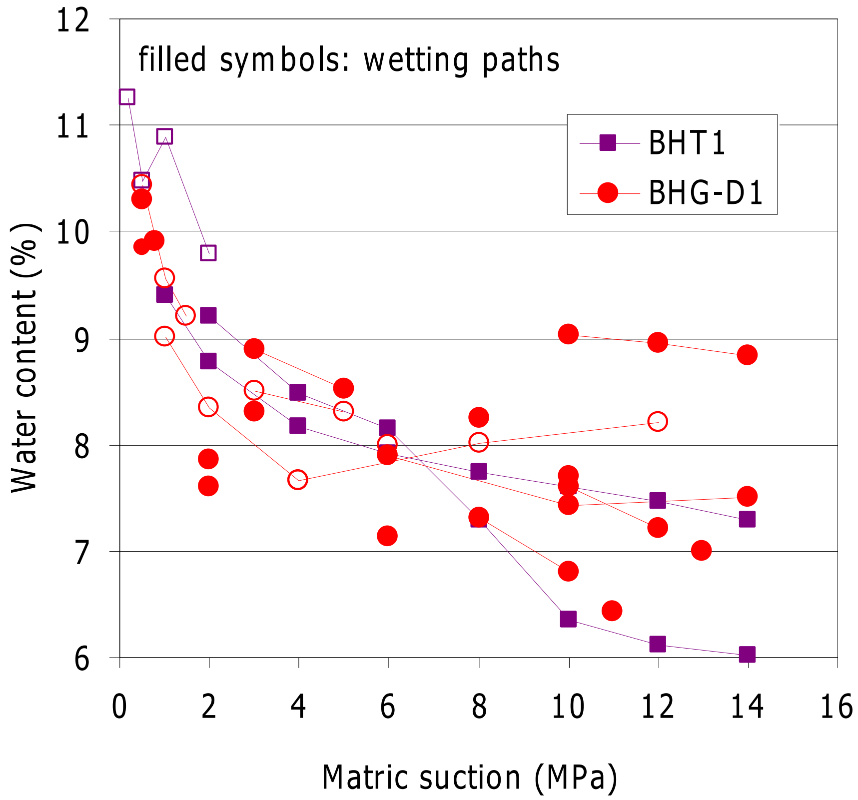

Figure 16: Water retention curves obtained in membrane cells in samples from two different boreholes following wetting and drying paths

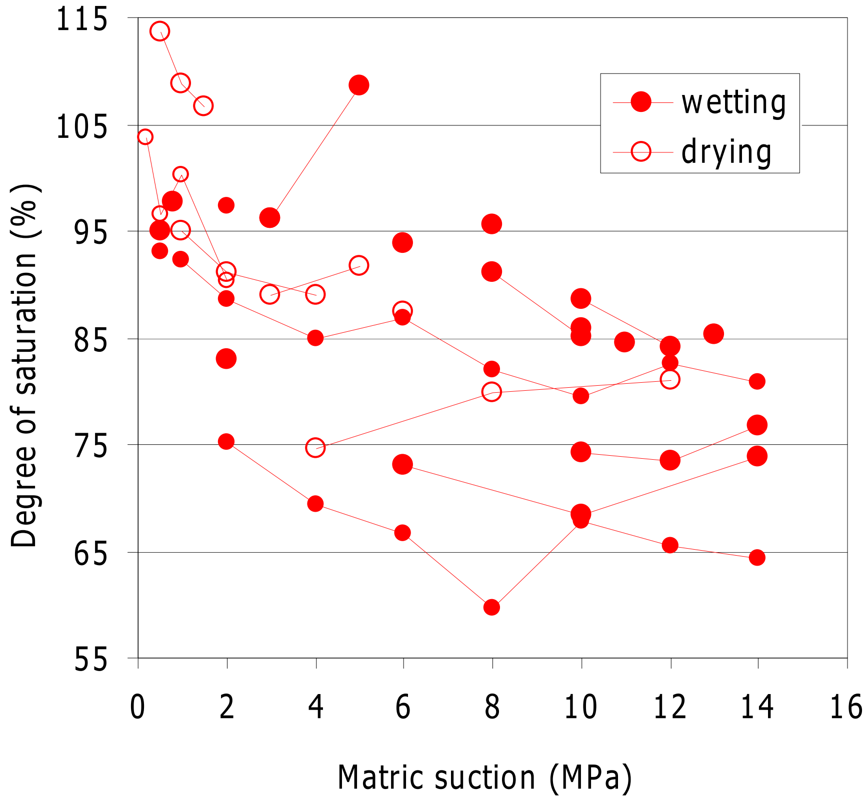

Figure 17: Water retention curves obtained in membrane cells in samples from two different boreholes following wetting and drying paths

4.3 ANALYSIS OF RESULTS

Water retention curves were obtained with the aim of determining the effects of vertical stress, hydraulic paths and kind of suction on the water retention capacity of the Opalinus clay.

Figure 18 compares the water retention curves obtained under vertical stress and under no vertical stress applying total suction, both in wetting and in drying paths and in terms of water content and of degree of saturation. There is a trend to find lower water contents for the same suction in samples tested under vertical stress, particularly towards the lowest suctions. However, when expressed in terms of degree of saturation this difference disappeared or even reverted in the case of the drying paths, what is due to the fact that in the tests performed in oedometers the average dry density of the samples was higher.

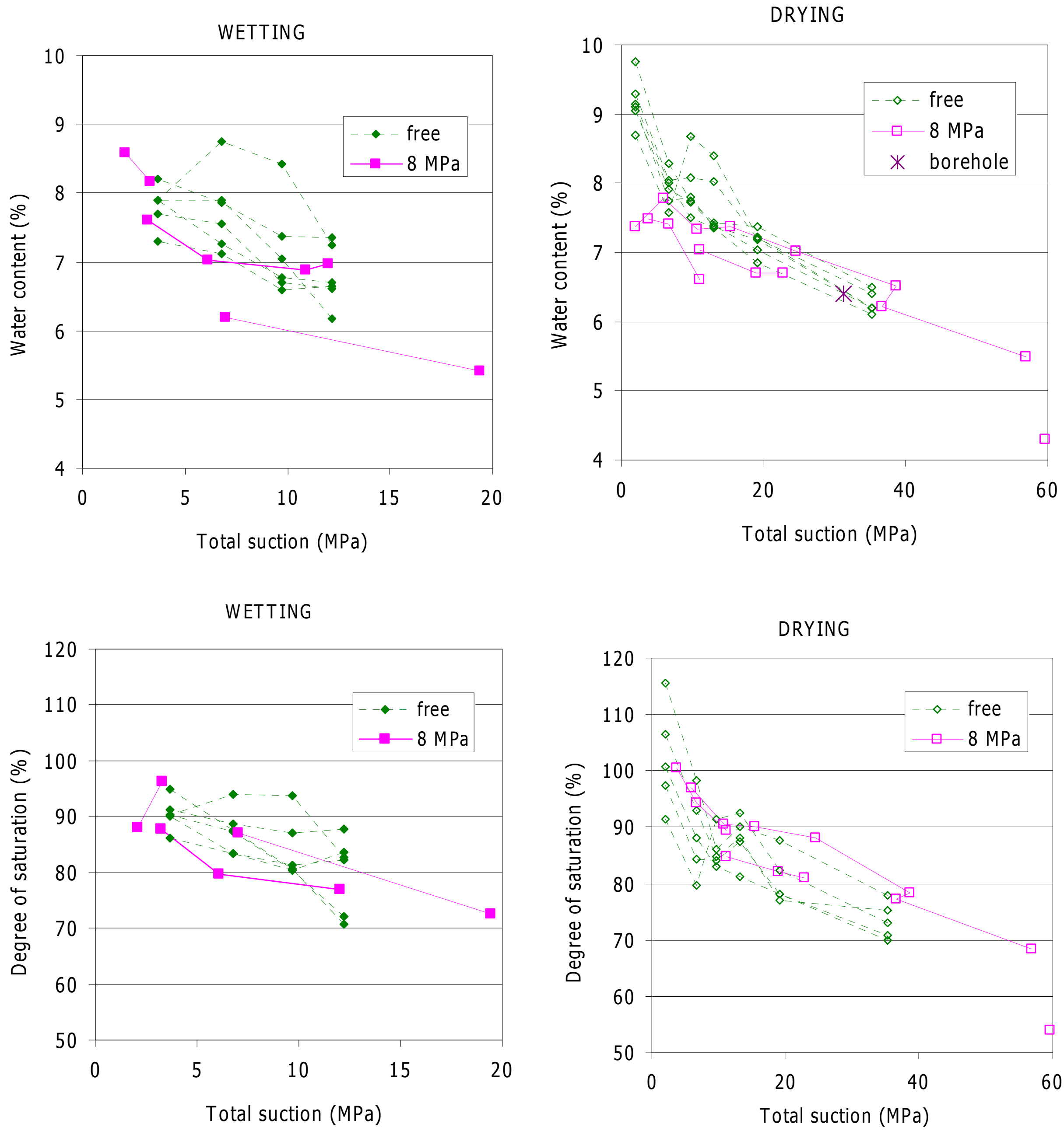

Figure 18: Water retention curves obtained applying total suction under 8 MPa vertical stress and under no mechanical stress following wetting (left) and drying (right) paths. The value directly measured in borehole BDR-1 is included in one of the graphs

In the curve in terms of water content corresponding to the drying path, the value measured by capacitive sensors inserted in borehole BDR-1 was also included (see section Material). This suction value agrees well with those obtained in trimmed samples, despite the fact that it has been obtained in a different borehole.

The same comparison is made in Figure 19 for tests performed applying matric suction. As in the case of the tests performed under total suction, the trend is for the water contents to be lower and the degrees of saturation higher when suction was applied under vertical stress.

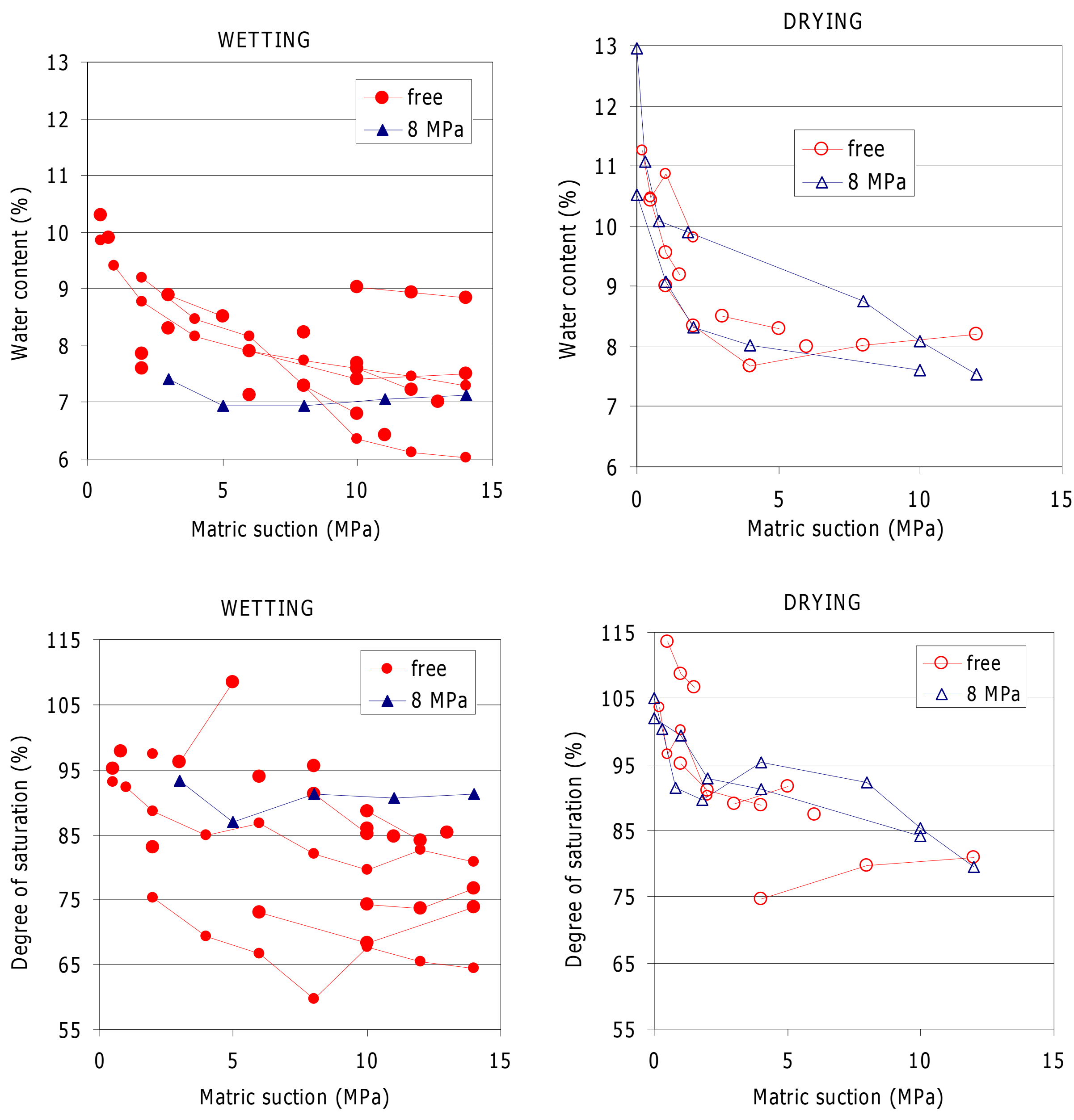

Figure 19: Water retention curves obtained applying matric suction under 8 MPa vertical stress and under no mechanical stress following wetting (left) and drying (right) paths

Figure 20 shows a summary of the results obtained under no stress conditions applying both matric and total suction. There is not a distinct difference between the results obtained under total or matric suctions. In the drying paths, both the water contents and the degrees of saturation tended to be higher when total suction was applied, however the reverse trend was observed for the water contents reached in wetting paths. As well, no clear difference was observed in the water retention curves obtained in oedometers applying total or matric suction (Figure 9, Figure 10), what points to the osmotic component of suction in Opalinus clay not being significant.

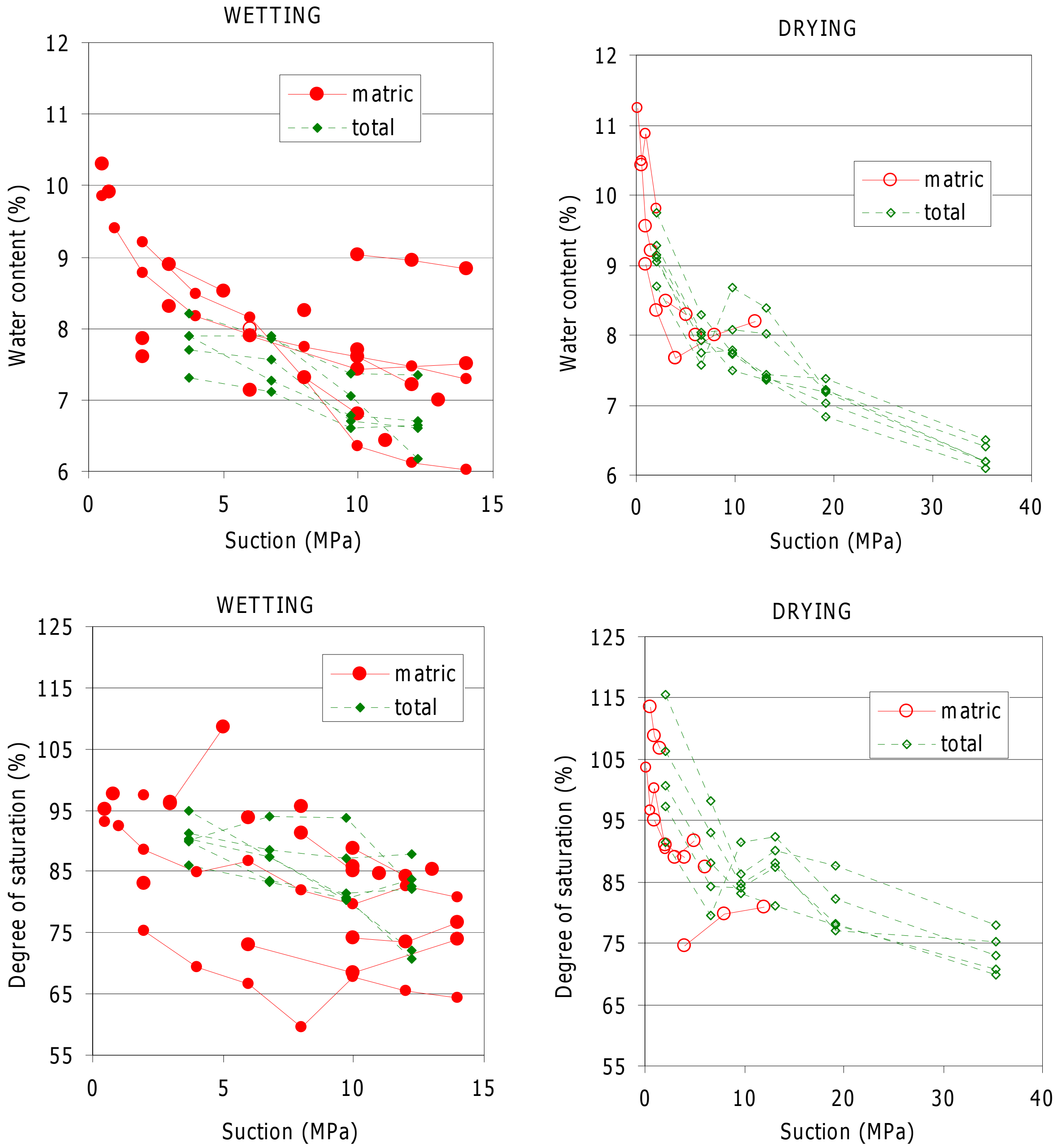

Figure 20: Retention curves obtained under free volume conditions in Opalinus clay samples from boreholes BHT1 and BHG-D1

4.4 FITTINGS TO THE VAN GENUCHTEN EXPRESSION

The results obtained have been fitted to the van Genuchten expression, in order to obtain the parameters that define the water retention curves:

$$S_e = \frac{S_l - S_{rl}}{S_{ls} - S_{rl}} = \left(1 + \left(\frac{P_g - P_l}{P}\right)^{\frac{1}{1-\lambda}}\right)^{-\lambda} \qquad [2]$$

where S_e is the effective degree of saturation ($0 \leq S_e \leq 1$), P is a material parameter related to the air entry value (MPa), λ is a parameter that controls the shape of the water retention curve, P_g-P_l is suction (MPa), S_{rl} is the residual degree of saturation and S_{ls} is the maximum degree of saturation. It was considered that S_{rl}=0 and S_{ls} =1, for which reason $S_e = S_r/100$, where S_r is the degree of saturation actually measured (appearing in Tables from Appendix 1). In clayey materials this relation can be modified by a correction function (Fredlund & Xing 1994) that aims at soothing the steep shape of the curve towards the high suction and forces the degree of saturation to be zero for suctions lower than 1000 MPa. The experimental results were also fit to this corrected form of the van Genuchten expression, but since all the results refer to suctions below 100 MPa no major differences with respect to Equation 2 were observed in the significant suction range (below 100 MPa), and consequently the fittings obtained for the corrected form of the equation are not presented here.

It has to be taken into account that the degrees of saturation were computed from the measurements of water content and dry density. The latter were particularly difficult to achieve and in some cases the values obtained were only indicative, because the dry density was not actually measured but just assumed or estimated. For this reason the dispersion of the values obtained for each kind of test was very high and the fittings obtained have a limited value.

Eight fittings were carried out: for the tests applying matric suction under free volume conditions (membrane cells), for the tests applying total suction under free volume conditions (desiccators), for the tests applying matric suction under vertical stress 8 MPa (membrane cells oedometers) and for the tests applying total suction under vertical stress 8 MPa (deposit cells oedometers), for each of them following both wetting and drying paths. The parameters found for each fitting are shown in Table IV.

Table IV: Parameters for the van Genuchten expression (Eq. 2)

	Matric, 8 MPa		Matric, free		Total, 8 MPa		Total, free	
Parameter	Wetting	Drying	Wetting	Drying	Wetting	Drying	Wetting	Drying
P (MPa)	25.66	15.27	6.35	8.59	10.75	34.93	11.78	21.14
λ	0.51	0.45	0.25	0.45	0.35	0.42	0.37	0.33

The P parameters obtained (related to the air entry values, AEV) tended to be higher for the samples tested under stress, as well as in drying paths and when total suction was used. The AEV obtained from the mercury intrusion porosimetry was 27.8 MPa (see section Material).

Figure 21 shows a comparison of the curves fitted for tests performed under free volume conditions and under vertical stress of 8 MPa. In the range of suctions tested, and when matric suction was applied, the degrees of saturation obtained for a given suction were higher for samples tested under confinement, but this trend was not so clear when total suction was applied. In the latter case and in wetting paths, the curves obtained under free or confined volume conditions were practically identical.

Drying paths recorded higher degrees of saturation than wetting paths when total suction was applied, which is the expected behaviour, but this was not the case when matric suction was applied. This is considered as due to the difficulty in fitting right curves to results showing a large dispersion.

Figure 22 shows the effect of the kind of suction on the WRC fittings. As it was discussed above, the effect is not clear since, although for the tests performed under free volume conditions the samples submitted to total suction tended to reach higher degrees of saturation, this was not the case for all the paths in the tests performed in oedometers.

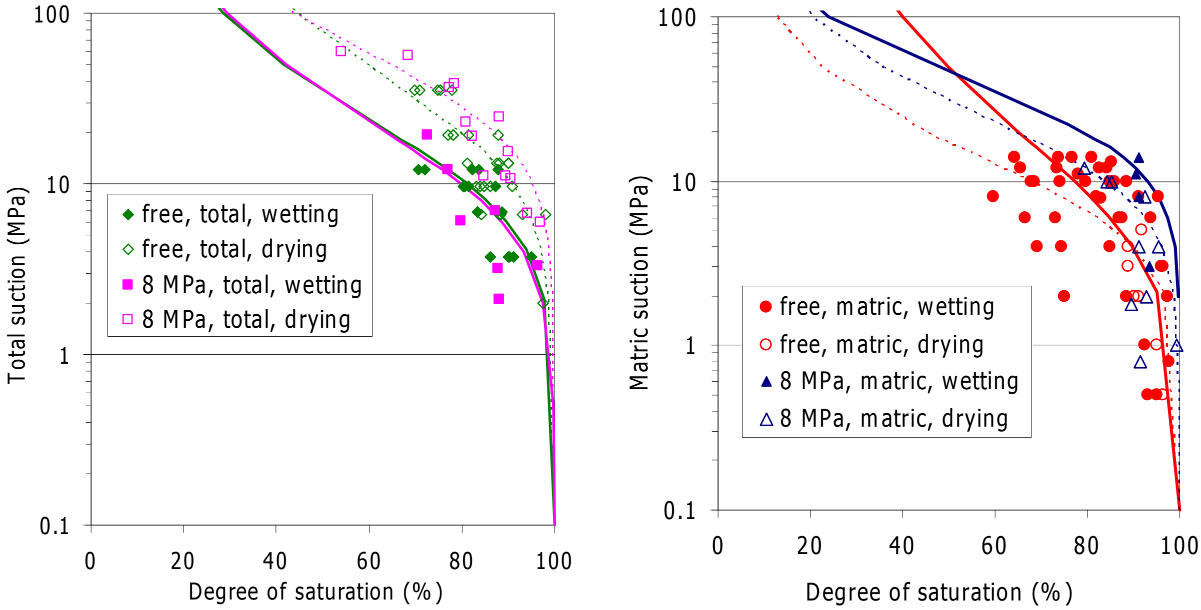

Figure 21: Fittings to the van Genuchten expression as a function of the confinement conditions for total and matric suction (the dotted lines correspond to drying paths)

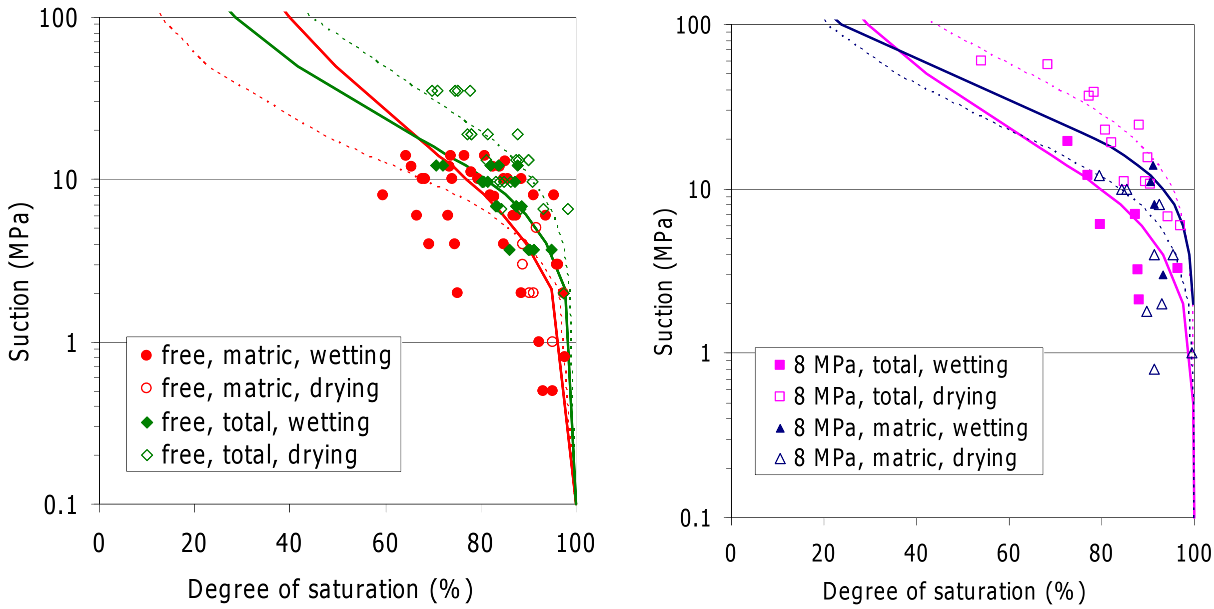

Figure 22: Fittings to the van Genuchten expression as a function of the kind of suction applied for different confinement conditions (the dotted lines correspond to drying paths)

5 Discussion

The determination of the Opalinus clay water retention capacity in terms of degree of saturation was difficult due both to the problems arisen during the preparation of the samples and to the inaccuracy of the intermediate measurements of dry density, as it has been pointed out above. In the case of the tests in oedometers the dry densities given, and consequently the degrees of saturation, could have been underestimated.

Nevertheless the approximate results obtained showed that in the tests performed in desiccators the dry density increased slightly in the first suction step and remained approximately constant during the subsequent steps. On the contrary, in the tests performed in membrane cells, the dry density decreased in the first step with respect to the initial value, irrespective of the value of suction applied.

Despite these experimental drawbacks, it was possible to fit the results to the van Genuchten expression and highlight the effect of confinement on the *P* parameter (related to the air entry value), which was clearly higher for confined samples in drying paths.

The results shown above have been compared with results obtained in samples taken from boreholes BVE-99 to BVE-102 (Villar et al. 2009) and BVE-1 (Muñoz et al. 2003) which were drilled in the context of the Ventilation Experiment.

Samples from boreholes BVE-99 to 102 were subjected to drying paths applying total suction in desiccators, under no confinement. The comparison with the results obtained using the same procedure in samples from borehole BHT-1 (this work) is shown in Figure 23. The suctions applied in BVE samples were higher, except for a narrow range of overlapping with the suctions applied to samples from borehole BHT-1, from 20 to 40 MPa. In this range of suction the water contents obtained in samples from borehole BHT-1 were higher, whereas when expressed in terms of degree of saturation (Figure 24) the results are quite similar, and the fitting to the van Genuchten expression for samples of BVE boreholes lies between those found for drying and wetting paths for BHT-1 samples.

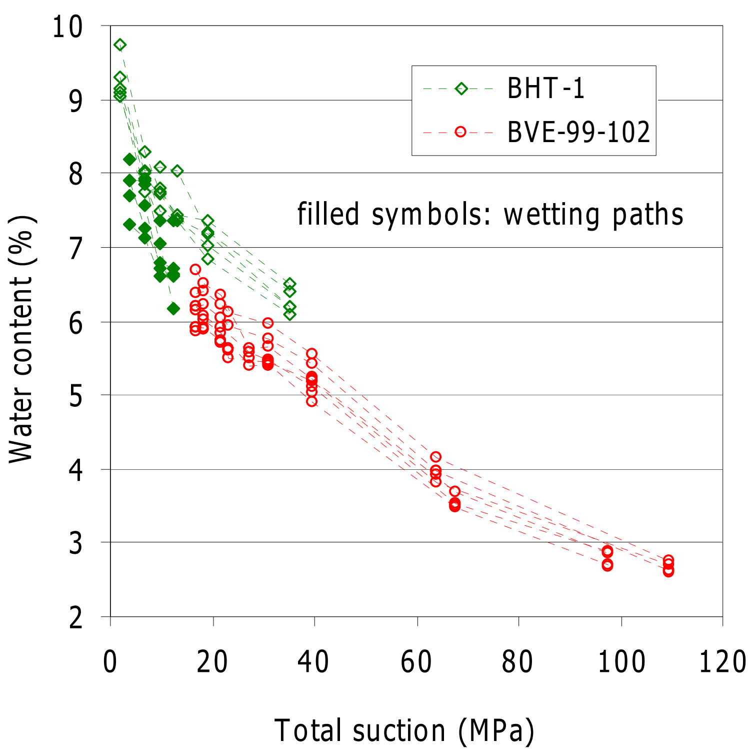

Figure 23: Retention curves obtained under free volume conditions in Opalinus clay samples from different boreholes (BVE-99-102: Villar et al. 2009)

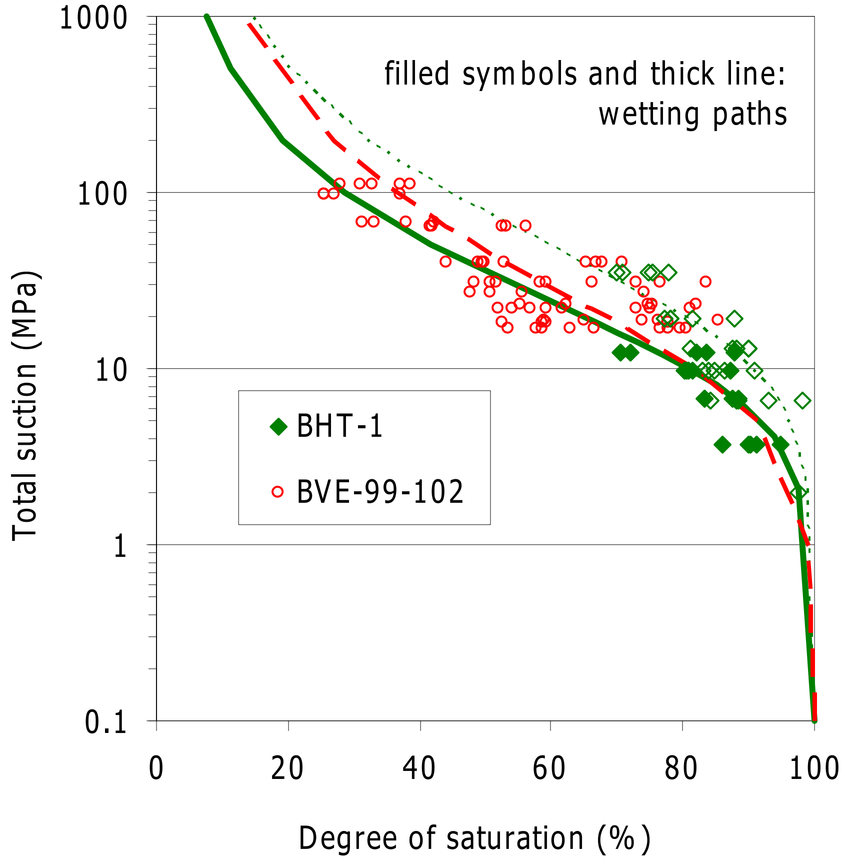

Figure 24: Fittings to the van Genuchten expression of WRC obtained under free volume conditions in Opalinus clay samples from different boreholes (BVE-99-102: Villar et al. 2009)

The samples from borehole BVE-1 were subjected to drying paths applying total suction (for suctions above 3 MPa) or matric suction (for suctions below 3 MPa) while keeping their volume constant in the oedometer (Muñoz et al. 2003). Before performing the drying path, the samples were saturated under constant volume conditions with Pearson water, what caused the average water content at the beginning of drying to be 9 percent. The results obtained have been plotted in Figure 25 along with the results obtained in oedometers in this work (previously shown in Figure 9). There is a good agreement between the two sets of results, both in terms of water content and of degree of saturation.

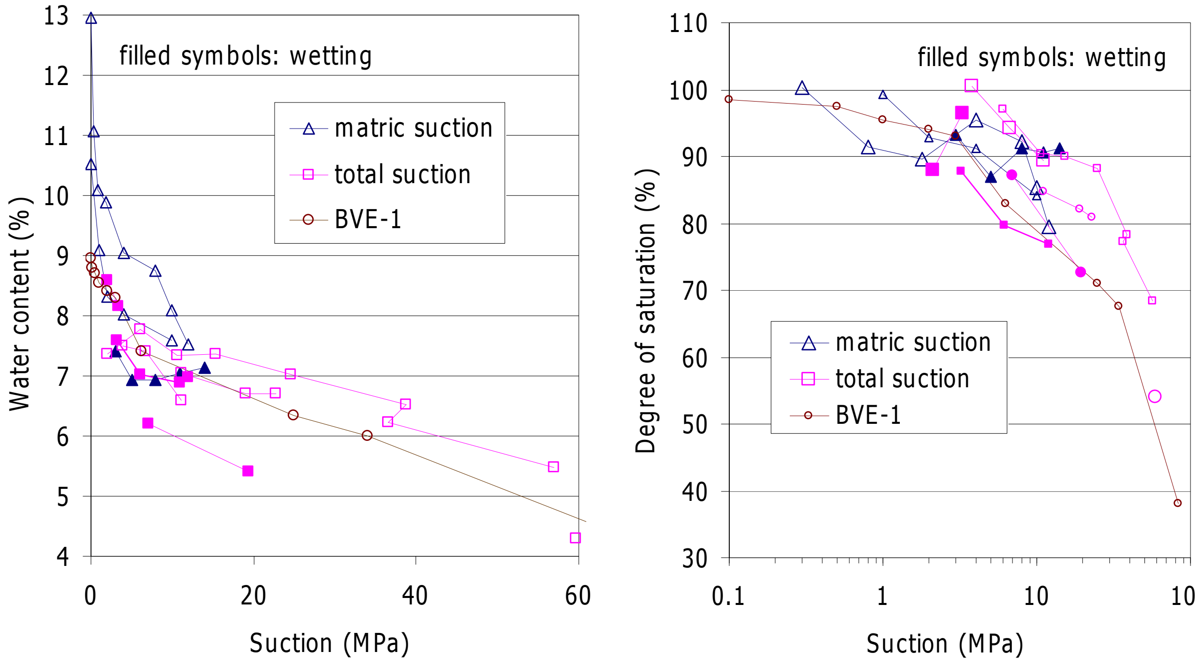

Figure 25: Water retention curves obtained under confinement (8 MPa vertical stress for samples in this work, constant volume conditions for samples BVE-1) in Opalinus clay samples from different boreholes (BHG-D1 and BHT-1: this work; BVE-1: Muñoz et al. 2003)

Conclusions

The water retention curve of Opalinus clay samples from boreholes BHT-1 and BHG-D1 was determined under different conditions: total and matric suction, stress or no-stress conditions, wetting and drying paths. Through the fitting of these results to expressions such as the van Genuchten one, it is possible to compute the P parameter, which is usually related to the air entry value, *i.e.* to the suction value above which air is able to enter the pores of the sample, and consequently, above which 2-phase flow can take place in the soil pore structure. This value can change according to the hydraulic history of the material, its porosity and confinement conditions and even the kind of suction considered, since in some soils the osmotic suction component can be important and thus the difference between total and matric suctions (those usually applied in the laboratory) large.

Because of the Opalinus clay bedding and foliation it was difficult to determine precisely the samples' dry density and degree of saturation evolution during the tests, what gave place to a large dispersion of results, for which reason the values given in this report concerning degrees of saturation must be taken as indicative.

The samples used in this research came from two different boreholes, BHT-1 and BHG-D1, but the behaviour of them did not depend on their location, what was confirmed by the fact that the results obtained agreed well with others obtained in samples from other boreholes analysed in previous researches, all of them drilled in the shaly facies. Consequently, to analyse the results all the samples were taken as a single material, Opalinus clay shaly facies.

There was not a distinct difference between the results obtained under total or matric suctions. In the drying paths, both the water contents and the degrees of saturation tended to be higher when total suction was applied, however the reverse trend was observed for the water contents reached in wetting paths. As well, no clear difference was observed in the water retention curves obtained in oedometers under matric and total suctions, what points to the osmotic component of suction in Opalinus clay not being significant.

Overall, the water contents were lower and the degrees of saturation higher when suction was applied under vertical stress, what would indicate that the water retention capacity was lower under 8 MPa vertical stress than under free volume conditions. This vertical stress value is slightly higher than the maximum *in situ* stress.

Also, the samples showed hysteresis according to the expected behaviour, *i.e*. the water contents for a given suction were higher during a drying path than during a wetting path.

The *P* parameters obtained were between 6 and 34 MPa, and tended to be higher for the samples tested under stress, in drying paths and when total suction was used. The air entry value calculated from the mercury intrusion porosimetry tests was 28 MPa. Gas injection tests currently running have shown that the breakthrough pressure is higher than 18 MPa.

Acknowledgements

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Appendix 1

RESULTS OF THE TESTS IN OEDOMETERS

Table V: Results of WRC in oedometer test EDN1_F1 (sample from BHG-D1 11.8-12.5 m)

Suction (MPa)	Duration (days)	Dry density (g/cm^3)	*w* (%)	S_r (%)
Initial		2.18	7.9	87
0	111	2.18±0.01	10.5	101
1.0	77		9.1	99
2.0	104		8.3	93
4.0	108		8.0	91
<10.0	25		7.6[a]	84

[a]equilibrium was probably not reached

Table VI: Results of WRC in oedometer test EDN3_F1 (sample from BHT-1 14.30-14.73 m)

Suction (MPa)	Duration (days)	Dry density (g/cm^3)	*w* (%)	S_r (%)
Initial		1.94	8.3	56
0	101	2.12	12.9[a]	105
0.3	82		11.1[a]	100
0.8	42		10.1[a]	91
1.8	57		9.9[a]	90
4.0	103		9.0	95
8.0	91		8.7	92
10.0	120		8.1	85
12.0	166		7.5	80

[a]during these steps the water pressure was 200 kPa and the density lower

Table VII: Results of WRC in oedometer test EDN5_F1 (sample from BHG-D1 11.8-12.5 m)

Suction (MPa)	Duration (days)	Dry density (g/cm^3)	*w* (%)	S_r (%)
Initial		2.14	7.5	76
14.0	50	2.24±0.01	7.1	91
11.0	60		7.1	91
8.0	71		6.9	91
5.0	72		6.9	87
3.0	96		7.4	93

Table VIII: Results of WRC in oedometer test EDS2_F1 (sample from BHG-D1 11.8-12.5 m)

Suction (MPa)	Duration (days)	Dry density (g/cm^3)	*w* (%)	S_r (%)
Initial		2.09	8.3	77
3.3	111	2.17±0.04	8.2	96
2.1	68		8.6	88

Table IX: Results of WRC in oedometer test EDS2_F2 (sample from BHG-D1 11.8-12.5 m)

Suction (MPa)	Duration (days)	Dry density (g/cm^3)	*w* (%)	S_r (%)
Initial		2.21	7.2	87
3.8	141	2.24±0.01	7.5	101
6.7	83		7.4	94
11.1	96		6.6	89

Table X: Results of WRC in oedometer test EDS2_F3 (sample from BHG-D1 11.8-12.5 m)

Suction (MPa)	Duration (days)	Dry density (g/cm^3)	*w* (%)	S_r (%)
Initial		2.21	5.6	68
19.4	90	2.26±0.01	5.4	73
7.0	102		6.2	87

Table XI: Results of WRC in oedometer test EDS3_F1 (sample from BHT-1 14.30-14.73 m)

Suction (MPa)	Duration (days)	Dry density (g/cm^3)	*w* (%)	S_r (%)
Initial		2.11	6.5	62
2.0	110	2.22±0.01	7.4	
6.0	84		7.8	97
10.7	92		7.3	90
15.4	63		7.4	90
24.6	111		7.0	88
38.7	92		6.5	78
36.6	95		6.2	77
56.9	91		5.5	68

Table XII: Results of WRC in oedometer test EDS3_F2 (sample from BHG-D1 11.8-12.5 m)

Suction (MPa)	Duration (days)	Dry density (g/cm^3)	*w* (%)	S_r (%)
Initial		2.14	7.3	75
59.6	87	2.22	4.3	54

Table XIII: Results of WRC in oedometer test EDS5_F1 (sample from BHG-D1 11.8-12.5 m)

Suction (MPa)	Duration (days)	Dry density (g/cm^3)	*w* (%)	S_r (%)
Initial		2.09	3.7	34
12.0	63	2.18±0.01	7.0	77
10.9	92		6.9	
6.1	96		7.0	80
3.2	90		7.6	88

Table XIV: Results of WRC in oedometer test EDS5_F2 (sample from BHG-D1 11.8-12.5 m)

Suction (MPa)	Duration (days)	Dry density (g/cm^3)	*w* (%)	S_r (%)
Initial		2.19	6.6	78
11.1	91	2.21±0.00	7.0	85
19.0	104		6.7	82
22.8	90		6.7	81

RESULTS OF THE TESTS IN DESICCATORS

Table XV: Results of WRC in desiccator following a wetting path (samples from BHT-1)

Reference	BHT1-1	BHT1-2	BHT1-3	BHT1-4	BHT1-9	BHT1-10
Depth (cm)	14.30-14.73			12.42-12.96		
Dry density (g/cm^3)	2.20	2.22	2.21	2.20	2.18	2.20
Initial *w* (%)	7.6	6.8	6.6	6.5		
Initial S_r (%)	91	85	80	74		
Suction (MPa)	12.2					
Dry density (g/cm^3)	2.20	2.22	2.21	2.20	2.18	2.20
w (%)	7.4	6.6	6.7	7.2	6.6	6.2
S_r %	88	84	82	83	72	71
Suction (MPa)	9.7					
Dry density (g/cm^3)	2.20	2.21	2.20	2.19	2.22	2.20
w (%)	7.4	6.6	6.8	8.4	6.7	7.1

Reference	BHT1-1	BHT1-2	BHT1-3	BHT1-4	BHT1-9	BHT1-10
S_r %	87	80	81	94	80	81
Suction (MPa)	6.8					
Dry density (g/cm^3)	2.18	2.19	2.19	2.17	2.20	2.18
w (%)	7.9	7.1	7.3	8.8	7.6	7.9
S_r %	89	83	83	94	87	87
Suction (MPa)	3.7					
Dry density (g/cm^3)	2.17	2.20	2.18	2.20	2.21	2.22
w (%)	8.2	7.3	7.9	7.9	7.7	7.9
S_r %	91	86	90	90	90	95

Table XVI: Results of WRC in desiccator following a drying path (samples from BHT-1)

Reference	BHT1-5	BHT1-6	BHT1-7	BHT1-8	BHT1-11	BHT1-12
Depth (cm)	14.30-14.73			12.42-12.96		
Dry density (g/cm^3)	2.20	2.14	2.17	2.18	2.18	2.14
Initial *w* (%)	8.1	7.8	7.2	6.4	6.3	6.1
Initial S_r (%)	96	81	80	69	70	61
Suction (MPa)	2.0					
[a]Dry density (g/cm^3)	2.20	2.15	2.17	2.20	2.20	2.16
w (%)	9.8	9.3	9.0	9.1	9.1	8.7
[a]S_r %	116	97	101	104	106	91
Suction (MPa)	6.6					
Dry density (g/cm^3)	2.20	2.15	2.17	2.20	2.20	2.16
w (%)	8.3	8.0	7.9	7.8	8.0	7.6
S_r %	98	84	88	88	93	80
Suction (MPa)	9.7					
Dry density (g/cm^3)	2.17	2.14	2.16	2.21	2.19	2.16
w (%)	7.8	8.1	7.7	7.8	7.5	8.7
S_r %	86	84	83	91	85	91
Suction (MPa)	13.1					
Dry density (g/cm^3)	2.21	2.16	2.17	2.22	2.22	2.18
w (%)	7.4	8.0	7.4	7.4	7.4	8.4
S_r %	90	87	81	90	88	92
Suction (MPa)	19.2					
Dry density (g/cm^3)	2.20	2.16	2.17	2.19	2.19	2.20
w (%)	7.4	7.2	7.0	7.2	6.8	7.2

Reference	BHT1-5	BHT1-6	BHT1-7	BHT1-8	BHT1-11	BHT1-12
S_r %	88	78	78	81	77	82
Suction (MPa)	35.3					
Dry density (g/cm^3)	2.20	2.16	2.18	2.22	2.23	2.21
w (%)	6.5	6.4	6.2	6.2	6.1	6.2
S_r %	78	70	71	75	75	73

[a]not actually measured

RESULTS OF THE TESTS IN MEMBRANE CELLS

Table XVII: Results of WRC in membrane cell following a drying path (sample from borehole BHT-1, reference BHT-1-1, initial ρ_d 2.21 g/cm^3, initial *w* 6.3%)

Suction (MPa)	Duration (days)	*w* (%)	ρ_d[a] (g/cm^3)	S_r (%)
0.2	89	11.2	2.10	104
0.5	35	10.5	2.10	97
1	119	10.9	2.10	100
2	65	9.8	2.10	90

[a]not actually measured

Table XVIII: Results of WRC in membrane cell following wetting paths (samples from borehole BHT-1)

Reference	Initial ρ_d (g/cm^3)	*w* (%)	Suction (MPa)	Duration (days)	*w* (%)	ρ_d (g/cm^3)	S_r (%)
BHT-1-2	2.22	7.2	14	54	6.0	2.16	64
			12	28	6.1	2.16	65
			10	42	6.3	2.16	68
			8	66	7.3	2.03	60
			6	34	8.2	2.03	67
			4	70	8.5	2.03	69
			2	70	9.2	2.03	75
BHT-1-3	2.20	7.0	14	49	7.3	2.18	81
			12	70	7.5	2.18	83
			10	44	7.6	2.16	80
			8	42	7.7	2.16	82
			6	42	7.9	2.18	87
			4	42	8.2	2.16	85
			2	53	8.8	2.14	89
			1	24	9.4	2.13	92

Table XIX: Results of WRC in membrane cell following drying paths (samples from borehole BHG-D1)

Reference	Initial ρ_d (g/cm^3)	*w* (%)	Suction (MPa)	Duration (days)	*w* (%)	ρ_d (g/cm^3)	S_r (%)
BHG-D1-6	2.16	6.9	4	85	7.7	2.11	75
			8	91	8.0	2.12	80
			12	91	8.2	2.12	81
BHG-D1-9	2.26	6.7	1	133	9.0	2.15	95
			2	77	8.3	2.16	91
			4	70	7.7	2.19	89
BHG-D1-11	2.23	7.0	3	120	8.5	2.15	89
			5	77	8.3	2.17	92
BHG-D1-18	2.25	5.2	0.5	77	10.4	2.16	114
			1.0	49	9.6	2.18	109
			1.5	70	9.2	2.19	107

Table XX: Results of WRC in membrane cell following wetting paths (samples from borehole BHG-D1)

Reference	Initial ρ_d (g/cm^3)	*w* (%)	Suction (MPa)	Duration (days)	*w* (%)	ρ_d (g/cm^3)	S_r (%)
BHG-D1-1	2.20	8.7	14	46	8.8	2.06	77
			12	62	8.9	2.03	74
			10	19	9.0	2.03	74
BHG-D1-8	2.15	7.2	14	34	7.5	2.12	74
			10	105	7.4	2.09	68
BHG-D1-10	2.23	6.5	6	77	7.9	2.09	73
			5	120	8.5	2.23	109
			3	56	8.9	2.16	96
BHG-D1-13	2.23	6.7	12	65	7.2	2.19	84
			10	84	7.6	2.19	89
BHG-D1-14	2.22	6.2	10	65	6.8	2.22	85
			8	63	7.3	2.22	91

Table XXI: Results of single-step WRC in membrane cell (samples from borehole BHG-D1)

Reference	Initial ρ_d (g/cm^3)	w (%)	Suction (MPa)	Duration (days)	w (%)	ρ_d (g/cm^3)	S_r (%)
BHG-D1-2	2.21	7.0	0.5	36	10.3	2.09	95
BHG-D1-3	2.22	4.4	0.8	50	9.9	2.12	98
BHG-D1-4	2.21	7.9	8	73	8.2	2.19	96
BHG-D1-5	2.20	6.5	2	73	7.9	2.15	83
BHG-D1-7	2.20	7.1	6	42	8.0	2.17	87
BHG-D1-15	2.19	7.3	10	87	7.7	2.17	86
BHG-D1-16	2.12	6.6	0.5	106	9.8	2.10	93
BHG-D1-17	2.24	6.1	2	87	7.6	2.23	97
BHG-D1-18	2.20	6.9	3	87	8.3	2.19	96
BHG-D1-19	2.23	6.3	6	78	7.1	2.24	94
BHG-D1-20	2.26	5.8	11	78	6.4	2.24	85
BHG-D1-21	2.23	6.8	13	78	7.0	2.21	85

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