Mont Terri Ventilation Test Phase II. Water Retention Curves Determined on Argillite Samples Taken before and after an in situ Ventilation Phase

M. V. Villar A. M. Fernández A. M. Melón

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Water Retention Capacity of Argullite from the VE Test - Phase II at Mont Terri: Effect of Ventilation

Villar, M^a. V.; Fernández, A. M^a.; Melón, A. M^a.

34 pp. 38 fig. 10 ref.

Abstract:

The VE (ventilation) test carried out at the Mont Terri underground laboratory in Switzerland intended to evaluate in situ the behaviour of a consolidated clay formation when subjected to alternate periods of flow of wet and dry air during several months. For that, a 10-m gallery was excavated in the Opalinus Clay formation and carefully instrumented. Before and after a second ventilation phase boreholes were drilled. Samples were taken from the drill cores and were analysed from mineralogical and geochemical points of view. Also, the retention curves of these samples were determined in the laboratory following drying paths performed under free volume conditions at 20°C, what is the content of this report. Although there are not large differences in the WRC of samples taken from different boreholes, at different distances from the gallery wall or before or after ventilation, those samples taken near the gallery wall and after ventilation tend to show a higher water retention capacity. This has been correlated to the higher salinity of the pore water of these samples, what increases their osmotic suction. This effect is attenuated towards high suctions.

Capacidad de Rentención de Agua de la Argilita del Proyecto VE - Fase II (Mont Terri): Influencia de la Ventilación

Villar, M^a. V.; Fernández, A. M^a.; Melón, A. M^a.

34 pp. 38 fig. 10 ref.

Resumen:

El ensayo VE (ventilación) llevado a cabo en el laboratorio subterráneo de Mont Terri en Suiza tiene por objeto evaluar in si tu el comportamiento de una formación arcillosa consolidada cuando se somete a flujo alterno de aire seco y húmedo durante periodos de varios meses. Para ello se excavó en la formación arcillosa Opalinus una galería de 10 m de longitud y se instrumentó intensivamente. Antes y después de una segunda fase de ventilación con aire seco se perforaron sondeos. De los testigos de estos sondeos se tomaron muestras para su caracterización mineralógica y geoquímica. También se han determinado en estas muestras sus curvas de retención de agua a volumen libre y siguiendo trayectorias de secado a 20°C, lo que constituye el contenido de este informe. Aunque no se han encontrado grandes diferencias entre la capacidad de retención de muestras provenientes de sondeos perforados antes o después de la fase de ventilación o tomadas a diferentes distancias de la galería, sí se ha podido constatar una tendencia a una mayor capacidad de retención de agua en las muestras tomadas cerca de la galería y después de la fase de ventilación. Esta tendencia desaparece para las succiones altas, y se ha correlacionado con la mayor salinidad de estas muestras, y por tanto con un aumento de la succión osmótica.

WATER RETENTION CAPACITY OF ARGILLITE FROM THE VE TEST – PHASE II AT MONT TERRI: EFFECT OF VENTILATION

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WATER RETENTION CAPACITY OF ARGILLITE FROM THE VE TEST – PHASE II AT MONT TERRI: EFFECT OF VENTILATION

1 INTRODUCTION

The VE phase II experiment was part of the integrated project NF-PRO (EC Contract FI6W-CT-2003-02389, Work Package 4.3), and it is the continuation of the VE phase I experiment, which was carried out between December 2001 and May 2004 as part of a project led by ENRESA and partly financed by the EC through contract FIKW-CT2001-00126. The aim of this project was to evaluate *in situ* the ventilation of a consolidated clay formation when subjected to flow of air during several months (ENRESA 2005).

The VE *in situ* test was carried out in a non-lined horizontal microtunnel of 1.3 m in diameter, excavated in the Opalinus Clay formation at the Mont Terri underground research laboratory (URL). A 10-m long section of the tunnel was sealed off by means of two double doors and monitored with 96 sensors to measure rock displacement, water potential, water content, temperature and conditions of air ventilation. The first ventilation period began in July 2003, and included a 2-month phase in which air with a relative humidity of 30 percent was forced into the testing area, followed by a 5-month phase in which the inflow air had a relative humidity of 1-3 percent. At the end of ventilation, the rock relative humidity was less than 95 percent (suction above approximately 7 MPa) only in a ~30-cm thick ring, although suction developed up to a distance from the gallery wall of about 2 m. Near the gallery wall the relative humidity was lower than 65 percent, corresponding to a suction of about 70 MPa. Afterwards, a 1-month resaturation phase followed, during which the relative humidity of the air forced was set to 100 percent. The description and main results of these phases are given in ENRESA (2005).

The VE phase II experiment started afterwards, with the ventilation equipment stopped during 16 months, and in June 2005 a sampling campaign took place, to asses the conditions of the clay before the second ventilation phase. During this campaign seven 77-mm diameter boreholes (68-mm diameter drill cores) with lengths between 1.40 and 2.00 m were drilled (boreholes BVE-96 to BVE-102). All of them were drilled vertically upwards, with sub-parallel orientation with respect to bedding, except borehole BVE-100, that was drilled perpendicularly to bedding. Boreholes BVE-101 and BVE-102 were drilled outside the ventilated area.

The second ventilation phase started in July 2005 and went on for 18 months. During this phase air with a relative humidity of 1-2 percent was forced in the microtunnel. After this phase, a second drilling campaign took place during which boreholes BVE-104 to BVE-111 (drill core diameter 68 mm and lengths between 0.47 and 1.78 m) were drilled. They were all drilled vertically upwards with a sub-parallel orientation with respect to bedding, except borehole BVE-107, that was drilled perpendicularly to bedding. Boreholes BVE-110 and BVE-111 were drilled outside the ventilated area.

A summary of the phases followed in the testing area is shown in Table I (ENRESA 2005). The average temperature of the rock was 15.5°C. The location of all the boreholes is shown in Figure 1. The fact that they were drilled following different orientations is relevant, since the permeability of the rock mass and consequently the water transport are highly anisotropic (Garitte & Gens 2007).

Description	Starting date	Duration	Air <i>RH</i> (%)					
Microtunnel excavation	February 1999	~41months	90					
Test section sealing	August 2002	~8 months	93					
Forced ventilation <i>RH</i> ~30%	July 2003	~2 months	47					
Forced ventilation <i>RH</i> ~1-3%	September 2003	~5 months	15					
Forced ventilation <i>RH</i> ~100%	January 2004	~1 month	92					
Without ventilation	March 2004	~16 months	95					
Sampling campaig	Sampling campaign: boreholes BVE-96 to BVE-102							
Forced ventilation <i>RH</i> ~1-3%	July 2005	~18 months	15					
Sampling campaig	n: boreholes BVE	-104 to BVE-	-111					

Table I: Summary of phases in the VE test



Figure 1: Location of boreholes drilled around the VE microtunnel

The core samples were placed in aluminium foil bags that were sealed after flushing with argon gas and application of vacuum. Packing took place 4-24 hours after drilling, for which reason some moisture changes could have taken place in the samples. The sampling at CIEMAT laboratories took place keeping at a minimum the exposure to air.

This report compiles the results obtained concerning the determination of the water retention curve in samples taken from these boreholes, *i.e.* in samples taken in June 2005, after the first resaturation phase and just before the second ventilation phase, and in October 2006, after the second ventilation phase. It complements Deliverables 4.3.5 and 4.3.6 in which the detailed physical, mineralogical and geochemical characterisation of these samples is given (Fernández *et al.* 2007a, Fernández & Melón 2007).

2 MATERIAL

The Opalinus clay is a dark-grey, fine-grained indurated clay with a quite homogeneous texture. The main mineralogical phases of this argillite are clays, quartz, calcite, other carbonates (ankerite, dolomite, siderite), feldspars, micas, pyrite, hematite, opal minerals and, in some samples altered by oxidation, gypsum. The clay minerals identified are kaolinite, chlorite, illite and an ordered illite/smectite mixed layer mineral (Pearson *et al.* 2003, Matray *et al.* 2006).

Both the observations in the laboratory and the modelling of the *in situ* measurements suggest the existence of an Excavation Damage Zone around the microtunnel due to excavation and possibly intensified during the ventilation cycles (Garitte & Gens 2007). A higher number of unloading fractures has been observed at distances less than 70-90 cm from the gallery wall (Fernández *et al.* 2007b).

Samples were taken along the boreholes drilled before and after the second ventilation phase in order to determine the physical, mineralogical and geochemical characteristics of the argillite, as well as the water retention capacity reported herein. The average water content of the samples taken inside the microtunnel before the second ventilation phase was 6.7 ± 0.4 percent, being higher in the boreholes drilled subparallel to bedding and decreasing with depth. After the second ventilation phase the average water content along the boreholes drilled inside the microtunnel was 6.6 ± 0.9 percent, showing a decrease towards the gallery wall, very sharp in the first 40 cm (Figure 2). This decrease in water content is coupled to a slight increase in dry density. The fact that the average water content around the microtunnel is approximately the same before and after ventilation, points to a continuous and important supply of water from the rock mass that evaporated through the surface of the gallery.

The average water content of the samples taken from boreholes drilled outside the microtunnel before the second ventilation phase was 6.7 ± 0.5 percent and after ventilation it was 7.6 ± 0.3 percent (Figure 3). It seems that the clay was not completely resaturated when the second ventilation phase started, and thus its water content continued to increase during the 18 months the second ventilation phase lasted. The trend for the water content to increase towards the gallery wall is probably linked to the decrease in dry density in this sense, in turn caused by decompression.



Figure 2: Water content along boreholes drilled parallel to bedding before (BVE-97 and BVE-99) and after (BVE-104 to BVE-109) the second ventilation phase inside the microtunnel. The lines mark the trend for the boreholes drilled before the second ventilation



Figure 3: Water content along boreholes drilled before (BVE-101 and BVE-102) and after (BVE-110 and BVE-111) the second ventilation phase outside the microtunnel

The main mineralogical phases of this argillite identified in samples taken from boreholes drilled both in June 2005 and in October 2006 are the same found by other authors in the Opalinus clay (Fernández *et al.* 2007b). The clay presents some carbonate layers or lens

parallel or subparallel to bedding. Calcite fillings appear quite homogenously distributed inside the clay matrix in BVE-101, BVE-102, BVE107 and BVE-111 drill cores. In the samples taken before and after the ventilation phase at distances lower than 7 cm from the microtunnel wall, gypsum has been observed by SEM-EDX analysis. Its presence has been linked to the oxidation of pyrites (Fernández & Melón 2007).

The average total exchange capacity of the samples from the boreholes drilled before the second ventilation phase was 15 meq/100g and 16 meq/100g afterwards, the main exchangeable cations being sodium, calcium, magnesium and potassium in both cases. In the 40 to 80 cm closest to the gallery wall there was a decrease in exchangeable calcium and an increase in exchangeable magnesium both before and after ventilation and inside and outside the ventilated area (Figure 4, Figure 5, Fernández *et al.* 2007a, Fernández & Melón 2007).

After the first resaturation period (*i.e.* before the second ventilation phase), despite the fact that the water content was quite homogeneous along the boreholes, there was a salinity variation along them, with ion concentrations (chloride, sulphate, sodium) higher in the 40-60 cm closest to the gallery wall (Figure 6). The magnitude of this increase was higher in the boreholes drilled inside the testing area (Fernández *et al.* 2007a), especially in the one drilled perpendicularly to bedding. The same trends were observed after ventilation (Figure 7).



Figure 4: Exchangeable Mg and Ca in samples taken along boreholes drilled before the second ventilation phase (BVE-99: inside gallery, subparallel to bedding; BVE-100: inside gallery, perpendicular to bedding; BVE-101: outside gallery) (Fernández *et al.* 2007a)



Figure 5: Exchangeable Mg and Ca in samples taken along boreholes drilled after the second ventilation phase (BVE-109: inside gallery, subparallel to bedding; BVE-107: inside gallery, perpendicular to bedding; BVE-110: outside gallery) (Fernández & Melón 2007)



Figure 6: Chloride content in 1:4 solid:liquid aqueous extracts of samples taken along boreholes drilled before the second ventilation phase (BVE-99: inside gallery, subparallel to bedding; BVE-100: inside gallery, perpendicular to bedding; BVE-101and BVE-102: outside gallery) (Fernández *et al.* 2007)



Figure 7: Chloride content in 1:4 solid:liquid aqueous extracts of samples taken along boreholes drilled after the second ventilation phase (BVE-109: inside gallery, subparallel to bedding; BVE-107: inside gallery, perpendicular to bedding; BVE-110: outside gallery) (Fernández & Melón 2007)

3 METHODOLOGY

The retention curve has been determined in samples taken from a position from borehole BVE-107, two different positions along boreholes BVE-99, BVE-101, BVE-102, BVE-108 and BVE-111 and from three different positions along borehole BVE-100 (Table II). Two parallelepipeds were trimmed from each sample, taking care not to disturb the initial water content of the argillite. The initial lengths of the faces of the specimens were between 13 and 48 mm. The average initial water content of these samples was 5.9 ± 0.5 percent in the case of samples obtained before ventilation and 6.5±0.5 in the case of samples obtained after ventilation. The aim of trimming regular-shaped samples was to allow the following up of the density variations by just measuring the length of the faces. However, this trimming caused an overall decrease in the density of the samples with respect to those determined in irregular fragments shown in Fernández et al. (2007a) and Fernández & Melón (2007). Besides, since the shape of the specimens trimmed was not perfectly regular (Figure 8), the dry density deduced from the measurement of the maximum dimensions is underestimated. To overcome this drawback, the successive calculated densities were corrected by taking into account the difference between the initial density determined by mercury immersion in adjacent samples and the initial density computed from the dimensions. In the case of the samples taken after ventilation, the correction was performed at the end of the tests, when the density of the samples actually used for determination of the water retention curve was determined by mercury immersion and then compared with that computed from the dimensions at the end of the tests.

Table II: Provenance of the samples used for determination of the water retention curve (campaign: before or after 2nd ventilation phase, location: inside or outside the ventilated area, orientation: with respect to bedding, distances: from the gallery wall along the borehole)

Borehole	Campaign	Location	Orientation	Distances (cm)
DVE 00	Dafara	Incido	Subporallal	35-40
BVE-99	Belore	Inside	Subparallel	100-105
				25
BVE-100	Before	Inside	Perpendicular	60-63
				162
DVE 101	Dafara	Outsida	Subparallal	40-45
BVE-101	Delote	Outside	Subparallel	155-160
DVE 102	Dafara	Outsida	Subparallal	60-63
DVE-102	Delote	Outside	Subparallel	133-135
BVE-107	After	Inside	Perpendicular	27-30
DVE 109	After	Incida	Subporallal	30-45
DVE-108	Alter	Inside	Subparallel	100-110
DVE 111	After	Outside	Subporallal	20-40
DVE-111	Aller	Ouiside	Subparallel	135-170



Figure 8: Appearance of the samples trimmed to determine the water retention curve

The specimens thus prepared were placed in vacuum desiccators with sulphuric acid solutions, so that to apply total suction to the samples by means of the control of the relative humidity (Figure 9). They were initially submitted to a suction of 17 MPa in the case of the samples taken before ventilation and 16 MPa in the case of samples taken after ventilation (intended to be 15 MPa in both cases, which, according to Muñoz *et al.* (2003), was expected not to imply a major change in the initial water content of the samples). Afterwards they were progressively dried by applying increasingly higher suctions. The specimens were not confined during the determination and their dimensions could slightly change. The samples were subjected to each suction for a period of time enough to guarantee equilibrium. After each suction step the samples were weighed and measured to determine their water content and dry density. Likewise, the final density of the sulphuric acid solution in the desiccator was checked, using a pycnometer. This gives the exact value of suction to which the samples were subjected, since the density of the solution is related to its water activity (or relative

humidity generated) and this in turn is related to total suction by the psychrometric law. The determinations were performed at 20°C.

Once the retention curve determination completed, the samples were measured and dried in an oven at 110°C for 24 hours to check their final water content. The density of the samples taken after ventilation was checked by immersion in mercury after drying in the oven and their dimensions were measured again. This allowed to check the difference between the actual density (determined by mercury immersion) and the density deduced by dimensions, and thus correct the interim values obtained. The differences found were between 3 and 9 percent (depending on the regularity of the dimensions of each particular specimen), and thus the densities obtained in the course of the water retention curve determination by measuring of the dimensions was increased by these factors.



Figure 9: Samples trimmed from the boreholes, placed in the desiccators

4 RESULTS

4.1 Before the second ventilation phase

Specimens trimmed from boreholes BVE-99, BVE-100, BVE-101 and BVE-102 were placed in desiccators at 20°C in which they were submitted to suctions increasingly higher from 17 to 110 MPa, *i.e.* the samples were subjected to a drying path. The samples remained under each suction condition for periods of time between 30 and 155 days. The time needed for reaching equilibrium under a given suction increased as the drying process proceed (Figure 10). After reaching equilibrium in each suction step the specimens were weighed and their dimensions measured in order to follow the water content and dry density evolution.



Figure 10: Time allowed for the samples to stabilise for each suction value (not accumulative)

The average initial water content of the specimens used to determine the retention curve – checked at the end of the tests– was 5.9 ± 0.5 percent, with values slightly higher at deep locations, except for borehole BVE-99, along which the water content is almost constant up to at least a depth of 100 cm. Overall the values are lower than the values measured in intact fragments of the boreholes and shown in Appendix IV of Fernández *et al.* (2007a), whose average was 6.6 ± 0.4 percent, with a trend to decrease with depth (Figure 11). The initial decrease in water content of the samples used to determine the retention curves could have been caused by the exposition of these samples to room conditions during their trimming. This decrease is particularly significant for the samples of borehole BVE-99.

The average initial dry density of the samples (after correction by taking into account the difference between the actual density measured by mercury displacement and the density computed by dimensions) was 2.28 ± 0.03 g/cm³ (Figure 12). The initial dry density of these samples has probably decreased with respect to the original value due to decompression during drilling and to the trimming process. In the samples taken along the boreholes there was a clear trend to find higher dry densities as depth increased (Fernández *et al.* 2007a) which is not so clear in the samples picked up for the determination of the retention curve.

The initial suction of 16.8 MPa was chosen based on the results of Muñoz *et al.* (2003), who determined an average initial suction value of 14.5 MPa in samples taken from borehole BVE-1. The initial average water content of their samples was 6.8 percent, higher than that of the samples used in this work. This might be the reason why when the samples were submitted to the initial suction, their water content increased in all the cases. The water contents of most of the samples continued to be higher than the initial average ($5.9\pm0.5\%$) even for suctions as high as 18.4 MPa.

The values obtained in the drying paths followed are plotted in Figure 13 in terms of water content and in Figure 14 in terms of dry density. There is not a clear difference among the values obtained for the different boreholes. Despite the dispersion of the dry density values, it seems that the dry density increases slightly with suction. There is an initial difference in the average dry density of the samples of each borehole (BVE-101 < BVE-99 < BVE-102 < BVE-100, Figure 12) that keeps along the drying paths.



Figure 11: Initial water content of the specimens trimmed for the determination of the retention curve. The lines indicate the trend of the water content from fragments of samples taken along the boreholes as shown in Fernández *et al.* (2007a)

Figure 12: Initial dry density (approximate) of the specimens trimmed for the determination of the retention curve. The lines indicate the trend of the dry density from fragments of samples taken along the boreholes as shown in Fernández *et al.* (2007a)

Figure 13: Evolution of water content in samples trimmed from boreholes drilled before ventilation following a drying path at 20°C

Figure 14: Evolution of dry density in samples trimmed from the boreholes drilled before ventilation following a drying path at 20°C

The average results of the duplicate samples obtained for each borehole are shown in Table III to Table VI. As it has been explained above, the dry densities and degrees of saturation given in the Tables must be taken as approximate values.

Depth (cm)	epth (cm) 35-40 100-105					
Suction (MPa)	w (%)	$\rho_d (g/cm^3)$	$S_{\rm r}$ (%)	w (%)	$\rho_d (g/cm^3)$	$S_{\rm r}$ (%)
Initial	5.3	2.29	73	5.3	2.24	63
16.8	5.9	2.20	67	5.9	2.19	63
18.5	5.9	2.19	65	5.9	2.16	59
21.8	5.7	2.19	62	5.7	2.18	59
23.3	5.6	2.14	55	5.6	2.21	62
30.9	5.5	2.19	58	5.5	2.20	59
39.7	5.2	2.15	53	5.2	2.13	50
63.8	3.9	2.18	42	4.0	2.19	42
109.6	2.6	2.22	31	2.6	2.19	28

Table III: Results of the retention curves following drying paths of samples from borehole BVE-99 (each value is the average of two specimens, the ρ_d and S_r values are approximate)

Table IV: Results of the retention curves following drying paths of samples from borehole BVE-100 (each value is the average of two specimens, the ρ_d and S_r values are approximate)

Depth (cm)	25			60-63			162		
Suction (MPa)	w (%)	ρ_d (g/cm ³)	Sr (%)	w (%)	ρ_d (g/cm ³)	Sr (%)	w (%)	ρ_d (g/cm ³)	Sr (%)
Initial	6.0	2.29	82	6.1	2.27	78	5.5	2.31	81
16.8	6.2	2.25	77	6.4	2.25	78	5.9	2.28	81
18.5	6.2	2.23	74	6.4	2.28	85	5.9 ^a	2.26	76
21.8	6.0	2.25	75	6.2	2.28	81	5.7	2.27	75
23.3	5.9	2.26	75	6.1	2.28	82	5.5	2.28	75
30.9	5.8	2.28	77	5.9	2.31	84	5.5	2.27	73
39.7	5.4	2.26	68	5.5	2.25	67	5.2	2.25	65
63.8	4.0	2.28	53	4.1	2.29	56	3.8 ^b	2.29	53
109.6	2.6	2.25	33	2.7	2.29	37	2.7 ^c	2.30	38

^a suction was 17 MPa

^b suction was 66 MPa

^c suction was 104 MPa

Depth (cm)		40-45			155-160		
Suction (MPa)	w (%)	$\rho_d (g/cm^3)$	$S_{\rm r}$ (%)	w (%)	$\rho_d (g/cm^3)$	$S_{\rm r}$ (%)	
Initial	6.9	2.22	78	6.2	2.27	80	
16.8	6.7	2.10	59	6.1	2.13	58	
18.3	6.5	2.12	59	6.0	2.15	59	
21.8	6.3	2.11	57	5.8	2.11	52	
27.3	5.6	2.11	51	5.4	2.10	48	
30.9	5.7	2.09	48	5.4	2.11	51	
39.7	5.2	2.13	49	4.9	2.11	44	
67.7	3.7	2.11	33	3.5	2.11	31	
97.6	2.9	2.13	27	2.7	2.14	26	

Table V: Results of the retention curves following drying paths of samples from borehole BVE-101 (each value is the average of two specimens, the ρ_d and S_r values are approximate)

Table VI: Results of the retention curves following drying paths of samples from borehole BVE-102 (each value is the average of two specimens, the ρ_d and S_r values are approximate)

Depth (cm)	60-63			132-135		
Suction (MPa)	w (%)	$\rho_d (g/cm^3)$	<i>S</i> _r (%)	w (%)	$\rho_d (g/cm^3)$	<i>S</i> _r (%)
Initial	6.4	2.31	92	5.6	2.31	81
16.8	6.2	2.26	80	5.9	2.11	53
18.3	6.1	2.26	78	5.9	2.10	53
21.8	5.9	2.24	73	5.7	2.13	54
27.3	5.6	2.27	74	5.5	2.16	56
30.9	5.5	2.24	66	5.4	2.13	52
39.7	5.1	2.29	71	5.0	2.14	50
67.7	3.5	2.24	42	3.5	2.18	38
97.6	2.9	2.27	37	2.7	2.13	26

The results are plotted as a function of the position of the samples along the boreholes in Figure 15 (borehole BVE-99) to Figure 18 (borehole BVE-102). Except for borehole BVE-99, in which the behaviour of the samples seems independent of their position, the samples taken from deeper positions along the boreholes reached lower water contents for the same suctions than those samples taken closer to the gallery. These differences tended to decrease as drying proceeded, *i.e.* for higher suctions. The evolution of dry density during the tests is shown in Figure 19, where it can be observed that it barely changed along the paths. The dry density of some samples increased slightly at the beginning of drying, and despite the dispersion, in general the final dry densities are higher than the initial ones.

Figure 15: Water contents of samples from borehole BVE-99 obtained in drying paths at 20°C. The distance from the gallery wall is indicated in the legend

Figure 16: Water contents of samples from borehole BVE-100 obtained in drying paths at 20°C. The distance from the gallery wall is indicated in the legend

Figure 17: Water contents of samples from borehole BVE-101 obtained in drying paths at 20°C. The distance from the gallery wall is indicated in the legend

Figure 18: Water contents of samples from borehole BVE-102 obtained in drying paths at 20°C. The distance from the gallery wall is indicated in the legend

Figure 19: Dry density of samples from boreholes obtained in drying paths (approximate values). The distance from the gallery wall is indicated in the legends

4.2 After the second ventilation phase

Specimens trimmed from boreholes BVE-107, BVE-108 and BVE-111 were placed in desiccators at 20°C in which they were submitted to suctions increasingly higher from 16 to 108 MPa, *i.e.* the samples were subjected to a drying path. The samples remained under each suction condition for periods of time between 98 and 212 days. The time needed for reaching equilibrium under a given suction increased as the drying process proceed. After reaching equilibrium in each suction step the specimens were weighed and their dimensions measured in order to follow the water content and dry density evolution.

The average initial water content of the specimens used to determine the retention curve – checked at the end of the tests– was 6.5 ± 0.5 percent, with values slightly higher at deep locations in the case of borehole BVE-108. Along borehole BVE-111, drilled outside the ventilation area, the water content has a constant value of 7 percent. Overall the values are slightly lower than the values measured in intact fragments of the drill cores and shown in Appendix I of Fernández & Melón (2007) (Figure 20). The initial decrease in water content of the samples used to determine the retention curves could have been caused by the exposition of these samples to room conditions during their trimming.

The average initial dry density of the samples computed from their dimensions was 2.03 ± 0.17 g/cm³, and after correcting this underestimated value as explained in section "Methodology", the value is 2.28 ± 0.05 g/cm³, in the order of the value measured in intact fragments taken

from contiguous positions along the same boreholes, which was 2.26 ± 0.02 g/cm³ (Fernández & Melón 2007).

Figure 20: Initial water content of the specimens trimmed for the determination of the retention curve (big symbols). The small symbols indicate the water content of fragments of samples taken along the same boreholes shown in Fernández & Melón (2007)

The initial suction of 16 MPa was chosen based on the results of Muñoz *et al.* (2003), who determined an average initial suction value of 14.5 MPa in samples taken from borehole BVE-1 of initial average water content of 6.8 percent. Since the second ventilation phase had taken already place when boreholes BVE-107 to BVE-111 were drilled, the initial water content of these drill cores was lower than the water content of the samples from borehole BVE-1, and consequently their suction was higher. Hence, during this first step the water content of the samples from borehole BVE-107 increased, what indicates that their initial suction was higher than 14.5 MPa. On the contrary, the water content of samples from borehole BVE-111 decreased, what points to an initial suction lower than this value. The differences in dry density between the samples from borehole BVE-101 –taken at a much longer distance from the gallery wall and neither subjected to decompression nor to ventilation or resaturation– could also contribute to explain the initial suction differences.

The values obtained in the drying paths followed are plotted in Figure 21 in terms of water content and in Figure 22 in terms of dry density. There is not a clear difference among the values obtained for the different boreholes. Despite the dispersion of the dry density values, it can be observed that the dry density increases slightly as suction goes above 30 MPa, *i.e.* once the water content starts to clearly decrease below the initial value. The average density of the samples taken from borehole BVE-111 is higher that for the other boreholes, maybe because it was drilled outside the ventilation area and the argiillite was less disturbed.

The average results of the duplicate samples obtained for each borehole are shown in Table VII and Table VIII.

Figure 21: Evolution of water content in samples trimmed from the boreholes drilled after ventilation following a drying path at 20°C

Figure 22: Evolution of dry density in samples trimmed from the boreholes drilled after ventilation following a drying path

Borehole **BVE-107 BVE-108** 28-30 30-45 100-110 Depth (cm) Sr Sr $S_{\rm r}$ Suction ρ_d ρ_d ρ_d w (%) w (%) w(%) (g/cm^3) (g/cm^3) (g/cm^3) (MPa) (%) (%) (%) Initial 5.9 2.27 86 5.9 2.27 86 6.7 2.28 94 93 93 94 16.0 6.5 2.26 6.4 2.25 6.7 2.26 90 23.6 6.3 2.26 6.2 2.25 90 6.4 2.25 90 25.9 6.2 2.26 89 6.1 2.27 89 6.3 2.26 89 61.9 4.2 61 4.1 2.25 59 4.0 2.29 57 2.28 108.2 2.9 2.29 42 2.8 2.30 40 2.7 2.31 38

Table VII: Results of the retention curves following drying paths of samples from boreholes BVE-107 and BVE-108 (each value is the average of two specimens, the ρ_d and S_r values are approximate)

Table VIII: Results of the retention curves following drying paths of samples from boreh	ole
BVE-111 (each value is the average of two specimens, the ρ_d and S _r values are approxim	ate)

Depth (cm)	Depth (cm) 20-40			135-170		
Suction (MPa)	w (%)	$\rho_d (g/cm^3)$	$S_{\rm r}$ (%)	w (%)	$\rho_d (g/cm^3)$	$S_{\rm r}$ (%)
Initial	7.0	2.28	94	7.0	2.31	95
16.0	6.6	2.27	87	6.6	2.29	90
23.6	6.3	2.28	84	6.3	2.31	86
25.9	6.2	2.28	82	6.2	2.32	84
61.9	3.9	2.29	52	3.8	2.34	52
108.2	2.7	2.32	35	2.6	2.35	35

The results are plotted as a function of the position of the samples along the boreholes in Figure 23 (borehole BVE-108) and Figure 24 (borehole BVE-111). For borehole BVE-108, the samples taken from deeper positions reached higher water contents for the same suctions than those samples taken closer to the gallery. This trend inverted for higher suctions. However, the behaviour of samples along borehole BVE-111 was different: for the low suctions no differences could be observed as a function of position, but for higher suctions the samples taken closer to the gallery wall reached higher water contents.

Figure 23: Water contents of samples from borehole BVE-108 obtained in drying paths at 20°C. The distance from the gallery wall is indicated in the legend

Figure 24: Water contents of samples from borehole BVE-111 obtained in drying paths at 20°C. The distance from the gallery wall is indicated in the legend

5 DISCUSSION

5.1 Analysis of results

The retention curves of samples taken from boreholes drilled before and after the second ventilation phase have been determined in drying paths performed under free volume conditions at 20°C. All of these samples had previously been subjected to wetting/drying cycles in situ (due to ventilation) in addition to the drying paths they experienced in the laboratory. Figure 25 is an attempt to reproduce the hydraulic history of the samples both in situ and in the laboratory in an idealised way. This history must be taken into account to evaluate the results obtained, since the water retention capacity is usually affected by hysteresis effects. Overall, near the gallery wall the samples were first subjected to drying due to the excavation and subsequent first ventilation phase. The forced and natural resaturation phase followed. The two curves describing these processes in Figure 25 would be the main drying and wetting curves. All the subsequent hydraulic evolution of the argillite should be represented by scanning curves inside the main ones. After resaturation the first set of samples was taken and they were subjected in the laboratory to the drying path described in section 4.1. Meanwhile, the argillite in situ experienced a new ventilation phase, which, according to the in situ RH measurements, gave place to a drying more intense than that reproduced in the laboratory. Afterwards, a new set of samples was taken and they were subjected in the laboratory to a new drying path, described in section 4.2. The initial suction of this drying path was lower than the suction at the end of the *in situ* ventilation, at least near the gallery wall, what implied in some cases an increase in argillite water content in the laboratory with respect to that at the end of ventilation.

Figure 25: Idealised hydraulic history of the argillite during the VE test and in the laboratory. Thin continuous lines represent the *in situ* evolution. Discontinuous lines represent the probable evolution of drill cores from its recovery to the beginning of testing at the laboratory. The thick continuous lines represent the controlled drying paths followed in the laboratory

Despite the fact that there were boreholes drilled outside and inside the testing section, no significant differences in the retention capacity of the samples from the different boreholes have been remarked. However, regarding the location of the samples along the boreholes, it has been observed that the samples located near the gallery wall reached higher water contents for the same suctions than those taken at a higher depth along the boreholes (except in borehole BVE-99 and at the beginning of drying in borehole BVE-108). This increase is more significant in the case of boreholes drilled inside the ventilated area subparallel to bedding. This could be a consequence of the higher osmotic suction of the samples near the gallery wall, since the salinity of the argillite pore water near the gallery increased considerably with respect to the background one, which is found at 40-60 cm from the gallery, depending on the borehole location and orientation (Figure 6, Figure 7). In other words, for a given water content, the total suction of samples with a higher salinity is higher, due to the contribution of osmotic suction. Nevertheless, for high suctions the differences among samples reduce, *i.e.* the retention curves are similar in the high-suction range (Figure 26, Figure 27), what would mean that the effect of osmotic suction attenuates as total suction is higher, *i.e.* as it affects smaller pores. There is another factor that could influence the retention capacity of these samples, and this is the change observed in the exchange complex (Fernández et al. 2007b), in particular the increase in exchangeable magnesium –a cation with high hydration energy- towards the gallery (Figure 4, Figure 5). As a matter of fact, the statistical analysis of the data shows that the water content of the argillite for the lowest suction (15-16 MPa) is positively correlated mainly to the content of chloride in the pore water and to the exchangeable magnesium.

Another possible reason for the differences among samples located at different positions along the boreholes is their different initial dry density, which is overall slightly lower near the gallery wall (see section MATERIAL), due probably to decompression. Actually, the average dry density of the samples taken near the gallery wall and tested before ventilation was 2.27 g/cm³, whereas those taken at deeper positions had an average density of 2.28 g/cm³. Overall there is a negative correlation between the initial dry density of the argillite and the water content reached at progressively higher suctions.

The retention curves obtained for the samples taken from all the boreholes analysed are plotted in Figure 28. At the beginning of drying, the water contents reached by the samples taken after the second ventilation are higher, especially for those samples taken at less than 40 cm from the gallery wall (Figure 29). This difference attenuates as drying proceeds. According to the hydraulic history of the samples (Figure 25), the water content reached by the samples taken after the second ventilation phase should be lower than that of the samples taken before the ventilation phase, whereas the contrary has been observed. The most likely explanation for this increase in the retention capacity is the increase in osmotic suction near the gallery wall after ventilation due to the salt concentration. In fact, in the samples taken outside the ventilated area (boreholes BVE-101, BVE-102 and BVE-111), where the salinity does not increase as significantly (Figure 30), the retention capacity does not increase after ventilation.

Figure 27: Retention curves at 20°C of samples from boreholes drilled after the second ventilation phase as a function of their distance from the gallery wall (each point is the average of two specimens)

Figure 28: Retention curves obtained in drying paths in samples taken before (BVE-99 to BVE-102) and after the second ventilation phase (each point is the average of two specimens)

Figure 29: Retention curves obtained in drying paths in samples taken before (BVE-99 to BVE-102) and after the second ventilation phase at distances lower than 40 cm from the gallery wall (each point is the average of two specimens)

Figure 30: Chloride content in 1:4 solid:liquid aqueous extracts of samples taken along boreholes drilled before (BVE-99: inside gallery; BVE-102: outside gallery) and after the second ventilation phase (BVE-109: inside gallery; BVE-110: outside gallery)

5.2 Comparison with results obtained in powdered samples

Water sorption isotherms were also determined in samples taken along the boreholes that were grounded to powder and dried to 150°C before being submitted to different relative humidities in desiccators (Fernández *et al.* 2007a, Fernández & Melón 2007). The *RH* was achieved by using saturated saline solutions and the tests were performed at 20°C. The results are not directly comparable with those presented above, although some analogous conclusions can be drawn from them. The water contents reached by the powder samples for a given suction are clearly lower than those reached by the trimmed specimens in the range of suctions analysed (Figure 31). This could be the expected result if we take into account the hysteresis effect caused by the fact that the powdered samples had experienced an intense drying at 150°C before being hydrated at different relative humidities, whereas the trimmed samples experienced a much less intense drying path without any previous laboratory treatment.

As in the case of trimmed samples described above, the powdered samples taken near the gallery wall after ventilation have a higher retention capacity than those taken deeper, whereas this behaviour is not so clear in the samples taken before ventilation (Figure 32, Figure 33). However, the higher retention capacity of the samples taken after ventilation is quite clear, especially for the lower suctions. Both facts can be explained by the relation between salinity and water retention capacity already discussed. In fact, the statistical analysis performed shows a correlation coefficient of 0.91 between the chloride content of the powdered samples and their water content for a suction of 4 MPa, and of 0.81 between chloride content and water content for a suction of 22 MPa. However, there is no correlation between the water contents reached by the powdered samples and the exchangeable magnesium content.

Figure 31: Water retention curves for samples taken from borehole BVE-100 at 25 cm from the gallery wall. The powder samples followed a wetting path after drying at 150°C, the trimmed samples followed a drying path from the original state after drilling

Figure 32: Water isotherms for powdered samples taken before (BVE-99) and after (BVE-109) ventilation at different distances from the gallery wall

Figure 33: Water isotherms for powdered samples taken before (BVE-100 and BVE-102) and after (BVE-107 and BVE-110) ventilation at different distances from the gallery wall. Boreholes BVE102 and BVE-110 were drilled outside the ventilated area, and BVE-100 and BVE-107 inside the ventilated area and perpendicular to bedding

5.3 Fitting to the van Genuchten expression

When the results are plotted in terms of the degree of saturation, it is not possible to group them as a function of the situation of the boreholes or of the position of the samples along the boreholes, neither for the samples taken before nor after the second ventilation phase. Thus, it has been decided to fit the results obtained for all the samples tested in each campaign to the van Genuchten expression (1980):

$$S_{e} = \frac{S_{l} - S_{rl}}{S_{ls} - S_{rl}} = \left(1 + \left(\frac{P_{g} - P_{l}}{P}\right)^{\frac{1}{1-\lambda}}\right)^{-\lambda}$$
[1]

where S_e is the effective degree of saturation ($0 \le S_e \le 1$), P is a material parameter related to the air entry value (MPa), λ the shape function for the retention curve, P_g - P_l is suction (MPa), S_{rl} the residual saturation and S_{ls} the maximum saturation. It has been considered that $S_{rl}=0$ and $S_{ls}=1$, and thus $S_e = S_r/100$, where S_r is the degree of saturation shown in Table III to Table VIII. Figure 34 and Figure 35 show the van Genuchten fit in terms of suction-degree of saturation that was obtained in the drying path for all the samples tested, taken before and after the second ventilation phase. The parameters obtained for this equation are shown in Table IX.

In order to improve the predictions of the analytical expression of the retention curve for suctions above 100 MPa –for which too higher suctions are predicted for the low water contents–, it is advisable to use a modification of the van Genuchten function that is more suitable for the highest suction values:

$$S_{e} = \frac{S_{l} - S_{rl}}{S_{ls} - S_{rl}} = \left(1 + \left(\frac{P_{g} - P_{l}}{P}\right)^{\frac{1}{1 - \lambda}}\right)^{-\lambda} \left(1 - \frac{P_{g} - P_{l}}{P_{s}}\right)^{\lambda_{s}}$$
[2]

where P_s and λ_s are two material parameters. Again it has been considered that $S_{rl} = 0$ and $S_{ls} = 1$. The curves obtained using these parameters are also shown in Figure 34 and Figure 35 and the parameters in Table IX.

Figure 34: Retention curves at 20°C of samples from boreholes drilled before the second ventilation phase and fittings obtained with Equation 1 (VG) and Equation 2 (VG mod) (each point is the average of two specimens)

Figure 35: Retention curves at 20°C of samples from boreholes drilled after the second ventilation phase and fittings obtained with Equation 1 (VG) and Equation 2 (VG mod) (each point is the average of two specimens)

Table IX: Values of the parameters for the van Genuchten (Eq. 1) and modified van Genuchten (Eq. 2) expressions for the retention curve (drying path) of samples taken before and after the second ventilation phase

	Before	e ventilation	After ventilation	
Parameter	VG (Eq. 1)	VG mod (Eq. 2)	VG (Eq. 1)	VG mod (Eq. 2)
P (MPa)	10.6±2.2	4	18.3±2.0	18.3±2.2
λ	0.31±0.03	0.162 ± 0.028	0.40±0.03	0.40 ± 0.08
$P_{\rm s}$ (MPa)		144±95		200
$\lambda_{\rm s}$		0.41±0.62		0.0±0.5

All the results obtained have been plotted in Figure 36, along with the fittings for the modified van Genuchten expression. The curves for the samples taken before and after ventilation differ only for the range of suctions that has not been actually tested (lower than 20 MPa), for which reason this difference is not considered significant. However, the same trend has been found when the results are plotted in terms of water content (Figure 28), *i.e.* a higher retention capacity after ventilation and a higher air entry value.

Figure 36: Retention curves obtained in drying paths in samples taken before and after the second ventilation phase and fittings for the modified van Genuchten expression (each point is the average of two specimens)

5.4 Comparison of results obtained for intact Opalinus clay

The results shown above have been compared with results obtained by UPC in samples taken from borehole BVE-1 (Figure 37). This borehole was drilled before the first ventilation phase started, and the samples used for the determination of the retention curve were taken at a depth of 9.5 m, *i.e.* much farther from the gallery than the samples used by CIEMAT. 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. Other samples taken from the same location were subjected to a wetting path under free volume conditions after having been slightly dried to a suction of 34 MPa, what gave place to an average initial water content at the beginning of wetting of 6.3 percent (Muñoz *et al.* 2003, Muñoz 2006).

For every suction, the water content reached by the samples of borehole BVE-1 is higher than for samples of the other boreholes. This can be a consequence of the different paths followed, since the samples show a hysteretic behaviour, and those from borehole BVE-1 were first saturated, what agrees with their higher water contents during drying. There are also other differences between the two set of samples that must be taken into account when analysing them:

- the different hydraulic history, since the samples from borehole BVE-1 had not been subjected to *in situ* wetting/drying cycles prior to the determination of the water retention curve, and
- the different salt content of the samples, since the samples from borehole BVE-1 would have a salinity equivalent to the background one at the Mont Terri URL, whereas the samples from the other boreholes, especially those taken close to the gallery wall, had much higher salinities, as it has been explained in section Material.

However, the differences found among boreholes disappear towards the high suctions (above 80 MPa). If the results are expressed in terms of degree of saturation (Figure 38), the results obtained in both set of tests are more similar, since the dry densities assumed by CIEMAT are higher than those measured by UPC (2.28 *vs.* 2.26 g/cm³). This fact causes also that the curve found by UPC with Equation 2 is very similar to that found by CIEMAT (compare Figure 34 and Figure 38).

Figure 37: Water contents obtained in the drying path followed by samples from boreholes BVE-99 to BVE-111 (CIEMAT results, each point is the average of two specimens) and in the drying after wetting and wetting after drying paths followed by samples from borehole BVE-1 (UPC results, Muñoz et al. 2003)

Figure 38: Degrees of saturation obtained in the drying path followed by samples taken before and after the second ventilation phase (CIEMAT results, each point is the average of two specimens), and in the drying after wetting path followed by samples from borehole BVE-1 (UPC results, Muñoz *et al.* 2003)

6 CONCLUSIONS

The retention curves of intact Opalinus Clay rock samples, taken from boreholes drilled before and after the second ventilation phase of the VE gallery at the Mont Terri URL and inside and outside the ventilated area, have been determined at 20°C. The technique of relative humidity control has been used to impose total suction to the unconfined samples until water content equilibrium following drying paths.

There were initial differences in the samples concerning their water content, dry density, salinity of the pore water and cations inside the interlayer. However, these differences are not clearly translated to the water retention behaviour and there are not significant differences in the behaviour of samples from different boreholes. Nevertheless, there are two trends of behaviour that could be remarked, although these differences tend to attenuate towards high suction, *i.e.* when lower pore sizes are involved. On the one hand, the water retention capacity of the samples taken near the gallery wall tends to be higher than that of samples taken at deeper positions along the boreholes. On the other, the retention capacity increases after ventilation for the low suctions, especially for samples taken inside the ventilated area and near the gallery wall. Three circumstances could explain both facts:

- The higher salinity of the pore water of the samples that show higher water retention capacity, since their osmotic suction is consequently higher.
- To a lesser extent, the increase in exchangeable magnesium in these samples could also contribute to their higher retention capacity, since this cation has high hydration energy.
- The slightly lower density of the samples taken near the gallery wall, due to decompression, which would increase their theoretical capacity of water absorption by the increase in porosity.

The first hypothesis is supported by the fact that the same trends have been clearly observed in powdered samples, in which density has obviously no influence, and whose water content for low suctions is positively correlated mainly to the content of chloride in the pore water, whereas it is not correlated to the exchangeable magnesium content.

The experimental results have been fitted to the van Genuchten expression, in which the value of the parameter related to the air entry pressure (P) is higher for the samples taken after the second ventilation phase.

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