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Determination of the water retention curve of bentonite from Georgia and from Wyoming (United States)

Experimental results using the jar method and assessment of available techniques for suction control and measurement

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Abstract

This report contains the water retention curve measurement of two different bentonites, the first one from Georgia and the second from Wyoming (United States). The water retention curve was measured using the jar method, where dried and wetted powder were emplaced into jars with different saturated solutions which controlled different relative humidity. The water retention curve was measured in drying and wetting paths.

The relative humidity of the different saturated solutions at the laboratory temperature, 21±1 °C , were obtained from the literature. The relative humidity was also measured with capacitive hygrometers.

After the test, the suction of the bentonite samples was measured with a chilled mirror dew point psychrometer and the results obtained were compared with the suction expected from the relative humidity controlled in jars, as the relative humidity is related with the suctions through the psychrometric law.

The curves presented in this report are relative humidity vs water content and suction vs water content. The results showed the hysteresis effect, where the bentonite presents larger water retention capacity in drying paths than in wetting paths.

The report also contains an assessment of the different methods for measuring and controlling the suction. The suction can be measured with tensiometers (low range: 0 to 1.5 MPa), psychrometers (dew point in the range 100 kPa to 300 MPa and thermocouple psychrometers in a range of 50 kPa to 6.9 MPa) and hygrometers (10 MPa to 500 MPa, the lower range can be extended but increasing the error). The suction can also be measured with other sensors where the suction is calculated from the thermal conductivity (heat dissipation sensor, range 10 kPa to 2.5 MPa) or the volumetric water content (equitensiometer, range between 10 kPa and 1.0 MPa). The suction can be controlled through the axis translation technique (0 to 10 MPa), the relative humidity control (10 to 400 MPa, the lower range can be extended if the temperature is well controlled) and the osmotic method (0 to 10 MPa). A new device based on climate chamber principle (7 to 480 MPa) which is still under development is also presented. The axis translation and the osmotic technique control the matric suction and the relative humidity technique controls the total suction.

Sammanfattning

Denna rapport innehåller mätningar av vattenretentionskurvan för två olika bentoniter, Georgisk och Wyoming (USA). Vattenretentionskurvan mättes med burkmetoden, där ett torrt eller vått pulverprov placerades i en burk med olika mättade saltlösningar som kontrollerade den relativa fuktighetsnivån. Vattenretentionskurvan mättes under torkning eller vätning.

Den relativa fuktigheten för de olika mättade saltlösningarna vid laboratorietemperaturen, 21±1 °C, erhölls från litteraturen Den relativa fuktigheten mättes också med en kapacitiv fuktighetsmätare.

Efter experimentet mättes sugkraften från bentoniprovet med en psykrometer. De uppmätta resultaten jämfördes med de förväntade sugeffektresultaten från burkarna i vilka den relativa fuktigheten kontrollerades, eftersom den relativa fuktigheten relaterar till sugtrycket genom den psykrometriska lagen.

Kurvorna som presenteras i denna rapport är relativ fuktighet vs vattenhalt och sugkraft vs vattenhalt. Resultaten visar effekten av hysteres. Bentonit hade en högre vattenretentionsförmåga under torkning än under vätning.

Rapporten innehåller även en utvärdering av de olika metoderna för att mäta och kontrollera sugkraften. Sugkraften kan mätas med en tensiometer (lågt område: 0 – 1.5 MPa), en psykrometer (daggpunkt mellan 100 kPa och 300 MPa och en termoelementpsykrometer mellan 50 kPa och 6.9 MPa) och en fuktighetsmätare (mellan 10 MPa och 500 MPa, den nedre gränsen kan förlängas, men felmarginalen ökar samtidigt). Sugkraften kan även mätas med andra sensorer där suget beräknas utifrån värmeledningsförmågan (värmeförlustsensor, med ett mätområde mellan 10 kPa och 2.5 MPa) eller med volymetriska vattenhalten (med den så skallade equitensiometer med ett mätområde mellan 10 kPa och 1.0 MPa). Sugkraften kan styras med den så kallade axis-translation tekniken (mellan 0 och 10 MPa), genom att kontrollera relativa fuktigheten (mellan 10 MPa och 400 MPa, det nedre intervallet kan utökas om temperaturen är väl kontrollerad) och med osmotiska metoden (mellan 0 och 10 MPa). En ny apparat baserad på klimatkammar-principen (7 till 480 MPa) som fortfarande är under utvecklingsfas presenteras också. Axis-translation tekniken och osmotiska metoden styr matrissuget och teknologin för relativ fuktighet styr det totala suget.

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1 Introduction

This report presents the water retention curves of two different bentonites, one from Georgia and the another from Wyoming (United States). The water retention curves were measured in powder samples in drying and wetting paths using the jars method, where the sample suction is controlled in an atmosphere where the relative humidity is controlled by saturated solutions. The relative humidity was measured by a hygrometer and the suction of the sample was measured by a chilled mirror dew point psychrometer. The main purpose was to assess this method, which is partially described in ASTM and EN standards.

There is a wide range of alternative methods for controlling and measuring the suction (Delage et al. 2008). This report presents some of these methods and analyses what methods would be suitable for the bentonites.

2 Materials and base variables

2.1 Materials

Two different bentonites were analysed, bentonite from Georgia and bentonite from Wyoming (United States). The grain densities (density of solids, ρ_{solids}) of Georgia's bentonite is 2805 kg/m^3 and the grain density of Wyoming bentonite is 2776 kg/m^3. The initial water content of Georgia's bentonite was 11.0 % and the initial water content of Wyoming bentonite was 10.3 %.

2.2 Base variables

2.2.1 Density and water content

The base variables are the density of solids (ρ_{solid}) and the water content (w), which are determined following the equations:

$$\rho_{solids} = \frac{M_s}{V_s} \tag{2-1}$$

$$w = \frac{M_w}{M_s} \tag{2-2}$$

Where M_s is the mass of solids, M_w is the mass of water and V_s is the volume of solids.

2.2.2 Relative humidity and suction

The relative humidity (RH) was measured with a capacitive hygrometer HMT330 (www.vaisala.com) which is usually calibrated by placing the probe inside a flask with relative humidity controlled by saturated solutions or partially saturated solutions. It is important to carry out the calibration at constant temperature.

The relative humidity is defined by the equation

$$RH = \frac{p}{p_s} \tag{2-3}$$

Where p is the partial pressure of water vapour and p_s the saturation pressure of the pure water at the same temperature. The relative humidity is related with the suction by the psychrometric law (Edlefsen and Andersson 1943).

$$s = -\frac{R \cdot T \cdot \rho_w}{\omega_v} ln\left(\frac{p}{p_s}\right) \tag{2-4}$$

Where T is the absolute temperature, ρ_w the density of water (998 kg/m^3 at 25 °C), ω_v the molecular mass of water (18 kg/mol) and R the universal gas constant (8.31432 J/(mol·K)).

The suction was measured with the chilled mirror dew point psychrometer WP-4, similar to WP-4C (www.metergroup.com) when the relative humidity of the jar was in equilibrium with the suction of the sample. The weight of the sample was measured during the equilibration process.

3 Methods

The method for measuring the water retention curve by the jar method was described by Wadsö et al. (2004). This method has been widely used for measuring the water retention curve of MX-80 bentonite (Johannesson et al. 2008, Dueck and Nilsson 2010, Svensson et al. 2019). MX-80 bentonite is also a bentonite from Wyoming (US).

The samples were emplaced inside glass jars (Figure 3-1 and Figure 3-2) where the relative humidity was controlled by saturated solutions (Table 3-1). The change of mass of bentonite sample was measured regularly without removing the sample from the jar by using the density determination kit for solid samples (ASTM D792 and ISO 1183-1, Figure 3-3).

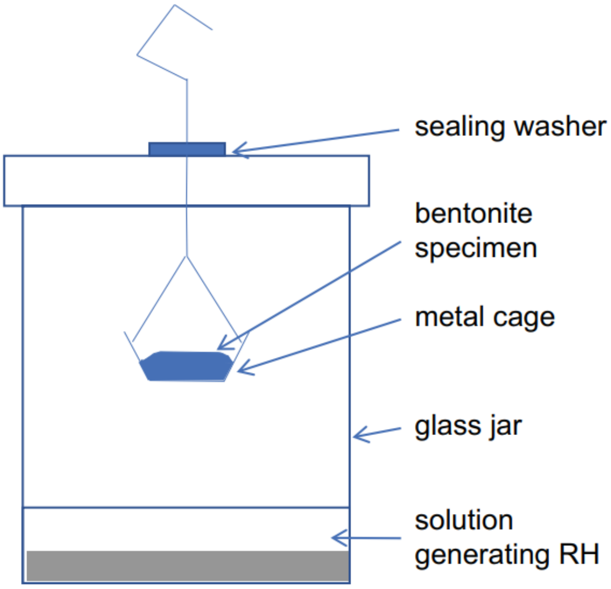

Figure 3-1. Glass jars for controlling the relative humidity (Johannesson et al. 2008).

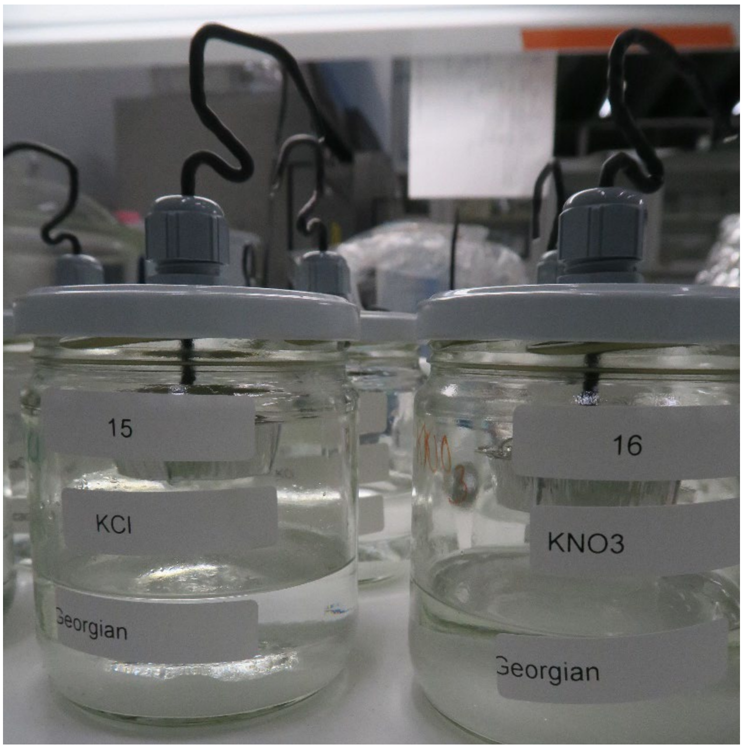

Figure 3-2. Glass jars in tests performed with bentonite.

Figure 3-3. Density determination kit for solid samples.

Table 3-1 Relative humidity imposed by saturated solutions.

#	Salt solution	Relative humidity at T=25 °C (%)	Suction at T=25 °C (MPa)	A (%)	B (K)
1	NaOH	6	386	5.48	27
2	LiCl	11	303	14.53	-75
3	$MgCl_2 \cdot H2O$	33	152	29.26	34
4	NaBr	58	75	20.49	308
5	NaCl	75	39	69.2	25
6	KCl	84	24	49.38	159
7	KNO_3	92	11	43.22	225
8	K_2SO_4	97	4.2	86.75	34

$RH(\%) = A \cdot exp\left({}^{B}/_{T}\right)$. Lide (1998)

3.1 Preparation of the samples

The grain size of bentonite material (powder) was less than 1.0 mm. The grain size of the powder is not the same as the size of the particles in the material, which is lower. An amount of around 5-15 g of powder was emplaced in the metal cage (Figure 3-1). The weight of the powder was designed to be a function of the size of the cage: the drier the powder was, the lower the capacity, so in wetting paths, the initial weight of the samples was smaller than the final weight. Two different paths were defined. In the wetting path tests (sorption path described in SS-EN ISO 12571:2021) the material was dried in the oven before starting the tests following the standard ISO/TS 17892-1.

In the drying path tests (desorption path described in SS-EN ISO 12571:2021), water was mixed with bentonite powder for increasing the initial water content in order to ensure quasi-saturated state before starting the test. For checking the quasi-saturated conditions, both bentonites were soaked at different water contents and the suction measured with the chilled mirror psychrometer following the method described in ASTM D6836. The relative humidity equivalent to the suction measured by the psychrometer was calculated with the psychrometric law (see equation (2-4)). The equation reads

$$RH(\%) = 100 \cdot exp\left(-\frac{s \cdot \omega_v}{R \cdot T \cdot \rho_w}\right) \qquad (3\text{-}1)$$

The results are presented in Figure 3-4 and Figure 3-5.

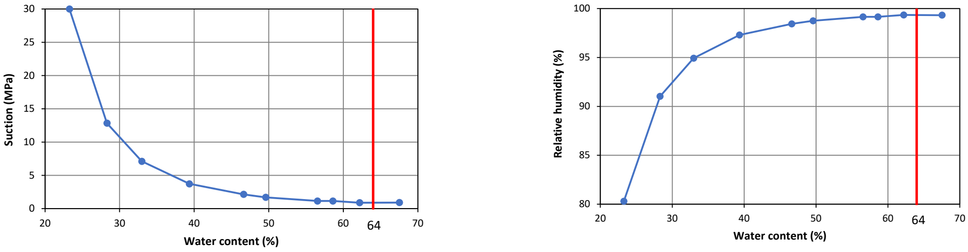

Figure 3-4. Suction vs water content (left). Relative humidity vs water content (right). Georgian bentonite.

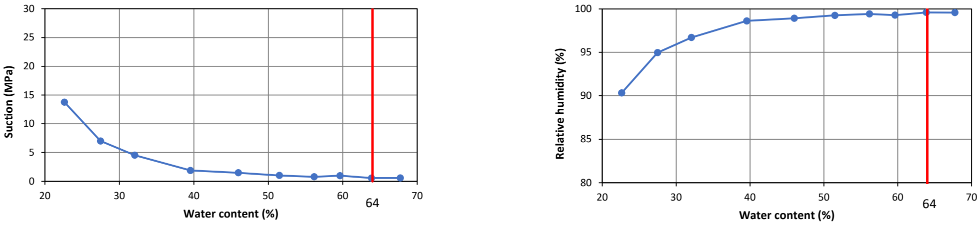

Figure 3-5. Suction vs water content (left). Relative humidity vs water content (right). Wyoming bentonite.

Quasi-saturated conditions can be considered when RH is 95% or larger (SS-EN ISO 12571:2021). These conditions were reached when the water content was 33% in Georgian bentonite and 28% in Wyoming bentonite. As the excess of water does not disturb the test (only causes delays), it was decided to start the drying path tests at the same water content as presented in Dueck and Nilsson (2010). The initial water content of both materials was 64% at the beginning of drying path tests.

3.2 Test program

Four test series were performed (Table 3-2): two wetting or sorption paths and two drying or desorption paths. A total of 36 tests (samples) were performed and each test had their own jar. The jars were acting as desiccators. The test was slightly different than the test proposed by SS-EN ISO 12571:2021, where the same sample is subjected to different relative humidity's during the wetting and/or drying paths. Due to the time necessary for reaching the steady state condition is large (around four months), it was necessary due to scheduling reasons to perform the tests in different jars each that have different relative humidity coincidently. The relative humidity was not measured continuously as it is indicated in SS-EN ISO 12571:2021 (Annex 4).

Table 3-2. Test program.

Serie	Material	Path	Sample	Salt solution	Relative humidity [%][1]
1	Georgian	Wetting (desorption)	1	NaOH	6
			2	LiCl	11
			3	$MgCl_2 \cdot 6H_2O$	33
			4	NaBr	58
			5	NaCl	75
			6	KCl	84
			7	KNO_3	92
			8	K_2SO_4	97
			9 (Duplicate)	$MgCl_2 \cdot 6H_2O$	33
2		Drying (sorption)	1	NaOH	6
			2	LiCl	11
			3	$MgCl_2 \cdot 6H_2O$	33
			4	NaBr	58
			5	NaCl	75
			6	KCl	84
			7	KNO_3	92
			8	K_2SO_4	97
			9 (Duplicate)	KNO_3	92
3	Wyoming	Wetting (desorption)	1	NaOH	6
			2	LiCl	11
			3	$MgCl_2 \cdot 6H_2O$	33
			4	NaBr	58
			5	NaCl	75
			6	KCl	84
			7	KNO_3	92
			8	K_2SO_4	97
			9 (Duplicate)	$MgCl_2 \cdot 6H_2O$	33
4		Drying (sorption)	1	NaOH	6
			2	LiCl	11
			3	$MgCl_2 \cdot 6H_2O$	33
			4	NaBr	58
			5	NaCl	75
			6	KCl	84
			7	KNO_3	92
			8	K_2SO_4	97
			9 (Duplicate)	KNO_3	92

[1] Relative humidity expected at 25 °C following the Table 3-1.

3.3 Test finishing

Termination criterion of tests was based on the standard SS-EN ISO 12571:2021, that recommends finishing the test when the change of mass between three consecutive weightings, each made at least 24 h apart, is less than the 0.1% of the total mass.

4 Results

Two different results are presented: the mass change (Equation (4-1)) with time in logarithmic scale to be able to check when the change of suction process reached the steady state conditions, and the water retention curves in drying (desorption) and wetting (sorption) paths. The Wyoming bentonite results are compared with the results obtained by Dueck and Nilsson (2010) for another bentonite from Wyoming (MX-80). The final water content was determined by drying the sample in the oven following the standard ISO/TS 17892-1.

The mass change is expressed as:

$$mass\ change\ from\ start\ (-) = \frac{m_s + m_{wf}}{m_s + m_{w0}} \tag{4-1}$$

Where m_s is the mass of solids and m_{wf} and m_{w0} are the mass of water after the test and at the beginning of the test, respectively.

4.1 Georgian bentonite

The summary of the results obtained in tests performed with Georgian bentonite samples is shown in Table 4-1 and Table 4-2.

Table 4-1. Georgian bentonite samples with wetting path.

Sample	Salt solution	Theoretical relative humidity at 25 °C [%]	Theoretical suction at 25 °C [MPa]	Initial water content [%]	Final water content [%]	Suction measured [MPa]
1	NaOH	6	386	0.0	1.4	332.61
2	LiCl	11	303		1.9	297.97
3	$MgCl_2 \cdot 6H_2O$	33	152		6.4	157.35
4	NaBr	58	75		13.4	77.65
5	NaCl	75	39		19.7	42.28
6	KCl	84	24		23.1	26.45
7	KNO_3	92	11		29.1	11.4
8	K_2SO_4	97	4.2		35.6	4.66
9	$MgCl_2 \cdot 6H_2O$	33	152		6.3	155.2

Table 4-2. Georgian bentonite samples with drying path.

Sample	Salt solution	Theoretical relative humidity at 25 °C [%]	Theoretical suction at 25 °C [MPa]	Initial water content [%]	Final water content [%]	Suction measured [MPa]
10	NaOH	6	386	64.2	5.2	297.33
11	LiCl	11	303		6.4	294.73
12	$MgCl_2 \cdot 6H_2O$	33	152		12.0	157.12
13	NaBr	58	75		19.8	77.00
14	NaCl	75	39		24.3	41.94
15	KCl	84	24		27.2	25.52
16	KNO_3	92	11		33.3	11.81
17	K_2SO_4	97	4.2		43.7	3.88
18	KNO_3	92	11		33.5	10.98

The evolution of the mass change in tests performed Georgian bentonite is presented in Figure 4-1 (drying path) and in Figure 4-2 (wetting path).

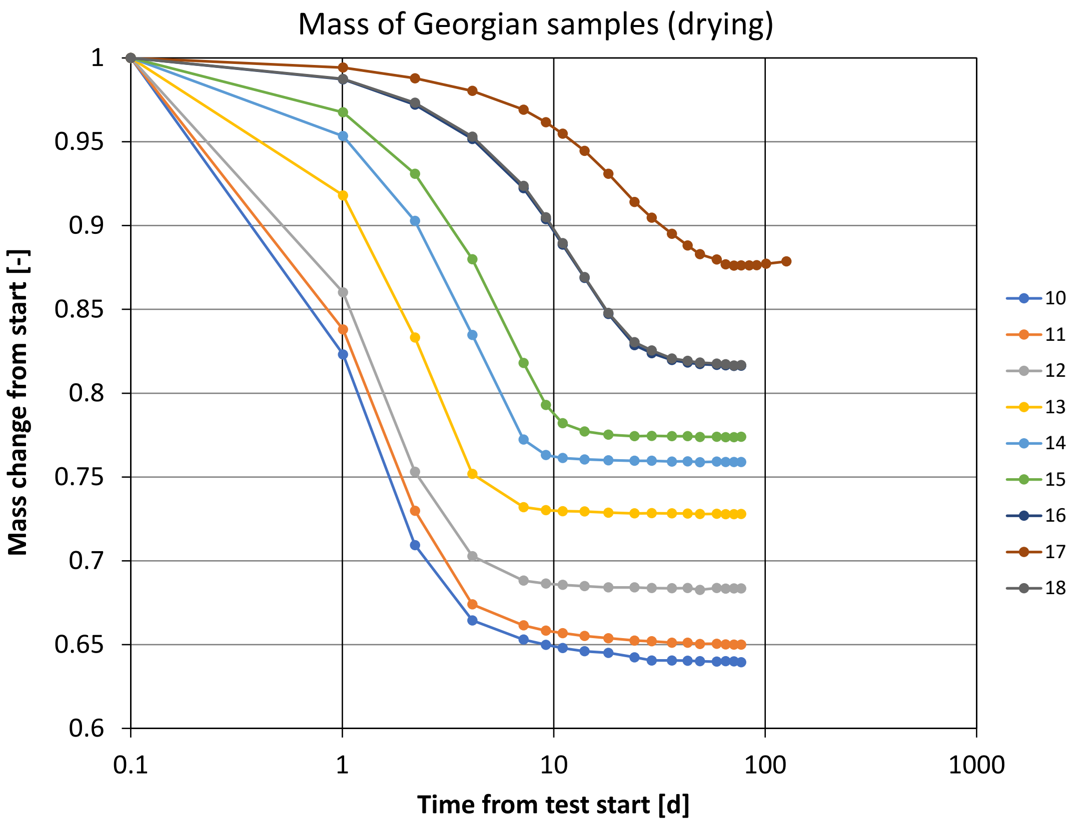

Figure 4-1. Evolution of mass change in drying paths. Georgian bentonite.

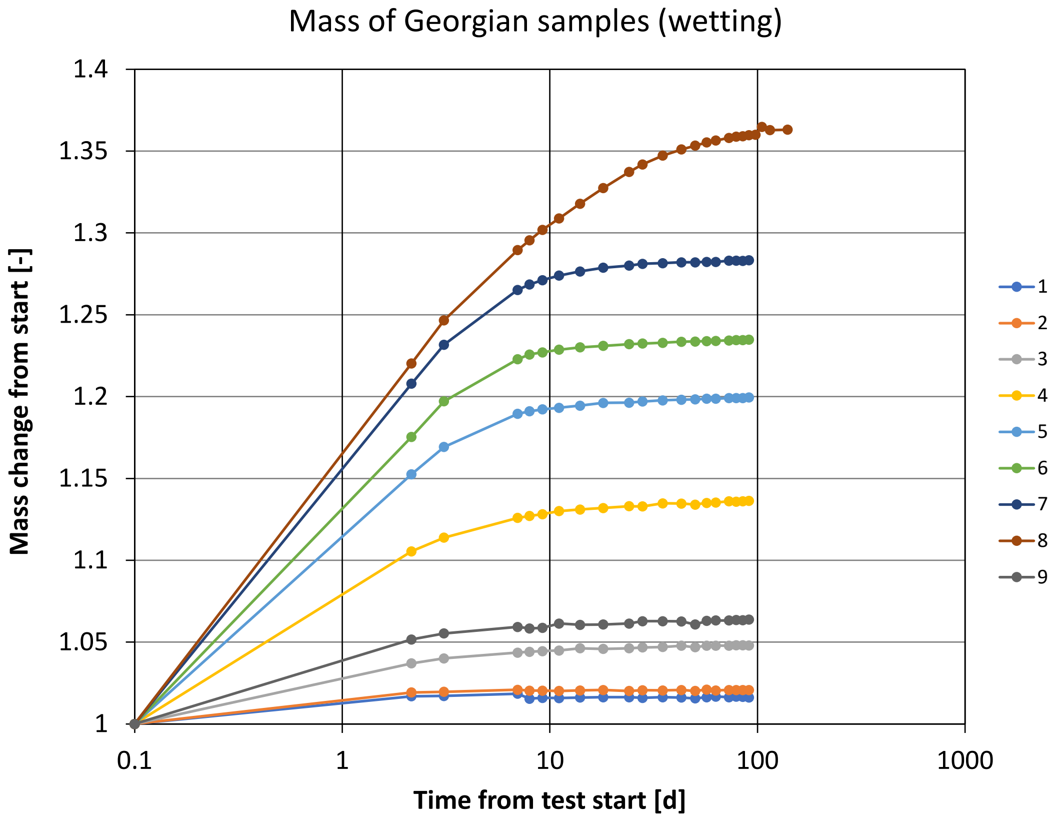

Figure 4-2. Evolution of the mass change in wetting path. Georgian bentonite.

The water retention curves of Georgian bentonite are presented in Figure 4-3 and Figure 4-4.

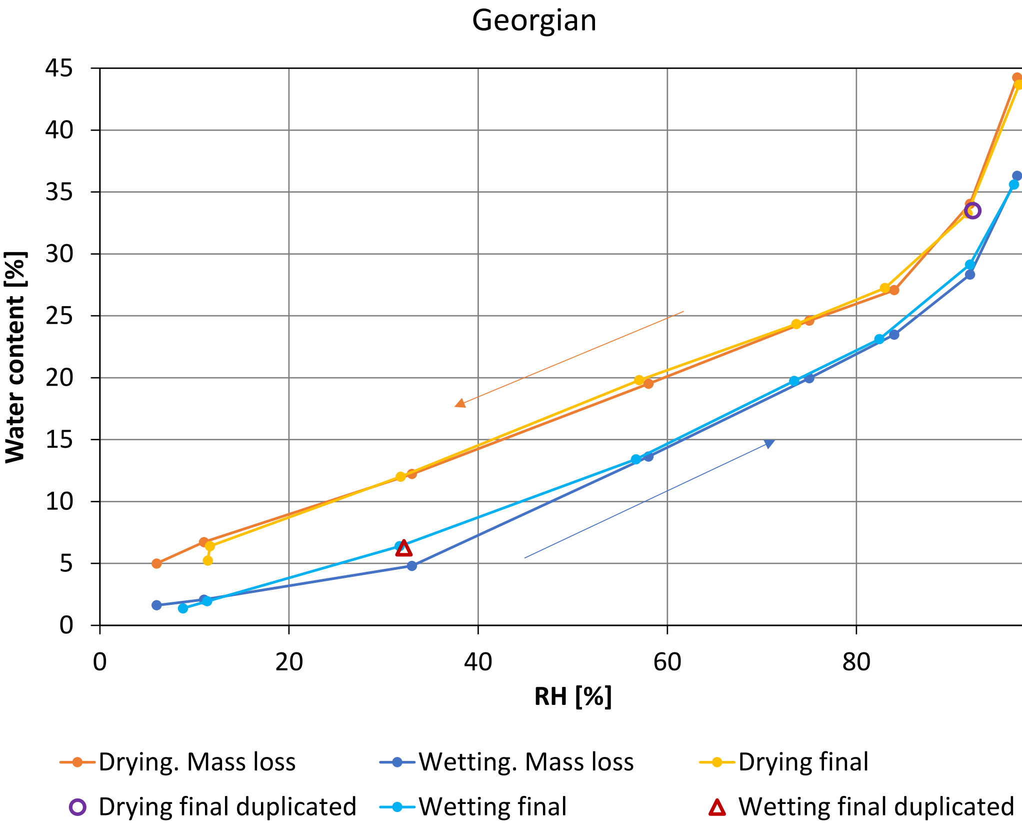

Figure 4-3. Water retention curve expressed as RH vs water content. RH is obtained from the theoretical values of the solutions (Table 4-1 and Table 4-2), and water content from the mass loss measurements (as indicated with 'Mass loss' in the legend). Also, RH is obtained from the suction measurement with the psychrometer following the psychrometric law, and water content is obtained drying the sample in the oven (as indicated 'final' in the legend). Georgian bentonite.

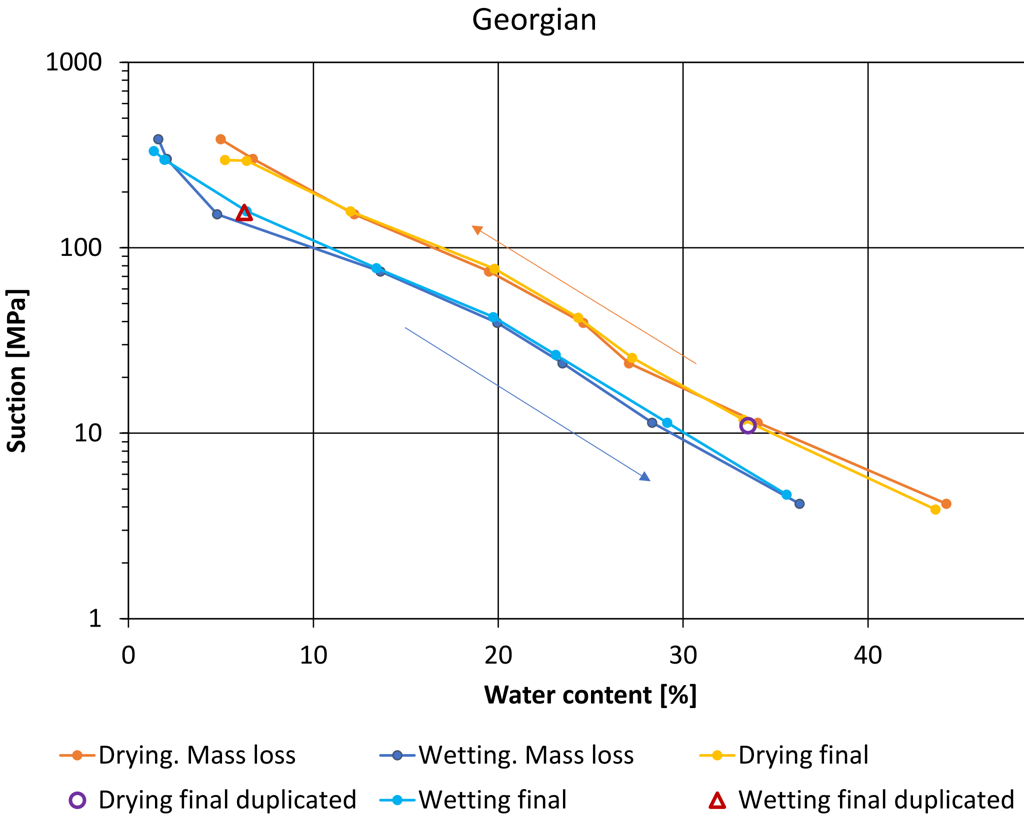

Figure 4-4. Water retention curve, expressed as water content vs suction. Suction is obtained from the theoretical value of the salt solutions following the psychrometric law (Table 4-1 and Table 4-2,) and water content from the mass loss measurements (as indicates 'Mass loss' in the legend). Also, suction is obtained from the suction measurement with the psychrometer, and water content drying the sample in the oven (as indicated 'final' in the legend). Georgian bentonite.

4.2 Wyoming bentonite

The summary of the results obtained in tests performed with Wyoming bentonite samples is shown in Table 4-3 and Table 4-4.

Table 4-3. Wyoming bentonite samples with wetting path.

Sample	Salt solution	Theoretical relative humidity at 25 °C [%]	Theoretical suction at 25 °C [MPa]	Initial water content [%]	Final water content [%]	Suction measured [MPa]
19	NaOH	6	386	0.0	0.5	346.18
20	LiCl	11	303		0.7	295.82
21	$MgCl_2 * 6H_2O$	33	152		2.2	160.35
22	NaBr	58	75		7.4	78.55
23	NaCl	75	39		13.7	42.77
24	KCl	84	24		17.1	24.99
25	KNO_3	92	11		21.3	11.06
26	K_2SO_4	97	4.2		29.5	4.08
27	$MgCl_2 * 6H_2O$	33	152		2.3	150.88

Table 4-4. Wyoming bentonite samples with drying path.

Sample	Salt solution	Theoretical relative humidity at 25 °C [%]	Theoretical suction at 25 °C [MPa]	Initial water content [%]	Final water content [%]	Suction measured [MPa]
28	NaOH	6	386	63.0	1.5	288.27
29	LiCl	11	303		2.7	279.32
30	$MgCl_2 * 6H_2O$	33	152		9.2	156.64
31	NaBr	58	75		16.5	76.71
32	NaCl	75	39		20.5	41.25
33	KCl	84	24		22.9	24.8
34	KNO_3	92	11		28.0	10.71
35	K_2SO_4	97	4.2		36.3	4.46
36	KNO_3	92	11		28.0	11.01

The evolution of the mass change in tests performed with Wyoming bentonite is presented in Figure 4-5 (drying path) and in Figure 4-6 (wetting path).

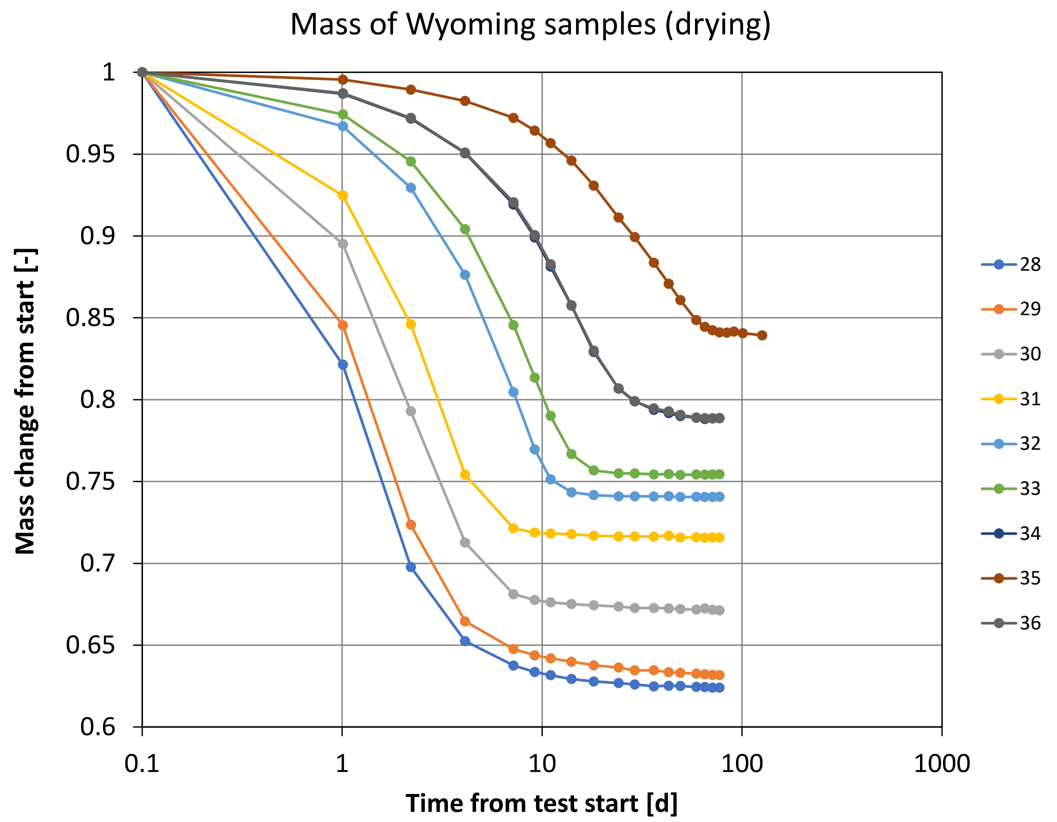

Figure 4-5. Evolution of mass change in drying paths. Wyoming bentonite.

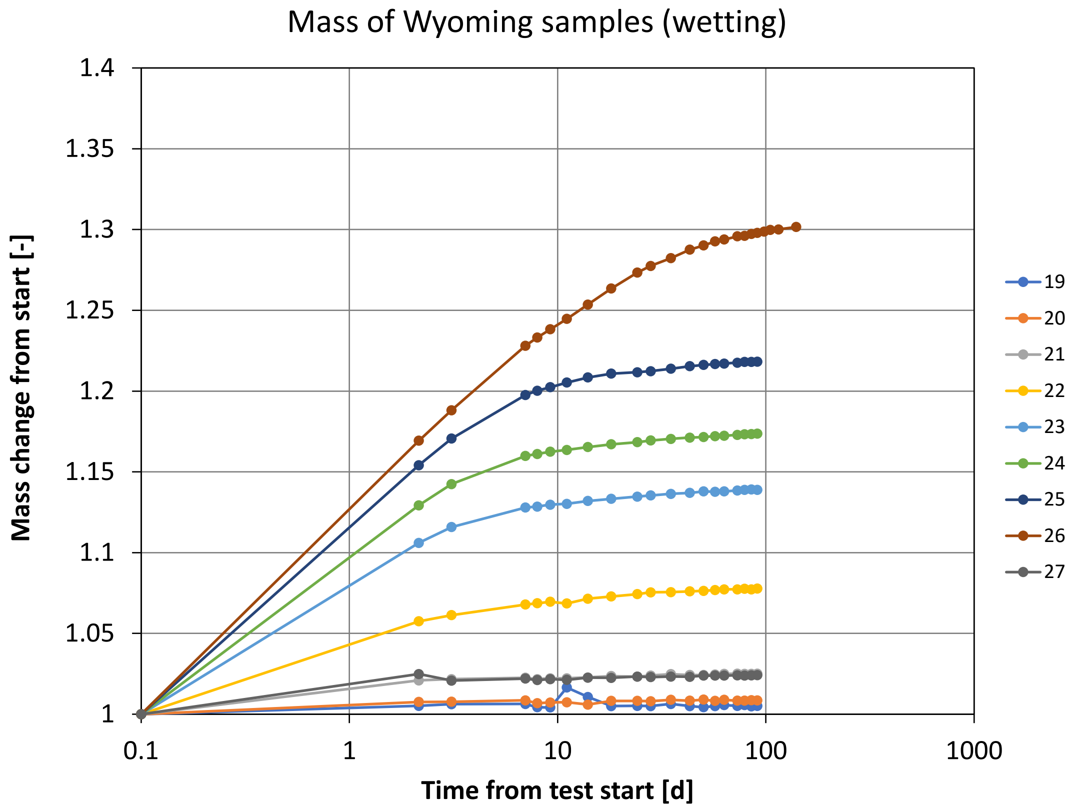

Figure 4-6. Evolution of the mass change in wetting path. Wyoming bentonite.

The water retention curves of Wyoming bentonite are presented in Figure 4-7 and Figure 4-8. Figure 4-8 includes the results obtained by Dueck and Nilsson (2010) with MX-80, a bentonite from Wyoming as well.

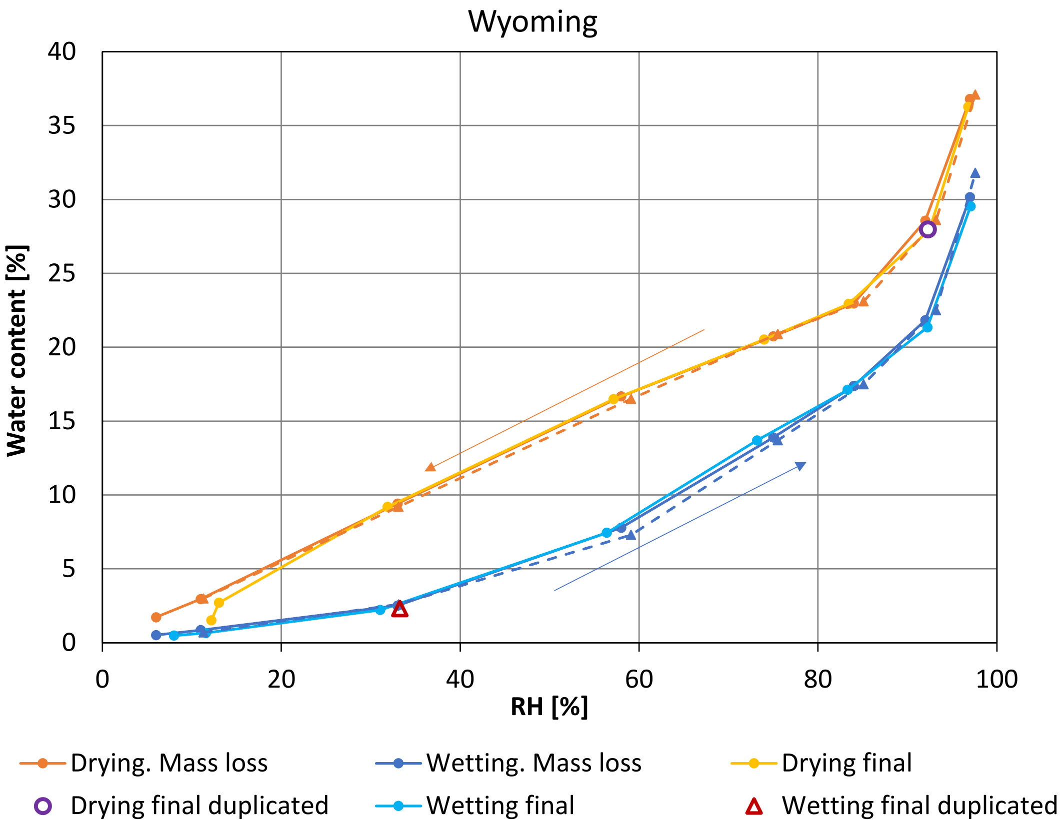

Figure 4-7. Water retention curve expressed as RH vs water content. RH is obtained from the theoretical values of the solutions (Table 4-1 and Table 4-2), and water content from the mass loss measurements (as indicated with 'Mass loss' in the legend). Also, RH is obtained from the suction measurement with the psychrometer following the psychrometric law, and water content is obtained drying the sample in the oven (as indicated 'final' in the legend). Wyoming bentonite.

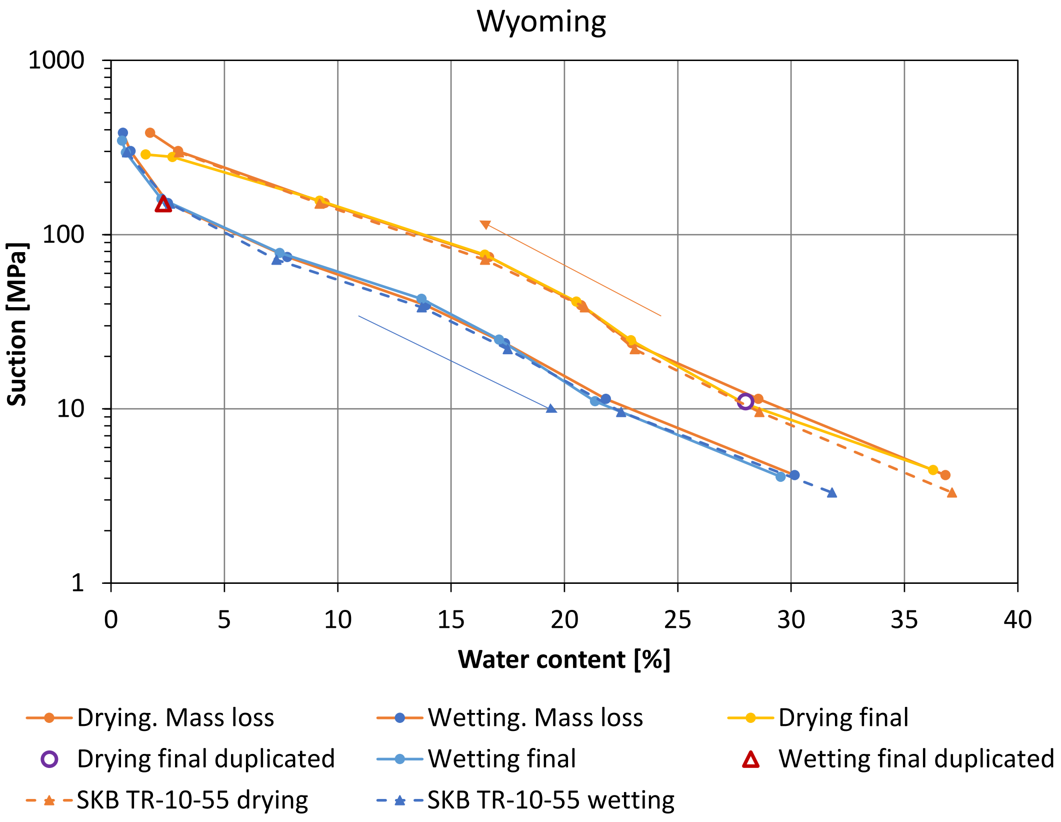

***Figure 4-8.** Water retention curve, expressed as water content vs suction. Suction is obtained from the theoretical value of the salt solutions following the psychrometric law (Table 4-1 and Table 4-2,) and water content from the mass loss measurements (as indicates 'Mass loss' in the legend). Also, suction is obtained from the suction measurement with the psychrometer, and water content drying the sample in the oven (as indicated 'final' in the legend). Wyoming bentonite. Results from Dueck and Nilsson (2010) are presented as well for comparison.*

5 Assessment of available techniques for suction control and measurement

This chapter contains the assessment of available standards and different test methods for determining the water retention curve and comparison of these methods with the method described in SKB report TR-19-25 (jar method, which was used in this work as well). The available standards for measuring the water retention curve are the ASTM D6836-16 and the SS-EN ISO 12571:2021.

The theoretical concept of soil suction was developed in the early 1900's and was related with the soil – water – plant system. The concept of suction in soil mechanics was introduced in the middle of the last century. More information and references concerning the development of the soil suction concept can be found in Fredlund and Rahardjo (1993).

Due to the concept of the soil suction was related at the beginning with the matrix suction and the presence of meniscus between soil particles, the suction was measured in relatively low ranges (order of magnitude of kPa's) and the methods for controlling and measuring were mainly developed for this range. The bentonites present a high air entry value, some MPa's, and the ranges of suction expected are large, till a few hundreds of MPa's. Due to the wide range of suctions expected in bentonites, the measurement of the water retention curve in bentonites might need more than one method.

The suction can be measured with different sensors. Like with the techniques for suction control, many of these sensors were developed for measuring the suction in low levels. The sensors were mainly designed for measuring the suction "in situ" although can also be used in the laboratory. Other devices were designed for measuring the suction in the laboratory.

The suction is related with the relative humidity (see the equations (2-3) and (2-4)), so some sensors are related with the meteorological sensors and as it has been pointed out before, the suction is also related with the soil – water – plant system, so other sensors were developed for the agronomy.

It is recommended to control the suction and after the test, once the steady state conditions of the system suction control – sample have been reached, to measure the suction in the sample.

5.1 Suction control techniques

The suction control consists in controlling the suction in one or both faces of the sample or in controlling the relative humidity around it.

5.1.1 Axis translation technique

Axis translation method fixes the liquid and gas pressures at certain level. Liquid and gas are phases and water and air are components. Usually, liquid (P_l) and water (u_w) pressure and gas (P_g) and air (u_a) pressure are considered the same. The air can be nitrogen or any other inert gas like helium. The suction controlled in this method is the matrix suction.

$$s = P_g - P_l = u_a - u_w \qquad (5\text{-}1)$$

Axis translation method is described in ASTM D6836-16. The hanging column apparatus used in the method fixes the gas pressure at atmospheric conditions and reduces the liquid pressure (Method A). The range of suctions is quite low, 0-80 kPa. Pressure chamber or pressure plate fixes the liquid pressure at atmospheric level and increases the gas pressure. In this case, the range can be up 1500 kPa if the test is performed with a high air entry value porous stone and up 10 MPa if the test is performed with a membrane (Methods A and B). During the method the high air entry value porous stone and the membrane remain saturated and water flows in or out from the sample. The change of water content can be determined by measuring the water flowing in or out (Method B, ASTM D6836-16) or weighting the sample after each step (Method C, ASTM D6836-16). The measurement of the water volume is less accurate than the measurement of the weight but it has the advantage that it is not necessary to remove the sample from the test set-up, avoiding disturbances. Axis translation method is also described in ASTM D2325-68(2000) in a range of 0-101 kPa and in ASTM D3152-72(2000) in a range of 101-1520 kPa.

The axis translation fixing the liquid pressure and increasing the gas pressure is widely used in suction controlled oedometer tests (Romero et al. 1999, Romero 1999, Villar 2002).

The method is not usual in bentonites due to the low range of suctions which can be controlled although it has been used by Villar (2002), Hoffmann (2005) and Alonso et al. (2011). Hoffmann (2005) and Alonso et al. (2011) have used the axis translation technique combined with relative humidity-controlled technique, explained at the next section. The high air entry value porous stones reach only 1500 kPa and the membranes 10 MPa (Figure 5-1). Testing with the membranes might be complicated and it is probable that the membrane breaks during the test.

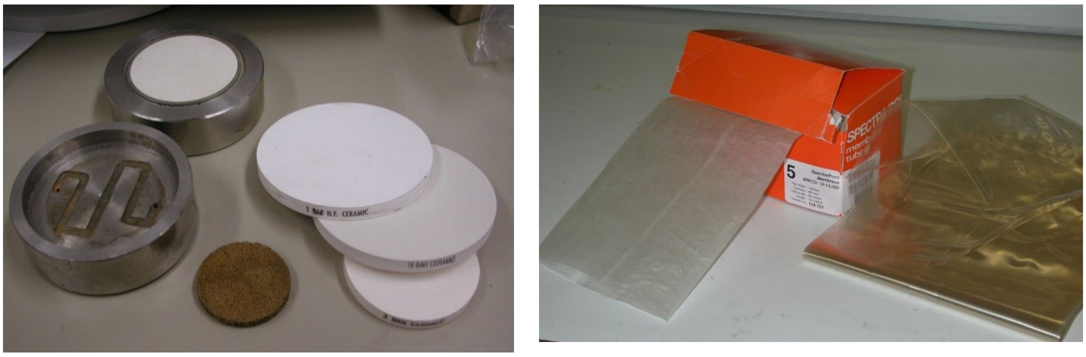

Figure 5-1. High air entry value porous stone (in left) and semi-permeable cellulose acetate membrane (in right) (Hoffmann 2005).

5.1.2 Relative humidity-controlled technique

Relative humidity-controlled method is described in the SS-EN ISO 12571:2021. This standard proposes two different methods for controlling the suction. The first one is with desiccators, where the relative humidity is controlled with saturated solutions as it is described in SS-EN ISO 12571:2021 (Annex A, relative humidity controlled by different saturated solutions) and SS-EN ISO 12571:2021 (Annex B, preparation of the saturated solutions). It could also be possible to use solutions at certain concentrations. The solutes might be sulphuric acid (Gmitro and Vermeulen 1964, Esteban 1990, Villar 2002) or sodium chloride (Lang 1967, MacMullin 1969, Romero 1999). The second method proposed is the climatic chamber but it will not be discussed in this report. The use of the climatic chamber would not be effective because the time for reaching the equilibrium can be of several weeks. The samples are small, so it is worth to carry out the test in smaller containers where the relative humidity is controlled. It is important to consider that the climatic chamber method controls the total suction.

Glass jars would also work as desiccators if they are properly sealed. Using a glass jar (as used in this work) is also described in SS-EN ISO 12571:2021 (Annex D).

The water exchange is performed through diffusion, so the time for reaching the steady state conditions might be some weeks. If the test is performed keeping the volume constant (Villar 2002), the time for reaching the steady state conditions can be some months. The SS-EN ISO 12571:2021 proposes to move the samples between desiccators where the relative humidity is different once the sample have reached the equilibrium with the relative humidity imposed, increasing or decreasing the suction. Due to the time for reaching the steady state conditions is long, it is recommended here instead to prepare multiple samples and desiccators or jars for fixing different relative humidity.

The water exchange can be forced if the air with the relative humidity controlled is pumped through the porous stones on top and at the bottom of the sample. Figure 5-2 presents the suction controlled oedometer test keeping constant volume and the Figure 5-3 the full instrumented cell, where the axial, radial swelling pressures and the evolution of the relative humidity are measured. This device was used by Dueck (2004), who presented a study about the variations of the relative humidity measurements as function of the position of the sensors. This device has also been used for measuring the swelling pressure (Karnland et al. 2006, Kiviranta et al. 2018) changing the relative humidity system by a water supply. Figure 5-4 presents the difference between the pure diffusion and when there is a forced convection along the boundaries in a suction controlled oedometer test.

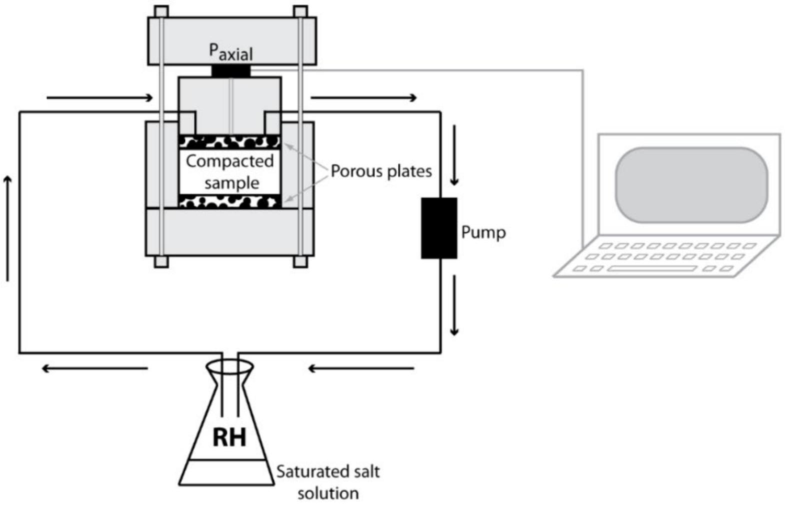

Figure 5-2. Schematic picture for measuring the water retention curve under constant volume. The swelling pressure is measured with a load cell. RH-controlled air flows through the upper or upper and bottom valves of the cell (Kiviranta et al. 2018).

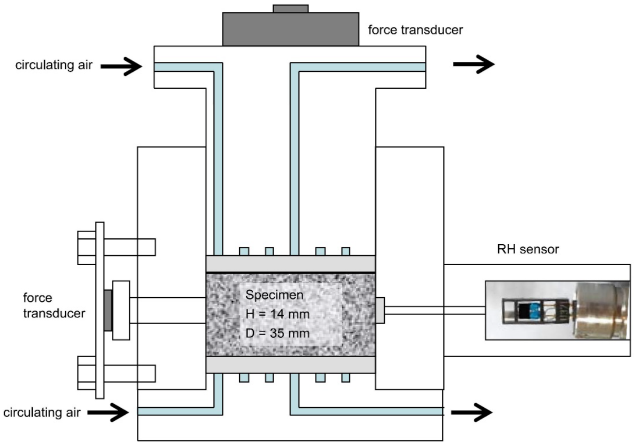

Figure 5-3. Schematic picture of the cell fully instrumented (Dueck and Nilsson 2010).

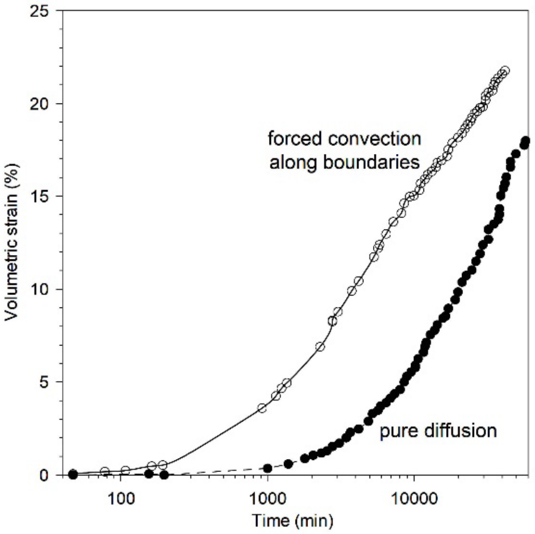

Figure 5-4. Evolution of volumetric strain on compacted bentonite in suction controlled oedometer tests using humid air flow along the boundaries of the sample (forced convection; Pintado et al. 2009) or controlling the air relative humidity inside a closed chamber (pure diffusion; Villar 2002) (Delage et al. 2008).

It could also be possible to force the convection through the sample. In this case, the water exchange is the fastest, but the suction is not constant in the sample (Pintado et al. 2009).

The relative humidity controlled with salt solutions is quite sensitive to the temperature when the relative humidity is larger than 95%, so small changes in temperature might mean large changes in relative humidity, which in turn causes large changes in suction.

Relative humidity-controlled method for controlling the suction is suitable for bentonites because this material presents large suctions. The jars method seems suitable when the samples are powder form. The method is also suitable for compacted samples if free swelling is allowed.

The suction controlled oedometer test forcing convection along the boundaries is suitable for compacted samples when measuring sorption paths (wetting paths). In desorption paths (drying paths), this method cannot be applied due to the volume cannot be kept constant due to the shrinkage. Nevertheless, it can be used as a free volume change test.

5.1.3 Osmotic technique

Osmotic method controls the suction adding a solute which decreases the water potential. The water with solute is in contact with the sample and there is a membrane which allows the water flow but retains the molecules of the solute, so if the soil potential (suction) is smaller than the water potential, water will flow from the soil to the water and if the soil potential is larger than in solution, water will flow from the solution to the soil.

The solute used in the method should have large molecules. Thus, a solute which has been used is the polyethylene glycol (PEG) with different molecular weights. The suction as function of concentration of the polyethylene glycol is presented in Figure 5-5.

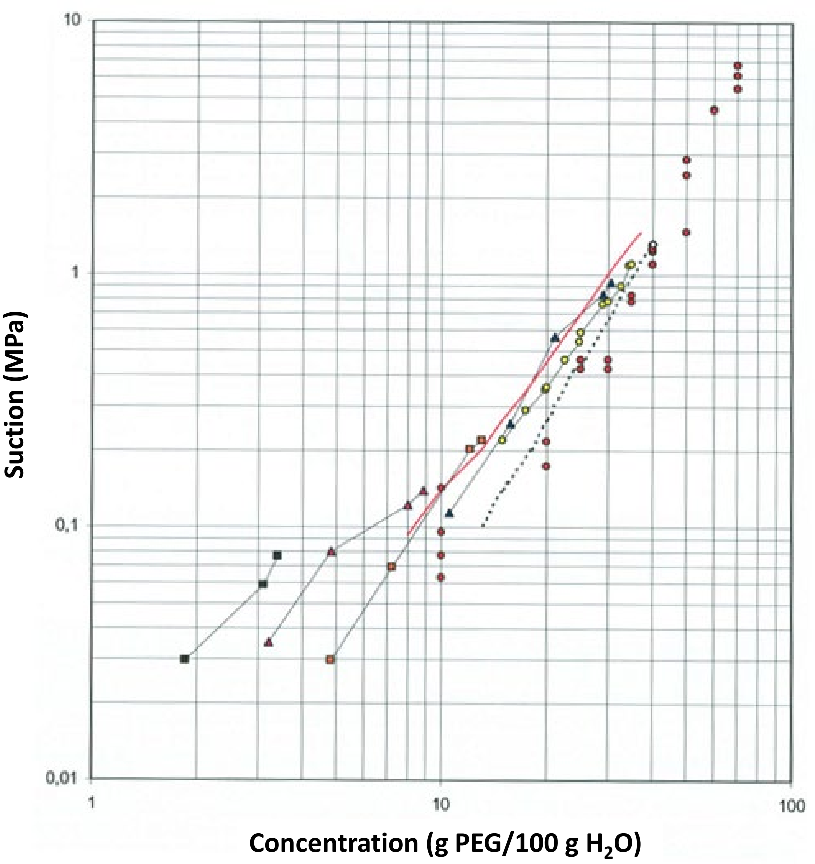

Figure 5-5. Suction vs concentration of polyethylene glycol (PEG 20000) in water (García Rodríguez 2006).

Like the relative humidity control method, it is possible to perform the tests in powder or in compacted samples. In powder samples or tests under free swelling or in drying paths (shrinkage), the tests can be carried out in the device showed in Figure 5-6.

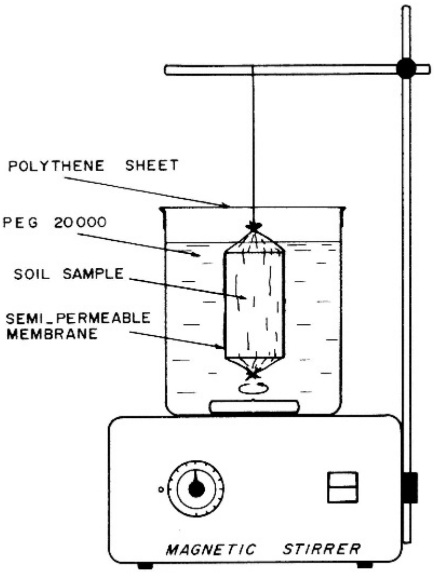

Figure 5-6. Use of osmotic technique for determination of the water retention properties of soil (Delage et al. 1998).

The test can also be performed keeping the volume of the sample constant or in oedometric conditions (Figure 5-7 and Figure 5-8).

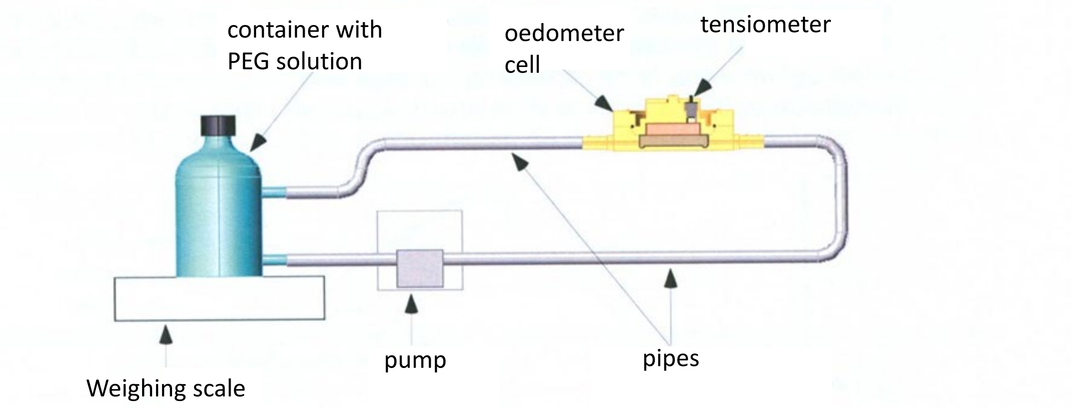

Figure 5-7. Osmotic suction controlled oedometer test (García Rodríguez 2006).

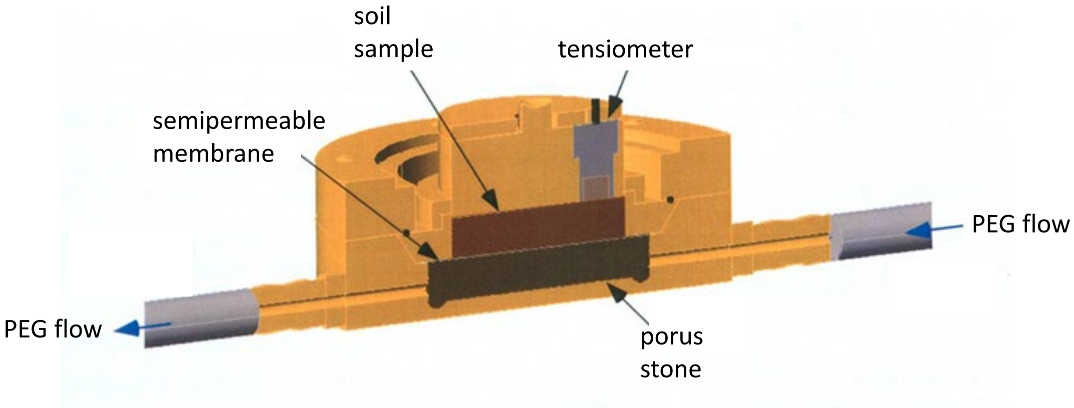

Figure 5-8. Cell in the osmotic suction controlled oedometer test (García Rodríguez 2006).

The osmotic suction has also used in the measurement of the water retention curve in bentonites (Yigzaw et al. 2016). The maximum range is up 10 MPa. This method could be interesting for measuring the last part of the water retention curve, when the suction drops from values close to the air entry value to saturation. The osmotic technique controls the matric suction, like the axis translation technique.

The osmotic suction and the axis translation techniques moist the samples with water that is in liquid phase and the relative humidity-controlled technique with water that is in gas phase. Yigzaw et al. (2016) have carried out tests using the osmotic and the relative humidity-controlled techniques showing that the differences in results are not only due to the differences between matric suction and total suction but also due to the different phase of water during the moist process.

5.2 Suction measurement techniques

The suction can be measured with different methods depending on the range of suction to be measured and the place where the suction will be measured ("in situ" measurements in the field or measurements at the laboratory conditions). The size of the sensor is important as well because there are sensors that are quite large which cannot be used at the laboratory unless the sample is large enough.

The sensors measure the suction indirectly except the tensiometer, which measures the water tensile stress directly. The sensors for measuring the suction are described below.

5.2.1 Heat dissipation sensor

The heat dissipation sensors have a cylindrical ceramic matrix which water absorbing characteristics of the matrix is similar to coarse soils like silts and sands. The suction (matric suction) is measured indirectly by measuring the temperature before and after giving a heat pulse through a resistor inside the ceramic matrix. The heat dissipation depends on the thermal conductivity and heat capacity of the ceramic matrix, which is related with the water content and this water content is related with the ceramic matrix suction, in equilibrium with the soil suction. These sensors must be calibrated individually because the heat dissipation depends on the joints and the size of the thermocouple and heat source host. This sensor can measure the suction in a range between 10 and 2500 kPa. The size of the sensor is 15×60 mm (Ø×l). The sensor is quite robust, so it is a good sensor for "in situ" tests but the range is too small for using in bentonites.

Figure 5-9 and Table 5-1 present the heat dissipation sensor CS229 from Campbell Scientific, INC.

Figure 5-9. CS229 sensor (suction measurement).

Table 5-1. Characteristics of the CS229 sensor (suction measurement).

Model	229 (Campbell Scientific Inc.)
Range	10-2500 kPa
Resolution	~1 kPa at matric suctions greater than 100 kPa
Temperature range	-5°C to +30 °C operating temperature range -40°C to +70 °C normal environmental temperature range
Sensor dimensions	60×15 mm l×Ø

5.2.2 Equitensiometer

The equitensiometer is a sensor which measures volumetric water content of a probe made in sintered material whose water retention curve is well known. The sensor measures the volumetric water content of the sintered probe with a frequency domain reflectometry (FDR) sensor. This sensor can measure the suction in a range of 0 to 1000 kPa. The size of the sensor is 40.5×181 mm (Ø×l). The sensor is also quite robust but too large, so it is a good sensor for "in situ" tests but the range is also too small.

Figure 5-10 and Table 5-2 present the equitensiometer sensor from Delta-T Devices Ltd.

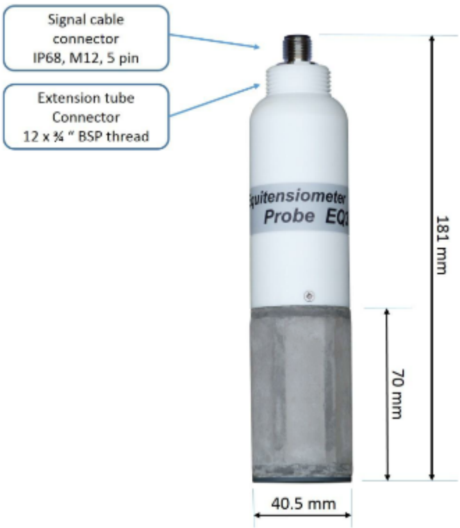

Figure 5-10. EQ3 sensor. Dimensions and connections.

Table 5-2. Characteristics of the EQ3 sensor (suction measurement).

Model	EQ3 (Delta-T Devices)
Range	0-1000 kPa
Accuracy	±10 kPa over 0 to 100 kPa 10 % reading over 100 to 1000 kPa
Temperature range	0 °C to +40 °C
Power	5 to 14 V DC,~18 mA for 1 s
Output	0-1 V DC
Sensor dimensions	181×40.5 mm l×Ø
Cable connection	5 pin IP68

5.2.3 Tensiometer

The tensiometer measures directly the suction of the soil measuring the tensile (capillary) pore water pressure. The tensiometers measured suctions up to 100 kPa but Ridley and Burland (1993) developed a high range tensiometer, which is able to measure suctions up to 1500 kPa. Recent developments allow to reach 2000 kPa (Lourenço et al. 2008) but this range is still far from the suction range required in bentonites. The tensiometers are not as robust as the previous sensors presented due to the risk of drying when the soil is in contact with the tensiometer that has large suction. The tensiometers are suitable for laboratory tests.

5.2.4 Hygrometers

The hygrometer is a device for measuring the amount of water vapour in the air or soil. Usually, the hygrometers provide the relative humidity (RH).

Capacitive hygrometer

The capacitive hygrometers measure the relative humidity measuring the variation of the capacitance of a capacitor. The capacitor has a polymer whose capacitance is function of the relative humidity. The capacitive hygrometer works well at low ranges of relative humidity (0-95% RH or 620 to 6.9 MPa of suction). The size of the sensor is 12×87 mm (Ø×l).

The capacitive hygrometer can be used in "in situ" tests and in laboratory tests. It is possible to connect the sensor to a datalogger for recording the relative humidity continuously. There are systems that allows to do the datalogging with wireless transmitters.

Table **5-3** present the capacity hygrometer manufactured by Sensiron. These sensors should be retrofitted (Figure 5-11). Amberg Infraestructuras S.A. can perform this retrofitting.

Table 5-3. Characteristics of STH85 Sensiron.

Model	Sensiron STH85
Measurement principle	Capacitive
Relative humidity range	0 % to 100% R.H. (not condensing)
Temperature range	-40 ºC to 105 ºC (adapting the connector, it can work up 125 °C)
Output	Digital 16 bit
Resolution	0.01% R.H. /0.01 ºC
Accuracy	±1,5% RH between 10 to 90%/ ±0,3 ºC
Dimensions	Diameter: 12 mm; Length: 87 mm (retrofitted)
Head casing material	Stainless steel 316L with steel filter (retrofitted)
Cable	RT47. Teflon jacketed. Screen threaded cable (retrofitted)
Nº of wires	4 × 0.3 mm^2, Teflon insulated (retrofitted)

Figure 5-11 presents the Sensiron STH75 V3 (previous version) retrofitted for carrying out the MPT "in situ" test (Pintado et al. 2018).

Figure 5-11. Capacitive hygrometer SHT75 V3.

Other capacitive hygrometers are the series HMT330 manufactured by Vaisala. The main advantage of the Sensiron hygrometer is the length of the cable, which can be up 100 m which is an advantage in "in situ" tests.

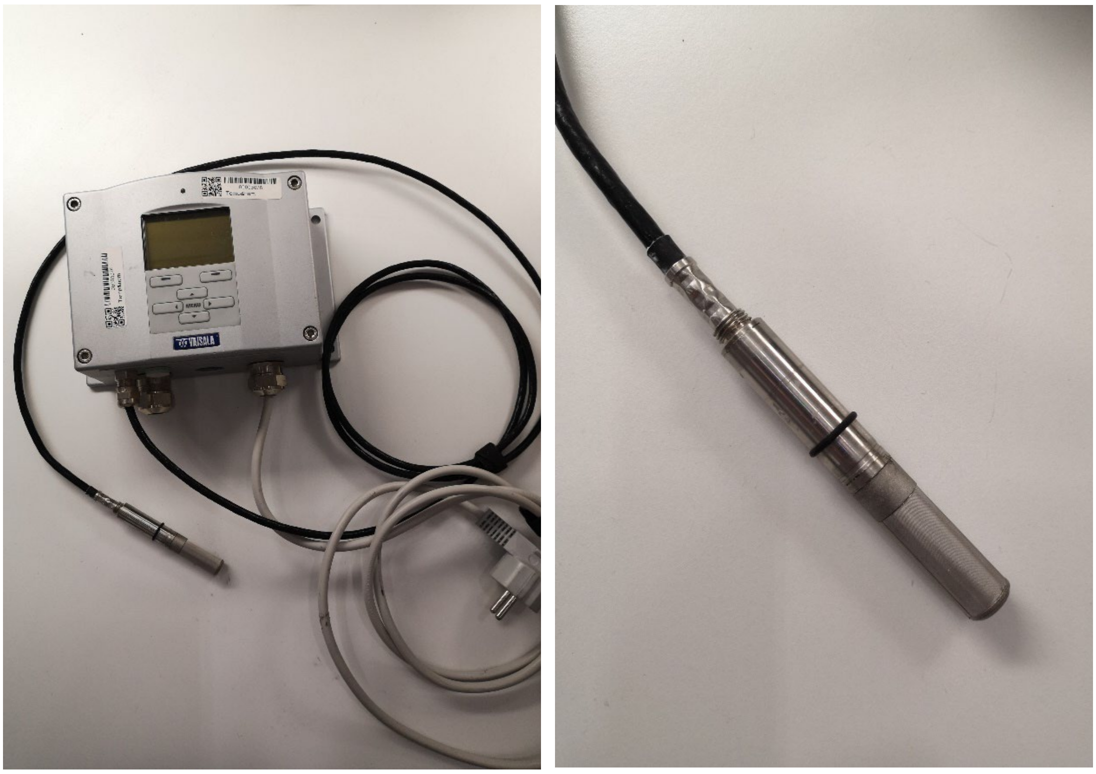

Figure 5-12. Capacitive hygrometer HMT330 (left) and the probe (right).

Figure 5-3 shows the measurement of the relative humidity evolution in a suction controlled oedometer test with a hygrometer. The measurement of the relative humidity with a hygrometer can also be done at the end of the test to compare the result with the suction calculated with the psychrometric law measured with a psychrometer. Note that this cell can also be used for measuring the water retention curve. With an accurate calibration, the measurement range can be extended and the accuracy increased (Oldecop and Alonso 2004).

Micro sensors

There are sensors with small dimensions (e.g. 18×6 mm, Ø×length) that allow to measure the relative humidity and the temperature. The measurements are recorded in a memory unit inside the sensor which should be removed from the soil. This type of sensors were not developed for bentonites, so they are brittle and should be protected with a casing for avoiding damage. This sensor can be used in the laboratory and in "in situ" tests.

Figure 5-13 and Table 5-4 present the micro sensor iButton manufactured by Measurement Systems Ltd.

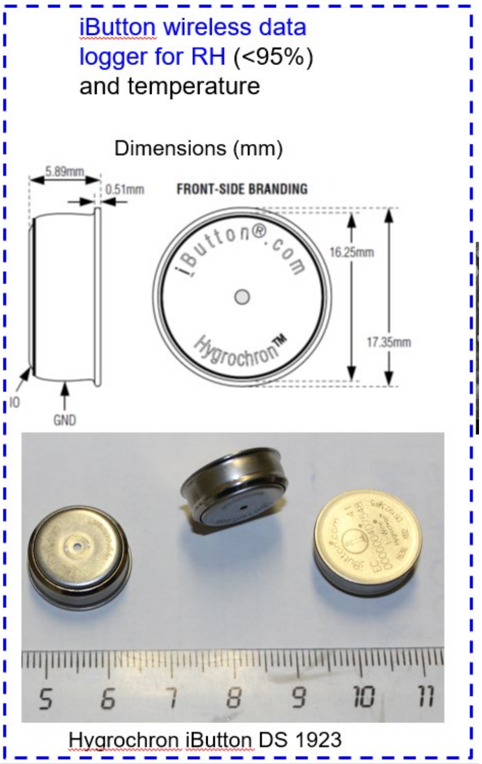

Figure 5-13. Wireless micro sensor for measuring temperature and relative humidity.

Table 5-4. Characteristics of the iButton sensor.

Model	iButton DS1923-F5# Hygrochron
Relative humidity range	0 % to 100% R.H.
Temperature range	-20 ºC to 84ºC
Output	Inner datalogger
Logging rate	1 sec to 273 hours (total readings: 8192 8 bit)
Dimensions	Diameter: 17.35 mm; Height: 5.89 mm
Head casing material	Stainless steel 316L with steel filter

5.2.5 Psychrometers

The psychrometer is defined as a hygrometer consisting of a wet-bulb and dry-bulb thermometers. The difference between both thermometers gives the relative humidity. The ASTM E337-15 is a standard for measuring the relative humidity with a psychrometer. The psychrometers are complex sensors and the supplier delivers them with proper instructions and if it is required, the sensors can be supplied with calibration certificates.

Psychrometer of transistors

The psychrometer of transistors consists of two bulbs, one is the wet bulb-bulb thermometer and the second is the dry-bulb thermometer. Both thermometers are inside a sealed chamber where the soil sample is also assembled. A drop of distilled water is used for wetting the wet-bulb. The time needed for performing the measurement is one hour.

Figure 5-14, Figure 5-15 and Table 5-5 present the psychrometer of transistors manufactured by Soil Mechanics Instrumentation (Australia).

Figure 5-14. Sample and container in psychrometer of transistors (in left) and preparation of the sensor adding a tear of pure water (in right) (Hoffmann 2005).

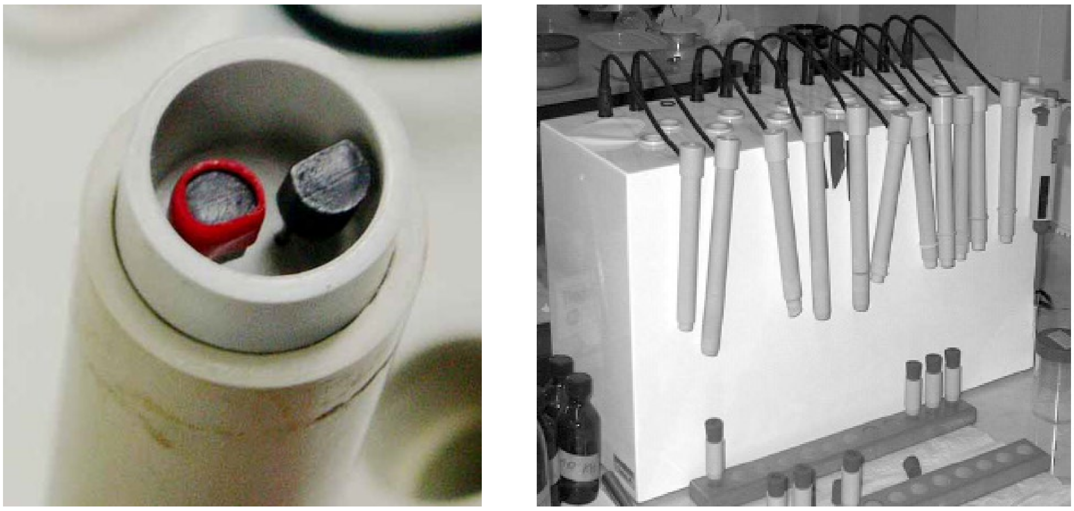

Figure 5-15. Thermocouples configuration (in left, Hoffmann 2005) and equipment with 12 channels (in right, Cardoso et al. 2007).

Table 5-5. Characteristics of the SMI psychrometer.

Model	SMI transistor psychrometer
Suction range	0-70 MPa
Output	Voltage
Accuracy	<±0.05 pF(1), ±0.01 pF (repeatability)
Dimensions of the sample	Diameter: 15 mm; Height: 12 mm

(1) pF: log(-water column in cm)

Thermocouple psychrometer

The thermocouple psychrometers can be used for measuring the relative humidity in a range of 95-99.96%, equivalent to a suction between 6.9 MPa to 50 kPa. In thermocouple psychrometry, the temperature depression of the sensing (wet) junction that is measured relative to the reference (dry) junction varies as a function of the relative humidity of air surrounding the sensing junction. A thermocouple is a double junction of two dissimilar metals. When the two junctions are subject to different temperatures, they generate a voltage difference (Seebeck effect). On junction of the thermocouple is suspended in a thin-walled porous ceramic or stainless screen cup in contact with the material, while another is embedded in an insulated plug to measure the ambient temperature at the same location.

This type of psychrometer is interesting since it can be used for measuring the suction in low ranges in "in situ" tests because the sensor can condensate water through a current, when the wet-bulb is created and the measurements can be recorded by a datalogger. The dry-bulb measures the temperature of the soil environment, so the difference between both temperatures gives the relative humidity. Smaller relative humidity's than 95% do not allow the condensation of water, so the wet-bulb cannot be created.

This type of thermocouple can also be used in the laboratory. The psychrometer of transistors requires to assemble the soil inside a sealed chamber, the thermocouple psychrometer has a probe which can be in contact with the soil samples. The size of the sensor is 12×87 mm (Ø×l).

Figure 5-16 presents the probe of the WESCOR type PST-55 psychrometer. Main characteristics of this psychrometer are listed in Table 5-6.

Figure 5-16. Configuration of thermocouple psychrometer (in left) and WESCOR type PST-55 sensor retrofitted to be used in "in situ" tests (in right).

Table 5-6. Characteristics of WESCOR type PST-55 psychrometer.

Model	WESCOR type PST-55 retrofitted
Temperature range	-200/350 °C
Humidity range	95 % RH to 99,96 % RH
Suction range	50 to 6900 kPa
Output signal	Two analogue outputs (µV). 0,47 µV/bar
Probe material	Stainless steel 316L with steel filter (retrofitted for "in situ" tests)
Cable connection	Welded and epoxy sealed
Nº of wires	1 cable with 3 conductors, PVC insulation
Dimensions	Diameter: 12 mm; Length: 87 mm

Amberg Infraestructuras S.A. can carry out the retrofitting and provide similar sensors. Besides, a new hardware based on the dew point method was developed in the EU project Modern2020 that provides a more accurate and stable measure.

This sensor can also be used for measuring the suction in a cell (Figure 5-3).

Chilled mirror dew point psychrometer

The chilled mirror dew point psychrometer measures the dew point temperature or condensation temperature. The sample is inside a chamber where there are a mirror and a detector that can detect the condensation on the mirror. The temperature of the mirror is measured by a thermoelectric (Peltier) cooler. The sensing of the condensation point is done with a photoelectric cell, which detects the presence of condensed water on the mirror through the reduction in the mirror's reflection. The condensation temperature is measured by a thermocouple attached to the mirror. The relative humidity is computed from the difference between the dew point temperature of the air and the temperature of the soil sample, which is measured with an infrared thermometer. A fan is included in the sealed compartment to speed up the equalisation period between the sample and the surrounding air. The time for performing the measurement is around five minutes. The ASTM D4230-20 describes the determination of the dew point temperature of ambient air by the condensation of water vapour on a cooled surface. This standard is not very useful due to the chilled mirror dew point psychrometers are complex devices which are supplied with detailed instructions for carrying out the measurements and also for carrying out the calibration. This is a recommended method for measuring the suction at the laboratory, it can be used with a large range of suctions and the measurement can be done in a few minutes.

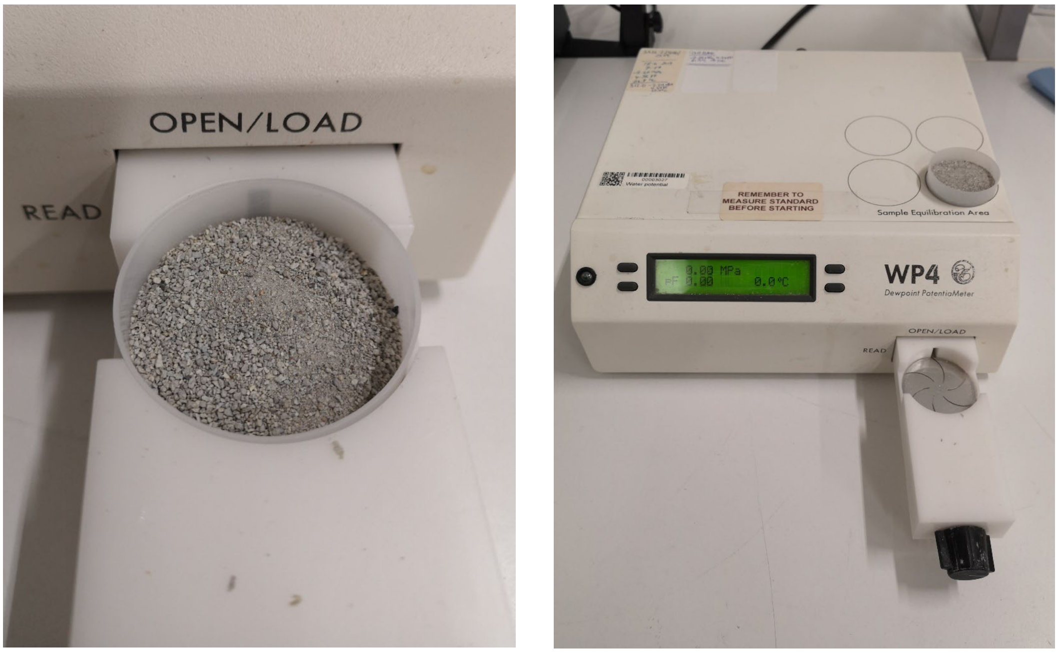

Figure 5-17. Sample (in left) and chilled mirror dew point psychrometer manufactured by Meter Environment (former Decagon Devices, Inc.) (in right).

Table 5-7. Characteristics of the Decagon chilled mirror dew point psychrometer.

Model	Chilled mirror dew point psychrometer WP4C
Suction range	0-300 MPa
Output	Suction in MPa and pF and temperature in Celsius
Accuracy	±0.1 MPa from 0 to 10 MPa and ±1% from 10 to 300 MPa
Dimensions of the sample	Diameter: 37 mm; Height: 7 mm

Cardoso et al. (2007) have done a study where the psychrometer of transistors is compared with the chilled mirror dew point psychrometer.

5.2.6 Vapour sorption analyser

The vapour sorption analyser (VSA) is an alternative for measuring the water retention curve in isothermal conditions. The device is described in http://www.metergroup.com/food/products/vsa. Basically, VSA is a climate chamber where the relative humidity can be varied between 3 and 95% (equivalent to a range of suctions between 483-7.1 MPa at 25 °C) and the temperature between 15-60 °C. Figure 5-18 shows the device and how it works.

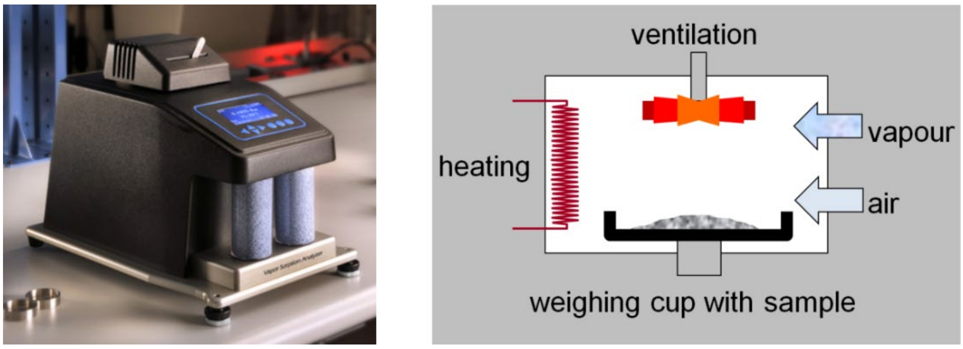

Figure 5-18. Image and working principle of the VSA (Kröhn and Kröhn 2020).

The VSA has been used by Kröhn and Kröhn (2020) for measuring the water retention curve in MX-80 bentonite (a Wyoming bentonite). The device is still under research although it presents interesting capabilities like promptness and continuous measurement of the sample weight carrying out the test with the same sample. It avoids using many desiccators with different salt solutions for controlling the relative humidity, providing a large amount of datapoints at different suctions. The main disadvantage is the capacity of the balance, which is very small, just 5 g.

5.2.7 Filter paper method

The filter paper method measures the total suction (if the filter paper is not in contact with the soil) or the matric suction (if it is in contact with the soil) measuring the water content of a piece of filter paper once it has reached the equilibrium with the relative humidity around the soil (total suction) or with the water in the pores of the soil (matric suction). This method allows to measure the suction between 0.01 and 100 MPa and it is described in the ASTM D5298-16.

The filter paper method was quite common in past because the psychrometers and hygrometers were not developed as nowadays but although the test is easy to do and it does not require specific equipment, it is not recommended because it requires a long period of time for reaching the equilibrium between the environment of the sample or the sample and the filter paper (some days). It should also be taken into account that the filter paper is not provided for suction measurements, it is just for filtering, so it would not be homogeneous enough for carrying out the tests and finally, the paper in contact with the soil might be contaminated with small particles of soil although the measurement paper is protected with other two papers, as a sandwich. Another important issue to consider is the calibration of the filter paper. The ASTM D5298-16 provides the relation between the suction and the water content of some filter papers but double checking the calibration is quite complex due to the small weight of the pieces of paper, which would dry or wet during the process of weighting for measuring the water content after the equilibrium with a saturated salt solutions in a desiccator or jar, following the SS-EN ISO 12571:2021. The filter paper calibration process is equivalent to obtain the filter paper water retention curve.

The Appendix 1 presents a detailed study about the filter paper method. More information can be found in Delage et al. (2008) and Muñoz-Castelblanco et al. (2010) and specifically related with the filter paper method and MX-80 bentonite, in Kuusela-Lahtinen et al. (2016).

5.3 Powder and compacted samples

The water retention curve in models is expressed as a relation between the degree of saturation and suction (Olivella et al. 2021). This relation depends on the temperature and the dry density (Villar 2002, Tang 2005, Seiphoori 2014). For the same suction, the larger dry density and lower temperature, the larger degree of saturation. The calculation of the degree of saturation is an issue due to the uncertainty in the measurement of the dry density and the water content. Controlling the density in small samples is not easy and there is also a certain dispersion at the same series because the dry density is not constant.

The water retention curve obtained in compacted samples at constant volume for different dry densities at laboratory conditions and the water retention curve obtained by Dueck and Nilsson (2010) and in this work in powder samples are presented in Figure 5-19 (Dueck 2004, Villar 2005, 2007, Tang 2005, Pintado et al. 2013, Seiphoori 2014, Kiviranta et al. 2018).

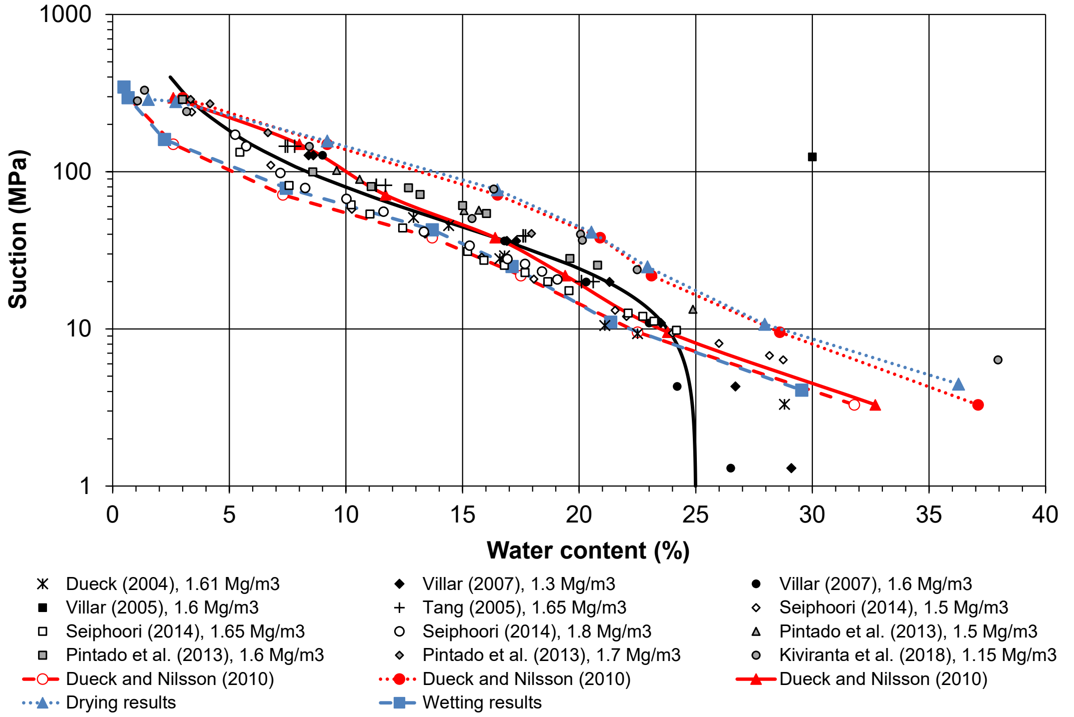

Figure 5-19. Water retention curve in compacted samples at constant volume and in powder samples. Black line represents the van Genuchten water retention curve.

There is a certain dispersion in the results obtained in samples at constant volume. The method for controlling the suction was the air convection (Figure 5-2) in some cases. This method is faster but creates gradients in the air pressure and the temperature of the air might be disturbed by the pump, which warms up during the test.

The water content is the sum of the water that fills partially the interaggregate voids (“macrostructural water”) and the water stored in the intra-aggregate voids (“microstructural water”). The capillary contribution from the water held in the macropores controls in the wetter range of the water retention curve and the adsorptive contributions control the dryer range. This means that the water retention curve keeps constant after certain value of the suction because the capillary contributions become negligible. Dueck (2004) shows this behaviour in MX-80 bentonite. The threshold might be fixed at 25 MPa and 30 MPa in MX-80. More information about this issue can be found in Navarro et al. (2015).

5.4 Conclusions

Some different methods for suction control and suction measurement are available. The relative humidity-controlled technique is the most suitable suction control method for bentonites because it covers the expected range of suctions. In addition, it can be complemented with the osmotic or the axis translation techniques for measuring the transition from suctions close to the air entry value to saturation. This transition can also be extrapolated from water retention curves (e.g. van Genuchten 1980).

The measurement of the water retention curve in powder is interesting because the method is well described in standards and there is previous experience using this method in bentonite. The measurement is easy to conduct and does not require special devices. The measurements show a repeatability which was not observed in tests carried out in compacted samples under constant volume conditions because the control of the dry density is quite difficult in small samples.

The measurement of the suction in powder provides information about the behaviour of the microstructure of the bentonite but not about the macrostructure, which should be obtained in compacted samples tested at constant volume condition, but the measurements of suction in powder would be useful in quality control in order to check that this parameter does not change between batches.

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6 Appendix 1

Implementation of test to determine total and matric suction with the filter paper method

6.1 Introduction

Currently determination of suction at Mitta Oy laboratory is done with chilled mirror dewpoint psychrometer (Decagon WP4) and capacitive hygrometer (HMT337 from Vaisala). A third option for determination of total suction between 0.01 and 100 MPa would be a filter paper method (e.g. ASTM D5298-10, Muñoz-Castelblanco et al. 2010) allowing not just determination of total suction but also matric suction. This technical memo includes description of implementation of the technique at the Mitta Oy laboratory. More information about suction measurement can be found in Delage et al. (2008) and specifically related with the filter paper method and MX-80 bentonite, in Kuusela-Lahtinen et al. (2016).

6.2 Method description

The filter paper method is very sensitive to errors in weighting and temperature fluctuations. Thus, only precision scale (with accuracy of 0.0001 g) was used, and care was taken that all vessels and tools were dust and moisture free, handled only with tweezers, and wearing disposable gloves in every step. Further ambient temperature of the laboratory was logged during the tests.

The following method description covers both measuring the calibration curve of the filter paper and performing a measurement on a clay sample, as the protocol is identical in both cases. Only the sample differs; the measurement of total suction from calibration sample (i.e. filter paper) is done above aqueous solution of known vapor pressure and the measurement of total suction from soil sample is done above the soil.

Calibration of filter papers can be done using saturated salt solutions as reported by Lide (1998, Table 6-1) or by unsaturated NaCl solutions (Horvath 1985, Romero 1999, Table 6-2).

Table 6-1. Examples of saturated salt solutions that can be used for calibration of the filter paper (Lide 1998).

Salt	Solubility in water at 25 °C [g/100 g water]	RH 25 °C [%]	Suction 25 °C [MPa]
KCl	35.5	84	23.9
$(NH_4)_2SO_4$	76.4	81	28.9
NaCl	36.0	75	39.4
NaBr	94.6	58	74.7
$CaCl_2$	81.3	29	169.7
NaOH	100.0	6	385.8

In this study the filter paper calibration was tested with two calibration samples (i.e. filter paper) using known relative humidity's imposed with unsaturated aqueous NaCl solutions (Table 6-2). The calibration was performed on two different filter papers from the same batch for each chosen NaCl solution. The results were compared to the calibration curve presented in ASTM D5298-10 for Whatman grade 42 to validate that the curve presented in the standard can be used with the filter paper available at Mitta Oy laboratory.

The relative humidity imposed with unsaturated solutions of NaCl for relatively low range of suctions (Horvath 1985, Romero 1999) was calculated as:

$$R_h(m,T) = 1 - 0.035m - m(m-3)(1.9772 * 10^{-3} - 1.193 * 10^{-5}T) \qquad (6\text{-}1)$$

Where m = molality of salt solution, and
T = temperature (°C).

This expression is valid for m>3 mol/kg. If the molality is less than 3 mol/kg, the temperature effect is negligible and the expression is:

$$R_h(m,T) = 1 - 0.035m - (1.9772 * 10^{-3})m(m-3) \tag{6-2}$$

The suction was calculated according to:

$$h = -\frac{RT}{v}\ln R_h \tag{6-3}$$

Where h = suction (kPa),
R = ideal gas constant (8.314472 J/(mol·K)),
T = absolute temperature (K),
v = volume of 1000 moles of liquid water (0.018 m^3), and
R_h = relative humidity expressed as a fraction.

Table 6-2. Examples of unsaturated NaCl solutions than can be used for calibration of filter paper (Horvath 1985, Romero 1999). The ones tested in this study are highlighted.

Concentration (NaCl g/kg H_2O)	RH 25 °C [%]	Suction 25 °C [MPa]
10	99.5	0.7
25	98.7	1.8
50	97.3	3.7
60	96.7	4.5
100	94.4	7.8
150	**91.2**	**12.5**
200	**87.8**	**17.6**
250	84.1	23.4
300	80.2	29.9

The calibration solutions were prepared by heating (to 60–70 °C) and stirring the solutions for 24 hours. The relative humidity above the prepared calibration solution was checked with a psychrometer (Decagon WP4).

Filter papers were dried in an oven at 110 oC for a minimum of 24 h prior to use, and then placed in a desiccant jar for storage until used.

The relation between water content of filter paper and suction presented in ASTM D5298-10 was first checked. Two filter papers of 55 mm in diameter previously dried at 110 oC were emplaced above an inert platform made of plastic, which was placed above selected salt solution (Figure 6-1a) in a glass container.

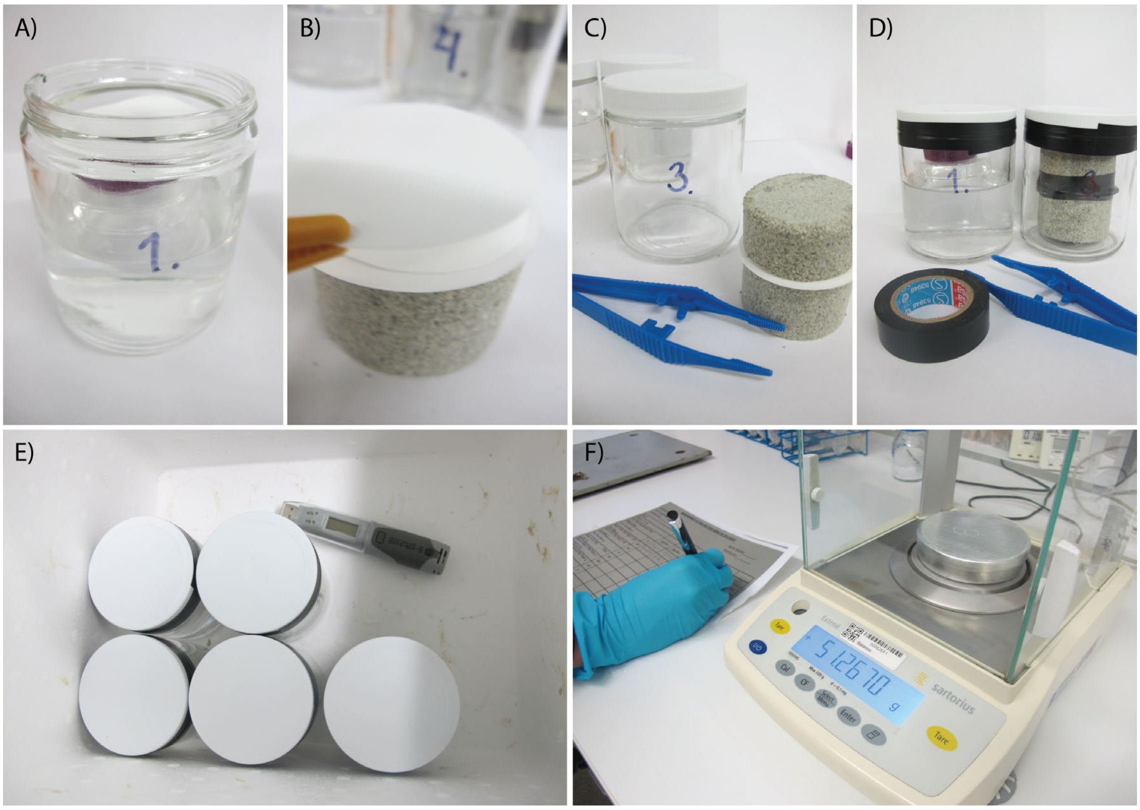

Figure 6-1. Sample set-ups for measurement of calibration sample (i.e. filter paper) and matric suction, and total suction of soil sample (upper row), and from actual measurement (lower row).

To measure the total suction in soil samples, one filter paper was placed on top of the soil sample, separated from it by a small sharp edged plastic lid with a minimum surface area, which was used as the inert platform for supporting filter papers above clay samples in order to avoid any contact between filter paper and soil sample. One edge of the filter paper was slightly bent with clean tweezers in order to hasten removal of the filter paper after equilibration.

To measure the matric suction, one filter paper was placed between two other filter papers (all previously dried at 110 oC), forming a sandwich. The middle filter paper was cut to a smaller diameter than the sample diameter, and smaller than the two outer filter papers in order to prevent contamination of the middle filter paper (Figure 6-1b). All three filter papers were placed in between two clay blocks so that the two outer filter papers became in contact with the clay blocks (Figure 6-1c). The top and bottom parts of the sample were joint and the filter papers therein was sealed with an electrical tape (Figure 6-1d).

Then glass containers were sealed quickly with lids and a minimum of one layer of electrical tape was wrapped around the lid to ensure it was airtight. The containers were then placed into an insulated chest (Figure 6-1e) with temperature variations less than ± 1 °C. The temperature variations were measured during the equilibrium period with a thermometer. The samples were let to equilibrate for a minimum of 7 days (but less than 14 days).

Before taking out the glass containers one by one from insulated chest, metal containers were weighted (Tc) carefully (with accuracy of 0.0001 g) one by one. The glass container was opened and the filter paper was transferred (using tweezers) to a pre-weighted container, and metal container was sealed with lid. This was done as quickly as possible (within three seconds) to avoid evaporation of moisture from the filter paper. The metal container with the filter paper was then weighted (M1).

Metal container was then placed into a pre-heated oven (110 °C) and unsealed (i.e. the lid was slightly ajar) in order to let moisture escape. The metal container was kept in the oven for 2-4 hours. Before the metal container was taken out from the oven, the container was sealed with a lid left in the oven for an additional 15 minutes (minimum) for temperature equilibration. Then, the metal container was removed from the oven and placed on a metal object (heat sink) for approximately 30 seconds. The mass of hot metal container with the filter paper (M2) was then weighted (Figure 6-1f). Immediately afterwards, the filter paper was removed from the metal container with the tweezers, and the mass of empty hot metal container (Th) was weighted.

After masses M2 and Th were determined, the filter paper was discarded. The mass of dry filter paper and mass of water in filter paper were calculated as:

$$M_f = M_2 - T_h \tag{6-4}$$

and

$$M_w = M_1 - M_2 + T_h - T_c \tag{6-5}$$

Where M_f = mass of dry filter paper (g)

M_2 = dry hot total (container + dry filter paper) mass (g)

M_1 = cold total (container + 'wet' filter paper) mass (g)

T_h = mass of the hot container (g)

M_w = mass of water in the filter paper (g), and

T_c = mass of the cold container (g).

Then the water content of filter paper was calculated as:

$$w_f = 100\frac{M_w}{M_f} \tag{6-6}$$

Where w_f = ratio of water to dry mass (%).

The water content of the filter paper was converted to the suction, h, of the sample by referring to the calibration curve shown in ASTM D5298 – 10 (Figure 6-1). The suction could also be determined using a calibration curve constructed from results of 6 filter papers imposed to different suctions (e.g. as in Table 6-1, or Table 6-2), and calculated from:

$$h = mw_f + b \tag{6-7}$$

Where m = slope of filter paper calibration curve ($\log_{10}$ kPa / w_f),

b = intercept of filter paper calibration curve ($\log_{10}$ kPa).

The osmotic suction was calculated after measurements of the total suction and the matric suction according to:

$$h_O = h_T - h_M \tag{6-8}$$

Where h_O = osmotic suction (log kPa),

h_T = total suction (log kPa), and

h_M = matric suction (log kPa).

After taking out the filter paper, the soil sample used for measurement of suction was sliced to 12 pieces. One third of the pieces were used to determine the water content of the soil sample (gravimetrically at 105 °C for 24 h), one third of the samples were used to measure the total suction with the psychrometer, and one third of the pieces were used to measure the density of the sample (with paraffin method).

Water content (w) was determined as follows (see details in Kiviranta et al. 2018):

$$w = \frac{m_w}{m_s} * 100\% \tag{6-9}$$

Where m_w = mass of water (g), and

m_s = mass of dry material (g).

Bulk density (ρ_b) was determined as follows (see details in Kiviranta et al. 2018):

$$\rho_b = \frac{m_{air}}{m_{air} - m_{fluid}} * \rho_{fluid} \tag{6-10}$$

Where m_{air} = mass of the specimen in air (g)

m_{fluid} = average mass of the specimen in fluid over 7 mass readings (g), and

ρ_{fluid} = density of the immersion liquid (g/cm^3)

Dry density was calculated from the measured values with following equation:

$$\rho_d = \frac{\rho_b}{1+w} \tag{6-11}$$

Where ρ_d = dry density (g/cm^3),

ρ_b = bulk density (g/cm^3), and

w = water content

Degree of saturation was calculated from the measured values with following equation:

$$S_r = \frac{w * \rho_b * \rho_s}{(\rho_s(1+w) - \rho_b) * \rho_w} \tag{6-12}$$

Where ρ_b = bulk density (g/cm^3),

ρ_s = grain density (g/cm^3),

ρ_w = density of water (g/cm^3), and

w = water content

6.3 Samples and sample preparation

The test matrix consisted of two calibration samples (in duplicate) to test if the calibration line defined in ASTM D5298-10 could be used with the filter paper available at Mitta laboratory, and of three compacted soil samples (from which one was measured as a duplicate) saturated with different salinity solutions (deionized water and 10 g/L with Na^+:Ca^{2+} 2:1). From soil samples both total suction and matric suctions were measured. The test matrix is presented in Table 6-3.

Material: Wyoming bentonite

Grain density: 2780 kg/cm^3

Target dry density: 1600 kg/m^3

Dimensions: Diameter 50 mm, height approximately 55 mm

The total suction expected (shown in Table 6-3) was calculated from the water retention curve for MX-80 presented in Pintado et al. (2017) for the reference case. More information about the results in MX-80 can be found in Kuusela-Lahtinen et al. (2016).

Table 6-3. Test matrix for samples testing the filter paper method.

Test	**Calibration sample/ soil sample**	**Water content [%]**	**Degree of saturation [%]**	**Saturating solution**	**Total suction expected [MPa]**	**Solution exposed**	**RH calculated [%] 25 °C**	**Expected suction 25 °C [MPa]**
1*	calibration	-	-	-	-	150 NaCl g/kg H_2O	91.2	12.5
2*	calibration	-	-	-	-	200 NaCl g/kg H_2O	87.8	17.6
3	soil	17.0	63.9	deionized water	36.2	-	-	-
4	soil (duplicate)	17.0	63.9	deionized water	36.2	-	-	-
5	soil	17.0	63.9	10 g/L**	35.8	-	-	-

** Average of two measurements from separate filter papers.*

*** 10 g/L Posiva reference solution with Na^+:Ca^{2+} 2:1.*

To assess whether the method was successful, the measured suction values were compared to previously published suction values on the same material and a few parallel measurements using a psychrometer.

Sample preparation

Initial material was first dried at 105 °C. Sufficient amount of deionized water (or other saturating solution) was added to reach the target water contents. The sample was compacted to target dry density with hydraulic press, and then cut to half in order to insert filter papers inside the sample for measurement of matric suction.

6.4 Results and discussion

6.4.1 Calibration samples

The results for calibration samples (i.e. filter papers) are shown in Table 6-4.

Table 6-4. Results of filter paper test. Wf is water content of filter paper (measured gravimetrically).

Test	Subsample	Total suction/ matric suction	W_f [%]	Suction filter paper [MPa]**	Average suction filter paper [MPa]**	Average suction of solution psychrometer [MPa]	Expected suction [MPa]
1	a	total	12.3	23.4	25.8	15.0*	12.5
	b	total	11.3	28.2			
2	a	total	10.4	32.6	33.1	20.7*	17.6
	b	total	10.3	33.5			

** Measured from the NaCl solutions used to create the desired RH% atmosphere.*

*** Calibration curve defined in ASTM D5298-10 was used (i.e. $h = 5.327 - 0.0779 \cdot w_f$ where h is $\log_{10}$ of the suction [in kPa] and the unit of w_f is %).*

Comparison of suction in subsamples (a, b) is well within limit of good quality measurement (< 0.5 log kPa) (ASTM D5298-10).

However, suction measured with filter paper method from calibration samples was in general 11-12 MPa higher than measured with psychrometer from NaCl solutions (Table 6-4), and 13-15 MPa higher than calculated from equation 7-3 (see Table 6-3).

The difference in the expected and measured suction values may be caused by errors in preparing the salt solution, uncertainties in psychrometer measurements or uncertainties in filter paper measurements. Therefore, the preparation of salt solutions, and the measurement of corresponding suction with psychrometer were repeated, and in addition, the salt content was also checked afterwards by drying a known quantity of salt solution in an oven at 105 oC. Based on the results, it was concluded that the psychrometer results are reliable, and the more probable reason for the differences are mainly errors in preparing the salt solutions, and uncertainties in the filter paper method. Possible uncertainties in the filter paper method are caused by e.g. drying of filter paper during dismantling the test and weighing; too short equilibration period; or some other unidentified reason.

6.4.2 Soil samples

The results from post-mortem analyses are presented in Table 6-5, and from filter paper tests in Table 6-6.

Table 6-5. Postmortem results of soil samples. W$_{clay}$ is the water content of clay sample (measured gravimetrically)

Soil sample	Subsample	W$_{clay}$ (%)	Bulk density (g/cm^3)	Suction$_{\text{psychrometer}}$ (MPa)	T (°C)	Dry density (g/cm^3)	Degree of saturation	Total suction expected (MPa)*
3	Top 3.1	14.5	1.76	63.94	25.3	1.54	0.50	50.6
	Top 3.2	14.5	1.73	59.53	25.5	1.51	0.48	52.5
	Bot 3.1	14.5	1.77	54.93	25.5	1.54	0.50	50.6
	Bot 3.2	14.9	1.76	54.82	25.6	1.53	0.50	49.9
4	Top 4.1	14.7	1.70	58.28	25.6	1.48	0.47	52.9
	Top 4.2	14.7	1.76	54.12	27.7	1.53	0.50	49.9
	Bot 4.1	14.9	1.77	52.55	25.8	1.54	0.52	47.2
	Bot 4.2	14.7	1.82	54.58	25.8	1.59	0.54	47.9
5	Top 5.1	14.5	1.81	56.28	25.9	1.58	0.53	48.6
	Top 5.2	14.5	1.77	61.21	25.9	1.55	0.51	49.5
	Bot 5.1	14.5	1.79	59.57	26.0	1.57	0.52	49.4
	Bot 5.2	14.5	1.82	56.71	26.0	1.59	0.54	47.9

** Expected total suction was evaluated from the measured degrees of saturations and the water retention curve for MX-80 reference case presented in Pintado et al. (2017).*

Post-mortem analyses of clay after filter paper tests show that there appears to be small scatter in the results of water content of quadruplicate subsamples (std in average 0.1 %, i.e. 0.7 % error (std divided with average)), but somewhat larger scatter in the results of bulk density (std in average 0.03 g/cm^3, i.e. 2 % error (std divided with average)) and even larger for suction (std in average 3.0 MPa, i.e. 5 % error (std divided with average)) measured with the psychrometer from post-mortem samples. Measured suctions from post-mortem samples were 5-10 MPa higher than what was expected for these samples based on previous studies (Pintado et al. 2017).

The total suctions measured with the filter paper method were however rather close to the values evaluated based on previous data (Pintado et al. 2017) and measured degree of saturations (Table 6-6): in sample 3, the measured total suction was six MPa smaller than expected, in sample 4 three MPa higher than expected and in sample 5 one MPa higher than expected. Thus, it can be stated that the filter paper method appears to work and produce similar kind of results for total suction as expected.

However, there were some drawbacks in the implementation of the filter paper method. Soil subsamples measured post-mortem with psychrometer might have dried slightly before the measurement, meaning that the total suctions measured with the filter paper were more reliable than suctions measured with the psychrometer. Further, total suctions should be larger than matric suctions. In all the three tested samples the matric suction was 2-4 MPa larger than the total suction. This erroneous result might originate simply from the measurement uncertainty, but also from other reasons, e.g. due to improper contact of filter paper with the sample (e.g. the matric suction filter paper diameter was not properly in its place), or because the removal and weighting of the filter paper was not performed sufficiently fast during the measurement. In addition, after the tests were performed, it was also realized that at the conditions and levels of suction reached in these tests the osmotic suction is expected to be low and negligible, i.e. the total and matric suctions should have been almost equal. Also, the difference in ionic strength between the samples 4 (water content adjusted with deionized water to 17 %) and 5 (water content adjusted with 10 g/L (Na$^+$:Ca^{2+} 2:1) to 17 %) was thought to be so minimal that the possible suction difference got lost in the uncertainty of the method, which is in accordance with results by Villar (2005) as they also did not see any effect in measured total suction when the salt content of saturating solution was changed.

Table 6-6. Results of filter paper test. Wf is water content of filter paper, and Wclay is the water content of clay sample (measured gravimetrically).

Test	Subsample	Total suction/ matric suction	W_f [%]	Suction filter paper [MPa]**	Average suction expected [MPa]***	Average suction from postmortem subsamples psychrometer [MPa]*	Average W_{clay} [%]*	Degree of saturation*	Average dry density [g/cm^3]*
3	a	total	8.6	45.1	50.9	58.3	14.6	0.50	1.53
	b	matric	8.3	47.7					
4	a	total	7.8	52.2	49.5	54.9	14.7	0.51	1.54
	b	matric	7.4	56.1					
5	a	total	8.0	50.2	48.9	58.4	14.5	0.52	1.57
	b	matric	7.7	53.2					

** Measured from the post mortem quadruplicate subsamples of soil (presented in Table 6-5).*

*** Calibration curve defined in ASTM D5298-10 was used (i.e. $h = 5.327 - 0.0779 \cdot w_f$ where h is $\log_{10}$ of the suction [in kPa] and the unit of w_f is %).*

**** Expected total suction was evaluated from the measured degrees of saturations (Table 6-5) and the water retention curve presented in Pintado et al. (2017).*

6.4.3 Comparison to previous results

The measurements of this study were compared with results from other authors (Figure 6-2), and the comparison shows that the results of this study (filter paper method) were in line with the results of previous studies.

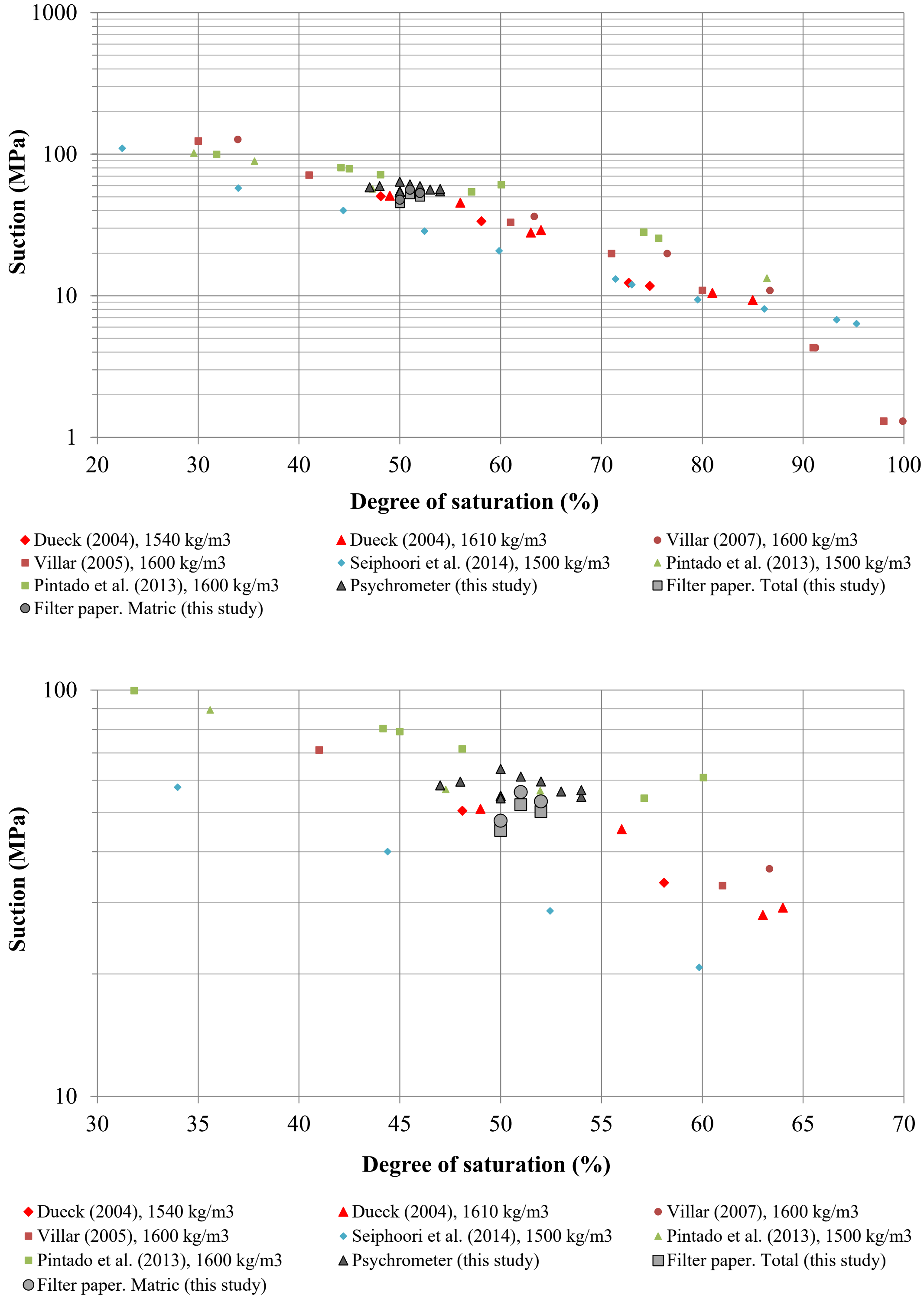

Figure 6-2. Suction measurements compared with results from various authors.

6.5 Conclusions

In this study, filter paper method for measuring suction was assessed. The main conclusions of this study were:

- The measured suctions of soil samples using the filter paper method were in line with the results of previous studies.
- Significant differences between matric and total suction were not detected, and after the measurements it was also concluded that the osmotic suction is expected to be low and negligible at the conditions and levels of suction reached in these tests, i.e. significant differences shouldn't have been detected at all, and the total and matric suctions should have been almost equal in all soil samples.
- The calibration measurements with filter paper were unfortunately not successful, i.e. the measured (psychrometer) suction caused by a salt solution and the suction determined from the water content of filter paper according to the calibration curve presented in ASTM D5298-10 did not correspond to each other. This could be caused by e.g. drying of the filter paper during the measurements or too short equilibration period.
- The quantity of performed calibration measurements, and soil sample measurements was very low, hence the accuracy of both could be improved a lot by rehearsing the measurements, as the fast performance of tests is significantly affecting the quality of results. Also some test conditions (e.g. heat sink to minimize errors caused by weighing of hot sample containers) could be further developed. In addition, in order to evaluate whether the differences between total and matric suction could be detectable with this method, the theoretical differences in total and matric suctions of bentonite samples should be studied further, and the theoretical differences should be compared to the uncertainty limits of this method.
- Finally, as currently the psychrometer measurement of total suction is relatively reliable, fast and cheap, it is recommended not to continue the filter paper method development any further, unless the possibility to observe differences between total and matric suctions is desired to be studied further (e.g. by trying to find sample types and conditions with distinct theoretical expectations of differing total and matric suctions).