

Retention behaviour of natural clayey materials at different temperatures

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Abstract The water retention capacity of geomaterials, and especially clayey soils, is sensitive to temperature changes as the physical mechanisms of retention, such as capillarity or adsorption, are affected by it. It is therefore a major issue to be able to define temperature-dependent behaviour of materials, especially for geo-energy and geo-environmental applications involving non-isothermal conditions. This paper presents results of experiments conducted on two representative materials: a hard clay (Opalinus clay) and a plastic clay (Boom clay), both of which have been considered as buffer materials for underground radioactive waste disposal, in Switzerland and Belgium, respectively. Two new devices were developed for this purpose to permit the analysis of water retention behaviour at different temperatures. The behaviour of these two materials at ambient (20 °C) and high temperature (80 °C) was observed and described through the evolution of the degree of saturation, the water content and the void ratio with respect to suction. It appears that the retention capability of the clays reduces significantly with an increase in temperature; on the other hand, the change in temperature had less of an effect on the total volume variation.

Keywords Natural soils · Non-isothermal conditions · Retention behaviour · Unsaturated soils

1 Introduction

Retention capabilities of soils use to be represented by the soil water retention curve (WRC). This fundamental curve links the variations of the liquid-phase potential energy, characterised by suction, to the variations of the saturation state, characterised by the water content or the degree of saturation. An understanding of the water retention features is an essential aspect in the field of hydro-mechanical unsaturated soil modelling. The development of geo-energy and geo-environmental applications involving non-isothermal conditions leads to the need for a better understanding of the retention capabilities of geomaterials at various temperatures [9, 12]. The trend in modern unsaturated soil mechanics is to quantify, with increasing accuracy, the soil water retention properties of soils in terms of: (1) the effect of suction on density [5, 18, 23], (2) the influence of soil compaction [25, 28, 30, 31] and (3) the hysteresis behaviour on wetting–drying paths [15, 17, 20]. Limited effort has been devoted until now to investigate the effect of temperature on the WRC. Experimental material characterisation is required as the basis to identify the mean physical mechanisms that govern the WRC behaviour; constitutive models could then be developed to simulate and predict the WRC behaviour at various temperatures [6, 26].

In the case of fine grained soils, the range of suction needed to be applied to cover the entire range of saturation states is wide, reaching values of several hundreds of MPa. To be able to test these materials under such conditions, several experimental techniques have to be involved. In this study, axis translation and vapour equilibrium methods

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are used, respectively, to apply low and high suction values. In addition, when dealing with different thermal conditions, the temperature is considered to be a variable of the test and the effect of temperature on the suction control has to be taken into account in the experimental set-up. For this study, two apparatuses were designed: a temperature-controlled pressure plate and a sorption bench [27].

In this paper, the WRC is determined by means of drying paths at different constant temperatures, allowing the quantification of several physical parameters such as the air entry value or the slope of the curve. Also, the investigation of the effect of temperature on the retention properties is made. In addition to the ambient temperature of 20 °C, we selected the temperature of 80 °C. This value is representative of the maximum temperature in some geo-engineering applications such as underground nuclear waste storage [3, 7, 14] and thermo-active geo-structures [13].

The Sect. 1 will be devoted to the presentation of the experimental set-ups and, in the Sect. 2, the experimental method used to obtain the various parameters is presented. A presentation of the experimental results is given for two different representative materials: a hard clay (Opalinus clay) and a plastic clay (Boom clay).

2 Experimental tools

The soils were tested under a large range of suction values using two different techniques: axis translation and vapour control. The first is the conventional air pressure technique allowing for a variation in suction from 0 to 1,500 kPa; this allows the matric suction to be controlled. The second technique is based on the equilibrium between the relative humidity of the atmosphere (imposed by a salt solution) and the water contained in the sample; this is achieved using a sorption bench. This technique allows the application of total suction in the range of 4–400 MPa.

2.1 Pressure plate apparatuses

For low suction values, the axis translation technique is used and involves the translation of the reference air pressure through an artificial increase in the atmospheric pressure in which the soil is immersed [11, 21]. The imposition of matric suction with such a technique relies on the fact that it is basically the difference between air and water pressures: by increasing air pressure and maintaining constant liquid pressure, suction is increased. This statement is valid as long as pore liquid continuity is ensured.

The axis translation technique is applied via a pressure plate extractor, consisting of a vessel connected to an air pressure inlet tube. The vessel encloses high air entry value

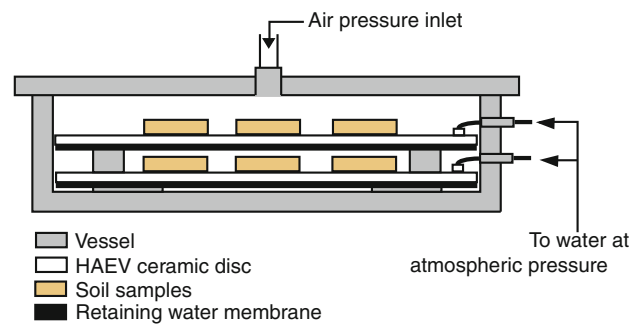


Fig. 1 Section view of the pressure plate extractor

(HAEV) ceramic discs connected to water outlet tubes. The air pressure range of the ceramic discs is limited by their HAEV; above this critical value, air penetrates inside the ceramic pores and rapidly cuts off the water phase continuity that is supposed to impose the water pressure. Two types of devices were used: a standard Soil Moisture Model #1500 (Fig. 1) for tests at ambient temperature and a temperature-controlled pressure plate, designed for this study, for tests at higher temperatures (Fig. 2).

The ceramic discs are saturated according to a procedure proposed by Fredlund and Rahardjo [8]. Water pressure cycles are applied, and the permeability of each ceramic disc is checked until a constant value is reached. Synthetic water is used in the saturation circuit. In this way, during the test, sample pore water and ceramic pore water are in osmotic equilibrium, which avoids unwanted and uncontrolled osmotic exchanges during the test. Two types of ceramic discs are used, with two different air entry values: 500 kPa (Soil Moisture 0675B05M1) and 1,500 kPa (Soil Moisture 0675B15M1). Due to the diffusion process (promoted by high air pressure and high temperature), dissolved air can pass through the ceramic discs and can accumulate beneath them. Therefore, it was necessary to perform a manual air flush. For the high temperature tests, the pressure cell is immersed in a thermo-regulated bath with the temperature regulation performed by means of an immersion heater. The temperature range is between 20 and 90 °C. The accuracy on the applied temperature is about 0.5 °C.

For the test itself, saturated samples of the different materials are put on filter paper sheets and placed on the saturated HAEV ceramic discs. Air pressure is applied inside the vessel, while the water pressure inside the ceramic discs and the samples is maintained constant and close to the atmospheric pressure. The main purpose of these apparatuses, in addition to their simplicity, is to place the sample under zero mechanical stress while its water retention properties are measured. Regular weighing of a control sample allows the operator to determine when equilibrium is reached. This mass determination requires

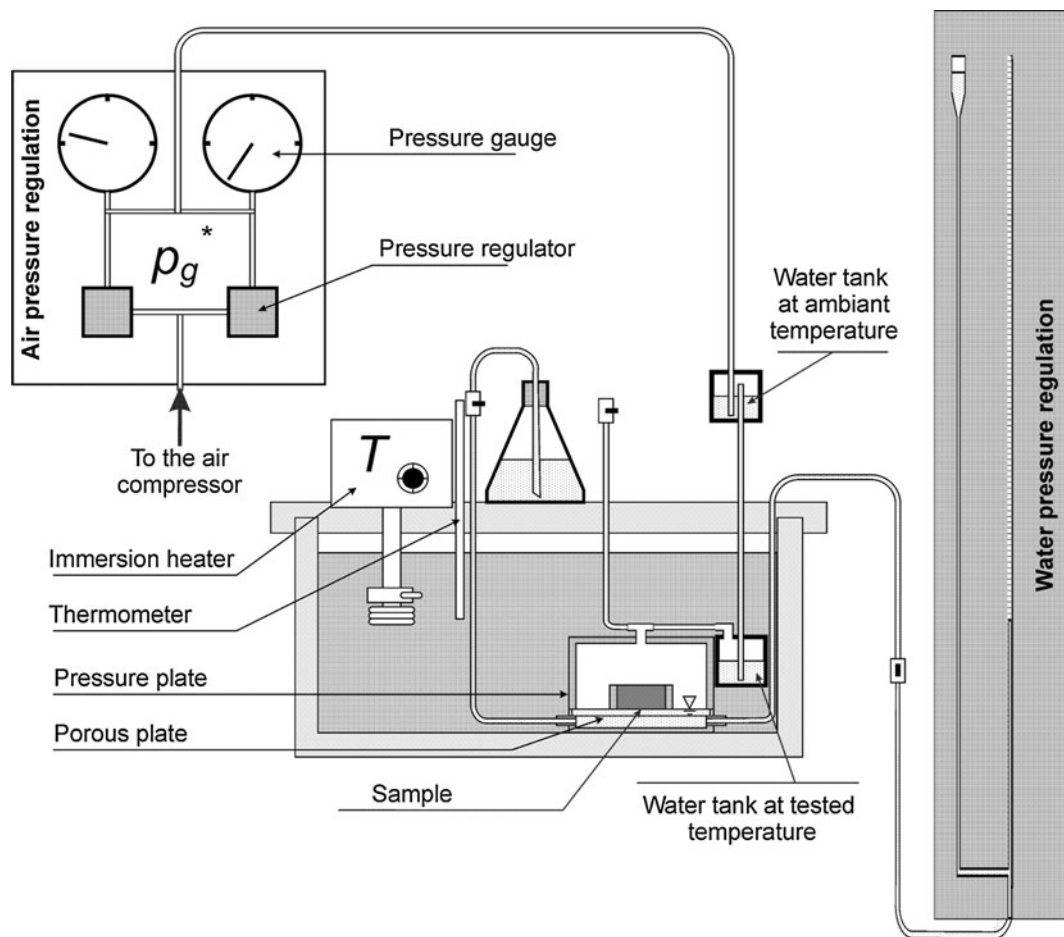


Fig. 2 Section view of the pressure plate extractor regulated in temperature

that the vessel is opened; however, this operation is shortened as much as possible in order to avoid air drying of the samples.

Measurement of water content and volume is undertaken at the time of equalisation. The new suction stage is then applied by increasing the air over-pressure. A benchmark exercise using this technique was recently published (El Mountassir et al. [4]).

2.2 Sorption bench

For experiments in the suction range between 4 and 400 MPa, the vapour equilibrium technique is applied. Vapour equilibrium consists of controlling the relative humidity of a closed system in which the soil is immersed. In this way, soil water potential is applied by means of the migration of water molecules through the vapour phase from a reference system of known potential to the soil pores, until equilibrium is achieved. The relationship between total suction and relative humidity is given by the psychrometric law known as Kelvin's law [29]:

$$s = -\frac{RT}{M_w} \ln(RH) \quad (1)$$

where s is the suction [Pa], R is the ideal gas constant [$\text{J mol}^{-1} \text{K}^{-1}$], T is the temperature [K], M_w is the water molar mass [kg mol^{-1}] and RH is the relative humidity of the atmosphere expressed as a percentage. The relative humidity of the reference system is controlled using various saturated salt solutions. This technique involves a very long equalisation time. The loss of thermodynamic equilibrium can occur due to temperature fluctuations.

This principle is applied for the WRC measurement by the means of the "sorption bench" equipment [27]. The sorption bench allows several samples to be tested at different suctions (i.e. in different closed vessels with imposed relative humidity). The required relative humidity is reached using saturated saline solutions that are poured directly into the vessel (Fig. 3). The desiccators are immersed in a thermo-regulated bath to control the temperature of the test. The saturated salt solutions used, corresponding to the imposed total suction values, are listed in Table 1.

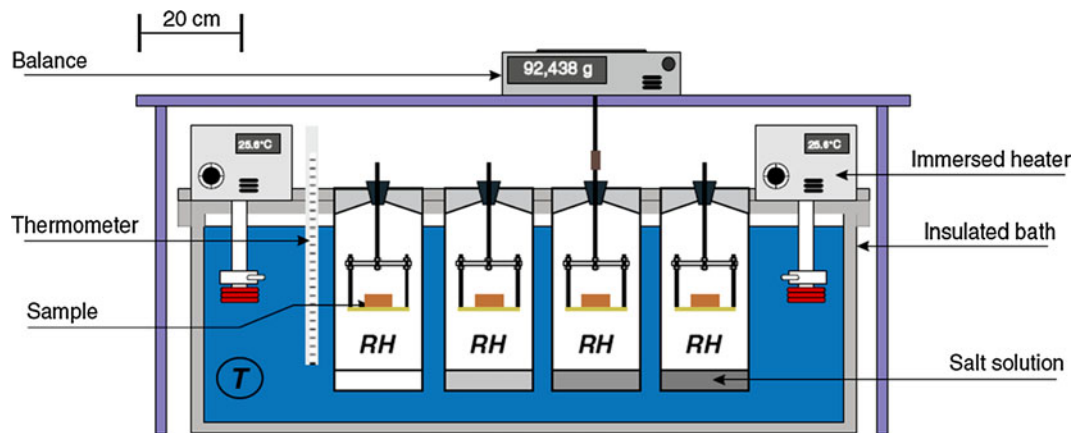


Fig. 3 Experimental layout of the sorption bench for the WRC test (RH stands for relative humidity)

Table 1 Salts and corresponding relative humidity at different temperatures and resulting suction (for temperatures of 21 and 80 °C)

T (°C)	Saturated salt solution	KOH	LiCl	MgCl ₂	K ₂ CO ₃	Mg(NO ₃) ₂	NaCl	KCl	K ₂ SO ₄
21	Relative humidity (%)	9	11.3	33.1	43.2	54.1	75.4	84.9	97.5
21	Suction (MPa)	326.8	295.9	150.5	114.1	83.4	38.3	22.2	3.4
80	Relative humidity (%)	5.3	10.5	26.3	43	37.4	76.1	79	94.3
80	Suction (MPa)	478.2	366.7	217.9	137.6	160.4	44.4	38.5	9.6

Values are given for ambient temperature ($T = 21$ °C) and for 80 °C. The salt saturation ensures a constant relative humidity; saturation could easily be checked by ensuring that solid salt is still present in solution at the end of the test. The relative humidity of the reference system is checked by a psychrometer sensor installed in the container solution. Inside each vessel, three samples are placed in a wire basket, hung from a rod. This rod protruded from the top of the vessel through a small opening which is plugged with an airtight removable seal. A moveable balance is placed above the bench (Fig. 4) which allows sample to be weighed regularly by removing the vessel's seal and attaching the balance to the protruding rod. This weighing method avoids sample disturbance such as handling or drying due to long air exposure. For a given vessel, equalisation is considered to be reached at mass stabilisation of the basket containing the 3 samples.

3 Experimental methods

3.1 WRC volume measurement

Once equilibrium has been achieved, the 3 samples are removed, either from the pressure plate extractor or from a given sorption bench vessel. At this point, the final soil characteristics are determined. The adopted procedure is based on the general method, which makes use of the fluid

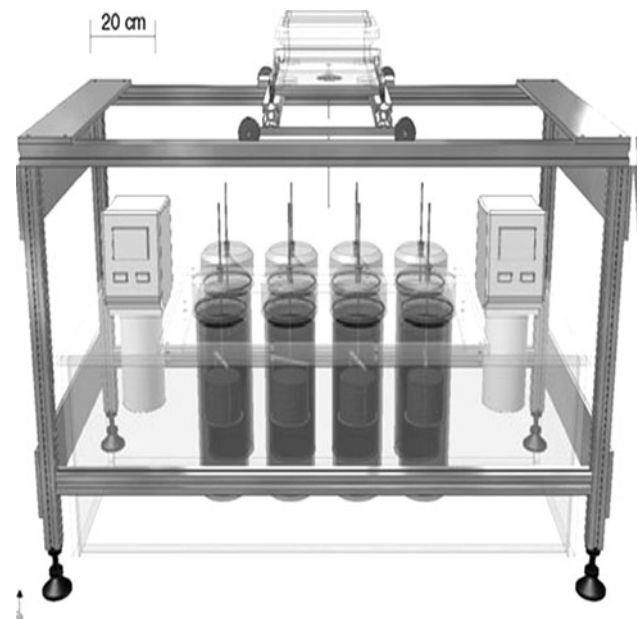


Fig. 4 The sorption bench with the balance for direct sample weighing

displacement technique. A description of this method was given by [10] and with further modifications and improvements by [19, 32]. In this method, a clean, dry pycnometer is first filled with Kerdane, an oil from which aromatics are extracted, and weighed (m_{p1}). Afterwards, each sample that has been set aside for volume

measurement is weighed, and its wet mass (m_1) is determined. The sample is then immersed in a bath of Kerdane to fill those voids that should be taken into account, despite their contact with the outside air. Kerdane is used because it is immiscible in water and invades the pore spaces filled with air without affecting the soil structure. At the end of this phase, the sample is removed from the bath and carefully wiped with absorbent paper. The sample is then re-weighed to obtain the mass saturated by water/Kerdane (m_2). Subsequently, the sample is placed into the empty pycnometer, which is then entirely filled with Kerdane, following the same filling technique used to determine m_{p1} and avoiding air bubble entrapment. Thus, the total volume of the sample is measured; the preliminary bath prevents Kerdane from penetrating the sample pore spaces during pycnometer filling. The filled pycnometer is weighed to determine the mass of the pycnometer with the Kerdane and the Kerdane/water-saturated sample (m_{p2}). In the last step, the dry mass of the sample (m_s) is determined by oven-drying. The sample volume V is calculated from the following relationship:

$$V = \frac{m_{p1} + m_2 - m_{p2}}{\rho_k} \quad (2)$$

where ρ_k is the specific mass of Kerdane. The corresponding void ratio is equal to:

$$e = \frac{V_v}{V_s} = \rho_s \frac{m_{p1} + m_2 - m_{p2}}{m_s \rho_k} - 1 \quad (3)$$

and the degree of saturation is

$$S_r = \frac{V_w}{V_v} = \frac{\frac{m_1 - m_s}{\rho_w}}{\frac{m_{p1} + m_2 - m_{p2}}{\rho_k} - \frac{m_s}{\rho_s}} \quad (4)$$

where V_v , V_s , and V_w are the volumes of the voids, solid particles and water, respectively; ρ_s is the specific mass of the solid particles; and ρ_w is the density of water.

The water content of the sample is deduced from oven-drying. It is emphasised that the present method is a destructive method. Therefore, each point corresponds to a given sample.

3.2 Sample preparation

3.2.1 Boom clay

The plastic clay involved in this work is the Boom clay from Mol, a site chosen for the Belgian high-level radioactive waste repositories concept. For the received core material, the initial matric suction was calculated using the contact filter paper method, while the initial total suction was measured by means of the non-contact filter paper method (Fig. 5). Both are indirect methods which allow the

suction value to be obtained through the measurement of filter paper water content in “hydraulic” equilibrium with the soil. Suction is subsequently deduced from the filter paper water content versus suction calibration curve. According to [22], this technique can be used for matric suction in the range of 30–30,000 kPa; ASTM standards D 5298-94 [1] were followed. A mean calibration curve for the most commonly used types of filter papers (i.e. Whatman No. 42 and S&S 589) was used, based on literature results [1] and the curve determined by [19]. The measurement was undertaken directly after removal from the core protection bag, by simply sawing three slices of the core sample. For each slice, the measurements of matric and total suction were done and were found to be 2.18 MPa and 3 MPa, respectively. It should be noted that the difference between the matric and the total suction is theoretically the osmotic suction.

The initial void ratio, degree of saturation and natural water content of intact samples from the cores were measured. Pieces of approximately 10 cm³ were used. The procedure is not detailed here; it corresponds to the general procedure described in the Sect. 3.1. It was found that the samples were saturated ($S_r = 100\%$) with a void ratio of 0.54 and a water content of 19.6 %.

A large piece of core was removed (approx. 30 cm length), wrapped in a plastic layer, to avoid water exchange, and sealed in mortar in order to firmly encase it. Three or four smaller cylinders (approximately 3 cm in diameter) were drilled from this large sample (in the axial direction). Finally, the thin cylinders were cut into 1 cm thick slices. More than 50 samples were fabricated in this way. About 30 % of the samples were partially broken, generally along discontinuity planes that followed the stratification.

The WRC was determined along a drying path; therefore, samples were all saturated (the suction was reduced to zero) before being dried. In order to saturate samples, they were placed on small coarse porous stones (the contact being ensured by a filter paper) and put into a water bath with water just to the level of the top of the porous stone. Synthetic water with an ionic composition similar to the in situ pore water was used, obtained as a solution of NaHCO₃ [1.17 g/l]. The bath was insulated with a plastic bag in order to create a saturated atmosphere. The synthetic water level was adjusted several times to keep it at the upper level of the porous stones.

The weight evolution of several reference samples placed in the bath was regularly determined. Samples were left saturating until the control sample weight was stabilised. The void ratio, degree of saturation and water content of the saturate samples were first measured, and these values were 1.15, 100 and 46.6 %, respectively.



Fig. 5 Phases of application of filter paper method: *left*, placement of a sandwich filter paper for matric suction measurement; *right*, removal of the non-contact filter paper for total suction measurement after 2 weeks equilibration time

3.2.2 *Opalinus clay*

The hard clay used in these experiments was the Opalinus clay. Tests were performed on specimens collected from undisturbed samples originating from the underground Mont Terry Laboratory (Switzerland).

Samples were obtained by re-coring and cutting the material in order to obtain small cylinders of 28 mm diameter and about 10 mm height. Coring was carried out without water, since this method caused less disturbance to the samples (initial void ratio and water content were close to those of the sealed cores) and the sample surfaces were carefully smoothed using sandpaper (to optimise the contact surface without significantly disturbing the samples).

The initial matric suction of the received Opalinus clay core was determined in the same manner as the Boom clay, by means of the contact filter paper method, while the initial total suction was measured using the non-contact filter paper method. The results are 12.13 MPa for matric suction and 17.3 MPa for total suction. A technique similar to the Boom clay tests was applied to measure the initial void ratio, degree of saturation and the natural water content of intact samples from the Opalinus clay cores; these were found to be 0.15 %, 74.3 % and 3.88 %, respectively.

In order to determine the main drying path, the samples were first saturated; 10 samples were used in order to investigate the saturation phase, where the aim is to find both the required time to reach saturation and that which can be considered as the initial saturated conditions. Synthetic water, namely Pearson's water [0.3 mol/l], was used. Final average values of around 10.9 % for the water content and 0.29 for the void ratio were found for the final saturated conditions of Opalinus clay.

4 Results

4.1 Drying kinetics

Figures 6 and 7 show the evolution of the water content of the samples for each desiccator during the dynamic drying processes. The notation (a) and (b) in the figures corresponds, respectively, to the tests performed at ambient temperature and at 80 °C. With time plotted on a logarithmic scale, the curves appear to have the same shape and the trends fit with the usual S-shaped curve. The time required to reach equilibrium increases with the increase in the relative humidity. At ambient temperature, the time varied between several days, for a relative humidity of 9 %, to more than 100 days at a relative humidity of 97 %. Comparing the kinetics for both temperatures indicates that the drying rate is strongly sensitive to temperature. For tests performed at 80 °C, equilibrium was reached after less than 2 days for all the desiccators. In addition, the gap between the drying kinetics at different suction values is less important. In fact, several phenomena explain the differences in the results at the two temperatures: (1) the evaporation rate increases strongly with temperature, (2) the vapour diffusion increases with temperature [2], (3) the permeability of the sample increases due to the decrease of the viscosity of the pore water [24] and (4) the physico-chemical forces resulting in the water retention capability decrease with the increase of temperature [16]. All these phenomena induce the variation of the soil–water properties with temperature and have a significant effect on the kinetics of the process.

4.2 WRC of both materials

The following graphs (Figs. 8, 9, 10, 11) sum up some aspects of the water retention behaviour of the two

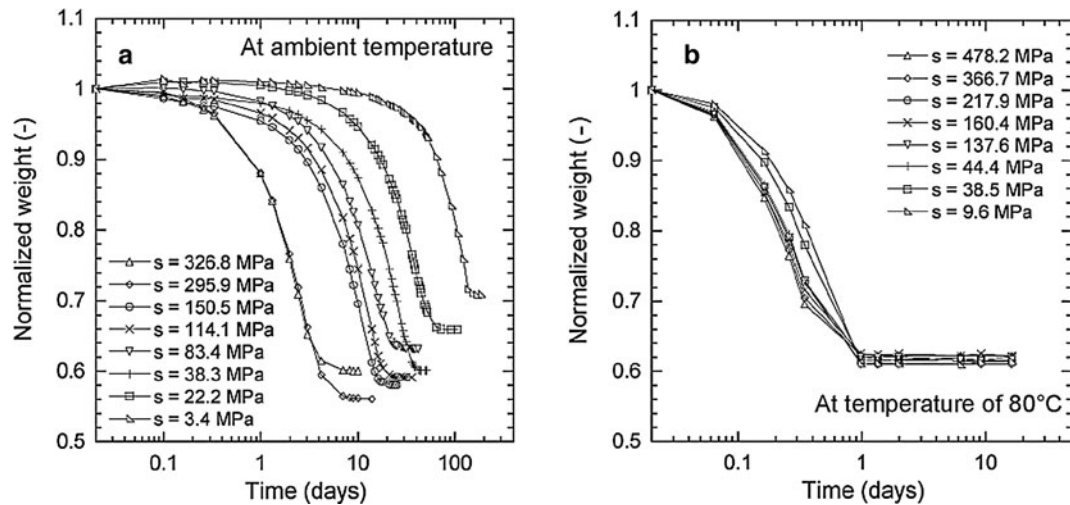


Fig. 6 Evolution of the normalised weight of the boom clay during the main drying path: for the ambient temperature (a), for temperature of 80 °C (b)

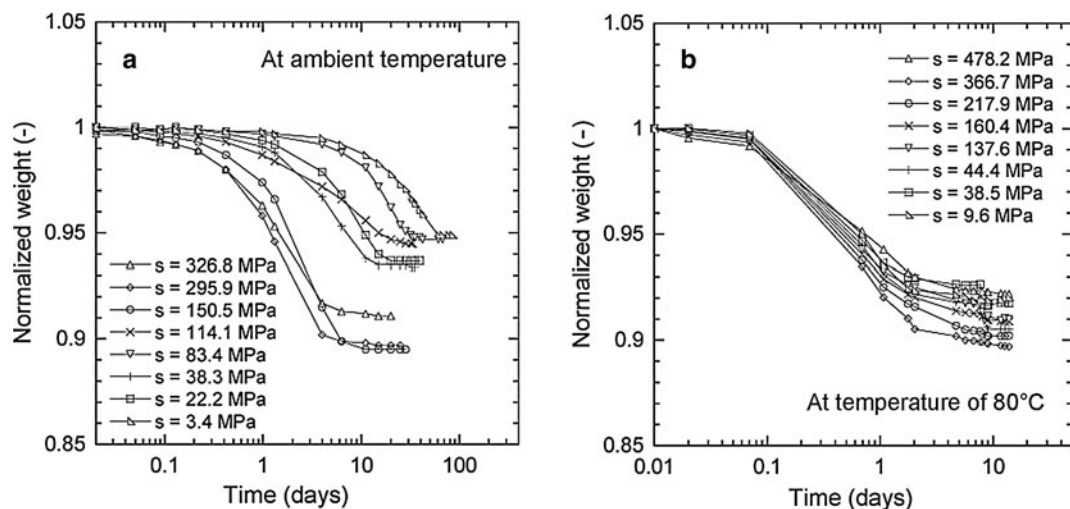


Fig. 7 Evolution of the normalised weight of the opalinus clay during the main drying path: for the ambient temperature (a), for temperature of 80 °C (b)

materials by showing the results obtained along the main drying path in terms of degree of saturation, void ratio, and water content versus suction. As mentioned previously, for each imposed suction, the measurements were carried out on three samples. The plotted results show that the measurements are reproducible. The initial state of the samples is also figured (light squared in the figure). It is emphasised that the WRC is measured by means of a destructive technique; therefore, it is not possible to obtain completely smooth evolutions. The more or less regular evolutions are related to the variability of the successive tested samples, which are themselves related to the heterogeneity of their natural origin. From this point of view, the present results can be considered to be of a very good quality, since clear trends are observed.

Figures 8 and 9 represent the WRC of Opalinus clay at ambient temperature and at 80 °C in terms of water content, void ratio and degree of saturation versus suction. The change in the degree of saturation with respect to the logarithm of suction is roughly bilinear, with a first part almost constant and a second part that shows a clear decrease of the values, down to very small values. The degree of saturation clearly starts to decrease from a suction value equal to 8 MPa. Therefore, the suction air entry value of the material is close to this value. The void ratio evolution with respect to the logarithm of suction is also bilinear and stabilises at a suction value of about 10 MPa. This value can be considered to be representative of the shrinkage limit of the material. The graphs in Fig. 9 present a comparison of the water retention behaviour of Opalinus

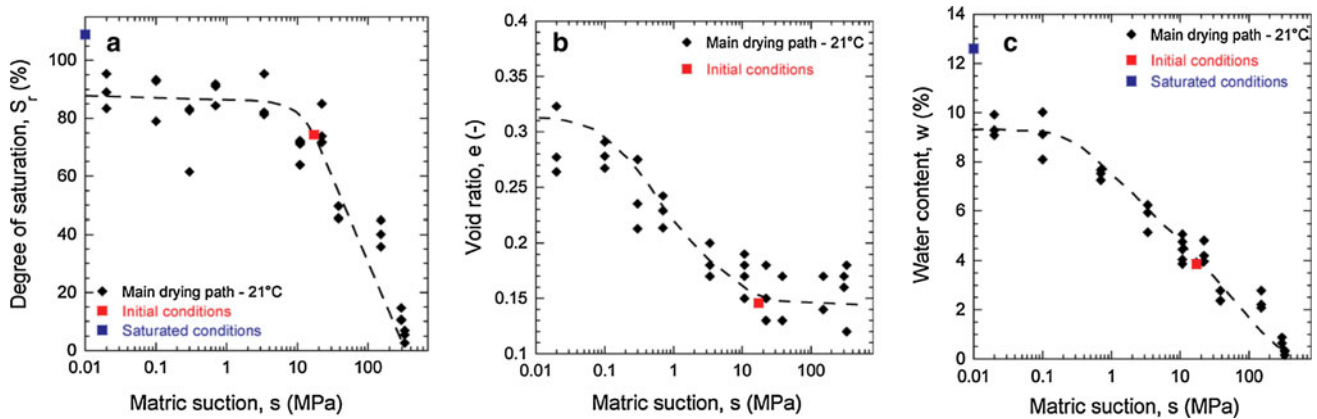


Fig. 8 Water retention curve of opalinus clay at ambient temperature; degree of saturation with respect to suction (a), void ratio with respect to suction (b), water content with respect to suction (c)

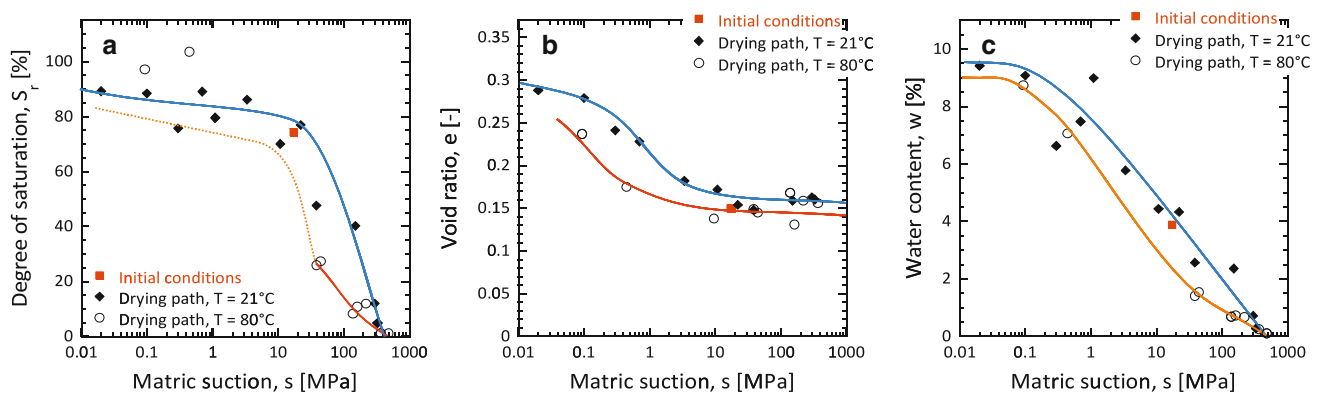


Fig. 9 Comparison of water retention curves of opalinus clay at ambient temperature and at 80 °C; degree of saturation with respect to suction (a), void ratio with respect to suction (b), water content with respect to suction (c)

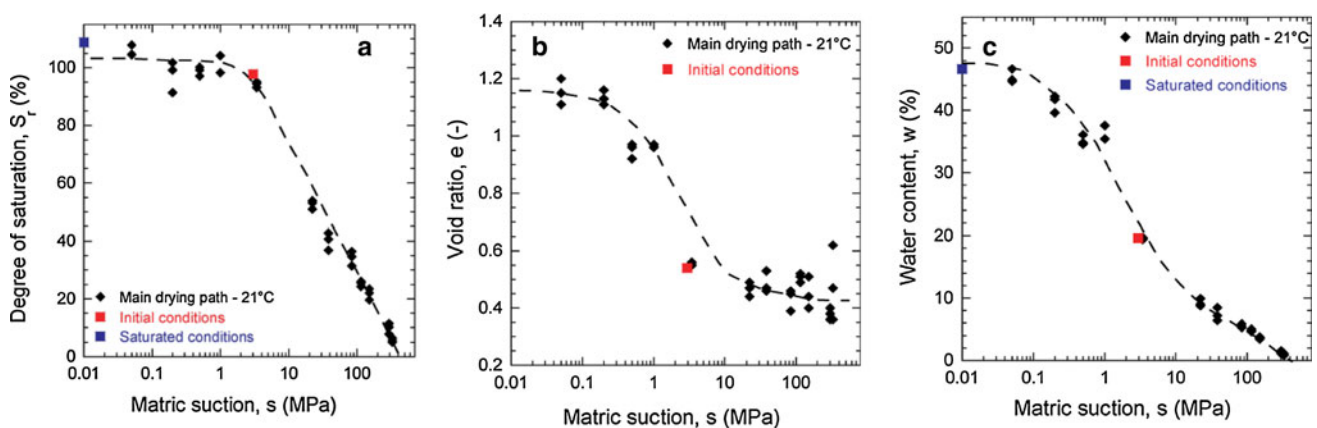


Fig. 10 Water retention curves of boom clay at ambient temperature; degree of saturation with respect to suction (a), void ratio with respect to suction (b), water content with respect to suction (c)

clay obtained at ambient temperature and at 80 °C in terms of water content, void ratio and degree of saturation. These results highlight that the water retention capability of the soil is strongly reduced by an increase in temperature. As for the water content and degree of saturation, the

difference is appreciable for $s < 200$ MPa. On the other hand, no clear differences appear concerning the void ratio variations.

Figure 10 represents the retention behaviour of Boom clay at ambient temperature. By analysing the results, it

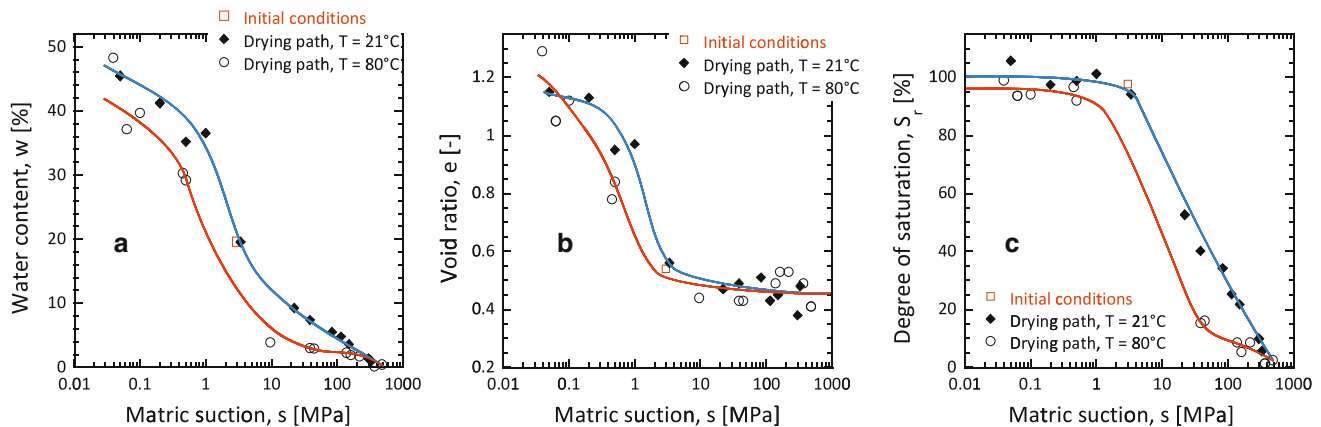


Fig. 11 Comparison of water retention curves of boom clay at ambient temperature and at 80 °C; degree of saturation with respect to suction (a), void ratio with respect to suction (b), water content with respect to suction (c)

can be inferred that, for values of suction lower than 0.1 MPa and higher than 20 MPa, the void ratio remains almost constant, with values around 1.15 and 0.45, respectively, and evolves in between. Finally, the air entry value for the material can be estimated at around 3 MPa. A comparison of the water retention of Boom clay at ambient temperature and 80 °C is presented in Fig. 11. The temperature clearly reduces the water retention capability of the material. In addition, three main observations can be made: (1) for the water content, the difference is appreciable for intermediate values of suction (in the range of the tested values), (2) for the void ratio, no appreciable differences are seen and (3) for the degree of saturation, a clear decrease with temperature is noted for $s > 1$ MPa.

5 Conclusion

In this paper, experimental investigations involving the thermal effects on water retention phenomena for two natural clayey materials have been reported. The methods and the tools related to these tests have been described. The large range of suction to be applied required the use of different techniques for suction loading: axis translation and vapour equilibrium. Due to the interaction with temperature, the design of experimental apparatuses devoted to water retention characterisation is tricky, but most of the requirements have been accomplished in this study. Finally, the characterisation of the water retention behaviour of the two materials has been presented through the evolution of the degree of saturation, the water content and the void ratio with respect to the suction at ambient and high temperatures. It should be noted that the retention capability of both soils is strongly reduced by an increase in temperature.

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