Suction in shales: consequences on triaxial testing

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Abstract. Significant interest has been devoted to claystones and shales in the context of geological radioactive waste disposal at great depth. The determination of their mechanical properties is needed for appropriate design of the underground galleries and tunnels. Given their high sensitivity to changes in water content, special care has to be taken so as to provide characteristics as close as possible to the (saturated) in-situ ones. Based on the hydromechanical path followed by samples from coring to trimming in the lab, that most often lead to some degree of desaturation resulting from evaporation and drying of the samples, some procedures aimed at minimising the resulting perturbations are described. The considerations presented are based on data obtained on two (swelling) claystones considered in Europe for deep geological disposal, i.e., the Callovo-Oxfordian claystone (France) and the Opalinus Clay (Switzerland).

1 Introduction

Due to their very low permeability and high capacity to retain radionuclides, claystones are considered as suitable host rocks for deep geological disposal of high activity radioactive waste in various countries including France (in the Callovo-Oxfordian claystone – COx) and Switzerland (in the Opalinus Clay). Intensive laboratory investigation has hence been conducted on both claystones so as to characterise their thermo-hydro-mechanical behaviour, based on isotropic, oedometer or triaxial compression tests [1 - 6].

Claystone samples are most often water saturated and extracted and trimmed from cores excavated at great depth (500 m for the Underground Research Laboratory – URL – run by Andra, the French Agency for the management of radioactive wastes, in Bure). Given their sensitivity to changes in water content, obtaining responses that are reasonably representative of the in-situ saturated claystone behaviour is a challenge that requires special care, that is described in more detail in this paper.

2 Materials

The investigation was carried out on both the Callovo-Oxfordian claystone (COx) and Opalinus Clay. Their main characteristics are presented in Table 1. Interestingly, both claystones have comparable characteristics.

Table 1. Characteristics of the Callovo-Oxfordian claystone [7] and Opalinus Clay [8].

	Opalinus Clay	COx
Dry density (Mg/m^3)	2.22 – 2.33	2.21 – 2.33
Clay content (%)	44 - 80	30 - 50
% Carbonate	6 - 22	12 - 28
% Quartz	10 - 27	10 - 28
Porosity (%)	13.5 – 17.9	13 - 19
Water content (%)	4.2 – 8.0	7 – 8.5
Young's modulus* (GPa)	4 - 10	6 – 10
UCS* (MPa)	4 - 22	20 - 30
Permeability (m/s)	1 – 5 x 10^{-13}	1 – 5 x 10^{-13}

** Measured values depend on the degree of saturation, see Fig. 3*

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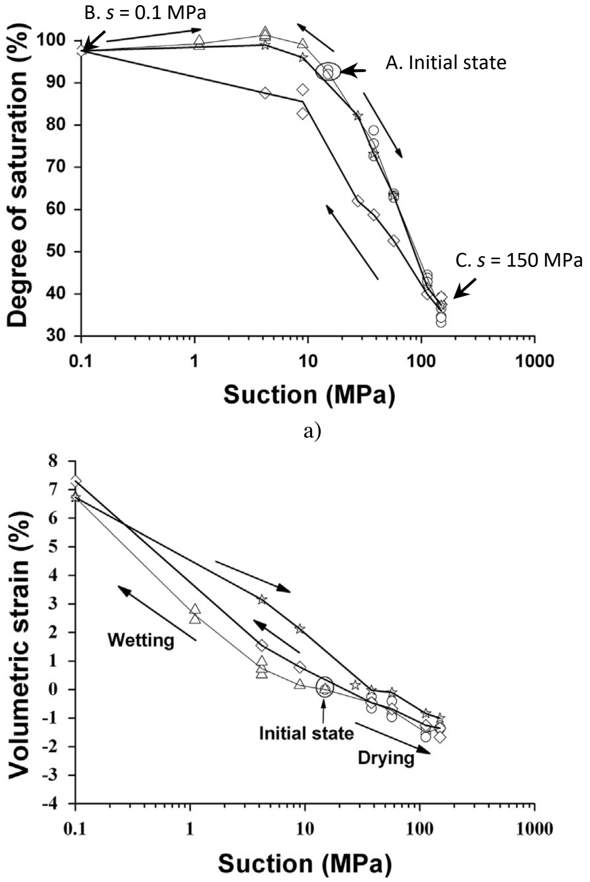
3 From coring to sample trimming

3.1 Water retention properties

It is known, since Skempton and Sowa [9], that saturated clay samples extracted at a given depth are not submitted to a stress release equal to the effective stress supported at that depth. In the case of "ideal sampling", it is admitted that, provided the sample remains saturated during extraction, the suction induced due to effective stress release should exert on the sample, provided it remains saturated, an equivalent effective stress. This is possible if the effective stress is smaller than the air entry value (AEV) of the sample. This was for instance observed in Boom clay (a stiff clay considered for deep geological radioactive waste disposal in Belgium) by Delage et al. [10].

Fig. 1 [11] shows the water retention curve of a sample of a COx sample cored at a depth of 500 m in the Bure URL. The curve has been obtained by using vapour equilibrium with saturated salt solutions (Table 1).



a)

b)

Fig. 1. Water retention properties of the Callovo-Oxfordian claystone (after [11]): a) changes in degree of saturation; b) change in volume.

The curve in Fig. 1a shows that the initial point has a suction of 18 MPa, corresponding to a degree of saturation $S_r = 92\%$. From this initial point, the sample has been submitted to decreased suction down to 0.1 MPa along a drying path, close to saturation. A subsequent wetting path was then followed up to a maximum suction of 150 MPa, followed by a drying path back to saturation.

The data show that the initial state of the trimmed sample in the laboratory was not fully saturated, due to the successive and cumulative effects of (air) coring, storage, transportation, core cutting and sample trimming in the lab [12, 13]. The shape of the curve also shows that the AEV of the COx sample is around 10 MPa. A significant hysteresis is observed.

Table 2. Salt solutions used, after [14].

Saturated soil solution	Suction (MPa)
$MgCl_2$	150.0
K_2CO_3	113.0
$NaNO_2$	57.0
NaCl	30.0
$(NH_4)_2SO_4$	27.5
KNO_3	9.0
K_2SO_4	4.2
PEG 20000 0.015 mol/l	1
Pure water	0

Observation of the curve of Fig. 1b) shows that significant volume changes occur upon wetting, with a volume increase around 6%. This swelling behaviour of the COx claystone, also observed on the Opalinus Clay, is well known. It is due to the presence in both claystones of a significant proportion of montmorillonite, in the mixed layer illite-montmorillonite that constitute the clay matrix, within which the detritic grains of calcite and quartz are embedded (see Table 1 and Fig. 2).

3.2 Sensitivity of shales to changes in water content

As seen in the SEM photo of Fig. 2 [15, 16], that was taken on a horizontal sample hydrated under zero suction and no stress, sub-horizontal swelling micro-cracks (lengths of dozens of μm and thickness of around 1 μm) are clearly apparent within the clay matrix. These micro-cracks would obviously result in some damage of the swollen claystone.

Some pyrite crystals can also be seen in the photo, together with the footprints of some detritic grains that have been pulled out during the freeze-fracturing set up of the observed plane.

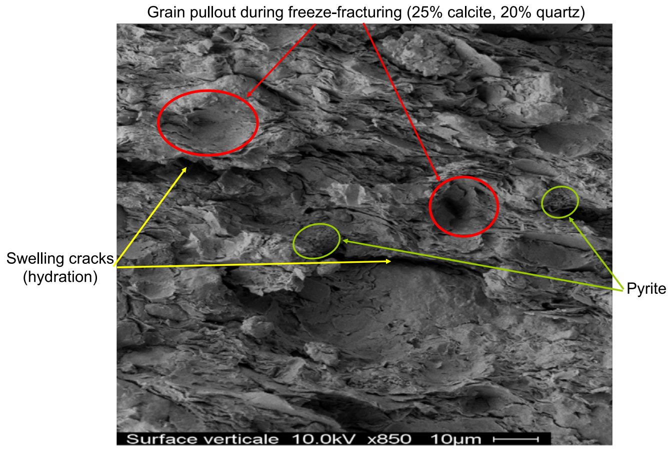


Fig. 2. SEM photo of a free swollen sample of COx claystone at zero suction, showing the sub-horizontal cracks [15, 16].

Together with swelling along wetting paths, claystones are also significantly sensitive to drying, that results in a significant increase in strength. This is shown in Fig. 3 with the data of unconfined compression tests run by Pham et al. [17] on COx samples submitted to various relative humidity between 98% (suction s = 2.5 MPa with a water content w = 5.24%, close to saturation according to Fig. 1) and 32 % (s = 141.3 MPa, w = 1.65%, S_r around 34%) through 76% (s = 35 MPa, w = 3.75%, $S_r \approx$ 80%) and 44% (s = 101.8 MPa, w = 3.2%, $S_r \approx$ 45%). The maximum unconfined compression strength (UCS) increases between 28 MPa close to saturation up to 58 MPa under 32% RH (s = 141.3 MPa).

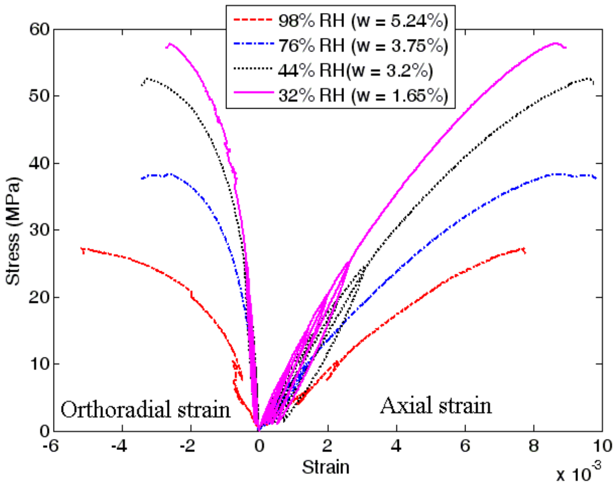


Fig. 3. UCS tests conducted under various relative humidity/suctions [17].

As a consequence, preventing as much as possible tested samples from evaporation starting from coring to trimming in the lab is of outmost importance. In this regard, the measurement of their degree of saturation is mandatory. Suction measurements, for instance by using a chilled mirror tensiometer (e.g., WP4 Decagon) are even better, due the high sensibility of suction to changes in S_r.

4 Testing claystones

4.1 Hydration phase

Given the swelling behaviour observed in Fig. 1b, the first rule to follow when testing a swelling claystone (either in oedometer, isotropic or triaxial compression) is to avoid any contact with water as long as the sample is not submitted to in-situ stress conditions. To do so, the sample has to be placed in contact with dry porous disks until the sample is under suitable stress conditions. Then, air has to be expelled by applying vacuum to the drainage system, prior to saturate the drainage lines with de-aired water [12]. Note, as initially shown by Mohajerani et al. in the oedometer [18] or Monfared et al. [12] in the triaxial apparatus, some swelling may be observed, even under in-situ stress conditions. This trend has been further investigated by Delage and Belmokhtar [19], who characterised the changes in swelling of the Opalinus Clay (shallow sample from Lausen extracted at a depth of 30 m) under hydration with respect to the isotropic applied stress (Fig. 4).

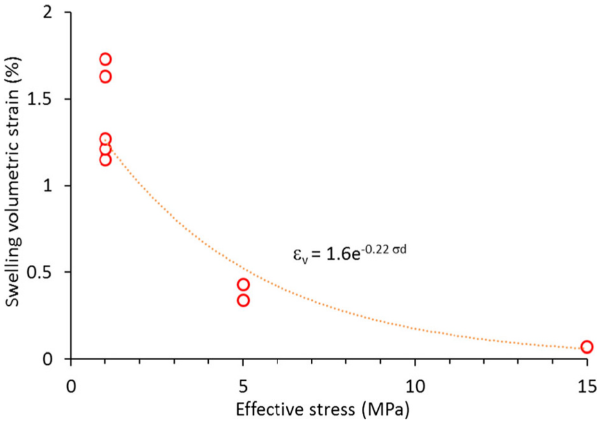


Fig. 4. Changes in swelling under hydration of Opalinus Clay with respect to the applied confining stress [19].

The curve clearly shows that significant swelling occurs under 1 MPa (the in-situ stress) and that an effective isotropic stress of 5 MPa is necessary to reduce swelling below 0.5%. No significant swelling is observed under 15 MPa. This shows that the properties of the sample at low stresses (below 5 MPa) may be significantly affected by damage.

4.2 Stress strain curves

Fig. 5 [19] shows the stress strain curves obtained under a confining stress of 10 MPa on a sample (38 mm wide, 76 mm high) with bedding perpendicular to the axis (contraction is positive). Strains were locally monitored by using strain gauges directly stuck on the sample at mid-level. The curves evidence some degree of anisotropy, with local radial strains significantly smaller than axial ones, a feature typical of shales. A significant difference is observed between local (green squares) and external (pink triangles) axial strains, confirming the need of local strain measurements, particularly for determining the Young modulus (that may be significantly underestimated when measured from external strain measurements). The volume change curve first exhibits a contracting phase, followed by a dilating one, with a transition at 13 MPa.

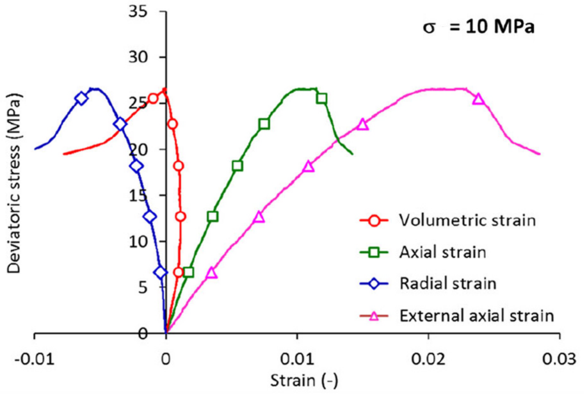


Fig. 5. Stress strain curves obtained under a confining stress of 10 MPa [19].

Fig. 6 shows the failure criteria obtained from drained triaxial testing of the shallow Lausen samples of Fig. 4, completed with i) the data of undrained samples carried out by Giger et al. [20] on the same Lausen samples, ii) drained tests carried out by Favero et al. [5] on Mt Terri samples and iii) undrained samples of Mt Terri by Wild and Amman [21].

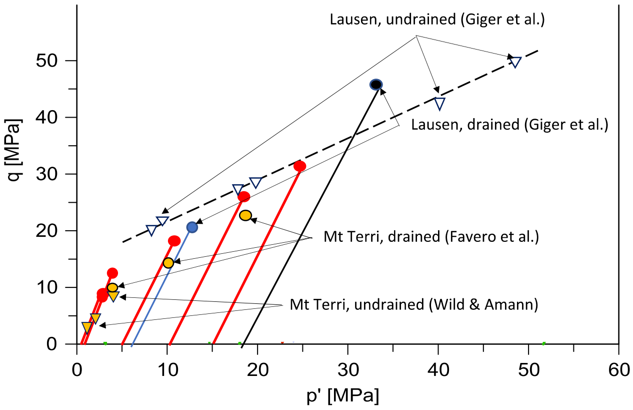


Fig. 6. Failure criterion of various samples of Opalinus clay from Lausen and Mt Terri [19].

The interest of undrained tests in shales carried out following Ewy et al. [22] approach is that samples keep their initial volume and are not affected by any swelling due to previous hydration (see [22] for more details). Fig. 6 shows quite a good agreement between Giger et al. [20]'s undrained tests and our drained data for p'> 10 MPa. For this stress, Fig. 4 shows that the swelling under hydration is equal to 0.2%. Our drained data at 5 MPa, with a swelling of 0.4%, exhibit a lower failure shear stress than that obtained through Giger's undrained tests, with an even larger under-estimation at lower stress. In spite of their different origin, the same observation apparently holds for the data obtained by Favero et al. [5] and Wild and Amman [21] on samples from the shaly facies of Mt Terri (these undrained tests were apparently not conducted following Ewy's approach [22]). As a consequence, it seems that the shape of the failure criterion at low stresses is not easy to determine, and that undrained tests at lower stress would be necessary to check whether the criterion keeps being linear.

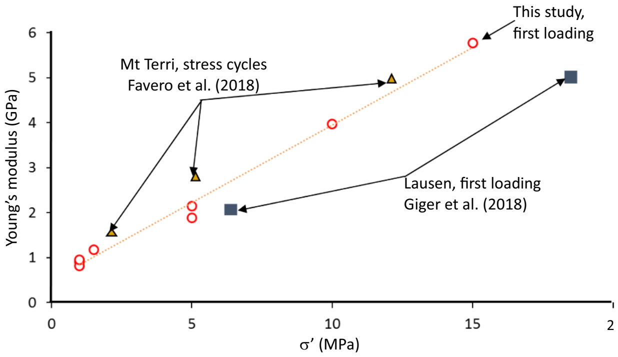


Fig. 7. Changes in Young's modulus of Opalinus Clay samples from Lausen and Mt Terri [5, 19, 20].

Fig. 7 shows the changes in Young modulus with confining stress of samples from Lausen and Mt Terri (both from first loading and stress cycles, that are known to provide a higher value). In spite of the different origins of the sample, a good comparability is observed between them. Based on the swelling data of Fig. 4, one can conclude that the data below 5 MPa probably under-estimate the Young's modulus. The linearity observed in the Figure is hence not guaranteed.

5 Concluding remarks

The sensitivity to changes in water content (and suction) of some claystones considered as possible host rock for the deep geological disposal of high activity radioactive waste in France (Callovo-Oxfordian claystone) and Switzerland (Opalinus Clay) has been taken into account to optimise the experimental characterisation of their mechanical properties (either through œdometric, isotropic or triaxial compression). Claystones are saturated in their in-situ state, but the successive operations of coring, storage, transport and lab trimming most often result in some degree of desaturation. Because of their sensitivity to changes in water content, special care has to be taken for mechanical testing, because the suction release occurring when putting the sample in contact with water in the cell should be compensated by applying a sufficiently high stress (that can be higher than the in-situ one). Based on this consideration, some data on the mechanical properties of these claystones have been discussed, focussing on possible under-estimation of their properties under smaller confining stresses.

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