Coupled stress-suction changes in the Callovo-Oxfordian claystone

Philipp Braun[1*], *Pierre* Delage[1], *Siavash* Ghabezloo[1] and *Jean* Talandier[2]

[1]Laboratoire Navier, Ecole des Ponts ParisTech, Univ. Gustave Eiffel, CNRS, Marne-la-Vallée, France
[2]Agence Nationale pour la Gestion des Déchets Radioactifs (Andra), Châtenay-Malabry, France

Abstract. Some (saturated) claystones, considered as host rocks for deep radioactive waste disposal, are sensitive to drying and wetting. Wetting may result in swelling and damage during the resaturation of specimens for mechanical testing. Given that, due to evaporation, specimens may have lost some pore water during coring, transport, storage and trimming prior to lab testing, it is required to re-saturate them by reinjecting the evaporated water content, compressing the pore space under constant water content, or by using a combination of both. It is important to characterize the hydromechanical processes involving suction changes under total stress loading in order to adapt preparation methods and minimize sample disturbances. We present a novel experimental setup to test claystone specimens under isotropic compression at constant water content, while measuring suction, and we discuss the data of three specimens testedat different initial suctions and degrees of saturation. Interestingly, the path followed in the (decreased) suction vs (increased) total confining stress diagram was observed to be close to the Gens-Alonso "Neutral Line" (a line inclined of 45° in a suction/mean total stress diagram, illustrating similar effects of changes of both variables). In other words, even in close to saturated conditions, changes in confining stress resulted in comparable changes in suction. The data obtained indicate that applying a total stress of 12.2 MPa during resaturation keeps the material on the compressive side of the Neutral Line, limiting swelling-induced sample disturbance.

1 Introduction

The hydromechanical properties of various claystones have been extensively investigated within the context of geological radioactive waste disposal at great depth. This concerns more particularly the Callovo-Oxfordian claystone (COx) in France and the Opalinus Clay in Switzerland. To determine, in the lab, the mechanical characteristics that are reasonably representative of the in-situ saturated conditions of the claystones, attention has to be paid to the hydromechanical conditions that the samples experience between extraction and laboratory testing. This is particularly important given the significant sensitivity of both COx claystone and Opalinus Clay to water, with strengthening upon drying (and desaturation) and weakening upon wetting, due to some possible damage resulting from swelling under low stress conditions (see [1] for more details).

Within the hydromechanical path followed by the claystone, starting from core extraction, transport, storage and finally sample trimming in the lab, this paper focuses on the re-confining stage of the sample, prior to subsequent drained triaxial testing. Experience showed that claystone samples, most often air cored during extraction, are received in the lab with degrees of saturation smaller than 100%. This results in the build-up of an initial suction within the sample, resulting from stress release during core extraction and possible further evaporation.

In order to tests the claystone under fully saturated conditions, it is hence necessary to either resaturate it by reinjecting the lost water content, reduce the pore space under constant water content by compression, or use a combination of both. Experimental evidence has shown that a first recompression to a stress of at least equal to the in-situ one is required, to avoid excessive swelling and the related damage when adding water afterwards [2].

This paper describes a new device developed to investigate the effect of mechanical compression at constant water content on the sample saturation and suction. The conclusions drawn on the coupling between stress and suction during the compression of nearly saturated COx samples also have a wider interest in the context of the mechanics of unsaturated media.

2 Materials and methods

2.1 Theoretical framework

It is well known in unsaturated soils mechanics that, as long as a specimen remains saturated while under suction (i.e., for suctions lower than the air entry value AEV), suction s has an effect equivalent to that of an isotropic confining pressure p. Hence, strains could be written as a function $f(s+p)$. Gens and Alonso [8] introduced the concept of Neutral Line (NL) in the suction/stress space, a line with a slope $\Delta s/\Delta p = -1$ (Fig.

* Corresponding author: philipp.braun@enpc.fr

1). If the suction-stress state remains on the Neutral Line, no deformations occur. States right of the NL induce compression, while states left of it induce swelling. Note that these authors stated that inclinations of the Neutral Line different from -1 could be found, depending on the microstructure of the material.

The knowledge of NL is essential if one wants to resaturate samples that have lost some pore water while minimizing swelling. To avoid swelling, paths left of the NL have to be avoided (Fig. 1). If the sample at state A is resaturated in unconfined state or under relatively low total stress (A-B-C), the path crosses the NL (B), resulting in swelling. If the sample is resaturated under constant volume condition (A-D), swelling is avoided. This procedure could require high stresses (D) that could result in plastic deformations of the material. The knowledge of NL and more precisely of the stress at state F is required to saturate the material under the minimum stress required to avoid swelling (A-E-F or A-D-F).

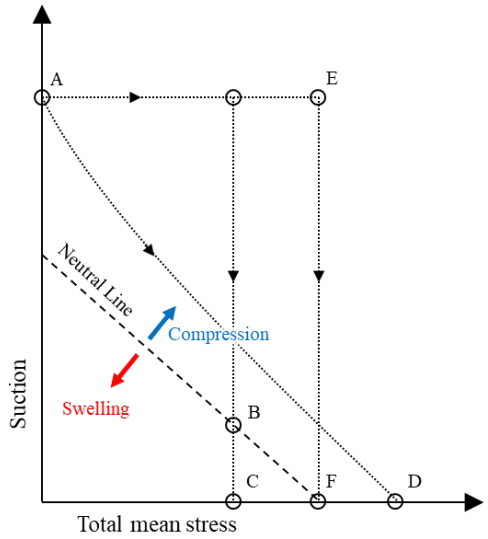

Fig. 1. Neutral Line concept [8], showing schematically different resaturation paths. Path A-B-C induces swelling during resaturation, while resaturation under different conditions A-D, A-E-F or A-D-F avoid swelling.

2.2 The Callovo-Oxfordian claystone

To investigate the potential host rock for the French disposal system, the French Agency for the management of radioactive waste (Andra) has established an underground research laboratory (URL) in the Callovo-Oxfordian claystone at 490 m depth. At his depth, the claystone has an average porosity of 17.5 %, an average water content of 7.9 % and an average clay content around 46% [3].

The stress state at a depth of 490 m has been estimated, with both the vertical σ_v and the minor horizontal total stress σ_h close to 12 MPa and the major horizontal stress σ_H close to 16 MPa. The pore pressure is p_f = 4.9 MPa [4]. This stress state is not too far from isotropic, resulting in a mean effective stress close to 8 MPa.

Fig. 2 shows the water retention properties of the COx claystone measured by [5] by controlling suction through the vapour phase by using saturated saline solutions [6].

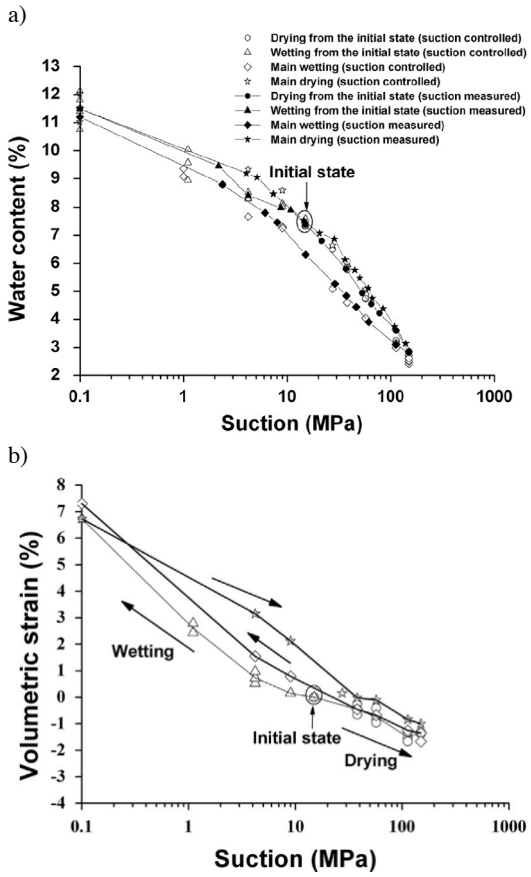

Fig. 2. Water retention properties of the Callovo-Oxfordian claystone (after [5]): a) changes in water content; b) change in volume.

The curve shows that the initial sample suction, measured by using a chilled mirror dew point tensiometer is equal to 18 MPa, which corresponds to a degree of saturation of 92%. An important feature illustrated by Fig. 2b is the significant swelling upon unconfined hydration, with an increase in volume of around 7%. Moreover, previous works have determined the air entry value (AEV) for COx claystone through gas injection (7-9 MPA) [9] and by back analysis from the predominant pore size of 32 nm (9 MPa) [10]. As a consequence, specific care has to be taken when running a triaxial test on this claystone, so as to avoid any sample-water contact before applying any confining stress (see [2] and [7] for more details). Samples have hence to be put in contact with dry porous stones and compressed under a constant initial water content prior to be re-saturated by injecting de-aired water, once the pore line (porous disk + ducts) of the system has been submitted to vacuum.

In this work, three specimens of COx claystone from cores EST53650 and EST57185 were investigated. The water content and the degree of saturation were measured on cuttings directly after opening the sealed core, using hydrostatic weighting in hydrocarbon and oven-drying at 105°C during 48 hours. The initial suction each sample was measured just before testing with a chilled mirror dew point tensiometer (WP4C Decagon). The characteristics of the three tested

samples are given in Table 1. Note that the rather high initial suction of sample A does not correspond to the high saturation which was measured when opening the core. The sample has been likely desaturated during storage.

Table 1. Characteristics of the tested samples after extracting them from the core.

	Core	Water content (%)	Degree of saturation (%)	Suction (MPa)
A	EST57185	7.9	95.3	37.4
B	EST53650	7.5	92.5	25.6
C				17.4

2.3 Experimental device

In the developed device, a cylindrical specimen is placed on a cylindrical steel base that contains a capacitive humidity sensor (Honeywell HIH-4000) as shown in Fig. 3 and 4**Erreur ! Source du renvoi introuvable.**. A porous steel disk is placed between the sensor and the sample i) to protect the sensor from any stress during compression and ii) to permit vapour exchanges and suction equilibration between the sample and the sensor.

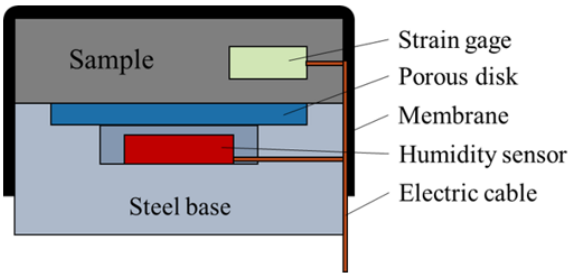

Fig. 3. Specimen sealed in a neoprene membrane, with a steel base that contains a capacitive humidity sensor. Strain gages are glued directly on the sample surface.

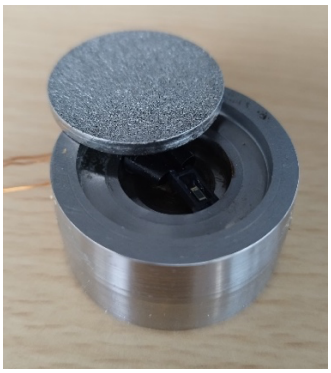

Fig. 4. Photo of the steel base without sample and membrane. The porous disk has been moved aside to show the humidity sensor below.

The assembly is sealed within a neoprene membrane to prevent any infiltration of the confining fluid during compression. The sample, trimmed with its axis perpendicular to bedding, is 38 mm in diameter and 10 mm high. Strain gages are attached to the sample surface using a special strain gage glue, along two directions, in order to monitor both axial and radial strain changes (changes in strains will not be commented in this paper).

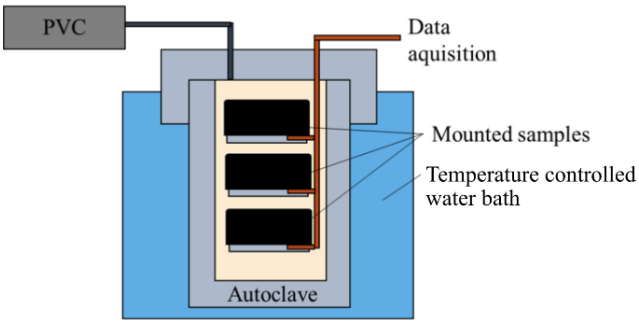

Fig. 5. Claystone specimens within an autoclave pressurized by means of a pressure volume controller (PVC). Temperature is controlled through a temperature-controlled water bath (25°C ± 0.1°).

Isotropic compression was carried out within a high-pressure autoclave (Fig. 5) pressurized by means of a pressure volume controller (PVC) up to 30 MPa (GDS Brand). The autoclave was kept at a constant temperature of 25°C ± 0.1° by means of a temperature-controlled water bath. As seen in the Figure, three devices with three different samples as shown in Fig. 3 could be placed and simultaneously tested in the autoclave. The autoclave was filled with silicone oil and tightly closed, while an electrical feed-through allowed to connect the sensors to the data acquisition system.

2.4 Testing procedure

Isotropic compression was carried out by stepwise increase of the confining pressure imposed by the PVC, with steps of 2 MPa. For sample A, a few steps with higher stress increments were carried out. An example of the loading sequence on specimen C is shown in Fig. 6, with the confining stress curve in red and suction in blue (both at same scale).

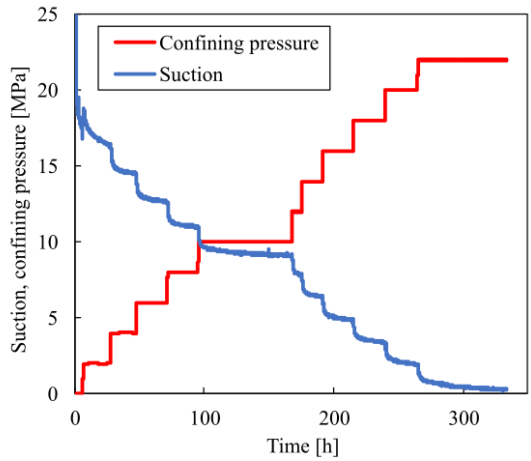

Fig. 6. Stepwise changes in confining pressure and monitored suction changes during the test on sample C.

After each pressure step, the recorded humidity change was monitored by the humidity sensor. Once the humidity stabilized under a given constant confining pressure, the next step was applied. The maximum isotropic stress reached for sample C was 22 MPa.

Observation of Fig. 6 shows that a period of time of around 5 to 10 h had to be waited until reaching suction stabilisation.

Specimens A and C were loaded until 22 MPa, while specimen B only reached 16 MPa. Note that tests on specimens A and B had to be stopped before reaching 100% relative humidity, due to leakage problems that resulted in a failure of the humidity sensor. As expected, it was not possible to reach 100% of relative humidity for these specimens, due to the initial loss of water. In these cases, the compression induced deformations couldn't compensate the deficit of pore water.

3 Results and discussion

The measured relative humidity *RH* was converted into suction *s* according to Kelvin's law, as follows:

$$s = R\,T\,\ln(RH)\,/\,V \qquad (1)$$

where *R* is the universal gas constant, *T* the absolute temperature and *V* the molecular volume of water.

The changes in suction with respect to applied confining pressure at equilibrium for each step are displayed in Fig. 7 for the three samples. One can observe that, in all three experiments starting from different initial suctions, suction linearly decreases with increased confining pressure. Moreover, the slope of the curves is steeper for samples with lower initial suction and higher degree of saturation: Sample A (initial suction s_i = 37.4 MPa) has a slope of -0.38, sample B (s_i = 25.6 MPa) a slope of -0.64 and sample C (s_i = 17.4 MPa) a slope of -0.82.

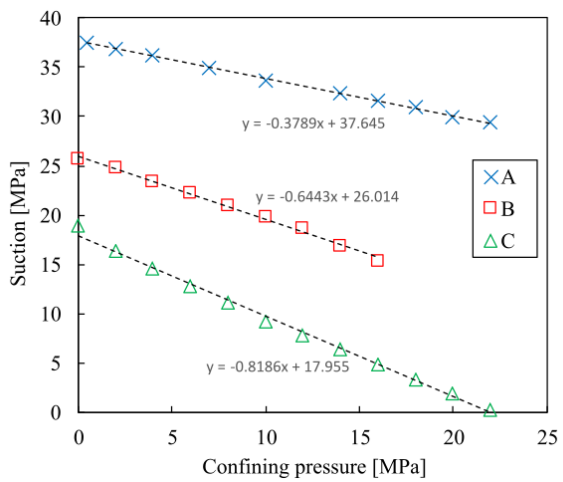

Fig. 7. Suction changes with respect to confining pressure for the three samples tested.

Interestingly, the data of Fig. 7 show an increase in slope with decreasing initial suction (corresponding to increasing initial degree of saturation), which would tend to a value smaller than -0.82 for 9 MPa (corresponding to the AEV, at which full saturation S_r = 100% is reached [5]). We suspect therefore that our experimental paths are located on the right side of the neutral line, where compressive strains are generated. An increase of the slope with decreasing initial suction also means that the rigidity of the pore space decreases with decreasing suction. The Neutral Line is significant for sample preparation, since crossing it to the left side, either by unloading the total stress or by wetting induced suction reduction, could generate unwanted swelling strains. According to [8], crossing the Neutral Line could be avoided by saturating under constant volume conditions or under total stress larger than the intercept of the Neutral Line with the mean stress (confining pressure) axis. Note that homogeneous constant volume conditions are difficult to maintain in conventional triaxial testing systems. To identify the minimum required stress to avoid swelling with the second method, we have to identify the NL. The first point of the NL can be placed at 9 MPa suction (AEV) for zero total stress, and by assuming a NL slope of -0.82, the second intercept of the NL at zero suction with p = AEV/0.82 = 11.0 MPa can be found (path A-E-F in Fig. 1). Further experimental data on samples with higher initial saturation could allow us to narrow down the location of the Neutral Line, w in lower required resaturation stress.

4 Conclusions

A new isotropic compression device with integrated suction measurement was presented. The experimental data on three claystone samples show that the initially desaturated material can be partially or completely resaturated through isotropic compression. The suction decreased linearly with increasing total stress, while the slope of decrease was larger for lower initial suction. Hence, the higher the initial saturation of a specimen, the higher the potential to reduce suction through compression. The lower the suction before wetting, the less water has to be added and less swelling strains are induced.

We suspect a Neutral Line for full saturation (9 MPa suction with a slope $\Delta s/\Delta p \leq -0.82$). Remaining on the right side of the neutral line, swelling and damage during resaturation could be completely avoided. These conditions can be ensured by applying a total stress p = 11.0 MPa during resaturation. Further experiments on samples with higher initial saturation and strain measurements could complement the presented study.

The work presented here has been carried out during the first author's PhD thesis, which was supported by Andra.

References

1. P. Delage, M. Belmokhtar, Acta Geotech., **17** (2021)
2. P. Delage, *Suction in shales: consequences on triaxial testing*, in Proceedings of the International Conference on Unsaturated Soils, 2-5 May 2023, Milos, Greece (2023)
3. N. Conil, J. Talandier, H. Djizanne, R. de La Vaissière, C. Righini-Waz, C. Auvray, C. Morlot, G. Armand, J. Rock. Mech. Geotech. Eng., **10**, 4 (2018)

4. Y. Wileveau Y, F.H. Cornet, J. Desroches, P. Blümling, Phys. Chem. Earth A/B/C, **32,** 8-14 (2007)
5. M. Wan, P. Delage, A.M. Tang, J. Talandier, Int. J. Rock Mech. Min. Sci., **64** (2013)
6. P. Delage, M. Howat, Y.J. Cui, Eng. Geol., **50**, 1-2 (1998)
7. M. Monfared, P. Delage, J. Sulem, M. Mohajerani, A.M. Tang, E. De Laure., Int. J. Rock Mech. Mining Sc., **48**, 4 (2011)
8. A. Gens, E.Alonso, Can. Geotech. J., **29**, 6 (1992)
9. R. De La Vaissière, J. Talandier. *Gas injection tests in the Meuse/Haute Marne underground research laboratory*, in Proc. Gas Generation and Migration Int. Symp. & Workshop, Luxembourg, (2012)
10. P. Delage, H. Menaceur, A-M. Tang, J. Talandier. Géotechnique Lett., **4**, 4 (2014)