Air-water interaction and time dependent compressibility of a subterranean quarry chalk

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AIR-WATER INTERACTION AND TIME DEPENDENT COMPRESSIBILITY OF A SUBTERRANEAN QUARRY CHALK

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ABSTRACT: In this paper the effect of suction and time on the mechanical response of a quarry chalk during eodometer tests is investigated. The water retention properties of the chalk, giving the changes in fluid content as a function of the suction applied, are studied and related to the changes in relative humidity in the quarry, chalk mineralogical composition and microstructure. Like in partially saturated geomaterials, it is recognised that chalk yielding is suction dependent, in that for decreasing suction (i.e. increasing water content) chalk softens. Preliminary results showing the effect of suction on the viscous behaviour of the chalk are also presented. The importance of the hydromechanical coupling associated to suction cycles and the induced cyclic changes in mechanical strength of chalk is emphasised, with regard to the long term behaviour of chalk pillars.

KEYWORDS: chalk, quarries, suction, viscosity, oedometer.

RESUME : On présente dans cet article les résultats d'une série d'essais de compressibilité réalisés à l'oedomètre pour étudier l'effet de la succion et du temps sur le comportement mécanique d'une craie de carrière. Les caractéristiques de rétention de la craie, donnant l'évolution de la saturation relative en eau en fonction du niveau de succion, ont été analysées et mises en relation avec les changements d'hygrométrie dans la carrière, la composition minéralogique de la craie et ses propriétés microstructurales. On montre que, comme dans les sols non saturés, l'écrouissage de la craie dépend du niveau de succion imposé, et qu'une réduction du niveau de succion (i.e. augmentation de la teneur en eau) augmente sa compressibilité. Des résultats préliminaires permettant de quantifier l'effet de la succion sur le comportement visqueux de la craie sont également présentés. On souligne l'importance du couplage hydromécanique existant dans cette craie, associé aux effets des cycles de succion sur sa résistance mécanique, en relation plus particulièrement avec le comportement mécanique à long terme des piliers des carrières.

MOTS-CLEFS : craie, carrières, succion, viscosité, oedomètre.

1. Introduction

Abandoned subterranean quarries constitute a potential risk in areas where their collapse may affect surface infrastructures and buildings. Quarries in chalk are quite numerous in France, for this reason a specific research is being conducted in the Estreux quarry (North of France) by INERIS, aimed at investigating the possible factors affecting the long term stability of the host chalk formation.

For mineworkings a number of collapse phenomena are documented in literature (among others: BLPC, 1973; Masson, 1973; Bonvallet, 1979; Bell et al., 1999; Talesnick et al., 2001; Lord et al. 2002; Grgic et al., 2003; Sorgi 2004). These collapses are mostly related to the destructuration (weathering) of the rock of the pillars due to external (i.e. physico-chemical processes, environmental conditions) or internal (intrinsic) factors. The timing of these collapses is not easy to predict. They often occur without any manifest precursory symptom or external action, indicating that time effects (weathering, aging) may have serious consequences on the structural stability of the rock.

It seems now well accepted that the collapse of mine pillars is mainly due to the aging and the progressive degradation of the rock material, submitted to a constant state of stress. In the case of Estreux quarry chalk, moreover, the average relative humidity (RH) varies cyclically between 100 and 80%. Due to the well known sensitivity of chalk to water action, it is believed that these changes in RH may affect dramatically chalk mechanical behaviour, increasing degradation phenomena and the risk associated to the occurrence of instabilities. It was then decided to investigate the retention properties of the chalk to check more in detail its state of saturation and its influence on the mechanical behaviour.

In this paper, preliminary results of oedometric tests on samples at various degree of water saturation are presented. Partial water saturation was imposed controlling the suction during the tests. It is shown that increasing water content (i.e. decreasing suction) decreases the yield stress and increases the time dependent sensitivity. Viscous behaviour under constant load (creep) and constant degree of saturation is also analysed. The examination of the experiments is proposed and results are discussed within the general framework adopted to describe the behaviour of unsaturated soils.

2. General framework

Chalk is considered as a multiphase geomaterial, and it is known from the mechanics of unsaturated soils that multiphase geomaterials behave quite differently as compared to geomaterials that contain only one fluid (saturated soils or porous rocks). In particular, they experience collapse when wetted under a constant load, indicating that an increasing proportion of wetting fluid, mainly located at the inter-granular contacts, soon causes failure.

Because pressure discontinuity through the interface between two immiscible fluids (e.g. air and water) gives rise to capillary pressure, strength loss due to wetting can be viewed as the consequence of the loss of capillary bonding between soil particles. Nonetheless, in clays for example, the chemical activity of the soil particles adds chemical fluid-mineral interactions. Neglecting the gravitational potential, the action of capillary and chemical interactions can be derived from the matric potential and the osmotic potential of the pore water, respectively. Both are part of the chemical potential μ_w of the pore fluid, its definition is derived from the Gibbs free energy (e.g. Sparks, 1998):

$$\mu_w = \mu_w^o + RT \ln a_w \qquad (1)$$

where μ_w is the chemical potential of the pore water solution, $\mu^o{}_w$ is the chemical potential of pure water, a_w is the activity of the pore water solution ($a_w = 1$ for pure water and $a_w \rightarrow 0$ increasing solutes concentration), T is the temperature (absolute Kelvin's scale) and R is the constant of the ideal gas (8.3143 $Jmol^{-1}K^{-1}$). When expressed in terms of pressure, the chemical potential is usually called total suction in unsaturated soil mechanics, and defined as

$$s = -\frac{\rho_w}{M_w} RT \ln a_w \qquad (2)$$

where the sign minus is introduced in order to ensure that s is strictly positive, ρ_w is the water density and M_w is the molecular weight of water (18 $gmol^{-1}$). For partially saturated states, equilibrium of pore water with its vapour phase allows to simplify in eq. (2) the expression of the activity coefficient a_w of pore water into the ratio between the partial vapour pressure p_w of the pore water and the partial vapour pressure p_{ow} of the pure water at a reference state ($a_w = p_w / p_{ow}$), this ratio defines the relative humidity RH (%) = $100a_w$. If we neglect the effect of dissolved solutes in pore water (accounted in the osmotic suction, s_o), the activity of pore water is only due to its pressure and the geometry of the pore space (i.e. matric suction s_m is equivalent to capillary bonding). Thus, total and matric air-water suctions are identical and are computed through

$$s = u_a - u_w = -\frac{\rho_w}{M_w} RT \ln(RH) \qquad (3)$$

where u_a is the air pressure and u_w is the water pressure.

To describe the mechanical behaviour of unsaturated soils the usual mono-tensorial approach based on the effective stress concept (Terzaghi, 1936) is no longer valid (e.g. Wheeler & Karube, 1996; Gens, 1996). Indeed, matric suction is considered as an independent stress variable (Coleman, 1962; Fredlund & Morgenstern, 1977), and is usually admitted to act as a pure hydrostatic stress tensor. The other possible stress variable is the net stress $\boldsymbol{\sigma} - u_a\mathbf{I}$, being $\boldsymbol{\sigma}$ the total stress tensor and $\mathbf{I}$ the unit tensor (both here are second order tensors). This hypothesis enables to account for hydromechanical coupling in the behavioural features of multiphase geomaterials. Its validity has been verified on multiphase reservoir chalks, where the non-wetting fluid is oil rather than air (Delage et al., 1996; Pasachalk1, 2001; De Gennaro et al., 2003 and 2004; Pasachalk2, 2004). Within this theoretical framework, based on equation (3), hydro-mechanical coupling associated to hygrometry changes (i.e. suction changes) has been considered has one of the main factors affecting the stability of the underground quarries in chalk. In this paper we discuss some of the experimental evidences corroborating the proposed general framework for chalks.

3. Tested chalk: characterisation and sample preparation

The chalk used in this study comes from Estreux quarry (north of France). Blocks of Estreux chalk were retrieved at 25 meters depth. At the temperature and the hygrometry inside the quarry, 11° C and nearly 100% of relative humidity, respectively, the chalk was almost completely water saturated (natural water content w = 20.7%). The SEM image and the MIP curves of Estreux chalk are presented in Fig. 1. SEM picture displays evidence of a clayey matrix (flat platelets) and microfossils (Fig. 1a). The clay fraction was identified as glauconite, which is often present in chalks from the north of France, giving a typical greenish colour (BLPC, 1973; Bonvallet, 1979). Probably due to the presence of microfossils, porosity of Estreux chalk is quite high, in the range 35 to 37 % (average void ratio e_o = 0.566). MIP curves identify a narrow family of pores, with an average pore entrance radius value in the range 500 to 800 nm (Fig. 1b). We note a certain scatter in the values of the intruded volume at higher pore entrance radii (> 2000 nm) which seems likely to depend on the natural variation of the porosity, and is amplified by the reduced dimension of MIP samples.

Samples used for laboratory tests were cored from a unique block (same orientation) and then resized on a lathe. For water saturated samples this procedure was performed as much quick as possible, in order to avoid evaporation, adding water if necessary. Samples of about 38 mm diameter and 20 mm high were used for the oedometer tests (excepted one test on dry chalk, performed on a sample 50 mm in diameter). For the retention tests, samples had a diameter of about 20 mm and their height ranged between 20 and 25 mm. Dry samples were obtained after a heating period of 24 hours at 105° C.

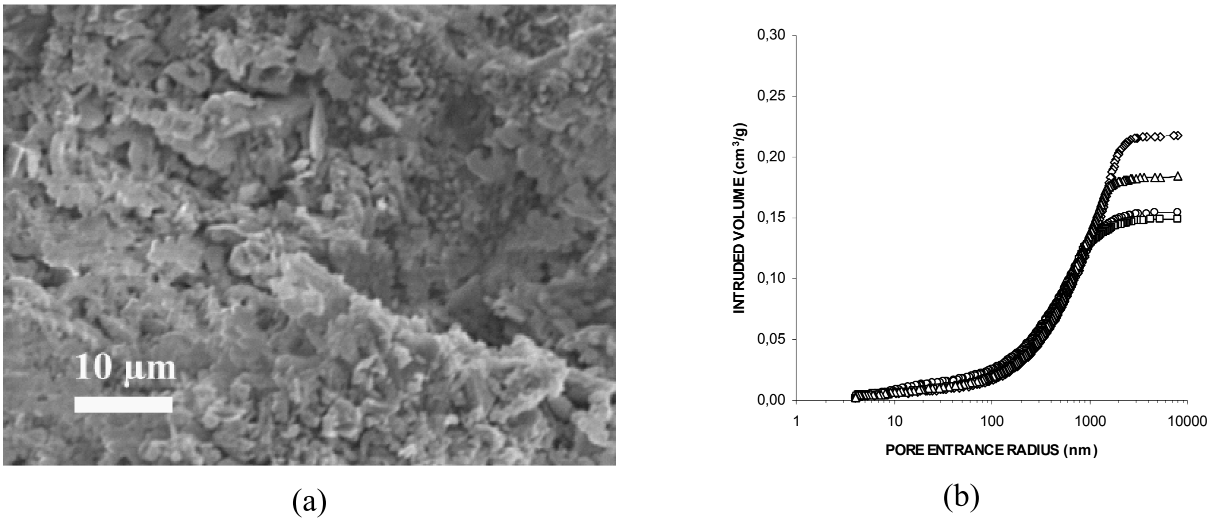


Figure 1. (a) SEM picture of Estreux chalk, (b) Mercury Intrusion Porosimetry curves.

4. Experimental techniques and apparatuses

Due to the key role played by suction on the behaviour of chalk, experimentation required a set of testing procedures allowing suction control. The two methods used were: (i) the osmotic technique, (ii) the vapour phase control.

4.1. The osmotic technique

The osmotic technique (Williams and Shaykewich, 1969; Kassiff & Benshalom, 1971; Delage et al., 1992; Dineen & Burland, 1995) allows to control suction by osmosis through a regenerated cellulose semi-permeable membrane behind which a solution at a given concentration of large molecules of polyethylene glycol (PEG) is circulated. The concentration of PEG solution define the corresponding value of suction applied, obtained by previous calibration. To apply the desired suction during experiments, apparatuses should allow the contact of the chalk sample with the semi-permeable membrane and the PEG solution.

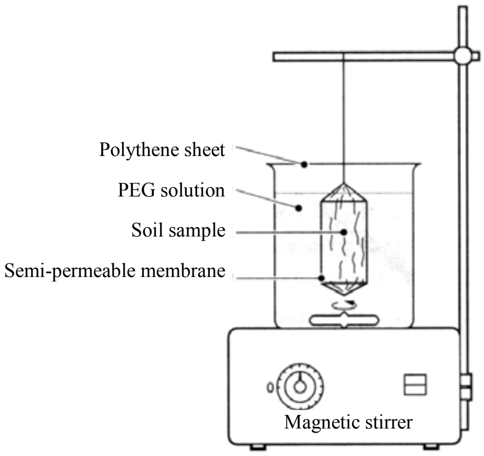


Figure 2. Experimental setup used to control suction by osmotic technique (Cui & Delage, 1995).

The layout of the experimental setup used to apply the desired suction level to chalk samples during water retention tests is shown in Fig. 2. The method (Cui & Delage 1995, De Gennaro et al. 2003) consists in inserting the chalk sample in tube-shaped semi-permeable membrane and in plunging it into a container full of PEG solution placed on a magnetic stirrer. For the determination of the imbibition curve, dry samples were inserted. Due to its wetting properties, water could infiltrate chalk under a controlled suction and expels a given volume of air. In the case of the drainage curve, the samples were saturated with water before being inserted. Due to the higher suction imposed by the concentred PEG solution, the pore water is expelled from the porous network of the chalk, crosses the membrane and is released into the PEG solution. In both cases the fluid transfer come to an end when the osmotic suction imposed by the solution and the matric suction of the sample are at equilibrium.

4.2. The vapour phase control

The vapour phase control allows total suction control by means of PEG solutions (Fig. 3a) or saturated salt solutions (Fig. 3b). Suction in this case is controlled by the ambience relative humidity imposed by the solution, taking care that isothermal condition is fulfilled. During all the retention tests presented in this paper a temperature of 20° C ± 0.5° C was ensured. The use of PEG or saline solutions depends on the level of desired suction. In this study PEG solutions were used for total suction levels lower than 2.5 MPa (RH = 98.2%), the minimum value of total suction attained by saturated saline solutions being 4.2 MPa (RH = 97% with K_2SO_4 salt).

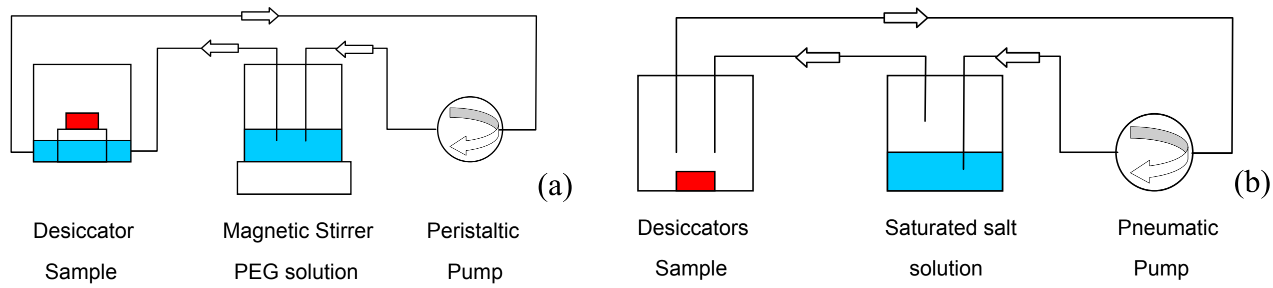


Figure 3. Vapour phase control: (a) using PEG solution, (b) using saturated salt solutions.

4.3. The suction controlled oedometer

One sample in this study was subjected to a mechanical test under suction controlled condition. This type of test was performed in the osmotic oedometer (Fig. 4). During oedometric tests chalk samples were subjected to incremental loading (applied instantaneously) and the evolution of the vertical strain during each loading stage was measured. This procedure is well established in soil mechanics. The value of the vertical strain is defined as the strain corresponding to the effective stress state (in saturated soil), i.e. the state of stress for which the excess pore fluid pressure is zero (end of primary consolidation). In a multiphase chalk, excess pore pressures of water during consolidation could modify the suction. Then, the actual vertical strain value should correspond to the condition of equilibrium of suction. In this device suction is controlled via the osmotic technique. The bottom of the sample is in contact with a semi-permeable membrane below which a solution of PEG is circulated (Kassiff & Benshalom, 1971; Delage et al., 1992). In order to control the suction level, water exchanges through the membrane were monitored through visual observation of the water level in a graduated tube placed in the bottle that contains the PEG solution.

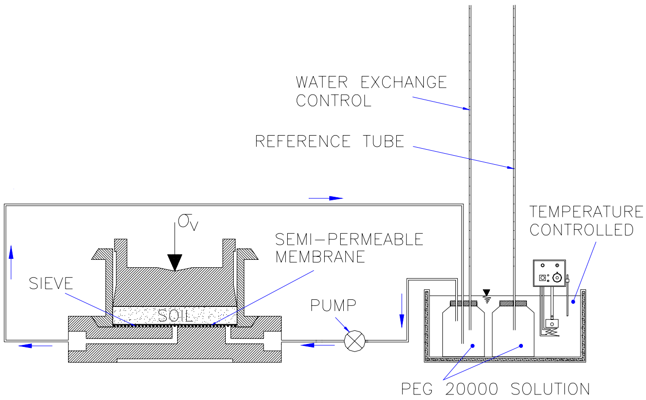


Figure 4. The osmotic oedometer.

In order to reach the desired net vertical stress, the sample was loaded using a high pressure oedometer frame with a double lever arm that allowed a maximum vertical stress of approximately 60 MPa on a sample 50 mm in diameter. At each loading stage, the stabilisation of the PEG level in the tube was checked.

5. Retention properties

The retention properties of Estreux chalk define the relationship between the degree of saturation S_{rw} and the suction s (the retention curve) in an air-water saturated chalk sample. Depending on the driving fluid used to modify the relative saturation of the sample, two curves can be established, following an imbibition path (water driving) or a drainage path (air driving). Retention curves have been obtained using one of the two techniques of suction control described previously.

MIP data were also used to obtain the retention curve following a drainage path. In fact, during mercury penetration the imposed pressure p_c is equal to the capillary pressure given by Laplace's equation : $p_c = (2\sigma\cos\theta)/r$, where σ is the surface tension, θ the contact angle between mercury and chalk (σ = 0.482 N/m and θ = 147°, respectively) and r is the pore entrance radius. The values of surface tension and contact angle in Laplace's equation for chalk in contact with water and air are σ_{aw} = 0.072 N/m and θ_{aw} = 0°. Using Laplace's equation, and imposing the equality of the pore entrance radius, air-water suction s can be derived knowing p_c values from MIP as follows

$$s = u_a - u_w = p_c \frac{\sigma_{aw}\cos\theta_{aw}}{\sigma\cos\theta} \quad (4)$$

Obviously this relation is valid admitting only capillary interaction between chalk, air and water. Retention curves of Estreux chalk are presented in Fig. 5. It can be seen that a suction level of 1.5 MPa is necessary to expel water and reduce the chalk water saturation degree following a drainage path. This value of suction is also called "air entry value". It is straightforward to observe that retention curve following a drainage path obtained using osmotic or vapour phase technique, give by far higher values of suction than the same curve derived from MIP data using Laplace's equation (i.e. admitting only capillary effects). It follows that air-water interactions of different nature probably exist in this chalk. These interactions are likely to be the consequence of the activity of the clayey matrix of Estreux chalk and may involve other physico-chemical processes (i.e. adsorption, dissolution, etc.). It is believed that the knowledge of the extent of these interactions is of great importance in defining the macroscopic mechanical response of this rock.

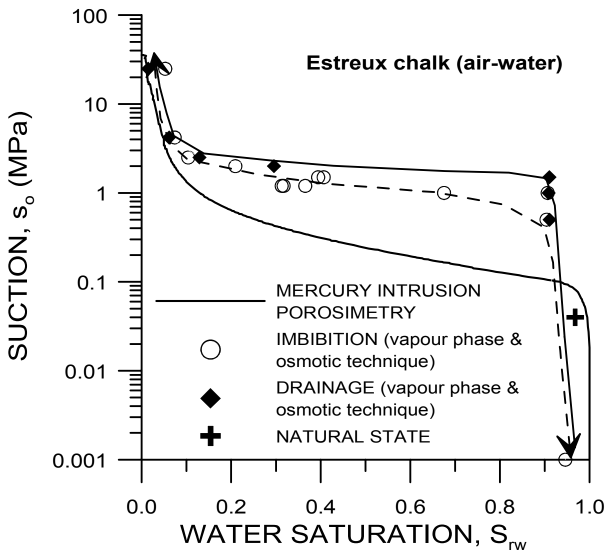


Figure 5. Retention curves of Estreux chalk.

Another indicator supporting the hypothesis of the existence of possible physico-chemical interactions is the small hysteresis observed in Fig. 5 between the imbibition path and the drainage path. In fact, significant hysteretic behaviour is often associated to capillary interaction and ink-bottle effect, leading to the entrapment of a given amount of water in the porous network, whereas physico-chemical interactions often produce reduced hysteresis. Note that, based on eq. (3), 1.5 MPa suction corresponds to a relative humidity of 99.8%.

Available measurements of the initial suction of Estreux chalk (at natural state) by filter paper (Fig. 5), gave values of about 40 kPa, in good agreement with the almost fully saturated state of the material. This corroborates also the in situ measurements of RH, indicating almost complete water vapour saturation at the time of the sampling. It is also interesting to observe that the residual water saturation is attained for suction values between 4.2 and 24.9 MPa. The former corresponds to RH = 97% (with K_2SO_4 salt), the latter to RH = 83,5% (with $(NH_4)_2SO_4$ salt). Thus, changes of few percentage points in RH may drastically reduce the relative saturation of the rock. Also, available measurements in the quarry gave minimum RH value of about 80%, which is sufficient, based on the retention curves plotted in Fig. 5, to attain almost complete de-saturation. Thus, the natural evolution of the relative humidity inside the quarry can lead to the two extreme saturation states (S_{rw} = 100% and S_{rw} = 5%), with changes in suction values between 0 and 24.9 MPa (i.e. the full range of attainable suction values). Consequently, due to the hydromechanical coupling, suction changes due to relative humidity could act as an additional load on the rock pillars and may affect, at long term, the stability of the underground quarry. The effects of these changes on material strength are quantified in the next section by means of suction controlled eodometric tests.

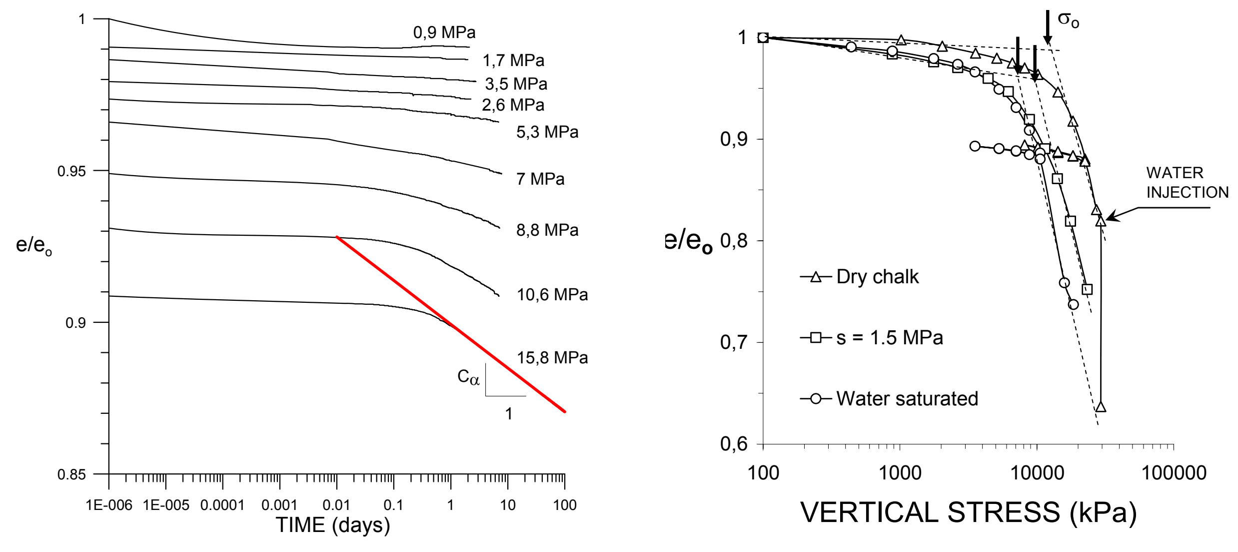
6. Results of oedometric tests

6.1. Compressibility curves

Figures 6 shows the results of three compression tests performed on chalk samples having various amounts of water: water saturated, dry and partially saturated under 1.5 MPa of controlled suction. The water saturation degrees (S_{rw}) of the samples and the corresponding level of suction can be estimated from the retention curve of Fig. 5. Thus, the water saturated sample has s = 0 MPa, the dry sample has s > 24.9 MPa and at 1.5 MPa of suction the chalk has $S_{rw} \cong 90\%$ (i.e. is almost water saturated).

The analysis of the consolidation curves (Fig. 6a) for the water saturated sample, using the usual graphical methods employed in soil mechanics, did not permit to identify any significant point where changes in excess pore fluid pressure were apparent. Indeed, these methods seem not reliable for chalk, which is characterised by consolidation curves showing continuous vertical strain of the sample as soon as the loading is applied. Because of the order of magnitude of the permeability in this soft rock is quite high (about 1×10^{-8} m/s) and the compressibility of the soil skeleton quite low (due to inter-granular bonding), it is believed that after 24 hours of consolidation any excess pore fluid pressure is reasonably dissipated. This observation is in agreement with the analysis proposed by Lade and de Boer (1997), about the concept of effective stress in soil and rocks, and the general results of poroelasticity (Biot, 1941; Rice & Cleary, 1976; Coussy, 1995), showing that in a porous medium, the amount of the applied total load transferred instantaneously to pore water pressure increases when the drained bulk modulus decreases. Thus, it seems likely that the reduced compressibility and the rather high permeability are sufficient in chalk to prevent excess pore fluid pressure generation during instantaneous loading (i.e. stepwise oedometric loading). If this holds true, the delayed volumetric strain of chalk, as observed during oedometric tests (Fig. 6a), should be essentially due to creep and the solid skeleton settles continuously, although the excess pore pressure is zero. This result should be corroborated by pore pressure measurements during applied loading.

In order to reproduce the compressibility curves for Estreux chalk, the time of consolidation was 48 hours for stress levels lower than σ_o (conventional yield stress, as defined in Fig. 6b), and it was extended to seven days for loading steps in the plastic regime. The rational behind this choice was to have an insight into the creep behaviour of the rock. The compressibility curves of the chalk show clearly strength dependency on suction. It is interesting to observe that since the imposed suction level in Estreux chalk gives a water saturation degree that is close to the fully water saturated state (Fig. 5), compressibility curve under suction control at 1.5 MPa is also close to that obtained on a fully water saturated sample (Fig. 6b).



(a) (b)

Figure 6. Results of oedometer tests : (a) Consolidation curves, (b) compressibility curves.

Nonetheless, the effect of suction is present, and it can be observed that the yield stress σ_o (the stress at which rock compressibility notably raises) increases with increasing suction, i.e. with lower degrees of water saturation.

Figure 6b also shows the collapse phenomenon of a dry chalk due to water imbibition. This was obtained by water injection under a vertical load of 29.3 MPa. In this test, the initial air-water suction (certainly higher than 24.9 MPa) was reduced to zero. Thus, based on the results shown in Fig. 5, the initial degree of water saturation of the dry sample, certainly less than 5%, was increased to the final value of 100% under zero suction. Water was infiltrated from the bottom to the top of the chalk sample, imposing few tens of centimetres of pressure head. The final point in Fig. 6b has been obtained after twelve days of continuous infiltration, and defines a total decrease of 0.183 in e/e_o ratio (vertical path). This variation of the void ratio corresponds to a compressive volumetric strain of about 6.6%. This collapse is significant, at the end of the process the final point is on the compression curve of the saturated chalk (Fig. 6b), in agreement with the double oedometer approach proposed by Jennings & Knight (1957) to assess the collapse susceptibility in unsaturated soils. The reduced amount of water menisci at the dry state doesn't seem to justify the magnitude of this breakdown as the consequence of the capillary loss solely. Consequently, it seems likely that the inter-granular links have a significant physico-chemical component, and are progressively weakened by the infiltration of water at the level of the inter-grain contacts. Pressure solution creep (Hellmann et al. 2002) is often mentioned to account for physico-chemical processes associated to rock weathering. This type of water-rock interaction bears a number of common features with the phenomena associated to suction. This point should certainly be the object of further researches, in order to better quantify the basic mechanisms at the origin of the chalk deformation.

The results presented so far are compatible with the water weakening effect in chalks, extending this effect to the partial saturation in both fluids under a controlled suction.

6.2. Viscous behaviour

Moving from the results presented in Fig. 6a, the analysis of the creep behaviour of Estreux chalk has been conducted quantifying the creep rate developing during each of the loading steps under oedometric conditions. The examination of the curves presented in Fig. 6a for the water saturated sample, plotting the evolution of the ratio e/e_o as a function of the logarithm of time, permits to define the classical creep coefficient C_α given by

$$\frac{de}{e_{oi}} = C_{\alpha i} d(\log t) \qquad (5)$$

where e_{oi} is the void ratio at the beginning of loading stage i, $C_{\alpha i}$ is the slope of the consolidation curve vs time at long-term (Fig. 6a). Since equation (5) is valid for each of the hydraulic configuration, $C_{\alpha i}$ coefficients were assessed for all the loading stages of the three tests in Fig. 6b. Results are summarised in Fig. 7, plotting the evolution of C_α as a function of the applied vertical stress for all the tests. It is straightforward to note that both suction and vertical stress influence the creep rate. The creep rate varies linearly with the applied vertical stress, while it decreases when the level of suction increases, moving from a fully saturated to a completely dry environment. Similar results are in agreement with findings by Alonso et al. (2005) on the creep behaviour of partially saturated rockfill.

Note that, since suction influences the yield stress of the material, it is very likely that the influence of suction on creep could be reconducted to the similar effect on yield stress, assuming a normalizing factor for the effect of suction. This is the object of ongoing studies.

Parameter C_α describes adequately the viscous behaviour of Estreux chalk. Indeed, changes in creep rate due to suction (i.e. relative humidity) and load changes are easily accounted for when considering the results shown in Fig. 7. It is believed that these preliminary results can be useful for both practical applications, such as the assessment of the long-term stability of chalk pillars under various mechanical and environmental conditions, and theoretical developments (modelling approaches).

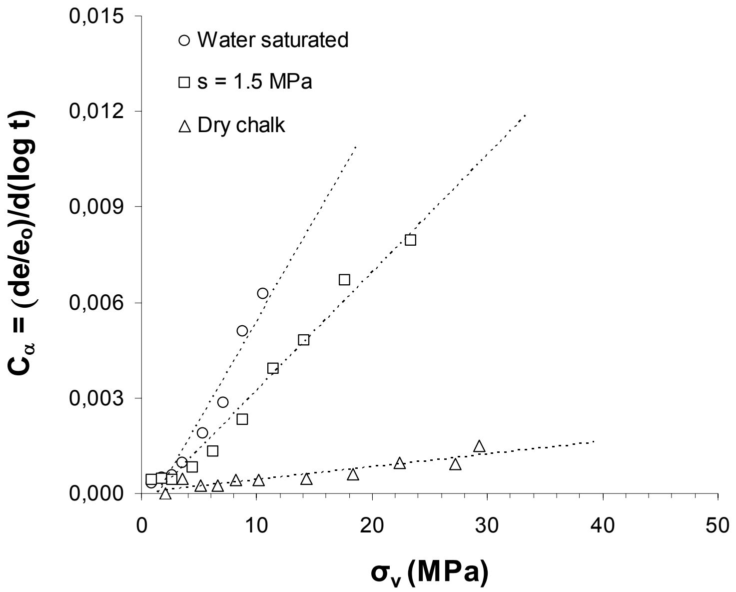


Figure 7. Evolution of creep parameter Cα vs vertical stress for various hydraulic configurations.

7. Conclusions

The experimental data presented in this paper are aimed at showing some effect of suction on the compressibility and the long term behaviour of a quarry chalk.

The role and the importance of the retention properties and their knowledge are emphasised, in order to better understand the coupled hydromechanical behaviour of partially saturated chalks. Compressibility and time-dependent behaviour have been studied in close relation with the influence of air-water suction. It has been shown that a number of common behavioural features exist between unsaturated soils and partially saturated chalks. Chalk is made stronger by increasing suction levels, which in turns means decreasing water contents. Its long-term deformation has consistently been related to the hygrometry changes by means of suction. The water weakening effect in chalk is considered here in a more comprehensive framework, introducing the air-water suction as an independent stress variable capable to account for the water-rock interactions.

8. Acknowledgements

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