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An Insight into the Fluid Retention Capabilities of Shales

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SUMMARY

The hydro-mechanical behaviour of shales is becoming one of the most important issues in modern geomechanics, largely driven by petroleum industries (i.e., the extraction of shale gas), the sequestration of CO₂ and the nuclear waste geological storage. In any such application, a deep understanding of the hydro-mechanical behaviour of the involved materials is of primary significance. The water retention mechanisms play a major role in either fluid trapping due to the capillary forces present in low permeability formations or in the resaturation of shale formations after desaturation. The paper presents different experimental methodologies that have been developed by the authors for the analysis of the retention behaviour of shales. They involve the direct control of the shale water content and the subsequent measurement of the suction at equilibrium by a psychrometer. A fluid displacement technique with a non-polar liquid is then used to assess the volume changes and compute the degree of saturation. Selected test results are presented for two shales from the northern region of Switzerland.

Introduction

Growing interest toward the geomechanical behaviour of shales is nowadays observed mainly due to the fields of application in which this material has been involved in the recent years such as petroleum industries, nuclear waste storage and sequestration of CO₂. In any such application, a deep understanding of the hydro-mechanical behaviour of shales is of primary significance.

The water retention mechanisms play a major role in either fluid trapping due to the capillary forces present in low permeability formations, as in the case of shale gas reservoir, or in the resaturation of shale formations after ventilation, as in the case of deep geological repositories. This paper presents an advanced experimental technique that have been recently developed by the authors for testing the water retention behaviour of shales as well as the main obtained results on 2 different shales. Different testing procedures are conceived for the determination of the wetting and of the drying paths of the shale water retention curves; the methodologies are based on the direct control of the shale water content and on the subsequent measurement of the total suction at equilibrium by a psychrometer. The swelling/shrinkage of shale is related to suction, or the degree of saturation variations; in order to investigate the volume change response of the material upon total suction variations a fluid displacement technique with a non-polar liquid is used. The technique allows to assess the changes in void ratio of the shale specimens and to compute the degree of saturation during drying and wetting episodes.

Tested shales

The two Mesozoic shales considered in the present study come from the northern region of Switzerland: the Opalinus Clay sample (OPA) was obtained from the Mont Terri Underground Laboratory and the “Brown Dogger”, from a deep geothermal well (depth of 766.67 and 778.30 m) near the village of Schlattigen in the Molasse Basin. The tested OPA was recovered in the Gallery 98 of the Mont Terri URL from the shaly facies, consisting of dark grey silty, calcareous shales with a typical clay content of 55-60%, 25-30% carbonate, 5-10 % quartz and 10-15% biotritus. The core samples of “Brown Dogger” (BD) originated from the Varian- and Parkinsoni-Würtembergica beds (Bathonian/Bajocian age), consisting of silty to clay-rich marls with a clay content of 25-45%, 30-50% carbonate and 20-25% quartz [1].

The geotechnical characterization consists in the determination of the particle density (ρ_s), the bulk density (ρ), the water content (w), the void ratio (e), the degree of saturation (S_r) and the Atterberg limits (the liquid limit w_L and the plastic limit w_P). The results of the geotechnical identification of the cores are reported in Table 1.

Table 1 Geotechnical characterization of the tested shales.

Shale	OPA	BD
ρ_s (Mg/m ³)	2.74	2.72
ρ (Mg/m ³)	2.46	2.55
w (%)	6.9	2.7 - 3.5
e (-)	0.21	0.09 - 0.11
S_r (%)	92	78 - 92
w_L (%)	38	25 - 29
w_P (%)	23	23

The pore size density (PSD) function of the tested shale cores is determined through the Mercury Intrusion Porosimetry (MIP); this technique consists in forcing the penetration of mercury into a sample and in measuring the intruded volume of mercury as a function of the applied pressure. The required pressure is inversely proportional to the size of the progressively filled pores.

The obtained results are presented in Figure 2. The PSD functions are found to be unimodal for both tested shales; they show pore diameter modes at approximately 20 nm and 15 nm for the OPA and the BD samples, respectively.

Figure 1 Pore size density function of the tested shales.

Testing methodologies

This section describes the experimental methodologies established to analyse the water retention behaviour of shales under unstressed conditions. Synthetic waters are used with the aim to reproduce the in-situ pore water composition [2, 3]. The osmotic suctions of the synthetic waters are measured by a dew-point psychrometer and result in values of 1.2 MPa for the OPA sample and 0.98 MPa for the BD sample.

Initially, slices measuring 7 – 8 mm in height are obtained from the shale core; they are divided into different specimens of about 2x2x1 cm³ and three specimens are immediately tested by means of a dew-point psychrometer (Decagon, WP4c) [4, 5]. After the determination of the total suction, the volume of the specimens is measured through a technique based on the fluid displacement concept. The technique uses pycnometers filled with kerdane; the latter is adopted because of its immiscibility with water and its ability to invade the air-filled pore spaces in the surface without affecting the soil structure [6]. Once the above procedure is performed, the initial water content, void ratio and degree of saturation can be evaluated thus determining the initial conditions of the tested shales. The following step consists in the determination of the main wetting and drying paths. The remaining shale specimens obtained from the core slices, are brought either to a complete saturated state or to a completely dry state thus reaching the initial point required for the determination of the main drying path and main wetting path respectively. Subsequently, target water contents are imposed using different techniques for the wetting and drying paths.

The drying path is obtained after the complete saturation of the shale specimens: the specimens are initially saturated by capillary action, placing them in a sealed-glass jar on filter papers, which in turn are placed on a porous stone filled by synthetic water. Volume changes of the specimens are allowed. The equilibrium is assessed by monitoring the weight evolution over time. Each part is then placed in a desiccator and dried for the amount of time necessary to achieve the desired water content. Subsequently, the specimens are packed hermetically to allow internal redistribution of humidity before the measurement of the total suction. Finally the specimen volume is measured.

The main wetting path is determined using shale specimens which are brought to a completely dry state: they are initially placed in a desiccator containing silica gel and left to dry until no significant change in their weight can be detected. The volume change is allowed during the drying. After the equalization, the target water content is applied using a syringe in order to have a better control on the mass of the added water. The time for the internal redistribution of the humidity is allowed and the total suction of each specimen is thus measured using the WP4c dew-point psychrometer. Finally the volume of the specimen is determined using the fluid displacement technique described above, and the water content, void ratio and degree of saturation can be evaluated.

Results

The water retention behaviour of the OPA and the BD core samples are reported in Figures 2 and 3, respectively. The results are depicted in terms of the water content, the void ratio and the degree of saturation versus the total suction.

Figure 2 Water retention behaviour of the Opalinus Clay core sample: the water content (a), the void ratio (b) and the degree of saturation (c) as a function of the total suction.

Both the considered materials appear to have an initial degree of saturation close to one, this observation is in agreement with the fact that both materials presented saturated in-situ conditions. The initial state is found for both the materials along the main drying paths; this observation is related to the effect of the desaturation which is a consequence of the coring process and of the exposure to atmosphere before preservation. The main drying and wetting paths are well distinguished, and hysteresis zones can be observed. The evolution of the void ratios with the total suction shows that the porosity changes are more significant for the lowest range suction variations. The void ratio corresponding to the lowest measured total suctions are in very good agreement with the results of the free swelling tests which have been carried out with the same synthetic waters used for determining the water retention curves. The shrinkage limits (assumed to be defined by $\Delta e/\Delta \psi < 0.1\%$) are identified at suction values of approximately 10 MPa for the OPA and 15 MPa for the BD. The corresponding water content values read along the main drying paths are 6.4% and 4.0% for the OPA and the BD, respectively.

Figure 3 Water retention behaviour of the Brown Dogger core sample: the water content (a), the void ratio (b) and the degree of saturation as a function of the total suction.

Conclusions

The abstract presents the experimental technique that has been developed for the analysis of the water retention behaviour of shales. Selected experimental results for two shales from the northern region of Switzerland have been analysed. The testing procedure for the determination of the water retention curves of shales consists in controlling the water content by the addition of synthetic water or by the dessication of the material in a desiccator. Total suction measurements are performed through the use of a dew-point psychrometer. After every suction measurement, the sample volume changes along the wetting and drying paths are systematically assessed; thus the computation of the degree of saturation and the evaluation of the shrinkage limit of each material can be achieved. The proposed method is proved to be suitable for describing the water retention behaviour along the main drying and wetting paths and for highlighting the hysteretic aspect of the retention behaviour of shales.

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