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## Experimental analysis of the water retention behaviour of shales

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### ABSTRACT

Shales are among the most commonly considered geomaterials in current energy-related geomechanical investigations, as they are involved in engineering applications such as the extraction of natural gas, CO<sub>2</sub> sequestration and nuclear waste storage. A deep understanding of their behaviour with regard to variations in the degree of saturation is of significance for such applications. With the aim to establish a sound protocol for shale retention behaviour analysis, this paper presents an experimental methodology that takes into account the specificities of shales such as small pore size dimensions and the large variations in water potential to be applied to induce significant changes in the degree of saturation. The technique involves 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 also used to assess the volume changes of the shale and to compute the degree of saturation. Selected test results are presented for various shale samples that have been cored at different depths. The results indicate that important features of the retention behaviour of geomaterials are fundamental aspects to be considered when analysing the retention behaviour of shales, such as the existence of main wetting and drying paths, the hysteresis domain, and the dependency of the air entry value on the void ratio.

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### 1. Introduction

The development of engineering activities involving shales such as the extraction of shale gas and shale oil, the geological storage of nuclear waste and CO<sub>2</sub> sequestration, has led to an increasing interest in the geomechanical behaviour of these geomaterials. In the context of such engineering applications, a deep understanding of shale retention behaviour is of primary significance. Water retention mechanisms play a major role in fluid trapping due to the capillary forces in the two-phase flows in gas reservoirs as well as in the resaturation of shale formations after ventilation, as in the case of deep geological repositories. Wetting and drying episodes also have significant impacts on shale volumetric behaviour: swelling/shrinkage of shales may be caused by changes in suction or variations in the degree of saturation; additionally, significant swelling pressures may develop when volumetric expansion upon wetting is prevented.

In general, the water retention behaviour of geomaterials is expressed as a relationship between the pore water potential and the amount of water stored in the material; water retention curves represent this relationship. Suction is often used as the variable

that expresses pore liquid chemical potential. Matric suction ( $s$ ) is the component of the potential that accounts for the effects related to the presence of the matrix (capillarity, osmotic mechanism and electrostatic forces) [1]. Osmotic, or solute suction ( $\pi$ ) expresses the component of the potential associated with the solute concentration in the aqueous solution that constitutes the pore liquid. Total suction ( $\psi$ ) is the sum of these two components and is directly related to the relative humidity and temperature established in the pore gaseous phase. The amount of water is quantified by the gravimetric water content (the ratio of the weight of the water to the weight of the solid phase,  $w$ ) or the degree of saturation (the ratio of the water volume to the pore volume,  $S_r$ ).

The main features of water retention behaviour are found in the existence of a main wetting path (starting from a dry condition) and a main drying path (starting from a saturated condition), which, for a given density, embrace all the possible hydraulic states of the material. Any hydraulic path that moves from a main drying curve to a main wetting curve (or vice versa) takes place inside this hysteresis domain and is defined as a scanning curve.

The determination of water retention curves is becoming a well-established practice for the characterisation of unsaturated soils. For clayey materials, the characteristics that are usually quantified are the air entry value (value of suction at which the material starts to desaturate in a main drying episode) and its

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dependency on the void ratio [2], the residual degree of saturation and the hysteresis upon wetting and drying cycling [3]. For compacted clays, attention has also been paid to the effects of the evolution of the microstructure on retention behaviour [4,5]. Numerical models have been developed to reproduce the retention behaviour that accounts for these highlighted features [5–8].

In spite of this information on unsaturated soil retention behaviour, knowledge on shale retention behaviour is still limited. Indeed, the strength and stiffness of shales are significantly influenced by the degree of saturation or, alternatively, by the suction, as highlighted by Valès et al. [9] for the case of Tournemire shale (France). Ramos da Silva et al. [10] reported a reduction in the UCS for Beringen shale (Belgium) to a fifth of the original value when the suction was decreased from 50 to 10 MPa.

Despite these recognised impacts, few experimental studies have been conducted to evaluate shale water retention properties in a comprehensive way. The limitation of the acquired information is even more evident when compared to the available experimental evidence on the retention behaviour of low-porosity compacted clays [3,11] and natural clays [12–15]. Schmitt et al. [16] presented data on the retention behaviour of Tournemire shale (France), which was obtained from porosimetry tests along with data on two different sandstones highlighting the higher suction range that is needed for the shale in order to observe appreciable variations in the degree of saturation. Ramos da Silva et al. [10] investigated the water retention curve for Beringen shale along drying paths and compared it to the curve for Lixhe chalk, asserting that suction of a magnitude greater by two orders was needed for the shale to dry the materials at a 90% degree of saturation. Information on the water retention behaviour of the Opalinus Clay shale (Switzerland) has been also provided. Muñoz [17] presented data on the evolution of the water content and the degree of saturation with total suction. Zhang et al. [18] compared the water retention curves of Opalinus Clay shale in free and constrained volume conditions and discovered that there is a significant increase in the water amount that can be stored in the material when it is allowed to swell during the wetting phase. Villar and Romero [19] determined the retention curve for Opalinus Clay shale in terms of matric and total suction for free and confined volume conditions; they reported air entry values in the range of 9–21 MPa for free volume conditions and 15–35 MPa for confined conditions. Romero et al. [20] reported values of the air entry value of Opalinus Clay shale at different depths to be equal to 13 and 18 MPa, as obtained from mercury intrusion porosimetry. From the analysis of the available data, it appears that no investigation has been conducted to compose a comprehensive picture, containing all relevant features, of shale retention behaviour. In particular, to the authors' knowledge, the retention behaviour along scanning paths has not been experimentally investigated. This paper aims to provide a robust and complete experimental framework to analyse shale retention properties, highlighting and quantifying all the features of retention behaviour, such as the air entry value and its dependency on the void ratio, the hysteresis phenomena and the volumetric response to suction changes.

Total suction has been selected for this study as the suction component for describing shale retention behaviour. This choice is justified, considering that shales have high air entry pressures (several MPa) and that a wide range of suction values need to be taken into consideration to observe significant changes in the degree of saturation. Indeed, typical values of dominant pore sizes for shales are in the range of nanometers or dozens of nanometers (e.g., 20 nm for the Pierre shale [21]); the Young–Laplace equation would allow to compute that matric suction values in the order of dozens of MPa (approximately 7 MPa for a pore size of 20 nm) would be required in order to remove the water from pores with characteristic size in this range. On the other hand, most techniques for controlling and measuring matric suction are limited to a

maximum suction of 1.5 MPa [22]. However, it is a common practice in petroleum engineering to gather information on the relationship between matric suction and degree of saturation by the analysis of porosimetry tests [21]. In order to compare results in terms of matric and total suction, the osmotic component must be assessed; this aspect has been poorly addressed for shales. Laloui et al. [12] reported values of 12.13 MPa and 17.3 MPa for the matric and total suction, respectively, for a sandy facies Opalinus Clay shale at a 74% degree of saturation. When comparing the water retention curves in terms of total and matric suction for Opalinus clay shales, Villar and Romero [19] pointed out that no clear difference is observed between the two curves; however, the scattering of the data did not seem to allow for a precise assessment of the osmotic component.

In the following section, the developed experimental protocol is described first. We then present and analyse selected results from shales from the northern region of Switzerland in light of the proposed experimental methodology. Complementary information is gained from the analysis of the porosimetry tests. The assessment of the osmotic suction component is performed by independently measuring the total and the matric suction; these measurements are used to explain the difference between the water retention curve that was obtained using the porosimetry technique and the methodology that is proposed in this paper.

## 2. An experimental technique to investigate the water retention behaviour of shales

The developed experimental technique for determining shale retention behaviour is based on the direct control of the water content and the subsequent measurement of the total suction. Different hydraulic paths are considered in order to analyse the response of the material to wetting and drying episodes. The following sections provide details on the specimen preparation, water content control, suction measurement and volume change detection.

### 2.1. Specimen preparation and water content control

The material is obtained from a shale core: slices of approximately 7–8 mm in height are cut with a diamond saw without using water, and they are immediately divided into smaller specimens with lateral sizes of approximately 20 mm. Three specimens are immediately tested to obtain the initial condition in terms of water content, void ratio and total suction, using the procedure detailed in the next sections. The initial measured void ratio is compared with the void ratio of the core in order to assess the quality of the obtained specimens. The main wetting and drying paths are sought first; to this aim the material is initially brought to a dry state or to a “zero-matric-suction” state, respectively. The dry state is obtained by placing the specimens in desiccators that contain silica gel and leaving them to dry for approximately three weeks (applied total suction of approximately 300 MPa) until no significant change in their weight can be detected. The “zero-matric-suction” condition is achieved by placing the specimens in a sealed-glass jar, within which a relative humidity of 100% is imposed; the specimens are wrapped in filter paper and rest on porous stones immersed in water; synthetic water (corresponding to the in-situ water composition) may be used for this initial wetting stage in order to preserve the initial osmotic suction of the material. The weight of the specimens is monitored using a precision balance (0.001 g), and the process is stopped once no further evolution is observed (usually in three weeks).

Once equilibration is achieved, the main wetting and drying paths can be determined. To obtain the main drying path, the equalized

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