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Evolution of water content and suction of Opalinus Clay from recovery at the drilling site to handling in the laboratory

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ABSTRACT

Advanced geotechnical engineering applications, such as shale gas extraction, CO_2 geological sequestration, and geological radioactive waste storage, often involve various types of shales located at significant depths. Shales exhibit mechanical properties that are highly influenced by their hydration state and are exposed to substantial stress relief during extraction from considerable depths. This results in the development of elevated total suction (free energy per unit volume of pore water). While water content measurements are conventionally employed for characterizing these materials, ongoing discussions and uncertainties persist regarding the relevance and representativeness of laboratory suction measurements, particularly in light of potential influences stemming from core extraction and conditioning processes. A recent extensive borehole drilling campaign has provided a unique opportunity to offer scientific insights into specimens of Opalinus Clay shale, extracted from various locations and depths. These specimens were examined in their freshly extracted on-site condition and their freshly opened condition in the laboratory. Notably, it has been observed that suction and water content measurements acquired on-site immediately after core extraction differ from those obtained in the laboratory. The evolution of suction and water content from the field to the laboratory is closely linked to the main drying water retention behavior of the geomaterial.

1. Introduction

In-situ core sampling involves a change in the total stress (from the in-situ value to zero) with simultaneous changes in the hydraulic boundary conditions. Due to their low permeability,[1–3] the unloading process in shales (typically extracted at a rate in the order of dozens of meters per day) can be usually considered undrained. Undrained unloading can induce negative pore water pressures in the extracted samples[4–9]; equivalently samples can have positive suctions. The forthcoming changes in water content and suction depend on the mechanical and hydraulic boundary conditions to which the material is subjected after extraction, resulting from the applied core sample preservation technique, and all activities after opening the cores. Ad-hoc protocols are necessary to ensure that specimens produced from core samples are tested under conditions that are relevant to the problem under investigation. For these reasons, in-situ core sampling, transportation to the laboratory, and laboratory activities (i.e., specimen preparation, storage, and testing) are designed to minimize disturbances of the geomaterial (e.g., changes in volume, and water content); the initial effective stress of the tested specimens (to be assessed by adopting a suitable effective stress concept[10]) in the experiments should be as close as possible to the in-situ one. Specific guidelines for core handling, preservation, sampling, and testing of shales are available e.g.,.[3,11–14] For instance, shales that are saturated in their natural state and under suction after specimen preparation, have to be subjected to positive pore water pressure trying to minimize the development of enhanced porosity (see methods proposed in the literature, such as the ones discussed in[1,3,13–17]).

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Literature[1,18,19] has highlighted discrepancies between in-situ and laboratory-measured water contents, as well as variations in measurements conducted within the laboratory itself. These disparities are contingent upon the specific stage at which the measurement is obtained, such as whether it is taken during the initial opening of the preserved cores or shortly before testing. The lower the density of the shales, the more significant the change in suction associated with a slight change in water content.[20]

In-situ core sampling, core handling, preservation, and laboratory activities have received much attention[3,11–14]; however, the impact of all of these activities on the suction of shales is not often fully traced. To this regard, a recent extended campaign of deep boreholes in Northern Switzerland has offered the opportunity to investigate the water content and suction of Opalinus Clay shale (OPA) at the following conditions: (a) immediately after in-situ core sampling; (b) immediately after opening in the laboratory cores preserved in various ways. These measurements provided the opportunity to understand how various activities – from on-site to the laboratory - affect water retention properties. For this purpose, a protocol for measuring suction and water content on cores of Opalinus Clay shale freshly extracted from different sites and depths was developed. A similar protocol was developed and followed for measuring suction and water content in the laboratory. Comparison with available data on the water retention behavior of the material allowed analysis of the changes in hydration and suction from on-site to laboratory conditions.

In the following, materials and methods are first described. Second, experimental results are presented. In light of the on-site measurements, the water retention behavior of Opalinus Clay is discussed.

2. Materials and methods

2.1. Tested shales

Opalinus Clay shale is currently under intense investigation as it is the selected host formation for the Swiss high-level waste geological repository. It is a sedimentary geomaterial of the Jurassic age, characterized by bedding planes.[21] Mineralogically, it consists mainly of silicates, carbonates, and quartz at different percentages.[22,23] Intending to identify the candidate site for deep geological repositories in Switzerland, the National Cooperative for the Disposal of Radioactive Waste (nagra) has conducted an experimental campaign on deep boreholes in the period 2019–2022, in three different areas, namely, the Zurich Northeast, the Lägern North, and the Jura East areas. This study refers to core and specimens of OPA extracted in the following boreholes: Trüllikon-1-1 (reaching a depth of 1310 m), Marthalen-1-1 (reaching a depth of 1099 m), and Bözberg1-1 (reaching a depth of 1037 m). Trüllikon-1-1 and Marthalen-1-1 belong to the Zurich Northeast area; Bözberg-1-1 belongs to the Jura East area. Details about the depths of OPA at the different sites are summarized in Table 1.

2.2. Preparation of specimens

For the present study, specimens were prepared on samples from selected cores 3-m long. Core sampling included the following steps: (i) drilling with a drill pipe containing an inner core barrel; (ii) recovery of the drill core (nominal diameter 95 mm and length about 3 m) using the wireline technique (when the core inside the inner core barrel reaches the length of 3 m, a cable with an appropriate core catcher is used to extract the inner core barrel to the surface); (iii) insertion of the inner core barrel within an auxiliary tube (Fig. 1(a)); (iv) ground-handling and extraction of the drill core from the inner core barrel; (v) removal of the drilling fluid from the outer surface of the drill core using water available on-site (limiting the amount of water and the duration of the wash to the minimum necessary) and quick manual external drying using a rag; (vi) metric and geological survey operations on the drill core (Fig. 1 (b)) and acquisition of core scans.

Table 1
Depths of Opalinus Clay shale at the different boreholes.

Borehole	OPA core depth intervals [m]
Bözberg-1-1	530–651
Marthalen-1-1	590–705
Trüllikon-1-1-	816–928

As soon as the operations mentioned above were completed, procedures for preparing the samples were started. Fig. 2 depicts the sequence of sampling and shows the different types of samples tested in this study. In particular, samples of about 10 cm in length were quickly obtained by cutting core segments (sometimes already detached from the core parallel to the bedding planes) using a dry-cut circular saw. Each 10 cm sample was further cut into two equal parts: one half - named OS sample (where “OS” stands for “on-site”) - was brought to an area a few meters away from the extraction zone, specifically equipped to perform the measurements; the other half – named V sample (where “V" stands for “vacuum-preserved”) - intended for the laboratory, was quickly preserved using plastic-coated thick Al-bag (aluminum bag), evacuated and heat-sealed. Additionally, 40–50 cm cores were kept in PVC tubes filled with a resin layer (cf.[14]) for further laboratory activities. From these cores – named R cores (where “R" stands for “resin--preserved”) - samples of about 2–5 cm in length (named R samples) were cut in the laboratory using a dry-cut circular saw.

OS, V, and R samples were used for preparing specimens (named according to the corresponding samples) for on-site and laboratory measurements.

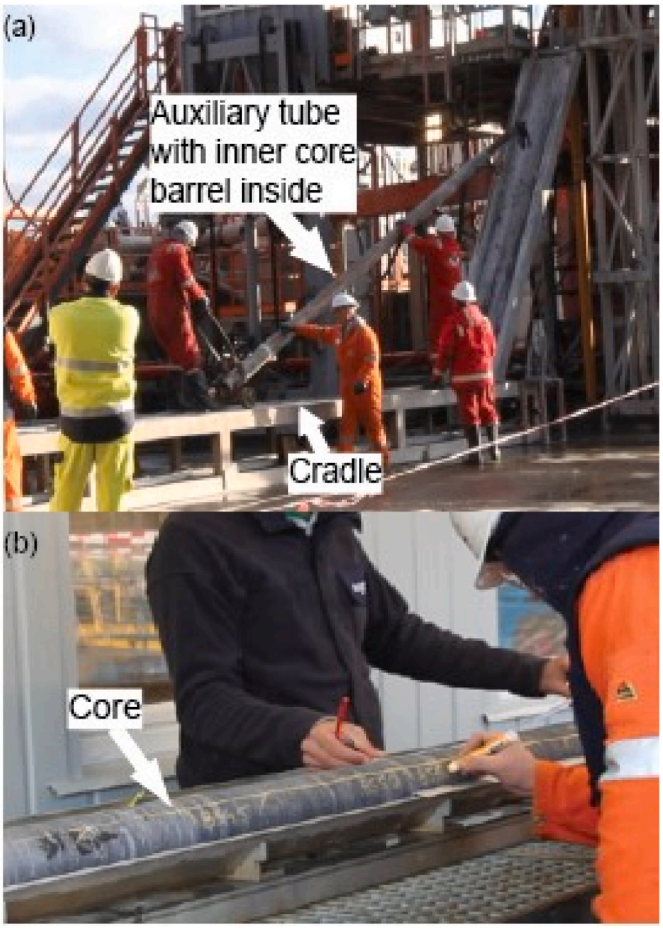


Fig. 1. Illustration of select surface operations in core sampling: (a) retrieving the inner core barrel with the auxiliary tube; (b) metric and geological survey procedures.

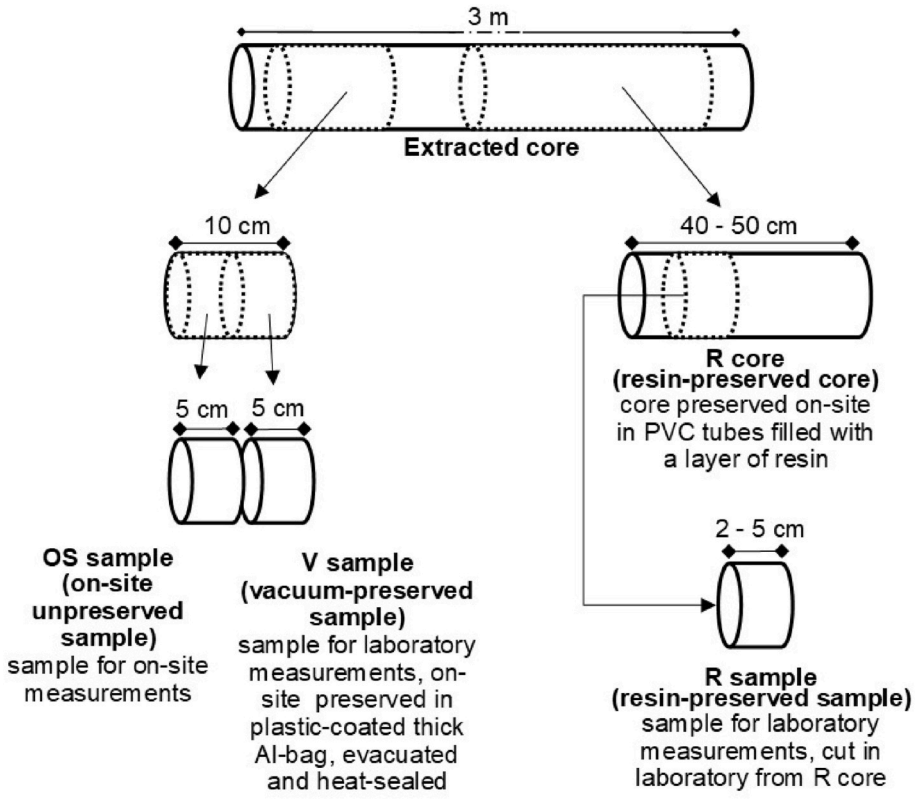


Fig. 2. Layout of samples obtained from the 3-m core.

Fig. 3 provides the timeline for on-site and laboratory activities. Protocols of on-site and laboratory activities were designed to reduce, at minimum, the exposure time of the samples to the atmosphere and other possible contaminants.

A schematic drawing and photographs summarizing the specimen preparation procedure are provided in Fig. 4. For each OS sample, OS specimens consisting of small fragments were extracted from the innermost area (longitudinally and diametrically) using a hammer and chisel; these fragments were immediately placed inside steel cups (sample cup capacity: 15 ml) closed with plastic lids and sealed with parafilm (type M, Bemis Flexible Packaging - NEENAH, WI 54956 8) (Fig. 3). All OS specimens were prepared within a time in the range of 35–88 min after extracting the corresponding core.

Similarly, two different types of specimens were prepared in the laboratory: (i) V specimens from V samples; and (ii) R specimens from R samples (Fig. 2). V specimens were prepared immediately after the opening of the corresponding V sample, following a protocol similar to the one performed on-site. In particular, the latter included the following steps: core opening, specimen preparation (using hammer and chisel), and preservation (steel cups with plastic lids and parafilm) (Fig. 4). Following the same strategy, R specimens were prepared immediately after obtaining the corresponding R sample. All efforts were made to obtain specimens even further from the outer edges to reduce possible local effects associated with the presence of resin. All V and R specimens were prepared within 10–48 min from the opening of the corresponding cores. Boundary temperature and relative humidity conditions were recorded manually using a digital hygro-thermometer. Also, larger portions of OS, V, and R samples were selected for further

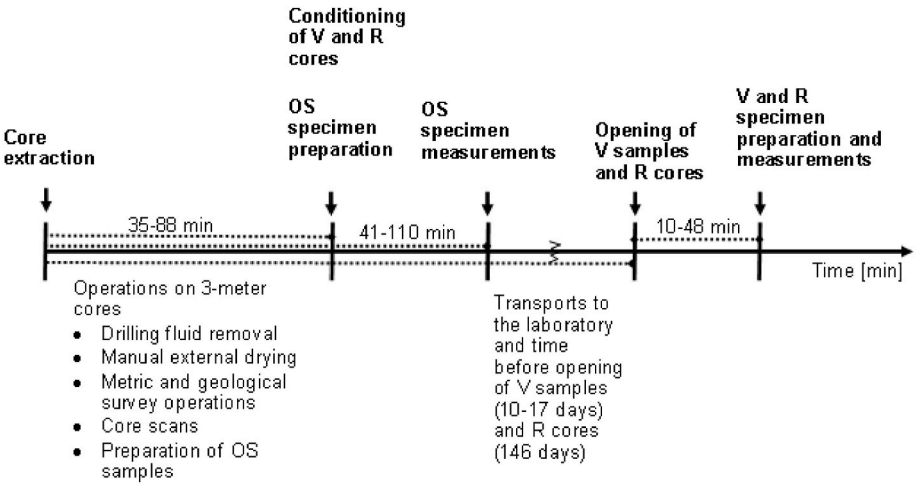


Fig. 3. Timeline of on-site and laboratory activities.

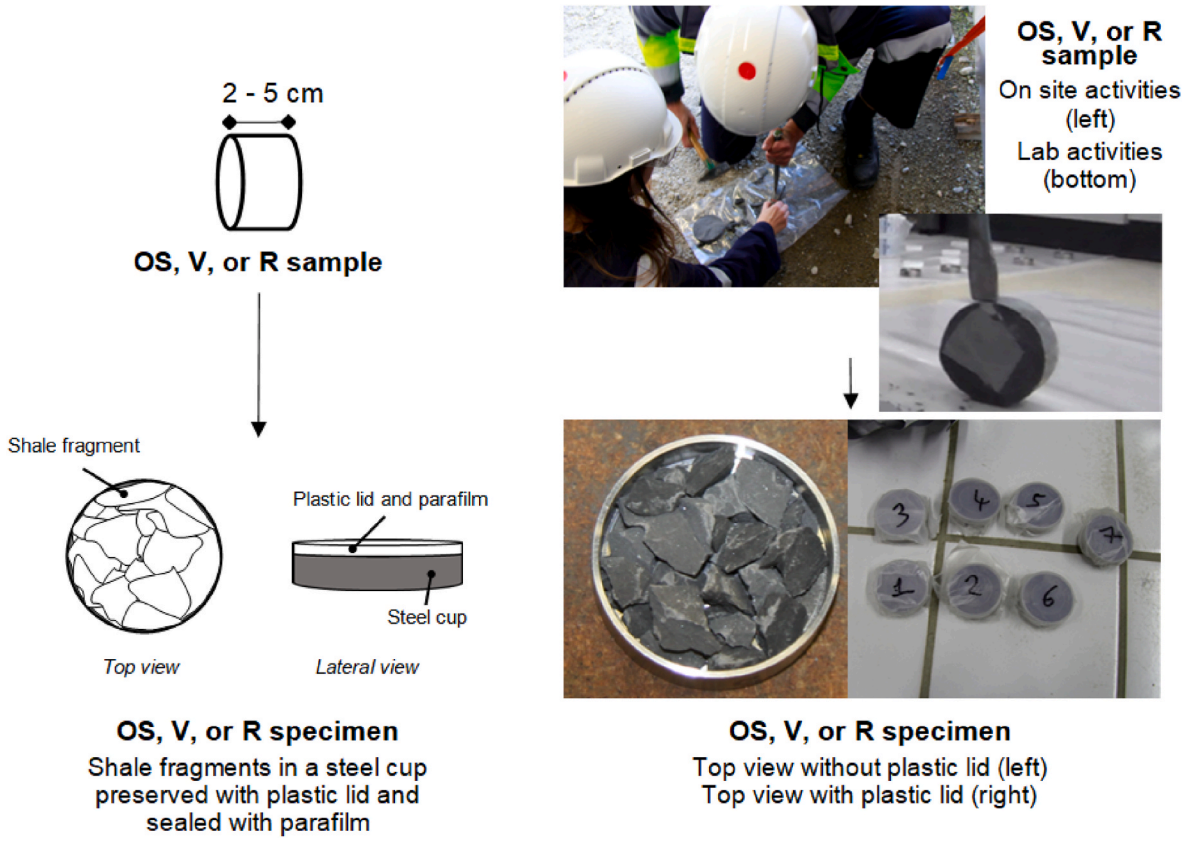


Fig. 4. Specimen preparation: schematic drawing (left) and photographs (right).

water content determinations.

2.3. Suction measurements

Table 2 summarizes the number of measurements performed on specimens retrieved from the different boreholes. For each deep borehole (Bözberg-1-1, Marthalen1-1, and Trüllikon-1-1), the following information is given: core depth (referred to the upper end of the core from which the specimens are prepared), type of measurements (on-site or laboratory) (i.e., specimens OS, V, R), and the number of tested specimens for each type of measurement.

Table 2
Overview of the testing program indicating the number of measurements of suction and water content carried out for each type of specimen.

Borehole	Core depth [m]	On-site measurements (OS specimens)		Laboratory measurements (V and R specimens)	
		Suction, ψ	Water content, w	Suction, ψ	Water content, w
Bözberg-1-1	551.73	7	8	7	8
Bözberg-1-1	554.47	8	9	6	7
Bözberg-1-1	557.25	6	7	5	6
Marthalen1-1	596.95	4	5	5	6
Marthalen1-1	622.12	4	5	5	6
Marthalen1-1	654.36	4	5	5	6
Trüllikon-1-1	845.43	–	–	4	5
Trüllikon-1-1	848.6	–	–	4	5
Trüllikon-1-1	851.7	–	–	–	3
Trüllikon-1-1	852.2	4	5	7	12
Trüllikon-1-1	855.47	2	3	10	17
Trüllikon-1-1	898.97	–	–	5	6

Total suction represents the free energy per unit volume of pore water and can be mathematically defined as follows at the absolute temperature of concern (T)[24,25]:

$$\Psi(T) = -\frac{RT}{\overline{V}_w^0} \ln \frac{p_w^v(T)}{p_w^{v0}(T)} \quad (1)$$

where $p_w^v(T)$ is the vapor pressure in equilibrium with the pore water; $p_w^{v0}(T)$ is the vapor pressure of the pure water, $\overline{V}_w^0$ is the molar volume of pure water, R is the universal molar gas constant. The dimensionless ratio $p_w^v(T)/p_w^{v0}(T)$ denotes the activity of the pore water. Determination of $p_w^v(T)/p_w^{v0}(T)$ involves bringing a sample into equilibrium with the vapor within a closed chamber using the chilled mirror method.

The pore water activity of each specimen was measured using the chilled mirror dew point psychrometer WP4C (suction measurement range: 0–300 MPa, accuracy as reported by the manufacturer: ±0.05 MPa in the range of 0–5 MPa and 1% in the range of 5–300 MPa). The instrument directly provides the total suction value; it repeats the measurements until two successive readings show a difference within a predetermined tolerance (0.03 MPa for suctions greater than 40 MPa, otherwise 0.3 MPa) (Decagon 2010)). The WP4C allows controlling the temperature in the range of 15.0–40.0 °C (±0.2 °C). To avoid the prevalence of condensation over evaporation inside the measuring chamber (a circumstance that may compromise the performance of the instrument), the temperature of the chamber was always set to be slightly higher than the temperature of the specimen in its immediately after-sampling condition (the latter was measured by taking advantage of the temperature sensor embedded in the WP4C instrument). Before

each on-site and laboratory measurement session, the device was appropriately calibrated using the 0.5 M KCl verification standard solution supplied by Decagon (Decagon 2010) (water potential of 2.14–2.32 MPa in the temperature range of 15.0–40.0 °C). Considering the manufacturer's suggestions, during the preparation phase of all specimens, care was taken to cover each base surface of the cup and reach a filling volume equal to about half of the available one (i.e., about 7.5 ml). Any inaccuracies related to this phase are considered to induce an error in measurements of a maximum of 1.1 % (as reported by Ferrari et al. (2014)[20] with investigations at a temperature of 25 °C using saturated solutions of NaCl and KCl). Generally, for a range of suctions between 4 and about 393 MPa (investigated using different saline solutions) and for temperatures between 25 and 40 °C, an accuracy of 3% has been highlighted in the literature.[20]

Each specimen was weighed immediately before and immediately after the total suction measurement (for the subsequent water content determination) using a precision balance (0.0001 g). Afterward, specimens were oven-dried at 105 °C for a minimum of 24 h and until a constant mass was reached. Water contents were then back-calculated for the different steps.

The following quality criteria were adopted when performing and processing the measurements.

(i) Only the measurements obtained within 2 h after core extraction (in the case of OS specimens) or core opening (for V and R specimens) were processed.
(ii) All experimental data for which technical problems were faced during the tests (e.g., the unsuitable temperature of the specimen compared to the chamber temperature, with consequent time intervals in which the specimen was exposed to the atmosphere under open-system conditions) are excluded from the analyses. Additionally, the analyses excluded total suction measurements for specimens that experienced water loss during the measurement phase.

3. Experimental results

In this section, first, the role of specimen preservation on suction measurements is assessed by reporting all the performed on-site and laboratory measurements. Second, the results of this study are compared with available water retention data to better analyze the changes in hydration and suction from on-site to laboratory conditions.

3.1. The role of specimen preservation on suction measurements

Fig. 5 collects the on-site and laboratory measurements performed in

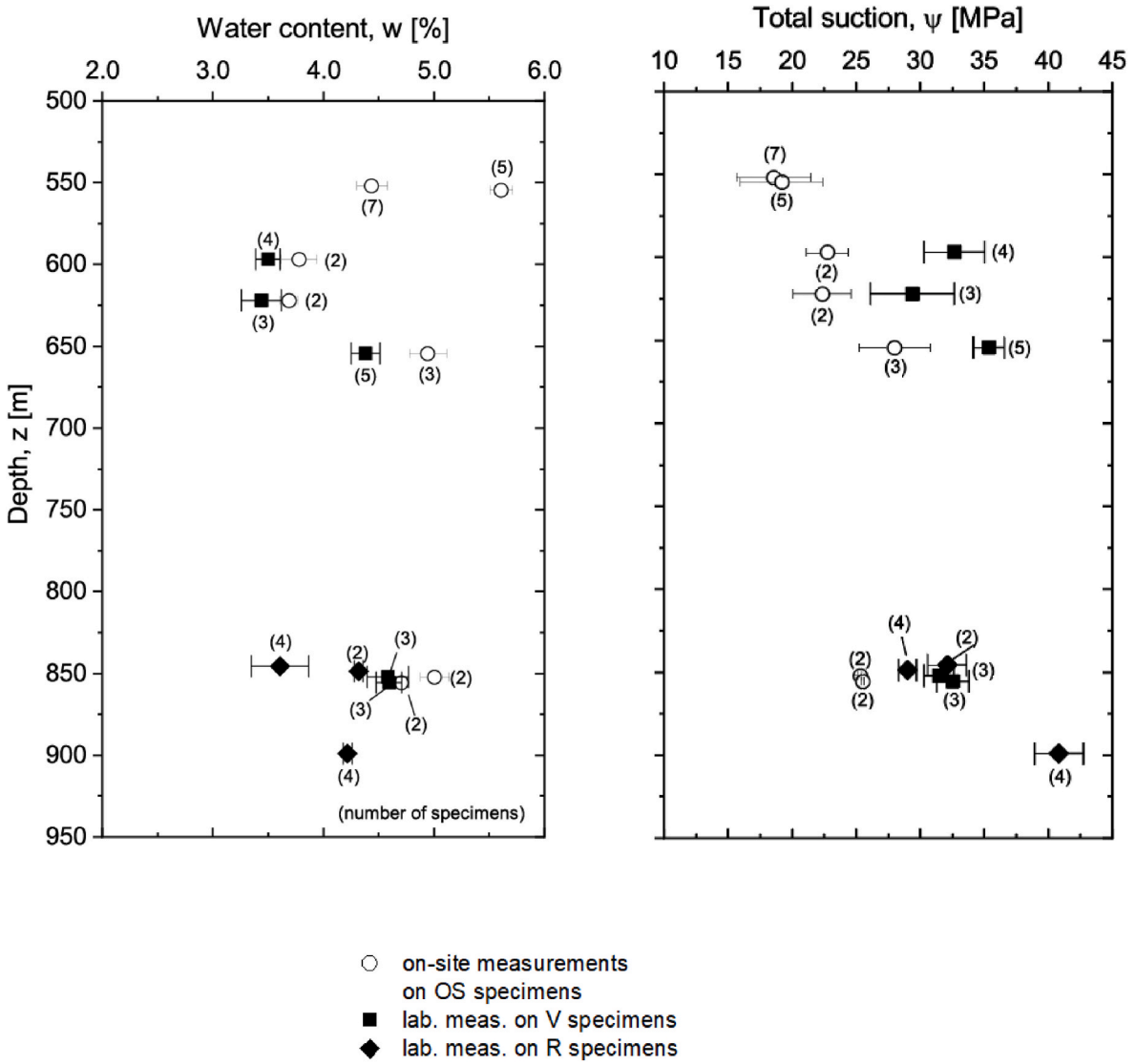


Fig. 5. On-site measurements at different boreholes: water content and total suction at varying depths.

the various boreholes on Opalinus Clay shale specimens. Average values and corresponding standard deviations of water content and suction against depth are provided. In brackets, next to each point, the number of tested specimens is reported.

Regarding the on-site measurements, an almost linear increase of total suction with depth can be detected up to 654 m, with values in the range of 18–28 MPa; afterward, similar values of about 25 MPa were also found at a depth of 855 m. Even if the measurements are performed on-site, a scatter of water content with depth can be highlighted and may be attributed to likely different mineralogy.[23]

For depths in the range of 596.95-654-36 m, average total suction in the range of 22.35–35.35 MPa and water content in the range of 3.5–5.1% were recorded. Comparing on-site and laboratory measurements, V specimens show a lower water content than the corresponding type OS specimens with differences of up to −1.42%; the total suction of V specimens is always higher than OS ones with maximum recorded difference values of 11.35 MPa.

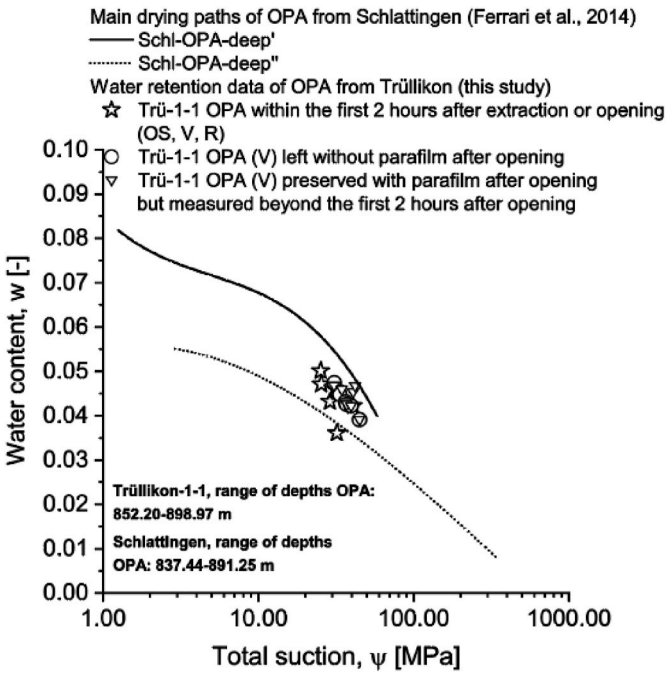
The average total suction is 18.56 MPa for a depth of 551.73 m, and 19.16 MPa for a depth of 554.47 m; the average water contents are 4.4% and 5.6%, respectively.

For depths in the range of 852.20–898.97 m, average total suction values in the range of 25.28–40.82 MPa have been measured on-site; the corresponding average water contents belong to the range of 3.6–5.0%. Comparing on-site and laboratory measurements, OS specimens are those with the lowest total suction and the highest water content values for depths comparable to the others. At a depth of about 852 m, an average suction difference of 6 MPa can be highlighted between V and OS specimens belonging to the same cores and obtained from the same sample of about 10 cm in length (see Fig. 2); in particular, the greater suction of the V specimens corresponds to an average water content of V specimens lower than the water content of OS specimens of 0.4%. For similar specimens obtained from a depth of about 855 m, the average suction difference between V and OS specimens is 7.06 MPa; V specimens have an average water content less than OS by 0.1%.

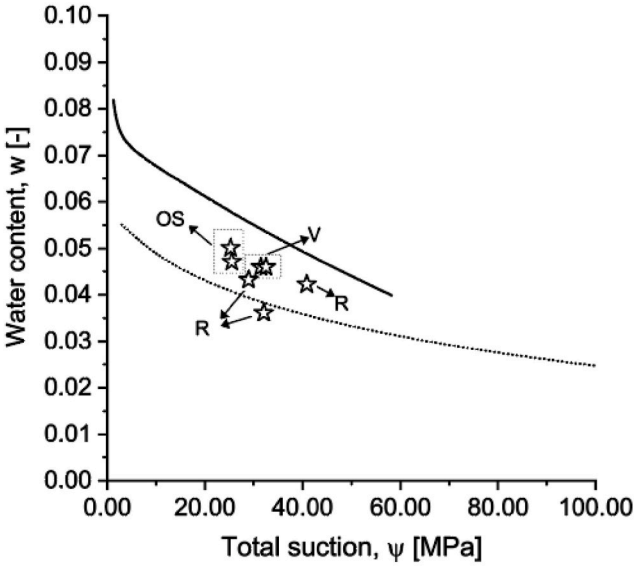
Overall, systematic differences between on-site and laboratory measurements were observed. In particular, on-site suction measurements are always lower than laboratory suction measurements (irrespective of the type of preservation implemented on-site) with differences varying between about 4 and 11 MPa. On-site water content measurements are always higher than laboratory measurements (irrespective of the type of preservation implemented on-site), with differences varying between about 0.1 and 1.4 wt%.

3.2. An insight into the water retention behavior

To shed light on the difference registered for the various types of tested samples, Fig. 6 (a) (range of total suction 1–1000 MPa in log axis) and Fig. 6 (b) (range of total suction 1–100 MPa in natural axis) provide (i) experimental points for OS, V, and R specimens from Trüllikon-1-1 representative of immediately after-sampling/after-opening states (these points have as coordinates the average of water content and total suction measurements of samples tested within 2 h after extraction or opening of the cores); (ii) single measurements of total suction and water contents for V specimens from Trüllikon-1-1, preserved with parafilm, and collected more than 2 h after the opening of the core (these specimens are those that were prepared afterward and therefore were most exposed to atmosphere before being preserved); (iii) single measurements of total suction and water contents for V specimens from Trüllikon-1-1 not preserved with parafilm. The collected results highlight a trend of increased suction and reduced water content for all Trüllikon-1-1 data following possible drying as a result of preservation and core handling. In general, the points seem to consistently describe a drained main drying curve, whose first points are the representative points of the material in its immediate after-extraction condition (i.e., the OS specimens). This observation is corroborated when comparing the experimental points with the main drying curves of Opalinus Clay from the



(a)



(b)

Fig. 6. Experimental data of OPA from Trüllikon-1-1 (this study) compared with main drying and main wetting paths of OPA from Schlattingen deep borehole (Ferrari et al., 2014)[20]: (a) water content plotted total suction in the range of 1–1000 MPa; (b) water content against total suction in the range of 1–100 MPa.

deep borehole in Schlattingen. The Opalinus Clay formation in Schlattingen exhibits two distinct densities and is accordingly referred to as Schl-OPA-deep' and Schl-OPA-deep'', (being Schl-OPA-deep'' the deepest one).[20]

A shift of the drying path of the water retention behavior for the considered OPA sample in Fig. 6 can be associated with the different void ratios (Schl-OPA-deep': 0.20, Trü-1: 0.13, Schl-OPA-deep'':0.12). In particular, higher void ratios are generally associated with higher water content for the same value of suction.[26]

Variations in suction between on-site and laboratory measurements always indicate water loss during preservation operations (under vacuum or with a resin-based technique), transport, and storage in the laboratory.

Interestingly, despite higher suctions and lower water content in the laboratory, suction and water content evolution from site to laboratory follows the main drying water retention behavior of the geomaterial. In this sense, the reconditioning of the specimens for the study of the hydromechanical properties (in contrast to what was argued in[27]) can be done with confidence within the known water retention behavior of the material. Consequently, the preservation technique used in situ - be it vacuum preservation or resin preservation - makes the corresponding specimens (V specimens and R specimens, respectively) suitable for experimental testing purposes.

4. Summary and concluding remarks

The possibility of planning an on-site experimental campaign and, in parallel, a laboratory campaign during the excavation of deep boreholes has been used in the present study. The study has provided scientific evidence about the state of specimens of Opalinus Clay shale extracted from different sites and depths, in their freshly extracted on-site condition and their freshly opened condition in the laboratory. These assessments were made through measurements of both total suction and water content. The results were discussed to get an overview of the hydration state of OPA under on-site and laboratory conditions for specimens preserved (on-site) with different methodologies.

The following main conclusions can be drawn.

- Total suction measurements performed immediately after in-situ core extraction always provide lower values than (for comparable depths) suction measurements performed immediately after opening the preserved cores in the laboratory. This is true whether the specimens are vacuum-preserved or resin-preserved.
- Water content measurements performed immediately after in-situ core extraction are always greater than the corresponding (i.e., relative to comparable depths) measurements performed "immediately" after opening the cores in the laboratory. Again, this observation is valid regardless of whether the cores are vacuum-preserved or resin-preserved.
- Vacuum-preserved and resin-preserved cores exhibit higher total suction and lower water contents than the corresponding on-site cores due to various contributing factors. These factors encompass the humidity levels present at the borehole site, the timing of conditioning, the conditions during storage and transport, as well as the duration of exposure and humidity upon opening the samples in the laboratory.
- Interestingly, it is demonstrated that measurements taken immediately on-site and in the laboratory of Opalinus Clay extracted at high depths correspond to a drained retention curve that is well placed compared to the drained main drying retention curves of shales extracted from other deep boreholes. This observation is fundamental in recognizing that during the transition from the site to the laboratory, the specimens did not suffer damage, which would have resulted in a change in the water retention behavior. Consequently, when reconditioned to in-situ conditions, core samples can be used for robust laboratory testing, whether in the case of vacuum or resin preservation techniques.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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