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ABSTRACT

This paper presents the development of a new high-pressure triaxial apparatus specifically prepared for inducing and tracking the degradation of clayey rocks. Total suction—used to induce the hydraulic degradation—is applied with vapor transfer technique by controlling the relative humidity of air in contact with the material. The evolution of the degradation process along different hydro-mechanical stress paths is continuously tracked with bender elements, as well as with air or water permeability measurements on partially saturated or saturated states, respectively. Relevant test results on a low porosity clayey rock (Lilla claystone, Spain) are presented to evaluate the main capabilities of the new equipment. The results bring up the high sensitivity to water of the material, which is evidenced by the important reduction of shear wave velocity induced on first wetting and drying at low confining stress, as well as by the significant increase in the air permeability of the degraded material (around four orders of magnitude larger than the intact material). Test results also showed clear differences in the volume change and shear strength behavior of undisturbed, saturated, and degraded samples, highlighting the relevance of degradation on macroscopic behavioral features.

Keywords

triaxial test, rock degradation, relative humidity, wetting-drying cycles

Introduction

Clayey rocks are very common in civil engineering works (slopes, foundations, underground excavations, to cite a few instances), and a frequent observation when working with these materials is

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their high degradation potential, which takes place mainly when they are excavated and subjected to environmental actions (e.g., Picarelli and Di Maio 2010; Alonso et al. 2010). Unloading and wetting-drying cycles have been broadly recognized to be the major contributors to degradation phenomena of clayey rocks in many geotechnical problems, which result in irreversible changes of the rock hydro-mechanical properties (loss in strength and stiffness, irreversible volume changes, increase in air permeability, loss of mass continuity induced by micro-cracking). These changes have been observed at both micro-structural scale (Montes et al. 2004; Gasc-Barbier and Tessier 2007; Pineda et al. 2010; Pineda et al. 2013) and at specimen scale (Alonso and Alcoverro 2002; Cardoso et al. 2012; Pineda et al. 2011; Pineda and Romero 2013).

One of the main challenges in the study of degradation of clayey rocks is to find adequate experimental techniques to induce and monitor in a continuous way rock alteration while maintaining stress conditions. This is not a straightforward task, since it requires the implementation of different instrumentation and experimental techniques—such as those used in unsaturated soil mechanics to control soil water potential—in high-pressure triaxial or oedometer cells, which have not been specifically designed to easily implement these techniques. This requires combining the technology recently gained in the design of high-pressure equipment (see for instance, Cuccovillo and Coop 1997; Bésuelle and Desrues 2001; Barla et al. 2010; Berre 2010; Alvarado et al. 2012) with the broad expertise gained in the control of water potential and already implemented in different mechanical cells (Oldecop and Alonso 2004; Chávez et al. 2009; Pintado et al. 2009a; Merchán et al. 2011).

In line with this challenge, this article addresses the development, calibration, and instrumentation of a new high-pressure triaxial apparatus specifically prepared for inducing and tracking degradation phenomena on clayey rocks. Two independent forced convection systems were included in the new setup to control the relative humidity of the air in contact with the material, and thus the total suction applied to the rock water. With this suction control technique, it was possible to progressively induce the degradation of a clayey rock along drying and wetting paths under a constant stress state. Different techniques were also implemented to monitor the progression of the rock damage, namely bender elements (shear waves propagation), and air or water permeability measurements on partially saturated or saturated states, respectively. Relevant test results showing the main capabilities of the new triaxial apparatus are presented, which bring up the important hydro-mechanical behavioral features observed on a clastone subjected to different degradation states.

High-Pressure Triaxial Cell, Instrumentation and Calibration

The apparatus comprises five main components which are indicated in Fig. 1: (i) the high-pressure triaxial cell (maximum radial stress 30 MPa) with different systems to apply and control axial and radial stresses (1, 2, 3, and 4 in the figure), (ii) the relative humidity control system (5), (iii) the shear wave velocity system (6), (iv) the air/water permeability system (7), and (v)

FIG. 1 General layout of the triaxial apparatus and setup.

FIG. 2 Cross-section of the high-pressure triaxial cell.

the acquisition data unit (8). The main properties of each component are described below.

The triaxial cell includes four main parts as shown in **Fig. 2**: (i) a stainless steel base in which the electric and hydraulic inlets/outlets are located, (ii) a rigid steel cylinder (23-mm thick), (iii) a cell top with hardened steel loading piston and a bronze piston casing, and (iv) a pair of rigid steel top and base plates that enclose the steel cylinder with four high-yielding steel bars (50 mm in diameter). The inner diameter of the cylinder is 200 mm with a height of 300 mm that enables the installation of internal instrumentation.

The stainless steel base—to avoid corrosion effects—contains eight inlets for electric connections and nine inlets/outlets for hydraulic connections. Two triaxial sample sizes (38 by 76 mm and 50 by 100 mm) can be tested using two exchangeable pedestal and top caps. As observed in **Fig. 3**, both

bottom and top caps include three independent inlets—two at the central part and a third external one—which can be used to apply controlled relative humidity or during saturation and air permeability measurements. The porous disks are constituted by concentric and polished stainless steel rings with separations around 0.2 mm between them and placed on the bases with circular and radial grooves.

The bronze piston casing allows minimizing friction effects between it and the loading piston. A reduced friction seal (Turcon Glyd Ring T38, 80 MPa of maximum pressure, which combines a Turcon based slipper seal and a nitrile O-ring) is used to avoid fluid cell leakages between the loading piston and its casing and to prevent stick-slip effects. The loading piston houses the bender elements connection through a 6-mm hole.

The stress state of samples tested is controlled by means of two high-pressure automatic pressure/volume controllers (GDS Instruments). A 64 MPa controller is used to apply cell pressure, whereas axial stress is generated using a 32 MPa controller connected to a stiff 500 kN load frame with a loading piston of 200 mm in diameter (**Fig. 1**).

The axial load and displacement instrumentation of the cell includes the following external and internal transducers: (i) an external load cell (133 or 233 kN range, Honeywell), (ii) an external linear variable differential transformer (LVDT) for axial displacement, (iii) two diametrically-opposed and pressure compensated (up to 20 MPa) LVDTs to measure local axial displacements (RDP model D5/300), and (iv) a pressure compensated local radial strain-gauge extensometer (± 5 mm range, Epsilon Technology Corp.). Both local transducers are physically attached to the specimen by top and bottom plastic supports adhered to the neoprene membrane (local LVDTs measuring the deformation of the central part of the sample, **Fig. 2**) and by self-supporting contacts that are held in place with a spring (local extensometer positioned at mid-height, **Fig. 2**). Transducers are continuously monitored and stored using a 16-channel data acquisition unit (USB NI-6210, National Instruments) connected to a PC. It is worth nothing that external readings of sample axial displacements are

FIG. 3 Schematic view of the triaxial base.

FIG. 4 Schematic view when working as an isotropic cell.

corrected by considering displacements of the cell elements at given confining stress. Displacements associated with the cell elements are registered by an external micrometer located on the rigid steel top plate. These displacements have been considered before computing the externally measured sample axial strain.

The triaxial equipment can be easily transformed into an isotropic cell without using a load frame to apply axial load. This is of particular interest when testing low-porosity clayey rocks, which require long-term hydraulic equalization periods. The loading piston and the bronze piston casing (Fig. 2) are replaced by a similar system but using a small transmission rod ($\phi_r = 8$ mm in diameter) with Turcon based slipper seal, which enables the external monitoring of the axial specimen deformation, as shown in Fig. 4. Cell fluid fills the upper cone-shaped chamber and acts on the top piston ($\phi_p = 38$ mm in diameter). Although the system does not apply a strictly isotropic condition, the error induced is small $\sigma_1/\sigma_3 = 1 - (\phi_r / \phi_p)^2 = 0.96$.

Since an external load cell is used, friction effects between loading piston and bronze casing should be considered. The following calibration protocol has been used to correct the measured external load. The triaxial cell has been assembled without using any natural or dummy sample. After filling the cell with silicone oil (the confining fluid usually used), the inner pressure was monotonically increased in steps using the automatic pressure/volume controller from 0 to 16 MPa. This pressure, acting on the loading piston ($\phi_p = 38$ mm in diameter) has been converted into force (applied load) and compared against values recorded by the external load cell (external load). Figure 5 shows the evolution of the axial load ratio (internally applied by the cell pressure/externally measured with the load cell) with the confining pressure. Ratios over one indicate some loss of efficiency in the transmission of load due to friction effects. For confining pressures lower than 1 MPa, friction

FIG. 5 External load correction factor due to friction effects.

effects are not constant and increase up to an axial load ratio of around 1.058. For confining pressures higher than 1 MPa, friction effects remain constant.

Another issue of concern, especially when dealing with water/air permeability determination, is the enhancement of the impedance to flow of the internal flexible pipes used when the cell pressure is increased. These polyamide pipes (4 by 2 mm, Legris) have been preferred to metallic ones to avoid a stiff connection between the base cell and the upper cap. To perform the calibration, two independent hydraulic inlets were internally connected using the polyamide pipe to allow the flow of water under different confining stresses. A constant water head equal to 10 kPa was applied to induce flow of water through the pipe, which was monitored for the different confining pressures applied. The outgoing flow was measured twice for the same confining stress. It was checked that no important flow impedance was originated up to 12 MPa confining pressure. A maximum flow reduction with respect to unconfined conditions of about 0.90 % was measured at this confining pressure. For higher confining pressures, stiffer (metallic) pipes should be used.

Auxiliary Systems

VAPOR TRANSFER SYSTEM

As indicated above, the vapor transfer technique was introduced in the experimental setup to apply controlled hydraulic cycles. The water potential is applied by the migration of water molecules through the vapor phase of a reference system, where the relative humidity is controlled, to the material pores until thermodynamic equilibrium is reached. The relative humidity

$RH = p_v/p_v^0$ is related to the total suction ψ by the psychrometric law:

$$(1) \quad \psi = -\frac{RT\rho_w}{M_{mw}} \ln\left(\frac{p_v}{p_v^0}\right)$$

where:

R = the gas constant,

T = the absolute temperature,

M_{mw} = the molecular mass of water,

ρ_w = the density of pure water,

p_v = the absolute pressure of vapor in the air, and

p_v^0 = the absolute vapor pressure at saturation and temperature T .

Figure 1 shows a scheme of the forced convection system, which was used to speed up the vapor transfer process from the reference vessels to the low porosity material. Two independent circuits were used—for top and bottom caps—each with an air pump, a vessel containing the reference saline solution to control relative humidity, and a hygrometer. The vessels are placed on electronic balances to register water mass transfer from or to the material, during drying or wetting, respectively. Distilled water ($RH \approx 100\%$) was usually employed for wetting, whereas lithium chloride solution (at around $RH \approx 20\%$) was used during drying.

The vapor transfer technique has been conventionally used on unsaturated clayey soils (e.g., Jotisankasa et al. 2007; Delage et al. 2008; Pintado et al. 2009a; Merchán et al. 2011), and to a lesser extent on very stiff clays (Lima et al. 2012), evaporitic rocks (Oteo et al. 1996), rockfills (Oldecop and Alonso, 2004; Chávez et al. 2009), and compacted marls (Cardoso et al. 2012).

SHEAR WAVE VELOCITY SYSTEM

Bender elements (Shirley and Hampton 1978) are included in the triaxial apparatus to determine the shear wave velocity—as a non-destructive way to evaluate the material stiffness—and to track its degradation state. Their implementation for testing clayey rocks under unstressed conditions was described in detail by Arroyo et al. (2010). Here, it has been extended to track the stiffness evolution of clayey rocks during the application of hydraulic and mechanical paths (i.e., under confined conditions).

In the bender element technique, two polarized piezoceramic transducers are used to generate and capture a shear motion that travels through the sample. The travel time between emitted and received signals t_s is used to determine the shear wave velocity $V_{s(ij)}$, in which the travel length has been taken as the tip-to-tip distance between piezoceramics (e.g., Viggiani and Atkinson 1995; Jovicic et al. 1996). The time domain analysis is used to determine the travel time, as the first significant deflection observed in the output trace. Bender elements are located on top and bottom caps, so that the shear wave velocity

in vertical direction $V_{s(vh)}$ is obtained. Thus, shear modulus $G_{0(vh)}$ is determined as

$$(2) \quad G_{0(vh)} = \rho V_{s(vh)}^2$$

where ρ is the total density of the material.

The experimental setup used is equivalent to the one reported by Arroyo et al. (2010), and is schematically shown in **Fig. 1**. A 4-mm slot for the bender beam is carved on specimens using a mini-drilling machine. A programmable function generator (TTi -TG1010A, Thurlby Thandar) is used to generate and emit the input signals. Output signals are acquired through a digital oscilloscope (TDS2024b, Tektronix). An ultrasonic pre-amplifier (OLYMPUS Panametrics 5656C, including a high band filter) is used for amplifying the output signals and to minimize the influence of the electrical current. To minimize random noise, signal stacking is systematically applied to all measurements with the number of stacked signals equal to 128.

WATER AND AIR PERMEABILITY SYSTEMS

As a complementary technique to track the degradation process of the material, both air and water permeability systems have been implemented in the general setup shown in **Fig. 1**. Air permeability is used to monitor the degradation process under unsaturated states (after a drying path), whereas water permeability is determined only under saturated conditions. Water permeability tests are performed under controlled-gradient conditions by applying a constant back-pressure at the bottom cap by a pressure/volume controller, while the top cap is maintained open under atmospheric conditions (**Fig. 1**). Water permeability is calculated under steady state conditions using the water volume inflow registered by the automatic controller.

On the other hand, air permeability tests are performed using the gas pressure decay method, as also depicted in **Fig. 1**. A pressurized tank of known volume V and at an initial absolute pressure u_{a0} (upstream vessel) is connected to the bottom cap, whereas the top cap is again maintained under atmospheric conditions (downstream absolute pressure at u_{atm}). The time evolution of the absolute air pressure decay u_a inside the tank is recorded, which can be used to estimate the mass of air flowing through the sample assuming perfect gas law:

$$(3) \quad \frac{dn}{dt} = \frac{V}{RT} \frac{du_a}{dt}$$

where:

n = the corresponding mols of air flowing through the sample,

R = the ideal gas constant, and

T = the absolute temperature.

The mass inflow rate dn/dt can be also estimated using the generalized Darcy's law for compressible fluid and the perfect gas law:

$$(4) \quad \frac{dn}{dt} = -\frac{A}{2RT} \frac{k_{ia}}{\mu_a} \frac{(u_a^2 - u_{atm}^2)}{L}$$

where:

k_{ia} = the intrinsic air permeability, which depends on different properties of the solid matrix (void ratio, pore size distribution, shape of pores, tortuosity) and reflects the changes undergone during matrix degradation at standard temperature and pressure, respectively,

A and L = the cross-sectional area and length of the specimen, and

μ_a = the air dynamic viscosity at standard temperature and pressure.

By using Eqs 3 and 4, the following expression is obtained for the air permeability k_a (see for instance, Yoshimi and Osterberg 1963)

$$(5) \quad k_a = \frac{k_{ia}\rho_a g}{\mu_a} = -\frac{2LV\rho_a g}{A(u_a^2 - u_{atm}^2)} \frac{du_a}{dt} = -\frac{2LV\rho_a g}{A(u_t + 2u_{atm})} \frac{d(\ln u_t)}{dt}$$

where:

$u_t = u_a - u_{atm}$ is the gauge air pressure just inside the specimen at the upstream end,

ρ_a = the density of air at standard temperature and pressure, and

g = the acceleration of gravity.

During air permeability tests, the system is connected to the specimen through the same inlets/outlets used for vapor transfer. The RH system is turned off during these tests, though it is re-connected afterwards to allow the equilibrium of the specimen before the next loading step.

Performance of the Triaxial Apparatus

MATERIAL TESTED

Triaxial tests were performed on a low porosity sulphate-bearing rock identified as Lilla claystone (Early Eocene to Late Miocene) from the Tertiary Lower Ebro Basin in Northeast Spain (Alonso et al. 2013). Lilla claystone has two main components: (i) the host argillaceous matrix composed by phyllosilicates (illite and paligorskite), dolomite and quartz, and (ii) the sulphated crystalline fraction constituted mainly by anhydrite and gypsum. The clay fraction, on average accounting for 60 %

mass fraction and dominated by illite, has low plasticity (liquid limit $LL = 23$ %, plasticity index $IP = 7$ %). Water content varies from 3.1 to 4.1 %. Initial void ratio ranges around 0.11 ± 0.01 , whereas the density of solids varies around 2.80 ± 0.02 Mg/m³.

Table 1 summarizes the initial conditions of the different samples. Additional details are given in Pineda (2012).

Triaxial samples (38 by 76 mm and 50 by 100 mm) were prepared under dry conditions—to avoid swelling and alteration of the material—from large diameter cores (110 mm) using a lathe. The samples were then stored in Pyrex containers and equilibrated at the specified relative humidity ($RH = 50$ %) using the vapor transfer technique. A salt solution was used to generate the specified relative humidity.

Figure 6 shows the water retention properties of undisturbed Lilla claystone (six specimens), which were determined with a chilled-mirror dew-point psychrometer (WP4, Decagon Devices). Up to three wetting-drying cycles were applied using vapor equilibrium, in which each equalization step was maintained for a minimum of 48 h—sample dimensions were kept to a minimum following the procedure described in Cardoso et al. (2007). The hydraulic behavior during RH cycling by vapor transfer of the undisturbed rock remained inside the main wetting and drying branches obtained during the first RH cycle. However, the water retention properties of specimens previously exposed to liquid water during the slaking tests (three cycles: liquid water contact and oven drying at 105°C) displayed a different hydraulic response, as shown in **Fig. 6**. Higher water retention capacity was displayed by the slaked material at the low suction range ($\psi < 8$ MPa). At high suctions, where the water retention capacity is mainly controlled by the specific surface of the clayey fraction, both undisturbed and slaked materials showed a similar response. This behavior highlights the importance of the exposure to liquid water at low stress levels in the degradation of Lilla claystone.

The main drying and wetting branches of the water retention curves were fitted to the model proposed by Romero and Vaunat (2000):

$$(6) \quad w = w_{\max} C(\psi - \psi_0) \left[\frac{1}{1 + (a(\psi - \psi_0))^n} \right]^m$$

with

$$(7) \quad C(\psi - \psi_0) = 1 - \frac{\ln \left[1 + \frac{(\psi - \psi_0)}{a} \right]}{\ln(2)}$$

TABLE 1 Initial conditions of specimens tested.

Specimen	Water content, w (%)	Total Density, ρ_t (Mg/m ³)	Total Suction, ψ (MPa)	Void ratio, e (–)
S3_1N	3.65	2.59	115	0.117
S4_3	3.10	2.60	130	0.119
S4	3.15	2.59	128	0.119

FIG. 6 Water retention curve along wetting and drying paths for undisturbed and slaked Lilla claystone.

where:

ψ = total suction,

w_{max} = the water content at saturation state or ψ_0 (being ψ_0 the total suction at saturation when the osmotic potential is not negligible),

Parameter a = the maximum suction at $w = 0$,

n = the slope of the inflection point whereas m refers to the residual water content, and

$1/\alpha$ = the air entry value of the rock (α in MPa^{-1}).

Equation 7 defines a correction function $C(\psi - \psi_0)$ included in this model to consider the linear relationship between $\log \psi$ and w at high suctions commonly observed in low porosity clayey materials. Solid and dashed lines in **Fig. 6** represent the main branches of the water retention curves for undisturbed and slaked material, respectively. There, $a = 1000$ MPa was adopted. Values of parameters α , n , and m have been also included in **Fig. 6**.

EXPERIMENTAL PROGRAM FOLLOWED

Two types of tests (Test series 1 and 2) were followed to check the capabilities of the triaxial system. Test series 1 refers to pre-conditioning tests, performed under low confining stresses to progressively induce and track rock degradation under isotropic stress state. Two long-term controlled wetting-drying cycles (around 300 days) at extreme relative humidity changes (between 100 % and 20 %) were applied on an initially undisturbed sample S3_1N at low isotropic stress ($p - u_a = 50$ kPa)

FIG. 7 Stress paths followed. (a) Test series 1 (b) Test series 2.

(**Fig. 7(a)**). The equalization stage of the relative humidity was considered complete when no appreciable sample volume change was observed during seven days. Axial and radial displacements, as well as relative humidity and temperature, were continuously monitored. Changes in rock stiffness were progressively tracked using bender elements measurements.

Test series 2 refers to high-pressure triaxial tests at different RH , performed on undisturbed samples S4_3 and S4, as well as on the previously degraded sample S3_1N. Two hydraulic states were studied for the undisturbed samples: (i) natural water content (sample S4_3 at $RH = 50$ %), and (ii) water saturated condition (sample S4 at $RH = 100$ %). **Figure 7(b)** shows the stress paths followed during the high-pressure triaxial tests. Saturation of sample S4 was carried out at $p - u_a = 1$ MPa. All samples were subjected to isotropic loading from $p - u_a = 1$ to 8 MPa. The loading rates were selected to maintain the stability of the pressure rate system, which may be affected even by small room temperature variations given the high volume of liquid used in the triaxial chamber and the piston of the pressure/volume controller. At specified steps (1, 2, 4, 6, and 8 MPa), the isotropic net stress was maintained constant (at least 24 h in unsaturated tests) to allow dissipation of any excess of pore pressure. As will

be explained below, each isotropic stress increment in saturated specimens was maintained constant until reaching, at least, the time corresponding to 90 % of the average consolidation (t_{90}). This way, isotropic loading should be considered as a combination of continuous and stepwise loading approaches. Air permeability tests (samples S3_1N and S4_3 at $RH=50\%$), water permeability tests (sample S4 at $RH=100\%$) and bender elements measurements were performed at the end of each equalization stage. Finally, specimens were subjected to shearing under drained conditions until reaching failure. As explained above, the shearing rates were selected to maintain the stability of the pressure rate system. Strain control but also stress control approaches were used during shearing. For saturated sample, a controlled axial stress rate criterion ($\approx 0.5 \text{ kPa/min}$) was adopted, since no important post-peak response was expected. On the other hand, an axial displacement rate criterion was preferred in unsaturated tests since a more brittle behavior was anticipated. The controlled displacement rate used ($3.33 \times 10^{-4} \text{ %/min}$ in axial strain rate) ensured a good stability of the rate along the shearing stage.

Experimental Results

TEST SERIES 1: RH CYCLING UNDER CONSTANT NET MEAN STRESS

Figure 8 shows the evolution of axial (ϵ_a), radial (ϵ_r), and volumetric (ϵ_v) strains during the application of two RH cycles on specimen S3_1N. Readings of RH and temperature are also included. Relative humidity varied from 99 to 20 % during wetting and drying, respectively. Temperature (solid circles) varied between 19.5 and 22°C. Measurements of RH were made with the air pump turned-off after an equalization period of 30 min to eliminate pressure gradients and thus minimize deviations in the target RH (Pintado et al. 2009a, 2009b). The equalization stage of the relative humidity was considered complete when volumetric strain rates lower than 0.004 %/day and 0.001 %/day were measured during wetting and drying paths, respectively.

Wetting induced volumetric swelling, whereas some shrinkage was observed during drying. It resulted in a definite volumetric expansion after each cycle. The volumetric strain at the end of the cycles was around -5.2% . Particularly, a significant expansion was observed during the second wetting, where ϵ_v increased from -2.0 to -5.9% . This behavior was attributed to an enhanced expansion mechanism facilitated by the development of new fissures created during the previous drying. Such an interpretation is based on the visual inspection of the specimen after Test series 1, where a clear fissuring pattern was observed at the sample boundaries in contact with the loading caps (see Fig. 9). The measured radial strain was slightly lower compared to the axial deformation, which suggests some anisotropic strain response. The comparison between external and local (internal) axial strains showed larger values in the former

FIG. 8 Temporal evolution of axial, radial and volumetric strains during Test series 1 on sample S3_1N.

case. It is attributed to the non-homogeneous deformation that took place along the specimen height during RH cycling (fissuring contributed to this non-homogeneous pattern). As observed in Fig. 9, the fissuring in specimen S3_1N was more pronounced at the boundaries of the specimen, which justifies the larger axial deformation registered by the external LVDT.

FIG. 9 Specimen S3_1N at initial state (undisturbed) and after Test series 1 (degraded). Fissuring pattern induced by RH cycling.

Figure 8 also shows that drying was more efficient (in time) than wetting to reach equilibrium (the rate of water absorption was always lower than water desorption). The rate of water transfer during *RH* cycling (measured externally using the digital balances) was very small and of the same order of magnitude as the evaporation rate measured in the reference vessels. For this reason, the analysis of the moisture transfer during Test series 1 was made in terms of the evolution of the relative humidity at top and bottom of the specimen (the criterion used to define “equilibrium” was based on the evolution of the volumetric strain, as explained above). The rate of moisture transfer dw/dt incoming/outgoing the sample during a change in relative humidity can be expressed as (e.g., Jotisankasa et al. 2007; Pintado et al. 2009b):

$$(8) \quad M_{\text{dry}} \frac{dw}{dt} = q_{da} x_0 (RH^{\text{reference}} - RH^{\text{sample}})$$

where:

M_{dry} = the constant dry mass of the sample,

q_{da} = the flow rate of dry air mass (related to the volumetric air flow delivered by the pump and assumed constant),

x_0 = the saturated mixing ratio (mass of water vapor at saturation per unit mass of dry air mass) that depends on temperature T (assumed constant) and dry air pressure u_{da} (assumed constant), and

RH = the relative humidity of the reference salt solution and the sample (measured at top and bottom caps).

The difference $(RH^{\text{reference}} - RH^{\text{sample}})$ will thus be representative of the rate of moisture transfer. The decrease in $(RH^{\text{reference}} - RH^{\text{sample}})$ as the relative humidity in the sample approaches the applied value $RH^{\text{reference}}$ is shown in Fig. 10(a) for top and bottom cap measurements during the first wetting path. It can be observed that 8 days were required to obtain values of $(RH^{\text{reference}} - RH^{\text{sample}}) < 5\%$ as indicated by the dotted horizontal line. Values below 2% were attained only after 30 days of wetting. This behavior highlights the progressive difficulty for vapor transfer in low porosity geomaterials as the degree of saturation approaches 1. The same evolution is shown in Fig. 10(b) for the first drying path. Only 3 days were required to achieve values lower than 5% and around 17 days to get values smaller than 2%. The faster equilibrium of the relative humidity under drying is in agreement with the higher moisture diffusivity coefficient obtained by Pineda (2012) during drying paths in specimens subjected to *RH* cycles under unstressed conditions. There, the diffusivity coefficient ranged from 2.2×10^{-10} to 1.5×10^{-9} m²/s during wetting and drying paths, respectively. It is worth noting that most of the volumetric strain registered during wetting but also along drying took place once the difference $(RH^{\text{reference}} - RH^{\text{sample}})$ was lower than 5% (see Fig. 10) (i.e., once *RH* has been almost completely equilibrated inside the system).

FIG. 10 Evolution of the *RH* and volumetric strain during Test series 1. (a) First wetting path. (b) First drying path.

Figure 11 shows an example of the output traces obtained from bender elements measurements following the experimental procedure described by Arroyo et al. (2010) and Pineda (2012). The apparent input frequency ranged between 30–50 kHz due to the high resonant frequency of the bender–soil system, which was defined by Arroyo et al. (2010) to be $f_{\text{res}}(\text{kHz}) \approx 25V_s$ (V_s in km/s). High-quality output traces, free from significant influence of the near field, were obtained in all cases. Figure 12 shows the evolution of the shear wave velocity during *RH* cycling in Test series 1. V_s decreased during the first wetting from 2200 to 1300 m/s (points A and B, respectively). V_s also reduced under drying due to fissuring, where a final value around 850 m/s was registered at point C. A slight increase in V_s , up to 1050 m/s, was observed during the second wetting. It may be attributed to some closure of

FIG. 11 Typical output traces obtained from bender measurements.

fissures—created during the previous drying—as a consequence of the swelling of the clayey matrix. V_s reduced to 640 m/s during the second drying (point F). Such an irreversible reduction in V_s (around 70 % regarding the initial, undisturbed value) demonstrates the strong influence of RH cycling on the degradation of the rock.

HIGH-PRESSURE TRIAXIAL TESTS: TEST SERIES 2. ISOTROPIC COMPRESSION AND SHEARING

High-pressure triaxial tests were performed during Test series 2 on specimens at different degradation states but also under different saturation conditions, as shown in **Fig. 7(b)**. Undisturbed specimens were maintained under constant relative humidity by connecting both top and bottom drainage lines to glass vessels containing saturated solution of sodium chloride ($RH = 50\%$). Air pumps were not employed in Test Series 2. During air permeability tests, top and drainage lines were connected to the air

FIG. 12 Temporal evolution of the shear wave velocity during Test series 1. **FIG. 13** Loading and equalization stages along the last isotropic compression step.

permeability system described above. Two paths will be discussed, namely isotropic compression and shearing.

Figure 13 presents the evolution of the volumetric strain during the last isotropic compression step, where the mean total stress increased from 6 to 8 MPa, for the three specimens tested (undisturbed S4_3 at $RH = 50\%$, fully saturated S4 and previously degraded S3_1N at $RH = 50\%$). Vertical dotted lines indicate the end of the stress controlled loading process and the beginning of pore pressure equalization at constant total stress. The degraded S3_1N underwent higher volumetric deformation on loading. A fast equalization, represented in terms of the evolution of the volumetric strain under constant total stress, was observed in unsaturated specimens (S3_1N and S4_3), irrespective of the degradation state. Only 12 h after loading were required to reach a volumetric strain rate lower than 0.001 %/h (a minimum of 24 h after loading was used in the unsaturated tests performed). In the fully saturated specimen S4, the equalization period after loading was much longer. Experimental results were used to estimate the time required to reach 90 % of consolidation, t_{90} , as shown in **Fig. 13**. The coefficient of volume compressibility of the previous loading step was equal to $m_v = 1.76 \times 10^{-6} \text{ m}^2/\text{kN}$. By considering a water permeability of $k_w = 1.05 \times 10^{-13} \text{ m/s}$ (see **Fig. 19**), the consolidation coefficient was equal to $c_v = 6.03 \times 10^{-9} \text{ m}^2/\text{s}$. Finally, the theoretical time required to reach 90 % of consolidation by assuming a drainage length equal to a half of the sample height, was around $t_{90} \approx 97 \text{ h}$. As observed in **Fig. 13**, the allowed maximum consolidation time in the saturated specimen was higher than this

FIG. 14 Compressibility curves of the three samples tested.

value and close to 118 h from the beginning of the loading process.

Figure 14 shows the compressibility curves for the three specimens tested. Rock compressibility showed a clear dependency on both degradation and saturation state. The maximum and minimum compressibility was obtained, respectively, for the degraded S3_1N and undisturbed S4_3 specimens (both at $RH = 50\%$). It can be noted that the maximum volumetric strain achieved during loading in the degraded specimen S3_1N ($\varepsilon_v = 2.90\%$ at $p - u_a = 8$ MPa) was lower than the maximum volumetric expansion caused during previous RH cycling ($\varepsilon_v = -5.90\%$ at $p - u_a = 50$ kPa; see **Fig. 8**). This highlights the strong influence of environmental effects— RH cycling—on the mechanical behavior of clayey rocks, particularly at low confining stresses. The maximum volumetric compression in the undisturbed sample S4_3 was 1.1% , about three times lower compared to the degraded sample S3_1N (both tested at $RH = 50\%$). The saturation of the specimen S4 was achieved under $p - u_a = 1$ MPa by applying a water back-pressure $u_w = 0.60$ MPa to the base of the sample during 20 days. This process induced a volumetric expansion equal to -0.28% . The total volumetric compression of the fully saturated specimen S4 was lower than the undisturbed and partially saturated sample S4_3. It has been attributed to the fact that specimen S4 has been saturated under a high mean net stress. However, the comparison between these two specimens shows a higher compressibility for the fully saturated rock, which is clearly observed for $p - u_a > 4$ MPa.

Figure 15 displays the output traces obtained from bender elements tests, at the end of each step along the isotropic loading stage. The arrival time, t_s , defined as the first significant deflection, has been included with a vertical line. Output traces showed good quality in all cases. Some high frequency noise

FIG. 15 Output traces obtained from bender elements tests during isotropic loading for the three samples tested. (a) Degraded. (b) Undisturbed. (c) Fully saturated.

FIG. 16 Variation of the shear wave velocity with the mean net stress during isotropic loading for the three samples tested.

was observed in the traces of the saturated specimen, although their quality was good enough to determine the arrival time. No evidence of important near-field effects was detected (similar to the previous results described by Arroyo et al. 2010). The maximum amplitude reduced in degraded samples as well as in fully saturated specimen, which is attributed to the change in the resonant frequency of the bender-soil system. The variation of V_s during isotropic loading is shown in Fig. 16. Values of V_s were computed using the current sample height at the end of each loading step. A clear increase in V_s with the isotropic loading was observed in all cases. V_s increased from 1915 m/s (unstressed condition) to around 3000 m/s ($p - u_a = 8$ MPa) in the undisturbed sample S4_3. The degraded sample S3_1N showed an increase in V_s from around 640 m/s (unstressed condition) to 1500 m/s at $p - u_a = 4$ MPa. Measurements for $p - u_a > 4$ MPa could not be obtained due to mechanical damage in the transmitter. It is interesting to note that the saturation process at $p - u_a = 1$ MPa reduced V_s (specimen S4) from 2500 to around 1500 m/s. Then, the isotropic loading increased V_s up to 1700 m/s at $p - u_a = 8$ MPa. Again the hydraulic path (wetting), even carried out at a higher confining stress (1 MPa instead of 50 kPa), induced rock degradation that was able to be detected with shear wave velocity measurements.

The temporal evolution of the air pressure inside the tank recorded during the air permeability tests—performed on unsaturated specimens at the end of each loading step—is shown in Fig. 17. The system impedance is indicated by a dashed line in Fig. 17(b). Results obtained from undisturbed S4_3 and degraded S3_1N specimens are presented in Figs. 17(a) and 17(b), respectively. At $p - u_a = 1$ MPa the initial air pressure was maintained equal to $u_t = 0.75$ MPa whereas for $p - u_a > 1$ MPa it increased to $u_t = 1$ MPa. Tank pressure ratios of

FIG. 17 Air pressure decay curves obtained during air permeability tests for unsaturated samples (RH = 50 %). (a) Undisturbed sample S4_3. (b) Degraded sample S3_1N.

$0.95 < u_t/u_{t0} \leq 1$ and $0.5 < u_t/u_{t0} \leq 1$ were used to evaluate air permeability (using Eq 5) on undisturbed and degraded specimens, respectively. A clear influence of the net mean stress was observed in the case of the degraded sample, which was not so important on the undisturbed material. Consistently, lower air pressure decay rates were detected on the undisturbed material.

Figure 18 presents the evolution of the water volume inflow registered by the automatic controller during the injection stages performed to determine the water permeability of the fully saturated sample S4. The duration of each injection stage was around 300 h, although only the last 200 h, corresponding to the steady-state flow, are shown in Fig. 18. The volumetric water flow rate decreased almost three times with increasing confining stress from 1 to 8 MPa.

FIG. 18 Steady-state water inflow during water permeability tests for sample S4.

Figure 19 summarizes the variation of both air and water permeability (k_a and k_w) during the isotropic loading stage. Air permeability ranged between 10^{-11} and 10^{-12} m/s in the undisturbed specimen S4_3, whereas the water permeability (sample S4) was around 10^{-13} m/s. It is worth noting the strong increase of the air permeability measured in the degraded specimen S3_1N as a consequence of the previous RH cycling. Air permeability increased around four orders of magnitude (10^{-7} to 10^{-8} m/s) compared with the undisturbed sample S4_3. Increasing isotropically mean total stress to 8 MPa did not significantly change the air permeability of the degraded sample.

FIG. 19 Variation of air and water permeability during isotropic loading for the three samples tested.

After the isotropic loading stage, all specimens were unloaded to $p - u_a = 1$ MPa and then subjected to axial compression until reaching failure using controlled axial displacement rate (unsaturated samples S3_1N and S4_3) and controlled axial stress rate (saturated sample S4). The stress-strain curves, in terms of deviatoric stress ($q = \sigma_a - \sigma_r$) versus shear strain ($\epsilon_s = 2(\epsilon_a - \epsilon_r)/3$), are shown in Fig. 20 (subscript “a” refers to the axial component, whereas “r” refers to the radial one). The evolution of the volumetric strain ($\epsilon_v = \epsilon_a + 2\epsilon_r$) during shearing has been also included. Similar volumetric compression patterns took place in all samples before achieving the maximum deviatoric stress. The strain rate adopted during unsaturated tests led to the achievement of the maximum deviatoric stress after around 13 days. On the other hand, the stress rate approach used during the saturated test caused the achievement of the maximum deviatoric stress after 18 days (sample failure was only observed after around 21 days). In both cases, the shearing rates employed were considered adequate to ensure

FIG. 20 Shearing stage. Stress-strain curves for the three samples tested.

“drained conditions” in these overconsolidated samples. A dilatant trend was detected in the degraded sample S3_1N around the peak. As observed by comparing unsaturated samples S3_1N and S4_3, the previous hydraulic degradation induced an important drop of the peak shear strength (from 46.6 to 9.9 MPa) and of rock stiffness. Undisturbed S4_3 and fully saturated S4 specimens displayed similar pre-failure response (rock stiffness), despite the different values of deviatoric stress reached at failure in each case (46.6 and 12.8 MPa, respectively). The strong reduction of the maximum shear strength can be attributed not only to the higher degree of saturation of S4, but also to the irreversible degradation induced during the saturation stage using liquid water. The degraded sample S3_1N showed a peak deviatoric stress slightly lower compared with the fully saturated specimen, but with a much lower pre-failure stiffness.

Concluding Remarks

The main characteristics of a new high-pressure triaxial apparatus, specifically prepared for inducing and tracking the degradation of low-porosity clayey rocks, have been described in this paper. Total suction—used to induce the hydraulic degradation by cycling the relative humidity—has been applied with vapor transfer technique by controlling the relative humidity of air in contact with the material on top and bottom caps. The evolution of the degradation process along different and long-term hydro-mechanical stress paths has been continuously tracked with bender elements (evolution of rock stiffness), as well as with air or water permeability measurements on partially saturated or saturated states, respectively. Test results obtained on a very low porosity claystone have been used to demonstrate the capabilities of the equipment along long-term hydro-mechanical paths. Experimental data showed clear differences in the permeability properties, volume change, and shear strength behavior of undisturbed, saturated, and degraded samples, highlighting the relevance of degradation phenomena on macroscopic behavioral features.

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