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Swelling experiments on mudstones

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Abstract: This paper studies the swelling of highly consolidated mudstones by theoretical considerations and laboratory experiments. A key assumption was made that saturated and uncemented clays behave as heavily dense colloid without direct contacts among solid particles. It leads to an important conclusion that the swelling pressure acting on adsorbed interparticle water-films is equivalent to the effective stress. This so-called clay-colloid concept is validated by various swelling experiments on two kinds of mudstones, the Callovo-Oxfordian argillite in France and the Opalinus clay in Switzerland. In the tests, water adsorption-desorption, swelling pressure and strain were measured on the samples at various suctions and load-controlled conditions. Results suggest that: (1) the mudstones can take up great amounts of water from the humid environment, much more than the water content in the natural and saturated states; (2) the swelling pressure increases with water uptake to high levels of the overburden stresses at the sampling depths of 230 to 500 m, indicating that the adsorbed water-films are capable of carrying the lithostatic stress; and (3) the large amount of water uptake causes a significant expansion of mudstones even under the lithostatic stresses.

Key words: clay; mudstone; adsorption; swelling pressure; expansion; suction; stress analysis

1 Introduction

Clay formations are being investigated worldwide as host medium for the disposal of radioactive waste because of the favorable properties such as very slow transport of fluids and high sorption capacity for most radionuclides. However, excavation of an underground repository produces fractures and cracks around openings, which may form pathways for fluid transport. During operation phase of a repository, the ventilation of the underground openings will lead to de- and re-saturation of surrounding rock, depending on changes in humidity of the ventilating air. The de- and re-saturation may result in swelling and shrinking and even fracturing in the clay. After backfilling and closing of repository, a gradual sealing process of the excavation-induced fractures is to be expected due to the compression by the rock deformation and the expansion of the fractured clay matrix by taking up water from the saturated far-field. The swelling may play a dominant role in the sealing process, depending on availability of water. Whereas swelling as a significant process occurring in clays such as bentonite-based buffer materials is broadly investigated, the swelling behavior of highly-indurated mudstones has been less characterized and was not well understood.

Based on the disjoining pressure concept that the swelling pressure is equivalent to the average disjoining pressure of water interlayers in clay, Horseman et al. [1] proposed that the isotropic effective stress in a compacted and uncemented clay, which behaves as perfect colloid without direct contacts among solid particles, was equal to the swelling pressure in the interparticle water-films. This also means that the adsorbed water-films in compacted clays are capable of bearing external loads and carrying the lithostatic stress. Obviously, this so-called clay-colloid model differs from the classic porous medium model, in which pore water is generally assumed to be free and physico-chemical interactions of water with clay minerals are not explicitly taken into account. So the application of the classic porous model encounters difficulties in interpreting some "anomalous phenomena" observed in mudstones [1].

However, the colloidal concept may be helpful for understanding of strongly-coupled hydro-mechanical processes in compacted clays and mudstones. Unfortunately, there are almost no direct experimental evidences for support of the colloidal concept.

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Recently, GRS has carried out various swelling experiments to validate the colloidal concept for mudstones. The Callovo-Oxfordian (COx) argillite in France and the Opalinus (OPA) clay in Switzerland have been tested. Both are the potential host rocks for the disposal of radioactive waste. This paper analyzes the relationship among total stress, swelling pressure and matric suction in compacted clays, and discusses the swelling experiments designed under the clay-colloid concept.

2 Effective stress in compacted clays

The natural and saturated clay may be regarded as a part of colloidal system [1, 2]. Most of the pores are interconnected and occupied with water and solutes. In highly consolidated clays, the pore size is mostly so small that a very significant portion of the water content is strongly bound on mineral surfaces. Only small amounts of free water may be included in relatively large pores [3]. In addition to clay minerals, there also exist other mineral particles and diagenetic bonds in natural clays. The properties and responses of the clay colloidal system are significantly determined by very complex interactions among water, solutes and clay surfaces. A conceptual model for the inner structure of a compacted clay may be assumed as illustrated in Fig.1.

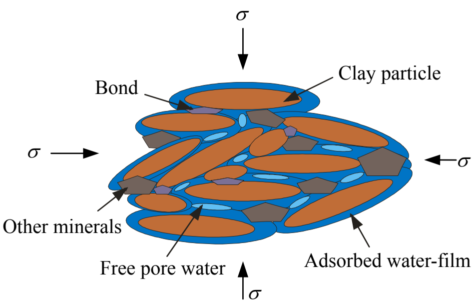


Fig.1 Conceptual model for consolidated clay.

Physico-chemical interactions of water with clay minerals cause adsorption of water on the internal and external surfaces of clay particles, forming electrostatic double-layer. In the narrow spaces between clay particles in compacted clays, the double-layer is overlapping. This generates local excess pressures to develop in interparticle water-films when the volume of the clay is kept constant by enclosing it in a rigid permeable box. This excess pressure is termed as the disjoining pressure [1] when equilibrated with the external bulk water. The disjoining pressure is the sum of a number of contributions which stem from electrostatic double-layer interactions, Van der Waal's dispersion forces, structural forces and solvation forces. The local disjoining pressure, Π_D, is related to the ratio of the vapor pressure of water-film, p_{vf}, to that of the bulk water p_{vp} by

$$\Pi_D = -\frac{RT}{v_w}\ln\left(\frac{p_{vf}}{p_{vp}}\right) \quad (1)$$

where R is the gas constant, T is the absolute temperature, and v_w is the partial molar volume of the water-film.

The average disjoining pressure over a wavy cross-section passing through the midplanes between particles is equivalent to the swelling pressure acting on the walls of the box. For uncemented clays which behave as perfect colloid so that there are no direct contacts between solid particles, the average disjoining pressure or the swelling pressure, $\bar{\Pi}_D$, is equivalent to the conventional isotropic effective stress σ_{eff}. Horseman et al. [1] suggested that an isotropic total stress σ applied to the clay-water system can be expressed by

$$\sigma = \sigma_{eff} + p_w - p_o = \bar{\Pi}_D + p_w - p_o \quad (2)$$

where p_o is the reference atmospheric pressure, and $p_w - p_o$ is the gauge pressure in the absence of osmotic effects. This equation clearly indicates that the total local pressure p_{fm} acting on an adsorbed water-film, is the sum of the disjoining pressure Π_D and the pressure p_w in the free bulk pore water. The negative measurable gauge pressure is defined as matric suction p_c [2]:

$$p_c = p_o - p_w \quad (3)$$

Substituting Eq.(3) into Eq.(2), the total stress can also be expressed as a function of matric suction:

$$\sigma = \sigma_{eff} - p_c = \bar{\Pi}_D - p_c \quad (4)$$

This equation implies that the matric suction of a saturated clay is simply the difference between the effective stress (or the swelling pressure) and the total stress. Rodwell et al. [2] illustrated the force balance acting on a water-film between two clay platelets, as shown in Fig.2. The analogue model (Fig.2(c)) demonstrates the coupling between the forces.

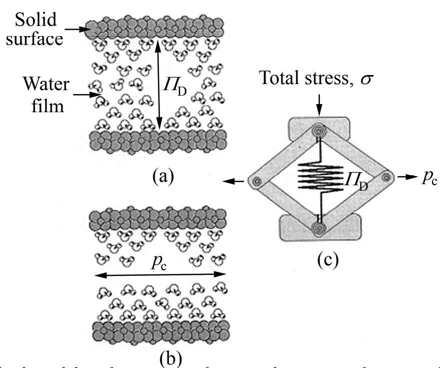


Fig.2 Relationships between the total stress, the matric suction and the disjoining pressure on a water-film among two clay platelets [2].

During undrained loading on a saturated clay, the water content or the thickness of the adsorbed water-films remains nearly constant, keeping the swelling pressure $\bar{\Pi}_D$ unchanged. So the matric suction inversely or the bulk pore water pressure proportionally varies with the total stress:

$$\left(\frac{\partial p_c}{\partial \sigma}\right)_{\Pi_D} = -\left(\frac{\partial p_w}{\partial \sigma}\right)_{\Pi_D} \approx -1 \tag{5}$$

During drained loading at constant pressure in the external water reservoir, i.e. p_w = const or p_c = const, the effective stress or the swelling pressure in the clay varies proportionally with the total stress:

$$\left(\frac{\partial \sigma_{eff}}{\partial \sigma}\right)_{p_c} = \left(\frac{\partial \bar{\Pi}_D}{\partial \sigma}\right)_{p_c} = 1 \tag{6}$$

This implies that the interparticle water-films in compacted clays bear external loads and are capable of carrying the lithostatic stress. When p_c = 0, the externally applied total stress or the lithostatic stress will be fully supported by the adsorbed water-films:

$$\sigma = \bar{\Pi}_D = \sigma_{eff} \tag{7}$$

Application of the total stress to a saturated clay compresses the adsorbed water-films, leading to desorption of water molecules from mineral surfaces. Reduction of the film thickness increases the disjoining pressure in it. If the total stress falls below the effective stress (or the swelling pressure), for instance, during rapid erosional unloading or destressing by excavation of underground openings or extraction of samples from a saturated clay formation, suction exerts an internal tensile stress among all pairs of neighboring mineral particles against the difference of the internal disjoining pressure and the external boundary load. Maintaining the total stress constant, the swelling pressure and the suction must change simultaneously:

$$\left(\frac{\partial \bar{\Pi}_D}{\partial p_c}\right)_{\sigma} = \left(\frac{\partial \sigma_{eff}}{\partial p_c}\right)_{\sigma} = 1 \tag{8}$$

When σ = 0, the matric suction of a saturated clay is equal to the average swelling pressure but not zero:

$$p_c = \bar{\Pi}_D = \sigma_{eff} \tag{9}$$

Thus, the matric suction and the average swelling pressure of compacted clays may be expressed by the same mathematical form as Eq.(1). Both are related to the ratio of the vapor pressure of the adsorbed water in narrow pores p_{vm} to that of the pure bulk water p_{vp} :

$$p_c = \bar{\Pi}_D = -\frac{RT}{v_w}\ln\left(\frac{p_{vm}}{p_{vp}}\right) \tag{10}$$

It is to be distinguished here that the disjoining (swelling) pressure is a generalized quantity that incorporates the full range of mechanisms affecting the chemical potential of water in the thin films of a compacted clay, while the matric suction is the reaction to the externally-applied boundary stress and the internally-developed excess pressure in the narrow gaps between particles [1, 2]. Matric suction is actually determined by the intermolecular strain of the pore water. When the matric suction is positive, the adsorbed water-films are in tension. As a suction threshold is exceeded, the water molecules evaporate from the clay and the hydrogen bonds of the interparticle water-films break down, resulting in collapse of pores. When the matric suction is negative, the water-films are in compression.

Up to now, compacted clays and mudstones have still been treated as classic porous media, in which all the pore water is assumed to be free without distinguishing the different effects of adsorbed and free waters. The application of the classic porous model to compacted clays and mudstones finds difficulties in interpreting some “anomalous phenomena”, for instance, anomalous pore water pressures observed in field mudstones [1] and in mudstone samples during loading [4]. In contrast, the above-mentioned colloidal concept may be suitable for the interpretation of such phenomena appearing in compacted clays and mudstones. However, this model must first be validated by experiments.

3 Studied mudstones

Both the COx argillite and the OPA clay are sedimentary mudstones and over-consolidated. For laboratory tests, COx cores were extracted from the Meuse-Haute-Marne under- ground research laboratory in France, while OPA cores from the Mont Terri underground rock laboratory in Switzerland. The COx argillite contains 40%–45% clay minerals, 20%–30% carbonates and 20%–30% quartz [5, 6], whereas the OPA clay has 58%–76% clay minerals, 6%–24% carbonates and 5%–28% quartz [6]. Both mudstones do not contain significant quantities of expansive clay minerals such as smectite, 13%–23% illite-smectite in COx argillite [6] and 5%–20% illite-smectite in OPA clay [7]. The basic properties of the mudstones were determined on the samples and are summarized in Table 1. The studied mudstones have similar properties. It is to be pointed out that the samples are more or less desaturated during sampling, storage and preparation.

Table 1 Basic properties of the studied mudstones.

Mudstone	Clay content (%)	Carbonate content (%)	Quartz content (%)	Water content (%)	Grain density (g/cm^3)	Dry density (g/cm^3)	Porosity (%)
COx	40–45	20–30	20–30	7.1	2.70	2.25	15.1
OPA	58–76	6–24	5–28	6.7	2.71	2.34	15.0

4 Measurements of swelling pressure

4.1 In volume-constraint conditions

As usual, the conventional method is adopted to measure swelling pressures of the COx and OPA mudstones in volume-constraint conditions. Figure 3 shows the schematic setup of a swelling cell in which a disk sample of 10 mm in thickness and 50 mm in diameter is inserted. The constrained sample is allowed to be wetted from the top and bottom with liquid water or water vapor at the controlled humidity.

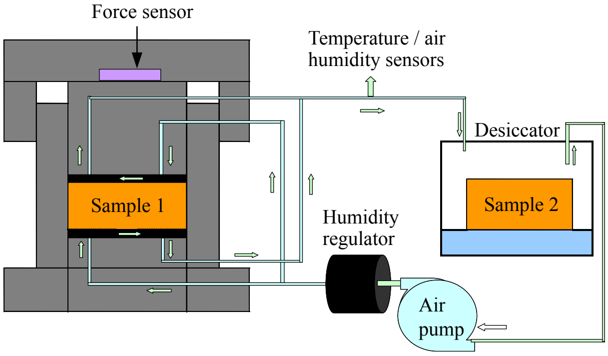


Fig.3 Schematic setup of a swelling cell.

Figure 4 illustrates the evolution of the swelling pressure measured on a partly-saturated COx sample which was preloaded to 0.5 MPa axially. The first wetting was performed by circulating wetted air at a relative humidity of 90%, resulting in an increase of the swelling pressure to about 1 MPa and then a slight fluctuation due to the temperature change. The following wetting by introducing synthetic formation water into the sample ended when the atmospheric pressure gave a quick rise of the swelling pressure up to 2.5 MPa and a gradual increase to 3.3 MPa over 4 months. Similar tests made by others on the same mudstones provided lower values below 2 MPa [5, 8]. Obviously, these values are not comparable with the expected ones according to Eq.(7), i.e. the swelling pressure should be comparable to the overburden stress of about 12 MPa at the COx sampling depth of 490 m [5] and the mean lithostatic stress of 6 MPa at the OPA sampling location [7]. One of the reasons for the measured low values of swelling pressure might be that clay minerals close to the entering water expanded

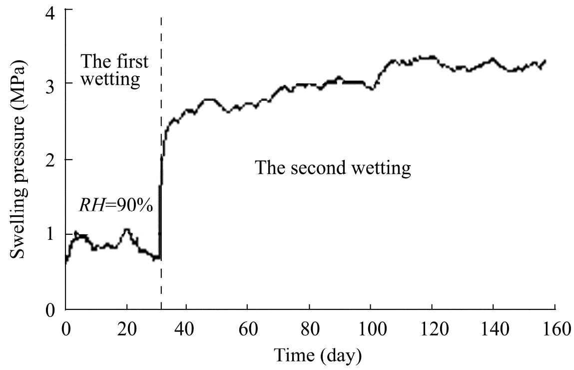


Fig.4 Swelling pressure measured on a COx sample in a cell with wetted air and synthetic formation water.

so highly that the local pore spaces were rapidly closed, making more water enter into the constraint samples more difficultly and even impossibly. Thus the swelling pressure in the samples could not homogenously develop.

4.2 In axially-fixed and laterally-unconstrained conditions

To minimize effects of rapid closure of the entry pores during wetting and achieve a homogeneous distribution of entering water within a sample, a new test method has been developed by the authors [9, 10] for determination of swelling pressure of hard clays. Figure 5 illustrates the principle of the so-called uniaxial swelling test. A clay sample is axially-fixed and laterally-unconstraint in a cell. Water molecules in vapor can easily access and move into the narrow spaces deeply, so that rehydration of the clay sample may be better made by circulating wetted air around

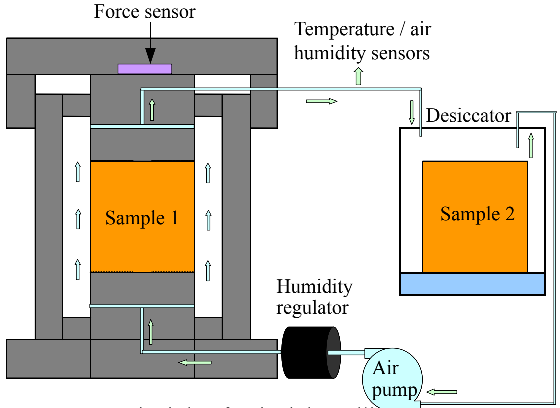


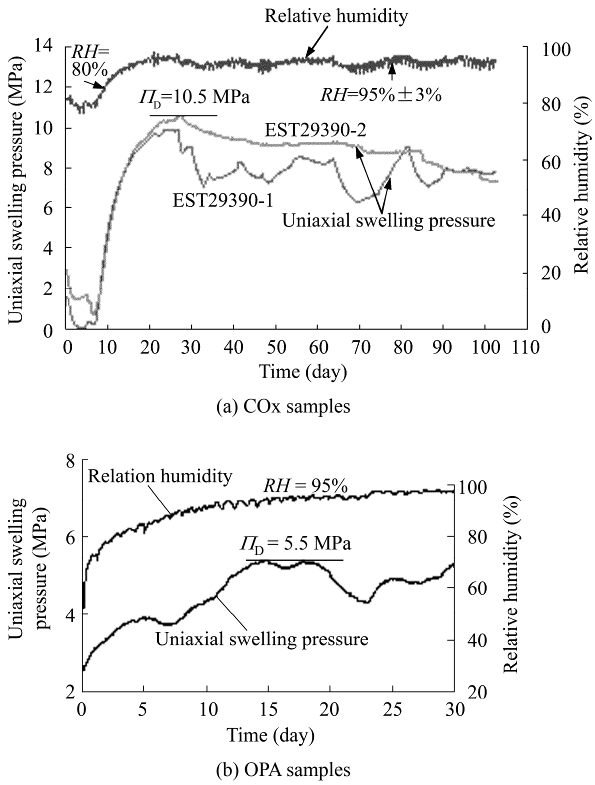
Fig.5 Principle of uniaxial swelling test.

the peripheral surface at the controlled relative humidity ($RH = p_{vm} / p_{vp}$) or suction (p_c). According to Eq.(10), the suction is related to the relative humidity as

$$p_c = -\frac{RT}{v_w}\ln\left(\frac{p_{vm}}{p_{vp}}\right) = -\frac{RT}{v_w}\ln(RH) \tag{11}$$

Variation of the air humidity leads to changes in water content (see Section 6) and thus in thickness of interparticle water-films, which determines the swelling pressure. A sufficiently high stiffness of a clay sample makes it possible to keep its stability without need of any radial confinement. Whereas the buildup of swelling pressure in the sample is measured in axial direction by the reaction of the rigid piston, it is also possible to monitor the swelling strain in radial direction.

Figures 6 presents the results of uniaxial swelling pressure measured on COx and OPA samples, respectively. The samples were prepared to a size of 40 mm in diameter and 50 mm in length. The initially saturated samples were more or less desaturated during preparation. They were axially preloaded to 2 MPa and then the axial strain was fixed.



(a) COx samples

(b) OPA samples

Fig.6 Uniaxial swelling pressure measured on COx and OPA samples as a function of relative humidity of surrounding air.

From Fig.6(b), it can be recognized that drying by circulating air of 80% relative humidity caused a quick drop of the axial stress tending to zero. The subsequent increase in relative humidity leads to a rising axial stress. At 95% relative humidity, the reacting stress in axial direction of the COx samples reached a maximum of 10–10.5 MPa while a peak value of 5.5 MPa was recorded for the OPA sample. Higher swelling pressures can be expected by elevating the boundary humidity. For instance, wetting another COx sample with water vapor at RH = 100% (or p_c = 0) led to a larger swelling pressure of 11 to 12 MPa, as shown in Fig.7. These maximum values of swelling pressure are almost equal to the overburden stresses of 12 and 6 MPa at the sampling positions of COx and OPA mudstones, respectively. This test result clearly confirms the theoretical stress concept (Eq.(7) or Eq.(4) at p_c = 0), which is indeed developed for uncemented clays behaving as perfect colloid. It is evident that the colloidal model with the associated stress concept is also true for the diagenetic mudstones. Because the studied mudstones do not contain significant quantities of expansive clay minerals such as smectite (13%–23% in COx argillite [6] and 5%–20% in OPA clay [7]), it seems likely that the swelling forces are developed between the external surfaces of closely-packed platy clay minerals such as illite (3%–20% in COx argillite [6] and 16%–40% in OPA clay [7]) by mechanisms similar to those operating during interlayer swelling [1]. The observed phenomenon of stress relief by drying and stress rising by wetting reflects that the adsorbed interparticle water-films in compacted clays and mudstones support the externally-applied loads and even the lithostatic stress. Additionally, the buildup of swelling pressure in the fixed axial direction without any lateral confinement may suggest that the pressure acting on the water-films among clay platelets is probably not a scalar quantity and should be represented by a second-rank tensor [1, 2].

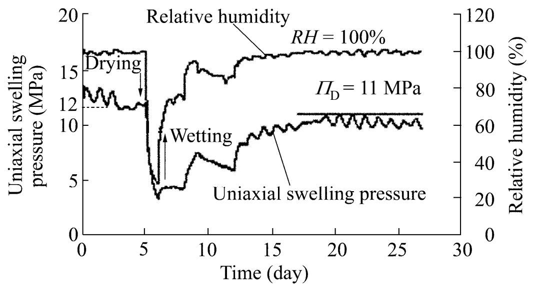


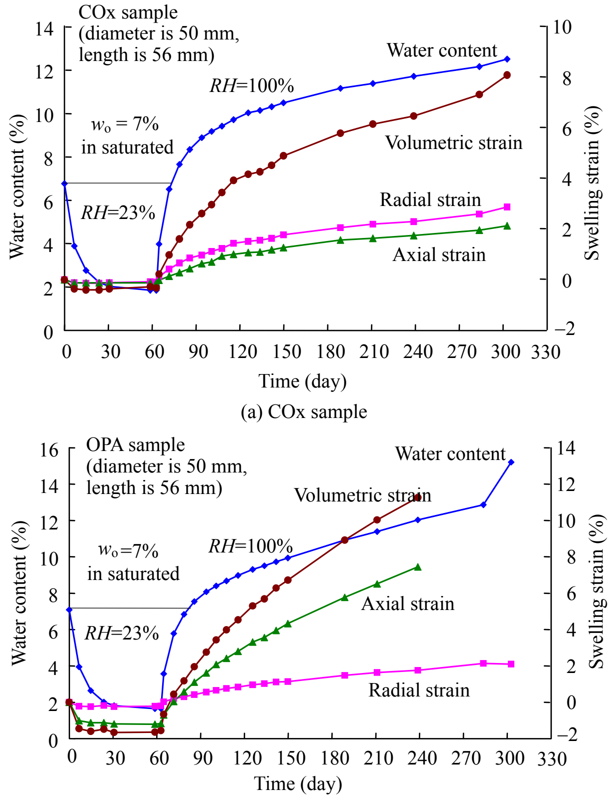
Fig.7 Response of uniaxial swelling pressure to drying and wetting recorded on a COx sample.

It is to be noted that the fluctuation of the measured swelling pressure may be caused by the local changes in the stiffness of clays during wetting, which decreases with the change of water content.

5 Measurements of swelling strain

In addition to the measurements of swelling pressure, the swelling potential of the mudstones has also been examined by measurements of swelling strain under various conditions.

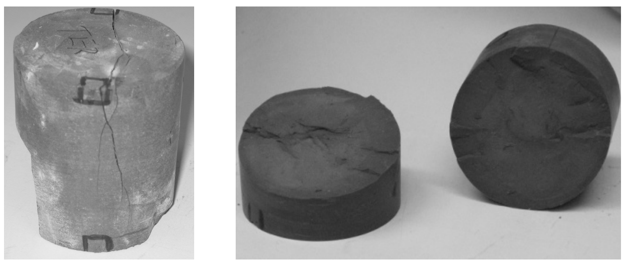
Free swelling tests were performed by wetting unconstrained samples with water vapor, during which the deformation and water content change were recorded. Figure 8 illustrates the evolution of measured strains on COx and OPA samples at a low relative humidity of RH = 23% (corresponding to the suction p_c = 200 MPa) for two months and 100% relative humidity (p_c = 0) for eight months, respectively. At the high suction applied externally, the water adsorbed on mineral surfaces evaporates and moves out from the pores, indicated by the reduction of the water content. The release of the pore water as stress-supporting element results in collapse of the pore structure and thus a macroscopic shrinkage. After reaching equilibrium during drying at RH = 23%, the remaining water contents in both samples are nearly the same (1.7%), but the shrinkages are different. A larger shrinkage of 1.6% was observed on the OPA sample with more clay content, about 5 times that of COx sample with less clay content. During the following wetting phase with water vapor at zero suction around the sample surface, the high suction potential of p_c = 200 MPa in the inner pores, which have been induced by the previous drying, drives water molecules to move into the pores, increasing the water content and hence the thickness of the water-films. The tests showed that the mudstones can take up great amounts of water, over 14%–18%, much more than that of about 7% in natural and saturated states. The increase of water content yielded a large volume expansion up to 8%–12%. More water uptake and volume expansion could be expected if wetting continued to reach equilibrium. This finding confirms that the matric suction dominated by the adsorption or swelling potential in the saturated mudstones is not zero after unloading (Eq.(9)). Additionally, it is to be pointed out that the swelling of the sedimentary mudstones is pronouncedly anisotropic due to bedding planes. A larger swelling strain occurs in direction perpendicular to the bedding plane (Fig.8). The anisotropic swelling is so strong that it is able to break down the mudstones along the bedding planes. Figure 9 shows the pictures of the samples after swelling.



(a) COx sample

(b) OPA sample

Fig.8 Free swelling strains measured on COx and OPA samples at various humidities (sample axis perpendicular to bedding plane).



(a) COx sample (b) OPA sample

Fig.9 Pictures of mudstones after free swelling.

On another COx sample, axial swelling was measured in a swelling cell by introducing synthetic formation water into the top and bottom at atmospheric pressure. The swelling curve is depicted in Fig.10. As the sample contacted with the water, a rapid expansion took place and then gradually increased with time up to 7% over 4 months. The swelling curve obtained during wetting with liquid water is quite similar to those observed during wetting with vapor (Fig.8).

The high swelling capacity of the mudstones was also confirmed by other tests under confining stresses. Figure 11 shows a triaxial swelling test on a COx sample under the originally lithostatic stress state of σ_1=

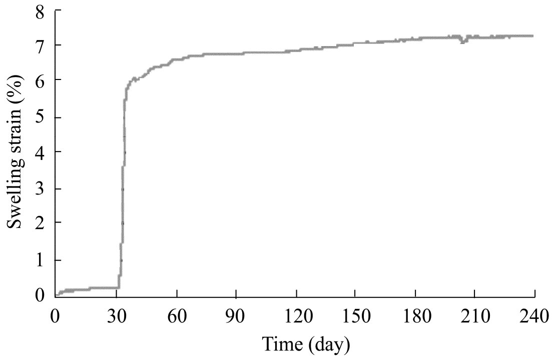


Fig.10 Axial free swelling strain measured on a COx sample with synthetic formation water.

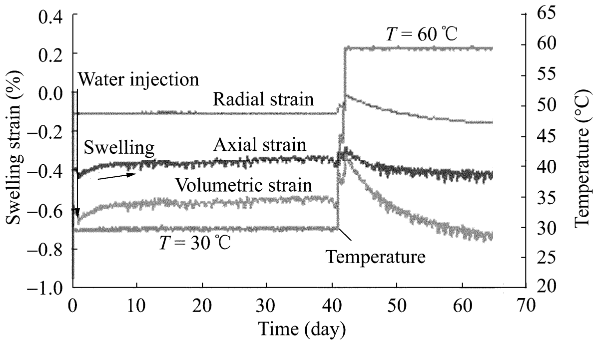


Fig.11 Swelling strains of a COx sample under high triaxial confining stresses.

15 MPa and $\sigma_2 = \sigma_3 = 12$ MPa. The in-situ water pressure $p_w = 4.5$ MPa was applied by injecting synthetic water to both ends of the sample. The strain curves show that a continuous expansion rather than compression occurred in axial direction even though the sample was subjected to the high effective compressive stress of 11.5 MPa in this direction. The gradual expansion suggests that the swelling potential (pressure) stored in the mudstone is larger than the difference between the externally applied total stress and the water pressure. The supporting capacity of the bound pore water, however, can be reduced by elevating the temperature. At high temperatures, the adsorbed water-films become thin due to the thermal-induced desorption of water. In drained conditions, the thermal-mobilized pore water is expelled from the sample under confining stresses, causing compaction of pores.

6 Water retention

The water retention function is a relation of matric suction with water content. Matric suction has two components, the first is associated with capillary forces acting gas-water interfaces, and the second with the adsorption forces of water mineral surfaces [1, 2]. The adsorption potential is an extremely important component of the total potential in compacted clays and mudstones.

The water retention curves of the studied mudstones were determined by measurements of water adsorption-desorption at various controlled relative humidities or suctions around samples. Figure 12 summarizes the relationship between the applied suction and the water content achieved after equilibrium on COx and OPA samples. By effect of high externally applied suction, the adsorbed pore water desorbs from the mineral surfaces and escapes from the pores. Conversely, when the external suction is lower than that of the inner pores, water migrates into the clay-water system. From Fig.12, it can be found that, at zero suction, the mudstones can take up large amounts of water up to 14% for COx sample and 18% for OPA sample, which were obtained over a period of 8 months when equilibrium was not yet achieved. The amounts of water uptake are significantly more than 7% around in the natural state, suggesting that if not all, most of the water content in the natural mudstones is bound on the surfaces of clay minerals. This reflects that the clay-water system is really a heavily dense colloid. It is also interesting to point out that the intersection of the natural water content of about 7% with the water retention curves gives the suction of about 6 MPa for OPA samples and about 15 MPa for COx samples, which are very close to the measured swelling pressures (Figs.6, 7) and also to the lithostatic stresses at sampling depths. This finding reveals again that the matric suction of the saturated mudstones is not zero but equal to the swelling pressure (Eq.(9)). In fact, the water retention function of mudstones is governed by the same adsorption-desorption phenomena that determine the swelling pressure.

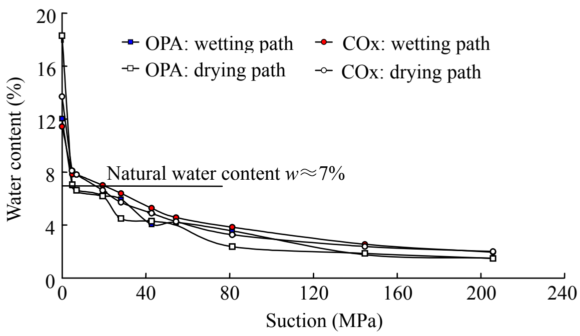


Fig.12 Water retention curves of COx and OPA samples.

Finally, it is to be noted that the adsorbed water can become free under certain conditions. The pore water in a saturated clay can be extracted by squeezing samples in laboratory [7]. In the underground laboratories at Bure in France and at Mont Terri in Switzerland, a small amount of water outflow from boreholes was observed, which may result from consolidation due to locally increasing stresses after borehole drilling. Another example is the observation of thermal-induced water flow during a heating experiment in the Opalinus clay [11].

7 Conclusions

Various swelling experiments were performed on the Callovo-Oxfordian and Opalinus mudstones. The tests included the measurements of water adsorption-desorption, swelling pressure and swelling strain on the samples in various suction-controlled and load-controlled conditions, from which the following conclusions can be drawn:

(1) The natural mudstones exhibit high adsorption potentials, under which a great amount of water can be taken up from the humid environment, much more than the water content in the natural and saturated states.

(2) The adsorbed water-films in the mudstones are capable of bearing external loads and carrying the lithostatic stress, indicated by the measurements of such high swelling pressures on the samples.

(3) The large amount of water uptake results in significant expansion of mudstones even under high lithostatic stresses and in-situ water pressures.

(4) All the test observations validate the colloid model and the associated stress concept, i.e. the swelling pressure acting on interparticle water-films is equal to the effective stress in compacted clays and mudstones.

Wide applications of the heavily dense clay-colloid model and the associated stress concept may be expected for improvement of understanding and modeling of strongly coupled hydro-mechanical processes in compacted clays and mudstones, for instance, water and gas transport, rheological deformation, self-sealing and healing of fractures in mudstones, etc..

Acknowledgements

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