Evaluation of structural modification of a hard deep clayey rock due to wetting/drying cycles

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EVALUATION OF STRUCTURAL MODIFICATION OF A HARD DEEP CLAYEY ROCK DUE TO WETTING / DRYING CYCLES

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1. ABSTRACT

New wetting / drying tests including cycles effects were performed on the same argillaceous deep rocks coming from Callovo-Oxfordien deposit from eastern France. Bulk volume and water content were measured in order to estimate the importance of physical changes. Then mercury injection tests were performed on dry and wet samples in order to quantify the influence of hydraulic history on fracture. Results showed that shrinkage and swelling originated micro-cracks which led to important volume variations, and thus strongly altered structures and organizations of the rock. Mercury injection tests performed on wet samples equilibrated with different hygrometric saline solutions allow describing unsaturated pores and complete classical water retention curves to explain changes in microstructure and related mechanical and physical properties.

2. INTRODUCTION

In underground structures different phenomena can occur with time. Considering construction and utilization phases, the rock can be potentially submitted to mechanical and hygrometric conditions (ventilation in order to let the workers breeze) which can lead to decrease the confining pressure and to desaturate the rock. Previous studies have been conducted on some French argillaceous rock in order to measure the influence of hygrometric changes during a triaxial test. The comparison of those results with classical swelling / shrinkage tests performed on unconfined samples showed that consolidation and reorganization of clay particles should be taken into account to explain the observed strain and encouraged further investigations [1].

3. PRESENTATION OF THE ROCK

The rock under study is a hard sandy which belongs to Callovo-Oxfordien and came from the east of France. It is found between 400 – 500 m depths.

3.1 Mineralogy and physical properties

Mineralogical analysis shows that the rock is composed by about 40 % of clay materials, 25 to 30 % of quartz and 20 to 30% of carbonates, mainly $CaCO_3$. Clay minerals are mainly interstratified illite-smectite minerals, but also contain illite and a small amount of kaolinite and chlorite. The clay mineralogy and content vary with depth.

Natural density is about 2.4, water content is quite small, between 5 and 9, void ratio is in the range of 15 to 20%. Permeability, measured in laboratory, is about 10^{-21} m^2 and in-situ measurements give a higher value -as usual-, about 10^{-19} m^2. Different types of microscopes were used to characterize the microstructure [2] and showed that the microstructure is quite important to explain the behavior of this rock: grains of quartz and calcite, which are about 200 μm in size, are surrounded by clayey particles. This previous study also showed that free

wetting and drying leads to micro cracks and voids of about 1 μm in size, at the periphery of quartz and calcite grains.

3.2 Mechanical properties

Triaxial compression tests were performed on undisturbed samples, in natural state and after 15 days of immersion in water [2]. The maximum strength and the Young modulus of the samples are reduced by a factor of one order of magnitude after immersion as it is shown on table 1.

	Natural state	After immersion
Max strength	40 MPa +/- 10 %	5 MPa +/- 10 %
Young modulus	4.2 GPa +/- 10 %	0.8 GPa +/- 20 %

Table 1 Comparison of mechanical properties consecutive to immersion in water

4. SITES AND METHODS

Two types of experiments have been realized both at the laboratory of soil science at the INRA Versailles (National French Institute for Agronomic Research) and at G3S (Group for the study of Underground Storage Structures) at the French Ecole Polytechnique.

4.1 Wetting – drying tests

Wetting-drying tests were performed at INRA in Versailles. Samples were subjected to various suction pressures: 1 kPa-1 MPa using pneumatic pressure cell (an original system described in (2) and Richard's apparatus). Beyond 1 MPa, equilibrium was obtained under 47 to 98% of relative humidity using saline saturated solutions. Four types of tests have been performed in order to submit the samples to various relative humidity and rates of wetting:

1. from the natural state, samples are both wetted and dried: the curve obtained can describe the reference sample,
2. from the natural state, samples are slowly wetted until to 1 kPa (Relative Humidity = 99.9993%) and then are subjected to a slow drying (slow means step by step, all the classical scale of partial pressure is used),
3. from the natural air dried state (about 47% of relative humidity) samples are wetted quickly (they are put into free water, HR=100%) and then they are slowly dried.
4. from the natural air dried state samples are slowly wetted, this is classical wetting / drying tests.

The samples used here are small pieces (about 1 or 2 cubic centimeter) provided from the same coring collected at 527 m depth). When equilibrated with each hygrometric state the water content was measured after heating in an oven at 105°C. Their volume was also measured by using the classical kerosene method explained in [3]. Thus the water content and other parameters were deduced. The results of the cycles are presented on figs 1 and 2.

4.2 Study on wet samples

Samples used in those tests provided from the same boring but not at exactly the same depth (491.5 m depth). These are also small pieces of core (less than 1 cubic centimeter). The experiment consists in putting samples in different small chambers -each chamber is submitted to a different relative humidity using the saline saturated solutions method-, and in measuring the evolution of the weight of the samples at differents times. Eight relative humidities are used: 98%, 95%, 90%, 81%, 79.5%, 66%, 32.5% and 20%. Four to six samples are tested for each relative humidity in order to have a range of results and to avoid strange behavior. Only the evolution of weight is measured on those samples (not volume), which can't give the water content and other parameters. This has been chosen to affect the sample as less as possible in order to study the microfissuration which is really obtained by wetting or

drying at different hygrometric steps and which not the microfissuration induced by introduction in an oven.

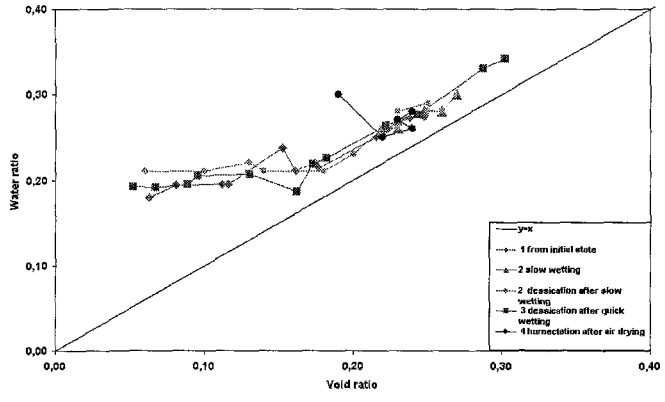

Fig. 1 : wetting / drying cycles – water ratio versus void ratio

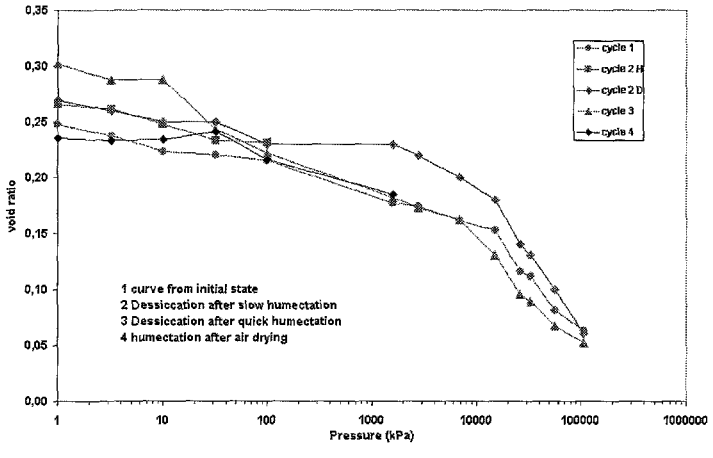

Fig. 2 : wetting / drying cycles – void ratio versus suction pressure

4.3 Mercury injection analyses

After those different tests, all samples were studied by mercury injection analyses. Those analyses were carried out on samples in their final states, i.e. on dry samples when they come from INRA and on wet samples when they come from G3S.

A ThermoQuest apparatus, Pascal 440, was used. It can reach a pressure of 400 MPa. Considering [4], it allows studying pore radii up to 0.35 nm, which is quite a good resolution but probably not completely reached in reality.

Wet samples were studied in order to characterize pores which were really free of water. We want to point out that those results must be used very carefully because of different possible artifacts which can occur when water is present in the sample (additional gravity forces, water compressibility at high pressure, etc.) but in this article we want to focus on qualitative results only.

5. RESULTS

5.1 Results on dry samples

Because of drying in oven, we will focus on porosimetric results dealing with macro-porosity only. We won't give all the porosimetric curves in order not to be too dense. Figs. 3, 4 give respectively partial results of mercury injection curves (cumulative volume versus pore radius) obtained for two of the four tests (so called, 2:"drying after slow wetting", 3:"drying after fast wetting").

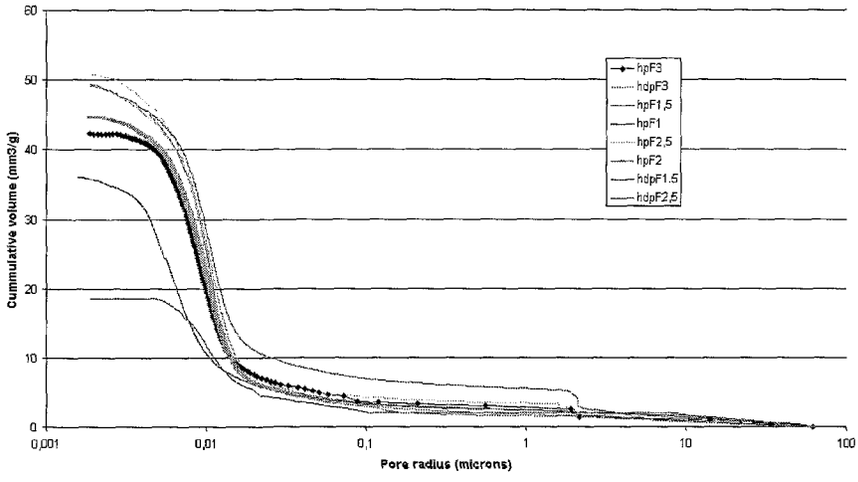

Fig. 3 : Mercury injection curve for humidity steps reach from drying after slow wetting (dry samples)

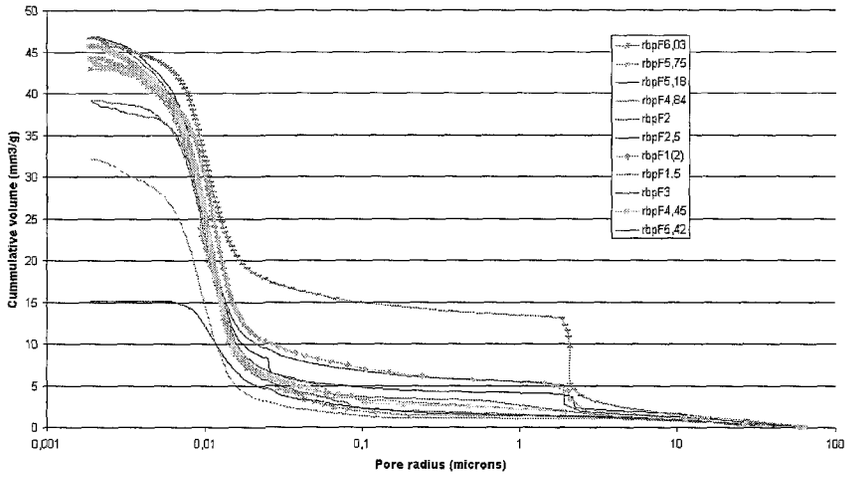

Fig. 4 : Mercury injection curve for humidity steps reach from drying after fast wetting realised on dry samples

Different aspects can be pointed out from the study of all the mercury injection curves :

- Repartition of pore radius seems to be nearly the same for each hygrometric step. it means that it seems neither to depend on the way the step is reached (quick or slow wetting) nor on the value of the hygrometric step (the range of the variation of the repartition of pore radius seems to be quite equal to dispersion results).
- Results obtained on "cycle 2" (desiccation after slow wetting) seem to show that the more the sample is wetted (small water pressure), the flatter the cumulative curve is,
- Two pore radius modes can be pointed out for 10-15 nm and for 2 μm.

5.2 Results on wet samples

Fig. 5 shows mercury injection curves obtained on wet samples. As we mentioned before, it is really hard to interpret quantitatively those curves. It has been partially done but it is not the purpose of our article. what we want to do is focus on the qualitative points of the method :

- If we don't take the RH 66 into account in the micro pores range, all the curves are in the expected order, i.e. the wetter the sample is, the less open the micro pores are to mercury injection. This fact illustrates that the method works,
- The range of the results is much more important on wet samples than on dry one. This could be attributed to the form of the pores (not perfect cylinders),
- We find the two same pore radius modes that in the dry samples even if the 2 μm one is not as clearly marked.

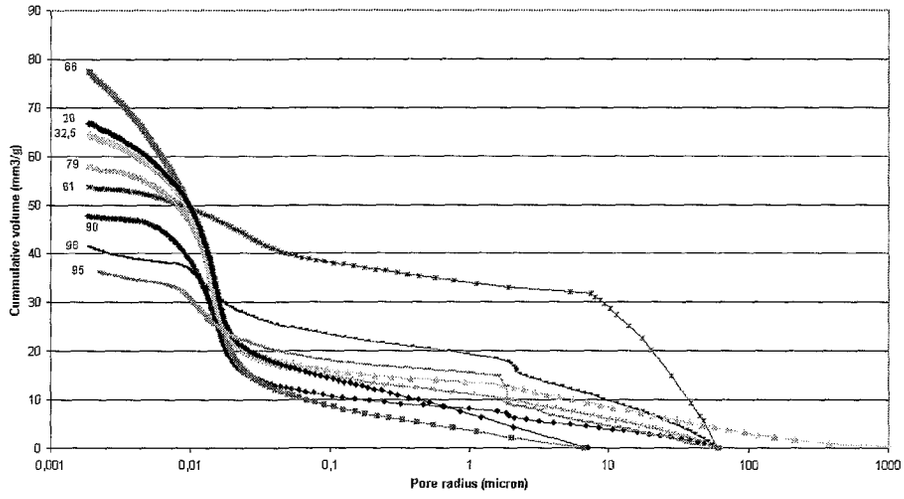

Fig. 5 : Mercury injection curves on wet samples

5.3 Partial conclusion

The first important result is that -even if it can't be seen on the curves- the samples which were submitted to fast wetting (immersed in free water) are much more fractured than the others. Those samples have often split in two or three smaller pieces. The second important point is that the sensitivity of the sample to flaking or fracturation depends on sample characteristics. For instance other samples on the same clayey rock but from an other bore presented in [2] were less sensitive to fracturation.

Another important result is that mercury injection shows two important pore radius modes: one at about 2 μm and the other at about 10 to 20 nm. Those pore radii can be better seen on dry samples but they are also characteristic of the wet samples. Thus those cracks

can be generated both by drying and wetting but they seem to be characteristic of the sample macrostructure and not only on the mean used.

6. DISCUSSION-CONCLUSION

The combination of different techniques allowed us to characterize a highly consolidated clayey rock at different scale of its structural organization. Water retention curves done on large energy range can be used to characterize a large spectrum of pores, i.e. from 150 µm at 1kPa to about 1nm at 50 % RH (100 MPa). Nevertheless two techniques have to be used, i.e. gas pressure cell and relative humidity equilibrium to build up those curves. Mercury injection also covers a similar pore size distribution. We attempted to use this method on samples prepared at different rates of saturation in order to characterize free water pores. A combination with classical mercury injection experiments on air-dry samples was made.

There is a very good similarity between water retention results and mercury injection curve. The two methods confirm that there are two main pore size modes in our clayey material, i.e. at about 2 µm and at about 15 nm. This feature is clearly demonstrated whatever the stress history and the preparation mode of the sample. Moreover mercury injections before and after desiccation showed similar pore sizes but the magnitude of each family varies to a large extent. Thus, those pores have to be considered as the main feature characterizing the microstructure of such a material, in any of its properties.

It is also interesting to point out that those pores can be really seen on the partly saturated samples, i.e. without desiccation in oven. Thus one of the main results of this work is to demonstrate that drying or wetting does not change the pore size distribution modes but only their magnitude.

The origin of the pores can then be discussed. It is known that 10 nm pores are rather characteristic of the clay fabric, especially for illitic materials. Our results show that there is a very good similarity between pore size distribution deduced from mercury injections and from water retention curves. For instance, between 1 and 10 MPa and on the basis of Kelvin's equation, only 100 to 10 nm are filled with water. In the same way, close to 100 kPa, pores filled with water are close to 1-2 µm. These pores are in fact those concerned with other levels of the structural organization of the material. As shown before [1], with a SEM and a confocal laser microscope, such discontinuities correspond to the packing voids of the coarse grain fraction, i.e. between the clay mass and the coarse particles such as quartz and calcite grains.

In other words, any change in properties is primarily due to changes in the microstructure of the rock at these two levels. We can notice that the changes can happen during or after drying or wetting. Thus any stress history should be considered in order to interpret material changes. This sort of result can be used to discuss properties especially coupled hydro mechanical properties

7. BIBLIOGRAPHICAL REFERENCES

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