LOW WATER PERMEABILITY MEASUREMENTS OF CLAY SAMPLE. CONTRIBUTION OF STEADY STATE METHOD COMPARED TO TRANSIENT METHODS

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ABSTRACT

Very low permeability geomaterials (order of nanoDarcy (10^{-21} m^2)), such as clays rocks, are studied for many industrial applications such as production from unconventional reserves of oil and gas, CO_2 geological storage and deep geological disposal of high-level long-lived nuclear wastes. For these last two applications, clay efficiency as barrier relies mainly on their very low permeability. Laboratory measurement of low permeability to water (below 100nD (10^{-19} m^2)) remains a technical challenge. Some authors [1] argue that steady state methods are irrelevant due to the time required to stabilize water fluxes in such low permeability media. Most of the authors (e.g. [2]) measuring low permeabilities use a transient technique called pulse decay. This study aims to compare objectively these different types of permeability tests performed on a single clay sample.

For the steady state method, a high precision pump was used to impose a pressure gradient and to measure the small resulting water flow rate at steady state. We show that with a suitable set-up, the steady state method enables to measure a very low permeability of 0.8 nD (8 10^{-22} m^2) in a period of three days. For a comparable duration, the pulse decay test, most commonly used for such low permeability measurements, provides only an average estimate of the permeability. Permeability measurements by pulse decay require to perform simulations to interpret the pressure relaxation signals. Many uncertainties remain such as the determination of the reservoirs storage factor, micro leakage effect, or the determination of the initial pulse pressure. All these uncertainties have a very significant impact on the determination of sample permeability and specific storage.

Opposite to the wide-spread idea that transient techniques are required to measure very low permeability, we show that direct steady state measurement of water permeability with suitable equipments can be much faster and more accurate than measurement by pulse decay, especially in very low permeability porous media. In fact, low compressibilities of water and clay result in fast propagation of pressure wave and it cannot be argued that steady state conditions are not reachable in a reasonable amount of time.

INTRODUCTION

In the last decades, new challenges appeared in the field of geosciences: radioactive wastes disposal, geological storage of CO_2 and gas and oil production from unconventional reservoirs. Amongst the candidate geological formations that are foreseen as potential host-rocks for radioactive waste disposals, clay formations are good candidates and have already been selected by some national agencies [3]. The very low permeability of these formations will prevent the radionuclides migration to biosphere for a long period of time. Potential site for CO_2 geological storage are selected from their storage capacity and from their cap rocks sealing property [4]. Permeability of cap rocks should be as low as possible to minimize any CO_2 leakage towards the surface. In oil and gas industry, new hydrocarbon sources represent new challenges: tight reservoirs or overpressure zones. Some overpressures, due to low permeability rocks unable to release water pressure from compaction [5], can lead to major drilling problems. Permeability is a key parameter since it controls fluid migration. However, laboratory measurement of low permeability (< 100nD (10^{-19} m^2)) remains a technical challenge. Most of the authors dealing with permeability measurements of clays use a transient method known as the pulse decay [2]. Its principle, illustrated in Figure 1, has been firstly proposed by Brace [6]. It consists in a sample bounded by two reservoirs initially at equal pressures. A pressure rise is suddenly imposed in the upstream reservoir and the pressure evolution is recorded in both reservoirs. Determination of the permeability is made on the transient phase leading to pressure equilibrium in the reservoirs. Pulse decay is almost always preferred to the steady state method, which consists in imposing a pressure gradient over a sample and measuring the flow rate out of the sample. Many authors argued that the steady state method leads to very long experiment durations compared to a pulse decay test, due to the long time required for water flow stabilization [1] [7] [8]. This opinion is shared by other authors dealing with different low permeability media such as cement pastes [9] [10].

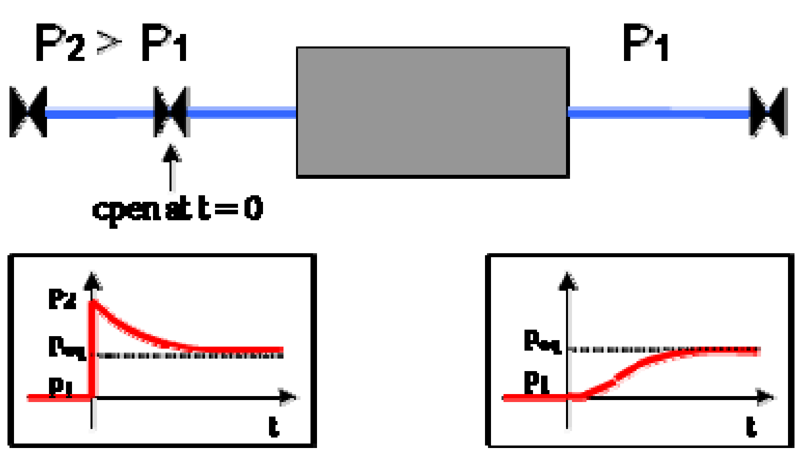

Figure 1. Principle of the pulse decay method

In the present paper, a specific clay sample was chosen for permeability measurement. Transient and steady state methods were performed and compared. This study focuses on demonstrating that the steady state method can be actually as fast as the pulse decay method. Additionally, special attention is given to compare the reliability of these two techniques.

MATERIALS AND METHODS

Experimental materials

The studied sample is an Upper Torcian argillite provided by IRSN (Institute of Radiation Protection and Nuclear Safety), extracted from well M6 of the Tournemire tunnel in Aveyron, France. The cylindrical core of 40mm diameter and 20mm length was placed in a Hassler cell with a confining pressure of 150 bar (15 MPa). The experimental set-up is presented on Figure 2.

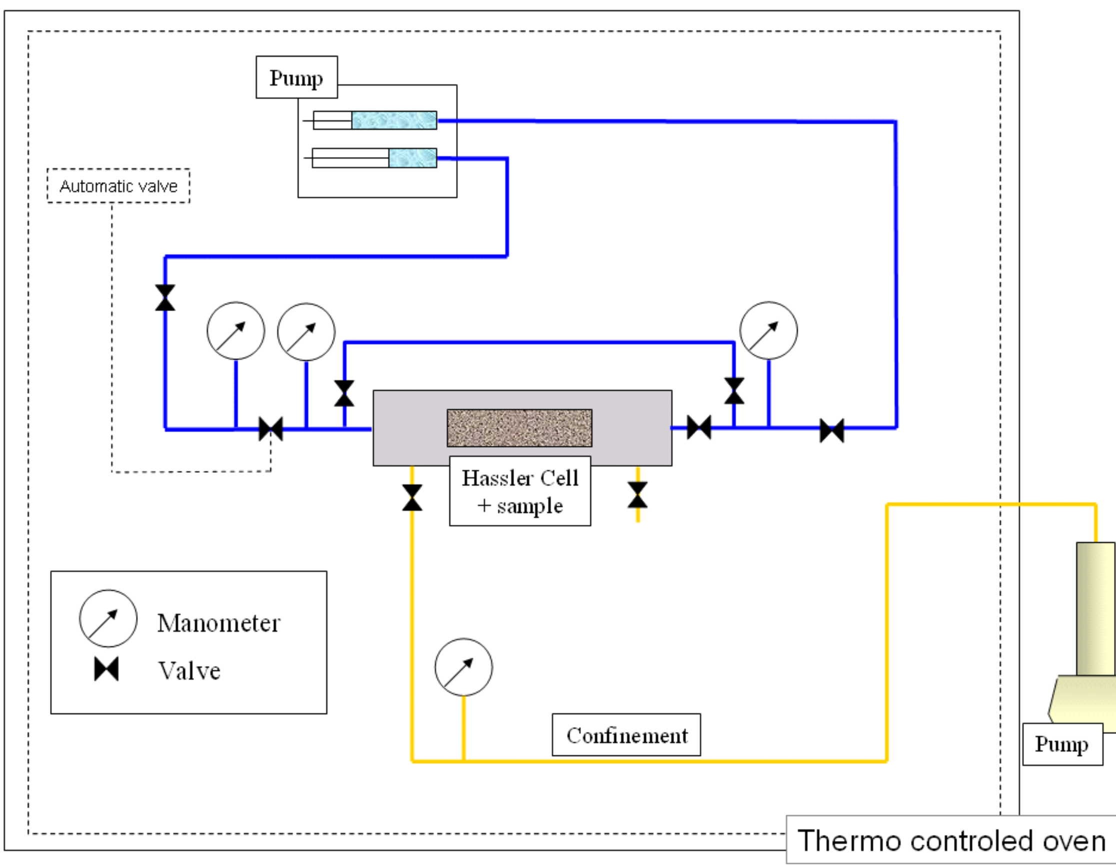

Figure 2. Experimental set up used for steady state and transient methods for permeability measurement (Upstream volume equal to 6.82 cc and Downstream volume equal to 4.74 cc).

The pump (QX 20K) is composed of two pistons cylinder with volume of 4 cc each. Each piston can be operated independently and monitored while imposing a constant flow rate or a constant pressure. One was connected to a side of the Hassler cell and the other one to the opposite side. The Quizix pump controlled water pressure in the whole system. Each experiment was carried out with an average water pressure of 100 bar (10 MPa). Water was initially equilibrated with clay fragments resulting from coring to prevent structural damages due to geochemical reactions. In closed reservoir configuration, temperature must be regulated carefully since water thermal expansion can affect measurements of small volumes [11] and pressures. In our closed reservoir configuration, a temperature fluctuation of 1°C would lead to a pressure increment of 5 bar (5 MPa), accordingly the temperature was maintained at 25°C +/- 0.1. Three different experiments were performed: first, a steady state experiment to measure directly the permeability (k_w in m^2); then, a Pore Pressure Transmission Test to obtain an estimation of the specific storage (S_s in m^{-1}); and finally, a pulse decay test to estimate both k_w and S_s.

Steady State method

Darcy's law describes the flow induced by a pressure gradient within a porous media:

$$q = \frac{k_w}{\mu_w} \cdot \nabla P_w , \tag{1}$$

where q is the Darcy velocity (m/s), k_w the permeability of the porous media (m^2), μ_w the water dynamic viscosity (Pa.s) and ∇P_w the water pressure gradient. The corresponding flow rate Q (m^3/s) is q times A, where A is the sample surface (m^2). The permeability can then be estimated from the relationship:

$$k = \frac{Q}{A} \cdot \frac{\mu_w \cdot L}{(P_u - P_d)} , \tag{2}$$

where P_u and P_d are respectively the upstream and downstream pressures (Pa), L being the sample length (m). Upstream and downstream pressures were maintained independently by each piston. Upstream pressure was set successively to 105, 107.5, 110 and 112.5 bar (10.5 MPa, 10.75, 11 and 11.25 MPa), while corresponding downstream pressure was set respectively to 95, 92.5, 90 and 87.5 bar (9.5, 9.25, 9 and 8.75 MPa) in such a way that the mean pore pressure was maintained at 100 bar (10 MPa); the imposed pressure gradients were thus 10, 15, 20 and 25 bar (1, 1.5, 2 and 2.5 MPa) and each pressure gradient step lasted about twenty hours. To accommodate the water flow induced by the pressure gradient, the pistons moved in a "push-pull" configuration (Fig 3.). The displacements of the pistons are plotted with time and once their evolutions become linear, the slopes corresponding to the upstream and downstream water flows are reported. Then the successive flow rates (Q) are plotted against the pressure gradient (P_u-P_d). According to (2), the four points obtained should align along a slope proportional to the permeability.

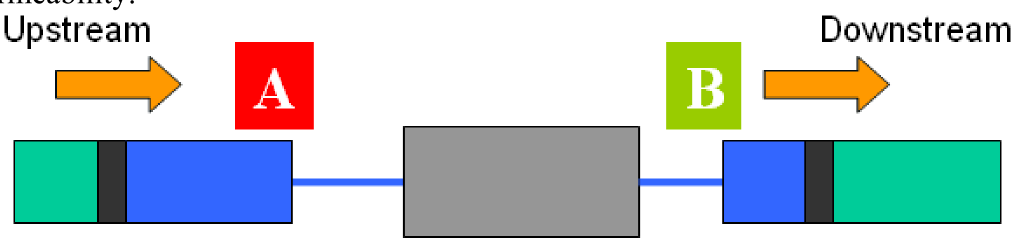

Figure 3. Steady state method by "push-pull" using a dual piston pump.

Up to now, such experiments using "push-pull" configuration, commonly carried out on porous media with intermediate to high permeability [12], have never been performed on very low permeability media.

Pore Pressure Transmission Test (PPTT)

A PPTT is nothing else than a pulse decay test with a constant pressure condition at one boundary. Prior to the tests, water pressure was maintained at 95 bar (9.5 MPa) for twelve hours. Then the downstream reservoir was closed and the upstream pressure was increased to 105 bar (10.5 MPa)(see Figure 4.).

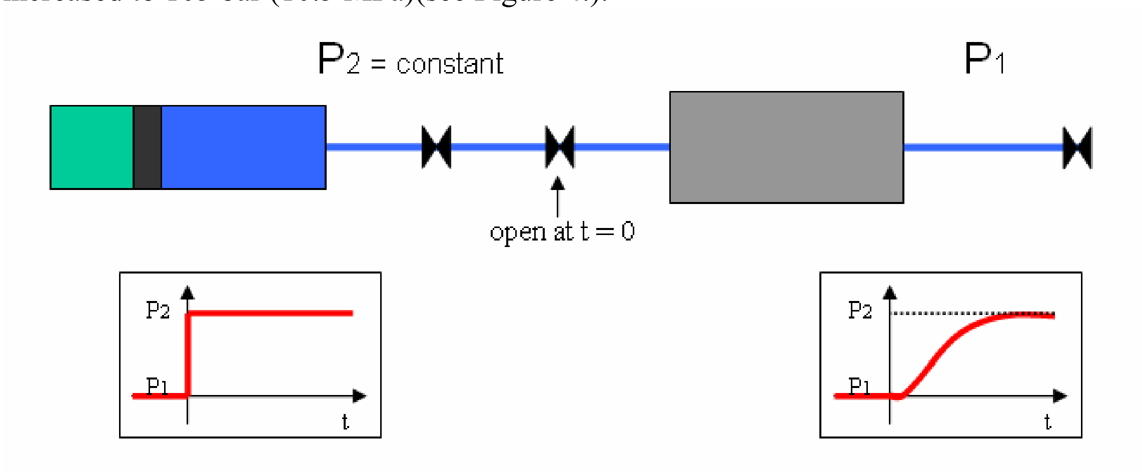

Figure 4. Principle of the PPTT.

Downstream pressure evolution with time was compared to simulations based on the following system of equations with the mass balance (3) and the two pressure boundary conditions (4) and (5) (Escoffier et al., 2005):

$$\beta_M \frac{\partial P_w}{\partial t} = -\nabla\left(\frac{k_w}{\mu_w} \cdot \nabla P_w\right), \tag{3}$$

$$\frac{\partial P_d}{\partial t} \cdot \frac{\mu_w \cdot S_d}{k_w \cdot \rho_w \cdot g} \cdot A - \left(\frac{\partial P_w}{\partial x}\right)_{x=L} = 0, \tag{4}$$

$$P_u = \text{constant}, \tag{5}$$

g is the gravity acceleration (9.81 m^2/s) and ρ_w the water density. β_M is the apparent compressibility (Pa^{-1}) of the matrix linked to the specific storage of the sample S_s by:

$$S_s = \rho_w \cdot g \cdot \beta_M \,. \tag{6}$$

S_s (m^{-1}) corresponds to the volume of water over the total volume of the rock which can be stored per unit of water head change [12]. S_d (m^2) in equation (4) is the storage factor of the downstream reservoir, which should be assessed before the test. Simulations were performed with the finite element commercial software COMSOL Multiphysics [14].

Pulse Decay test

A pulse decay test was performed on the experimental set-up presented in Figure 2. An automatic pneumatic valve placed in the upstream reservoir generates once closed a pressure rise of the order of 10 bar (1 MPa). Pressure evolution in the system was compared to simulations based on the system of equations (3), (4) and (7):

$$\frac{\partial P_u}{\partial t} \cdot \frac{\mu_w \cdot S_u}{k_w \cdot \rho_w \cdot g} \cdot A + \left(\frac{\partial P_w}{\partial x}\right)_{x=0} = 0 , \tag{7}$$

where S_u (m^2) is the storage factor of the upstream reservoir.

Storage Factors

Prior PPTT and pulse decay tests, S_d and S_u are important parameters that should be assessed. They correspond to the volumes of water that are required to increase pressure within the reservoirs:

$$S_{(u,d)} = \rho_w \cdot g \cdot \frac{dV_{(u,d)}}{dP_w} . \tag{8}$$

To measure S_u and S_d, the sample was replaced by a steel cylinder (protocol similar to Larive's work [15]). Confining pressure was set to 350 bar (35 MPa). Water pressure was increased first by steps of 20 bar (from 20 to 100 bar (2 MPa to 10 MPa)) and then by steps of 50 bar (from 100 to 300 bar (10 to 30 MPa)) and the volume of water needed to pressurize each reservoir was recorded (pistons displacement). S_d and S_u were estimated for each step by:

$$S_{(u,d)}\left(\frac{P_{w,i+1} + P_{w,i}}{2}\right) = \rho_w \cdot g \cdot \frac{V_{w,i+1} - V_{w,i}}{P_{w,i+1} - P_{w,i}} . \tag{9}$$

RESULTS AND DISCUSSION

Storage Factors of reservoirs

Figure 5 shows the evolution with pressure of the upstream and downstream storage factor. Upstream storage factor decreases from 2.10^{-10} m^2 to 6.10^{-11} m^2 and downstream storage factor from 1.10^{-10} m^2 to 4.10^{-11} m^2. Reservoirs are highly compressible at low pressure. Measurement at pore pressure lower than 80 bar (8 MPa) would thus lead to major uncertainties on the interpretation of transient experiments since storage factors would clearly not remain constant during the experiments.

To assess transient experiments, S_u and S_d were set respectively to 7.5 10^{-11} m^2 and 4.5 10^{-11} m^2 in the model which are the average values at water pressure of 100 bar (10 MPa). It corresponds to an average compressibility of 10^{-9} Pa^{-1} for the two reservoirs. Compressibility of the reservoir is half part due to the water compressibility ($\beta_w = 5\ 10^{-10}$ Pa^{-1}) and half part due to the material compressibility.

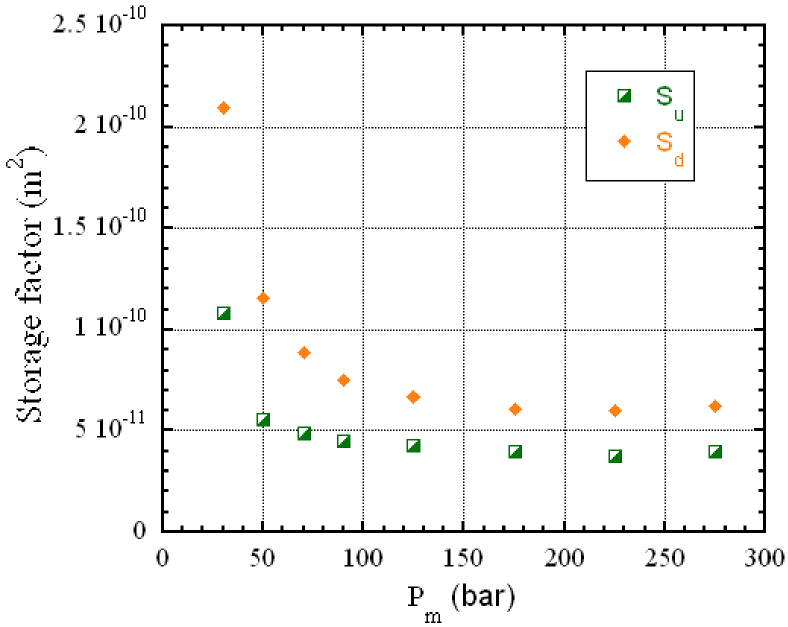

Figure 5. Upstream and downstream reservoirs storage factors evolutions with pressure.

Steady State experiment

Figure 6 shows the displacements of the pistons A and B during the four imposed pressure gradients, lasting three days. For each gradient, less than three hours were necessary to get the water flow stabilized. Flow rates are estimated on the linear parts of the displacements. Each pressure gradient was maintained twenty hours; we estimated that with the resolution of the pump, the flow rates could be determined fairly well within five hours; naturally, longer experiment would provide a better precision on the flow rates. Measured flow rates range from 0.005 cc/day to 0.01 cc/day. Figure 7 displays the water flow rates against the imposed pressure gradients. The linear regressions of Q versus ΔP for upstream and downstream displacements give respectively a permeability of 0.78 nD ($7.8\ 10^{-22}\ m^2$) and 0.87 nD ($8.7\ 10^{-22}\ m^2$). An average value of 0.82 nD ($8.2\ 10^{-22}\ m^2$) is chosen for further calculations.

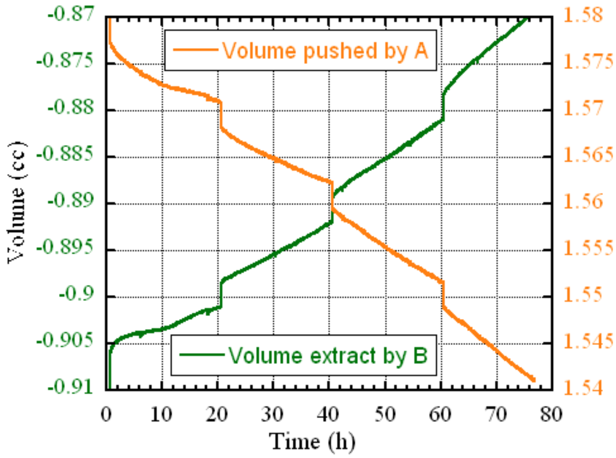

Figure 6. Measurement of the piston displacements during "push-pull".

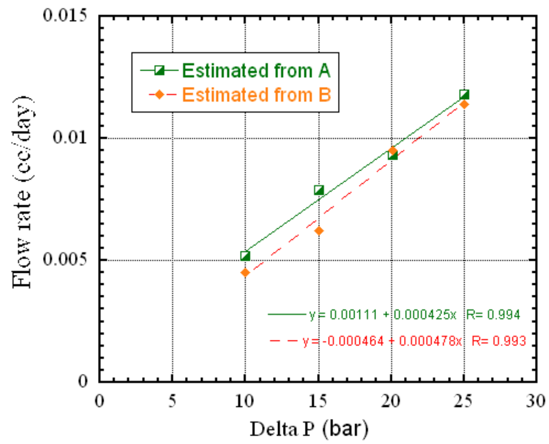

Figure 7. Evolution of the water fluxes with the pressure gradient.

Measurement of the storage factor

The PPTT lasted six days. This was not the necessary time for the downstream pressure to reach equilibrium (see Figure 8). Yet, the experimental data are representative enough and exploitable for interpretation before this equilibrium. Simulated curves, for different set of (k_w, S_s) values, can all describe the experimental data reasonably and up to this point there is no unique solution. In fact, such experiment would require a downstream reservoir volume large enough to distinguish the independent effects of the parameters k_w and S_s on the shape of the pressure increase, but larger volume would also involve longer experiment which is unrealistic. The permeability of 0.82 nD (8.2 10^{-22} m^2) previously determined, provides a S_s value of 3.4 10^{-6} m^{-1}.

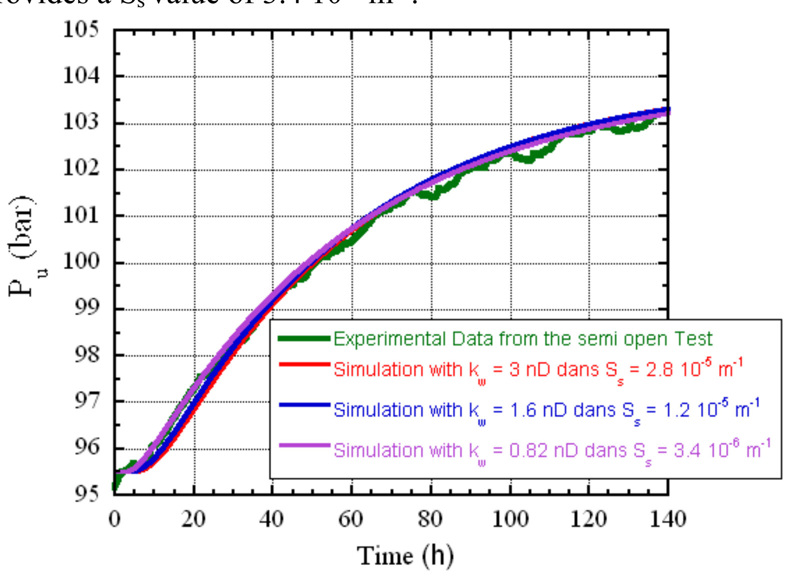

Figure 8. Downstream pressure increase during PPTT. Comparison with simulations.

Interpretation of pulse decay test

The pulse decay test lasted three days. The experimental data are compared with simulations made with the set of (k_w, S_s) values estimated respectively from steady state experiment and PPTT. As shown on Figure 9, simulation (red curves) does not match the experimental data (green curves). In fact, P_u relaxed at a lower value than P_d which might be due to a micro leak localized in the upstream reservoir. A second simulation (blue curves) was therefore carried out by integrating a constant leak of 1.2 Pa/s in the upstream reservoir allowing simulated curves to match the experimental data. A drop of 1.2 Pa/s represents a leak of 5 10^{-4} cc/day which is undetectable with our experimental set-up. Figure 10 shows simulations performed to fit independently P_u or P_d. k_w lies between 0.2 nD (2 10^{-22} m^2) and 2 nD (2 10^{-21} m^2). Thus, pulse decay test provides the order of magnitude of the permeability, however due to very small leakage inherent to such low permeability set-up, k_w values cannot be estimated without major uncertainties.

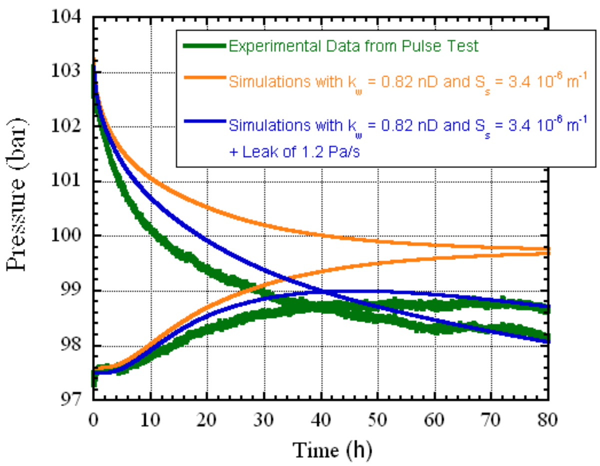

Figure 9. Downstream and upstream pressure evolution during pulse decay test. Experimental data compared to simulations (with and without leakage).

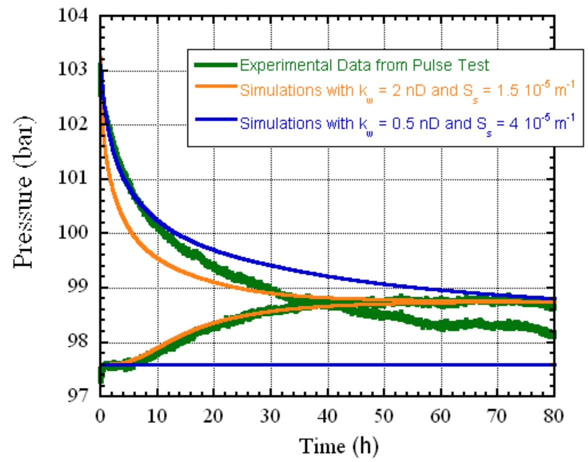

Figure 10. Experimental data compared to the simulations made on each pressure evolution during pulse decay test.

The additional uncertainties making difficult a proper estimation of k_w are:

- The accurate estimation of the initial pore pressure induced by the pulse at the upstream reservoir.
- Since the estimation of k_w is not direct and coupled to S_s, uncertainties on S_s lead to uncertainties on k_w.
- The uncertainties on the specific storage factors of the two reservoirs, S_u and S_d, have also a direct impact on (k_w,S_s) estimation.

DISCUSSION

Duration of the experiments

Few steady state experiments have been reported in the literature on low permeable media [16] [11]. In general, pulse decay method is preferred since the duration of the experiment seems shorter. However, this study shows that the two methods can be comparable in time. Flux stabilization is the result of pore pressure equilibration within the sample. Pressure wave propagation is known to be fast in porous media and is proportional to k_w/S_s (in [6] it is supposed to be instantaneous). The pore pressure equilibriums within the sample are presented in Figure 11 for steady state and pulse decay tests. During a transient test, the downstream pressure can only start to increase when the pressure wave has propagated through the sample. On Figures 8 and 9, the pore pressure increase happened after three to five hours of test which is consistent with the time required for flow stabilization observed on Figure 6 and Figure 11. Thus, pulse decay cannot be preferred to steady state experiments on the statement that to establish steady state conditions, very long periods of time are required.

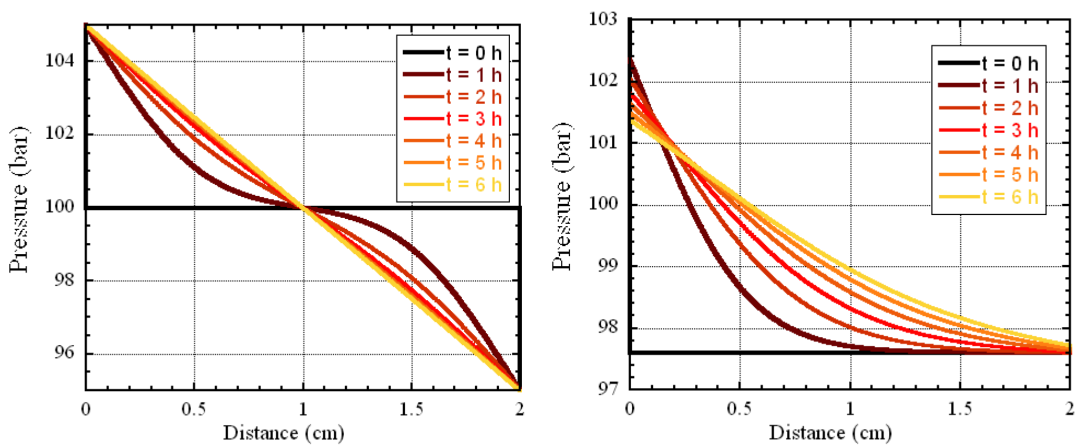

Figure 11. Pressure stabilization after 6h within the clay sample at 0.82 nD (steady state and pulse)

On Figure 11, we observe that pore pressure stabilization is fast. Duration of a steady state test is therefore only depending on the time required to be able to measure the water flow rate. The water flow is estimated on the volume that accumulates at the downstream reservoir. The duration of the test then depends only on the resolution of the pump; the smaller the volumes measurable by the pump, the shorter the time required to evaluate the water flow.

General recommendations

The measurement of a permeability of 0.8 nD (8 10^{-22} m^2) lasted three days by the steady state technique. It would have been possible to shorten the experiment duration to one day if only one pressure gradient was applied or if a sample of larger diameter was used (large diameter leads to large flow rate, easier to measure in a shorter time period). However, it is recommended to perform at least three different pressure gradients in order to verify the Darcy's law (debatable assumption in nD porous medium) and to avoid any problems due to possible leakages. Meanwhile, even with a leak, the linear regressions on Figure 7 remain proportional to k_w.

Nevertheless, transient experiments such as pulse decay test have in principle others advantages in comparison to steady state methods:

- First, S_s can be estimated. However, examples on Figure 10 show that the estimation may be poor. PPTT would be preferred (it can be shortened by stopping the experiment after the first increase of downstream pressure).
- It is also possible to highlight the effects of heterogeneities [17].
- Finally, the applied effective stress is more homogeneously distributed within the sample. Indeed, in steady state, effective stress is different between the top to the bottom of the sample and is more important ($\Delta P_{max,SS}$=30bar > $\Delta P_{max,PD}$=10 bar).

Pulse decay test can be done easily when permeability is larger than 10 nD (10^{-20} m^2), without being impacted by uncertainties linked to leakage and temperature variations. Pulse decay test should be done in a range of pore pressure where reservoirs storage factors are known to be constant (> 80 bar in our set-up). As piston displacements can quantify very small variations of water volume, it was possible to determine the volume of water drained out of the sample when the confining pressure was increased. The "push-pull" configuration allowed us to determine the mechanical equilibrium after each confining pressure increase. The permeability measurement could be performed only once the strains were stabilized, otherwise it would have led to erroneous measurements. For the considered shale sample, a delay of one day was required after each confining pressure increase, before the permeability measurement. This stabilization delay should be assessed if the evolution of permeability with stress variations is investigated.

CONCLUSION

This study shows that, for very low permeability media such as clays, the steady state method should be preferred to transient techniques like pulse decay tests. With an appropriate set-up, the experiments can be shorter and also more reliable. Steady state experiment duration depends mainly on the time needed to estimate the water flow and not on the time required for flow stabilization. This measurement duration becomes short as soon as a high volume resolution pump is used. Pulse decay test can only provide an estimation of the order of magnitude of the permeability. Indeed, the permeability estimated from a pulse decay test depends on many uncertainties such as leaks, proper pressure pulse estimation and determination of the reservoir specific storages.

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