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Contribution of the Steady State Method to Water Permeability Measurement in Very Low Permeability Porous Media

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Résumé — Contribution de la méthode stationnaire dans les mesures des très faibles perméabilités à l'eau — Les milieux très peu perméables (k < 10 nD (10^{-20} m^2)) comme les argiles sont étudiés dans le cadre de problématiques très diverses telles que le stockage du CO_2, les surpressions en forage profond ou le stockage des déchets radioactifs. La caractérisation pétrophysique de ces roches, et notamment la mesure de leur faible perméabilité, est difficile. La technique en laboratoire la plus répandue est celle du *pulse decay*. Cette technique consiste à imposer un *pulse* de pression en amont de l'échantillon puis de suivre l'évolution des pressions amont et aval. La durée de l'essai est supérieure à la journée pour des échantillons d'un nanoDarcy. Le signal est fortement tributaire des fuites dans le système ou des variations de température. La méthode stationnaire consiste à mesurer directement le débit d'eau à travers l'échantillon pour un gradient de pression donné. Cette technique est rarement utilisée pour les milieux très peu perméables car réputée très longue (Hsieh *et al.*, 1981, *Int. J. Rock Mech. Min. Sci. Geomech. Abstr.* **18**, 245-252). Des essais en laboratoire ont été réalisés sur trois échantillons. La méthode stationnaire ainsi que des méthodes transitoires (dont le *pulse decay*) furent employées. La limitation principale des méthodes transitoires est la difficulté d'interpréter correctement les profils de relaxation. La méthode stationnaire s'est montrée aussi rapide (trois jours pour un échantillon de 0.8 nD (8×10^{-22} m^2)), voire plus rapide (moins d'une journée pour un échantillon de 2.6 nD (2.6×10^{-21} m^2)), que la méthode du *pulse decay*. La perméabilité a été plus simple à déterminer et cette méthode peut être rapide avec un équipement adapté. En réalité, la faible compressibilité de la roche permet une propagation rapide des ondes de pression. De ce fait, il ne peut pas être soutenu que les conditions de régime permanent ne soient pas atteintes en un temps raisonnable. La méthode *pulse decay* reste une alternative intéressante à la méthode stationnaire lorsque la perméabilité est supérieure à 50 nD (5×10^{-20} m^2).

Abstract — Contribution of the Steady State Method to Water Permeability Measurement in Very Low Permeability Porous Media — *Very low permeability geomaterials (order of nanoDarcy (10^{-21} m^2)), such as clay rocks, are of interest for many industrial applications including production from unconventional reserves of oil and gas, CO_2 geological storage and deep geological disposal of high-level long-lived radioactive waste. In these last two applications, the efficiency of clay, as a barrier, relies on their very low permeability. Yet, laboratory measurement of low permeability to water (below 100 nD (10^{-19} m^2)) remains a technical challenge. Some authors (Hsieh* et al.*, 1981, Int. J. Rock Mech. Min. Sci. Geomech. Abstr.* **18***, 245-252) argue that steady state methods are irrelevant due to the time required to stabilize water fluxes in such low permeability media and prefer* *a transient technique called pulse decay. This study aims to perform and compare transient and steady state techniques on three samples. Regarding the steady state method, a high precision pump was used to measure water flow rate through the sample. We show that with a suitable set-up, the steady state method enables us to measure a very low permeability of 0.8 nD (8×10^{-22} m^2) over a period of three days and 2.6 nD (2.6×10^{-21} m^2) over a period of one day. While the pulse decay test provides only an average estimate of the permeability for a comparable duration. Many issues are raised in pulse decay tests: determination of the reservoirs storage factor, micro leakage effects, determination of the initial pulse pressure, 2D mechanical effect. Contrary to the widespread belief 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. In fact, low water and rock compressibilities 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. Still, pulse decay remains an interesting alternative to steady state methods when permeability is higher than 50 nD (5×10^{-20} m^2).*

INTRODUCTION

In the last decades, new challenges have appeared in the field of geosciences such as radioactive waste disposal, geological storage of CO_2 and gas and oil production from unconventional reservoirs. Regarding radioactive waste disposal, some national agencies have selected clay formations as potential candidate to host a possible radioactive underground repository (ANDRA, 2005). The very low permeability of these formations will prevent radionucleides from migrating to the biosphere for a long period of time. Potential sites for CO_2 geological storage are selected on the basis of their storage capacity and their caprock sealing properties (Bachu, 2008). Caprock permeability 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 that are unable to release water pressure from compaction (Horseman *et al.*, 1996), can lead to major drilling problems. Permeability is thus the key parameter since it controls fluid migration. However, laboratory measurement of low permeability (< 100 nD (10^{-19} m^2)) remains a technical challenge.

Most of the authors dealing with very low permeability measurements use a transient method known as pulse decay (Escoffier *et al.*, 2005). Its principle, illustrated in Figure 1, was first proposed by Brace *et al.* (1968). It consists in a sample bounded by two reservoirs that are 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, in most cases, 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 long experimental durations compared to pulse decay tests, due to the long time required for water flow stabilization (Hsieh *et al.*, 1981; Suri *et al.*, 1997; Jones and Meredith, 1998; Yang and Aplin, 2007). This opinion is shared by other authors dealing with different low permeability porous media such as cement pastes (Roy *et al.*, 1993; Scherer *et al.*, 2006).

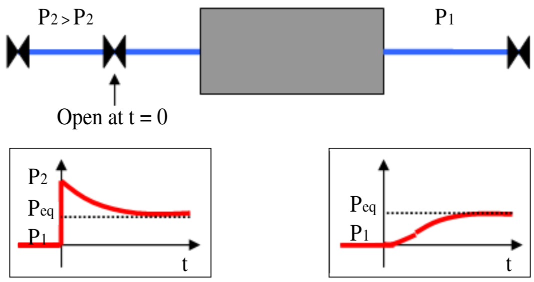

Figure 1

Principle of the pulse decay method.

More water permeability measurement techniques exist but they are less commonly used in very low permeability rocks:

- the Pore Pressure Transmission Test (PPTT) will be detailed further in this paper;
- the device described by Scherer (2006), which is similar in principle to the Darcygas device described in Carles *et al.* (2007) for gas, can quickly measure water permeability. The sample is placed in a small volume and is surrounded by water. Pressure is increased by a small change of volume. Pressure rises in the device, and then it decreases due to its propagation within the sample. Pressure decay is proportional to permeability. Due to

the motion of water in the sample in all directions, the experiment can be fast for low permeable porous media. However, uncertainties remain due to the fact that no overburden stress applied in the sample (some samples with no overburden pressure can loss their integrity when they are immersed in water) and due to the fact that permeability is not the only parameter that controls pressure decay. Parameters are strongly interdependent;

- the oscillation method was first proposed by Kanz *et al.* (1990). This technique is preferentially used to measure microDarcy (10^{-19} m^2) permeability (Larive, 2002) and to observe permeability change over the time, for instance during compaction (Suri *et al.*, 1997). The oscillation technique consists in imposing an oscillating upstream pressure and monitoring phase and amplitude differences between upstream and downstream signals. Theses values are related to permeability. Yet, in extreme low permeability (nanoDarcy - 10^{-21} m^2), downstream oscillations can be hard to notice;
- there are two steady state methods: one is based on a constant pressure gradient and is studied here, and the other one is based on a constant water flow rate (Olsen *et al.*, 1966). Pressures are recorded. Upstream pressure increases due to continual injection of water until equilibrium. This equilibrium can be very long due to reservoir compressibility. The application of the oscillation method and of the second steady state method in extreme low permeability porous media will be discussed further in the study.

The pulse decay technique was first proposed by Brace *et al.* (1968). The interpretation is based on the exponential decrease of the pressure difference ($P_{usptream}$ - $P_{downstream}$) with time. Based on the hypothesis of instantaneous equilibrium of pore pressure within the sample, log(P_u-P_d) *versus* time follows a linear regression. The slope is proportional to the permeability. This interpretation is still relevant nowadays for its simplicity of use in Chenevert and Sharma (1993), in Kwon *et al.* (2001) or in Fedor (2008). Hsieh *et al.* (1981) went further in the interpretation of pulse decay signals and reject the hypothesis of instantaneous pressure equilibrium within the sample. In fact, due to the storage capacity of the sample (proportional to the specific storage of the sample S_s), propagation speed of pressure wave within the sample is proportional to S_s/k_w. In very low permeability porous media, this travel time is not negligible. Equations are available in Hsieh *et al.* (1981) and their practical application can be found in Neuzil *et al.* (1981). The Heish's expressions are also used, for example, in Escoffier *et al.* (2005). A sensitivity analysis is available in Zhang *et al.* (2000). Waldert and Nura (1986) claim that motion of water in pulse decay test is not a 1D process, and its interpretation as such can lead to uncertainties. In fact, during pulse decay, pore pressure is non uniform within the sample and changes with time. The lateral deformation of the sample, due to effective pressure change, involves motion of water in the radial direction. This effect has been studied in homogeneous porous media (Giot *et al.*, 2011) and also in the case of anisotropy (Giot *et al.*, 2010). It seems that interpreting pulse decay signal as a 1D problem, as Hsieh *et al.* (1981) did, can lead to major uncertainties on the specific storage S_s of the sample but lower uncertainties on the permeability value k_w (Giot *et al.*, 2011). However, those approaches can require additional parameters related to the sample geomechanical deformations. Pulse decay experiment durations last from 2 hours for a 90 nD sample (Roy *et al.*, 1993) to 5 hours for a 14 nD sample (Escoffier *et al.*, 2005). It is thus a fast method for permeability measurements.

Few experiments of steady state method are available for extreme low permeable rocks (nanoDarcy). Jones and Meredith (1998) measured a permeability of 6 nD in 15 hours. Morrow and Locknet (1997) measured permeabilities of 0.1 to 100 nD in one to three days. El-Dieb and Hooton (1995) measured a permeability of 1 nD in 160 hours. In all those experiments, only one pressure gradient was applied on the sample. Permeability measurement is based on only one flow rate estimation.

Zhang *et al.* (2002) compared the constant pressure and constant flow steady state experiments with the pulse decay in microDarcy samples. The permeability values obtained were fairly similar but discrepencies were observed when pulse decay experiments are too fast (few seconds). Chenevert and Sharma (1993) compared the steady state and the pulse decay methods in low permeable media. On a 0.4 nD sample the steady state experiment lasts 170 hours for one pressure gradient applied. The pulse decay experiment is faster: 20 hours on a 0.6 cm thick sample. The conclusion is that the steady state method is the most straightforward permeability measurement method but a high pore pressure difference is required to measure enough water out of the sample in a reasonable time.

In the present paper, three specific samples are chosen for water permeability measurement: one with low and two with a very low permeability. This study focuses on demonstrating that the steady state method can actually be as fast as the pulse decay method. The specific device developed for the measurement of water permeability by steady state and pulse decay is described here. Additionally, special attention is given to compare the reliability of these two techniques.

1 MATERIALS AND METHODS

1.1 Experimental Materials

The first sample used (mentioned as sample A) comes from the Weyburn site extracted from the Watrous formation, Canada. The cylindrical core of 50 mm diameter and 25 mm thick was placed in a Hassler cell with a confining pressure at 24 MPa. The second sample, B, is an Upper

Torcian argillite provided by IRSN (Institute for Radiation Protection and Nuclear Safety), extracted from well M6 of the Tournemire tunnel in Aveyron, France. The cylindrical core was 40 mm in diameter and 20 mm thick. The third sample, C, is a Clay sample provided by ANDRA (the French National Radioactive Waste Management Agency) extracted within the framework of the TAPSS 2000 program (Bure, France). The sample comes from the EST433 borehole drilled by ANDRA and extracted at a depth of 593 m in the Callovo-Oxfordian formation. The core was 40 mm in diameter and 17.4 mm thick. Samples B and C were placed in a Hassler cell with a confining pressure at 15 MPa. The experimental set-up is presented in Figure 2. All experiments were performed perpendicularly to the bedding plane of the samples.

The pump QX 20K is composed of two piston cylinder with a 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 15 MPa for sample A and 10 MPa for sample B, 6 MPa for sample C. Water was initially equilibrated with sample fragments resulting from coring to prevent the 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 (Morrow and Lockner, 1997) and pressures. In our closed reservoir configuration, a temperature fluctuation of 1°C would lead to a pressure increment of 0.5 MPa, accordingly temperature was maintained at 25°C ± 0.1. After sample saturation, 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.

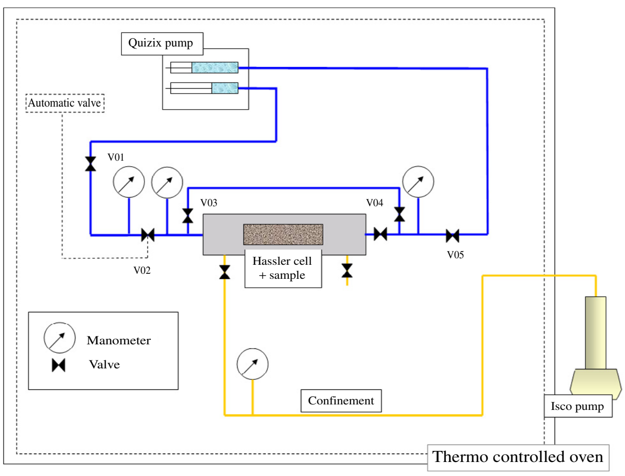

Figure 2

Experimental set up used for the steady state and transient methods for permeability measurement (upstream volume equal to 6.82 cc and downstream volume equal to 4.74 cc).

1.2 Saturation

Prior to test, the experimental set-up was vacuumed for ten minutes. Then water was injected in the device. Pore pressure was imposed by the pump, both at the upstream and the downstream sample boundaries, at a constant value. The amount of water that was injected into the sample was recorded by the pump. Once no more water was injected, sample saturation was considered to be finished. Sample A was initially dried and two days were required to inject an average value of 5 cc into the sample. Samples B and C were initially preserved and it took less than four days for a complete saturation. Small Water leaks were localized by salt deposition on the experimental set-up connections. The main leaks were usually localized on the connection from the Hassler cell and the experimental device, and were checked once the sample mounted. They were fixed before the sample characterization tests. Smaller leaks were difficult to assess.

1.3 Steady State Method

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

$$q = -\frac{k_w}{\mu_w}\mathrm{grad}P_w \tag{1}$$

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 gradP_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:

$$k_w = \frac{Q}{A}\frac{\mu_w * L}{(P_u - P_d)} \tag{2}$$

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. For example, on sample B, upstream pressure was set successively to 10.5, 10.75, 11 and 11.25 MPa, while corresponding downstream pressure was set respectively to 9.5, 9.25, 9 and 8.75 MPa in such a way that the mean pore pressure was maintained at 10 MPa; the imposed pressure gradients were thus 1, 1.5, 2 and 2.5 MPa and each pressure gradient step lasted about twenty

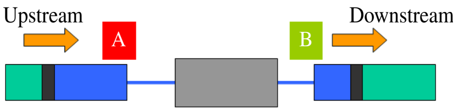

Figure 3

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

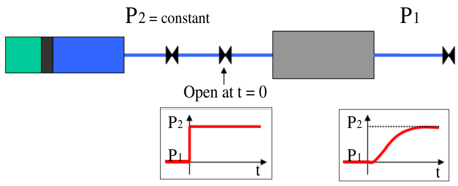

Figure 4

Principle of the PPTT.

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 were plotted with time and once their evolutions became linear, the slopes corresponding to the upstream and downstream water flows were reported. Then the successive flow rates (Q) were plotted against the pressure gradient ($P_u - P_d$). According to Equation (2), the four points obtained should align along a slope proportional to the permeability. For samples A and C, three pressure gradients were used.

Such experiments using a "push-pull" configuration, commonly carried out on porous media with intermediate to high permeability (Fabbri *et al.*, 2008), have similarities with Jones and Meredith's (1994) experiments on a 6 nD sample. Up to now, the push-pull method with multiple pressure gradients (three different pressure gradients applied to obtaine darcy plot) has never been performed on nanoDarcy permeability porous media.

1.4 The Pore Pressure Transmission Test

A PPTT is nothing else than a pulse decay test with a constant pressure condition at one boundary. Prior to test, water pressure was maintained for a certain duration in order to ensure pressure equilibrium within the sample. For example on sample B, pore pressure was maintained at 9.5 MPa for twelve hours. After that, the downstream reservoir was closed and upstream pressure was increased to 10.5 MPa *(Fig. 4)*.

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

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

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

$$P_u = \text{constante} \tag{5}$$

g is the gravitational 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:

$$S_s = \rho_w * g * \beta_M \tag{6}$$

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 (Schwartz et Zhang, 2002). 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 (COMSOL, 2005). The main assumptions are a 1D propagation of the pressure wave and that all parameters (S_s, S_u, S_d, k_w) remain constant over the experiment. Parameters used for simulation are available in Table 1.

TABLE 1

Experiments summary / parameters used for simulations of the transient methods

From the experiments			
Parameters	Sample A	Sample B	Sample C
K_w (nD)	275	0.8	2.6
S_s (m^{-1})	8.5×10^{-7}	3.4×10^{-6}	1.4×10^{-5}
Confining pressure (MPa)	24	15	15
Average pore pressure (MPa)	15	10	6
COMSOL Parameters			
Upstream volume (cc) V_u	5.64	6.82	6.82
S_u (m^2)	5.78×10^{-11}	7.64×10^{-11}	9.07×10^{-11}
Downstream Volume (cc)	5.23	4.74	4.74
S_d (m^2)	6.12×10^{-11}	4.64×10^{-11}	5.02×10^{-11}
Initial pore pressure (pulse decay - MPa)	14.532	9.760	5.923
Upstream pulse pressure (pulse decay - MPa)	16.723	10.326	8.164

1.5 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 open, a pressure rise of the order of 1 MPa. Pressure evolution in the system was compared to simulations based on the system of Equations (3, 4) and (7):

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

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

1.6 Storage Factors

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

$$S_{(u,d)} = \rho_w * g * \frac{V_{u,d}}{dP_w} \qquad (8)$$

To measure S_u and S_d, the samples were replaced by a steel cylinder (protocol similar to Escoffier (2002)). Confining pressure was set to 35 MPa. Water pressure was increased first by steps of 2 MPa (from 2 MPa to 10 MPa) and then by steps of 5 MPa (from 10 to 30 MPa) and the volume of water needed to pressurize each reservoir was recorded (pistons displacement). After that, the pressure was decreased the same way. At the end of the pressure cycle, volumes were checked to account for any possible leaks. The volume variation was very small and therefore the storage factor estimation was not affected by any leakage issues. S_d and S_u were estimated for each step by (Amaeful *et al.*, 1986):

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

1.7 Error Estimation

Error estimation in the PPTT and the Pulse decay tests would require a precise sensitivity analysis of each parameter. It was not investigated in this work. The error analysis in steady state measurements is straightforward. If water flow rate Q function of pressure gradient is approximated by a linear regression:

$$Q = a\Delta P + b \qquad (10)$$

With (Speigel, 1972)

$$s(a) = \sqrt{\frac{\sum \frac{1}{(Qs(Q))^2 + (\Delta Ps(\Delta P)A)^2}}{\sum \frac{1}{(Qs(Q))^2 + (\Delta Ps(\Delta P)A)^2} \sum \frac{(\mathrm{grad}P)^2}{(Qs(Q))^2} - \left(\sum \frac{\mathrm{grad}P}{(Qs(Q))^2}\right)^2}} \qquad (11)$$

where $s()$ is the confidence interval of each value. The interval of confidence of the water flux Q depends of the time t (s) where water volume is pumped and V (m^3) the amount of water pumped for this delay:

$$s(Q) = Q\sqrt{\left(\frac{s(t)}{t}\right)^2 + \left(\frac{s(V)}{V}\right)^2} \qquad (12)$$

The longer is this delay, the more precise is the pump and the more precise is the water flux measurement. The error on water permeability is estimated from Equation (2):

$$s(k_w) = k_w\sqrt{\left(\frac{s(a)}{a}\right)^2 + \left(\frac{s(A)}{A}\right)^2 + \left(\frac{s(L)}{L}\right)^2 \left(\frac{s(\mu_w)}{\mu_w}\right)^2} \qquad (13)$$

2 RESULTS

2.1 Reservoir Storage Factor

The upstream reservoir storage factor decreased with pressure from 2×10^{-10} m^2 to 6×10^{-11} m^2 and the downstream reservoir storage factor from 1×10^{-10} m^2 to 4×10^{-11} m^2 *(Fig. 5)*. Reservoir storage values correspond to an average compressibility of 10^{-9} Pa^{-1} for the two reservoirs. Compressibility of the reservoir is half due to the water compressibility ($\beta_w = 5{\times}10^{-10}$ Pa^{-1}) and half due to the material compressibility. This result is consistent with compressibilities obtained by Chenevert and Sharma (1993) and Escoffier (2001). The exponential evolution of the reservoir compressibility with pressure is not reported in theses studies. It can be explained by potential highly compressible air that dissolves itself at high pressure. However, reservoirs were vacuumed and water was degassed prior to the compressibility measurements. Presence of air was unlikely. Reservoirs seemed highly compressible at low pressure. Measurement at pore pressure lower than 8 MPa would thus lead to major uncertainties on the interpretation of transient experiments since storage factors would not remain constant over the experiments.

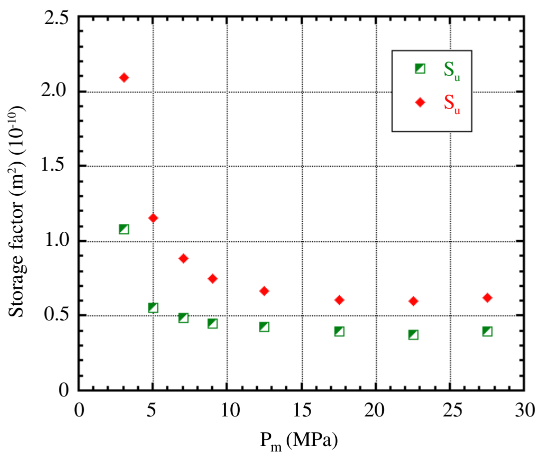

Figure 5

Evolution of upstream and downstream reservoir storage factors with pressure.

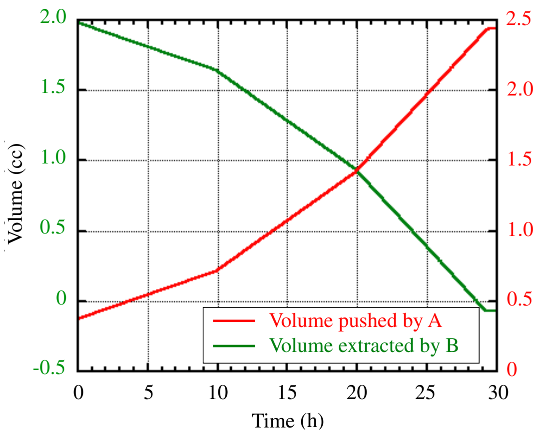

Figure 6

Piston displacements throughout the steady state experiment on sample A.

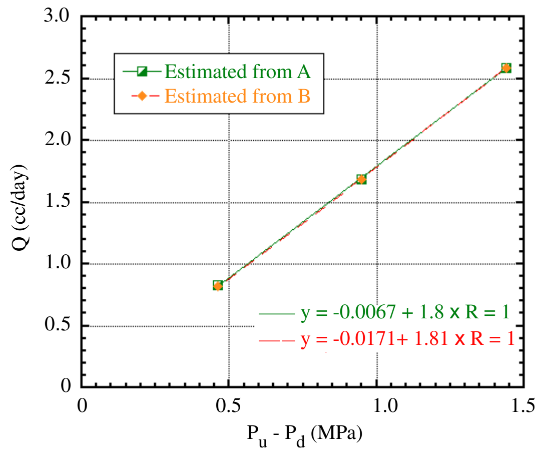

Figure 7

Evolution of the water flow rate with pressure gradient on sample A.

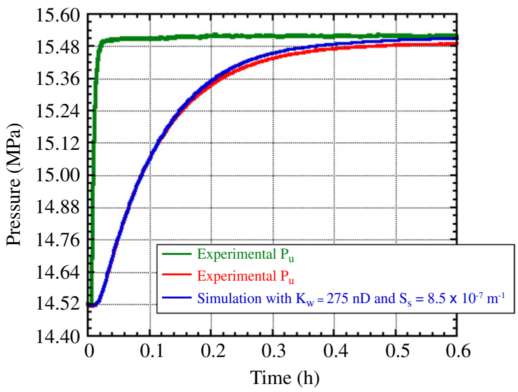

Figure 8

The PPTT on sample A. Comparison between experimental and simulation curves.

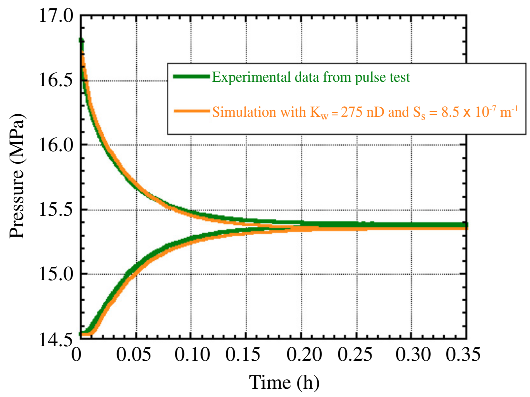

Figure 9

Pulse decay on sample A. Comparison between experimental and simulation curves.

2.2 Sample A - Experiments on a 275 nD Sample

The steady state methods on sample A lasted thirty hours. Three pressure gradients were applied, each of them lasted ten hours. The piston displacements were recorded *(Fig. 6)*. For each pressure gradient, the water flow rate was estimated and is reported in Figure 7. There was an exact match between upstream and downstream water flow rate. Darcy's law was verified, the permeability measured was 275 nD (2.75×10^{-19} m^2). The PPTT was used to estimated the storage factor S_s of the sample. Figure 8 presents the experimental data on sample A. Downstream pressure reached the imposed upstream pressure after forty five minutes. The simulation curves obtained with COMSOL fitted the experimental data for a S_s value of 8.7×10^{-7} m^{-1} *(Fig. 9)*. The pulse decay experiment lasted twenty minutes. The experimental data and the simulation curves fitted for k_w = 275 nD and $S_s = 8.7{\times}10^{-7}$ m^{-1}. In this case, the transient techniques and the steady state method are comparable.

2.3 Sample B - Experiments on a 0.8 nD Sample

Figure 10 shows the displacements of the pistons A and B through the four imposed pressure gradients. For each pressure gradient, less than four hours were necessary to get the water flow stabilized. Flow rates were estimated on the linear part 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 after five hours; naturally, longer experiment would provide a better precision on the flow rates. Measured flow rates ranged from 0.005 cc/day to

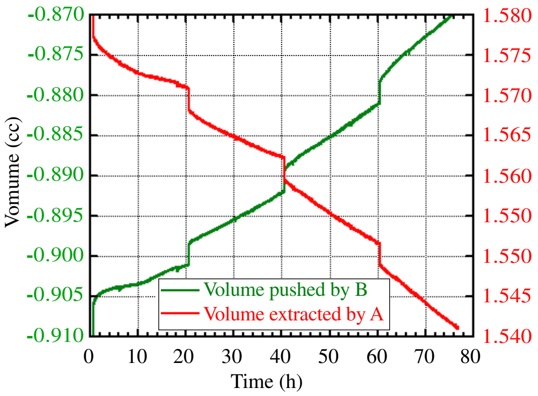

Figure 10

Piston displacements throughout the steady state experiment on sample B (in this case B is linked to the upstream reservoir, B pushed, A pulled).

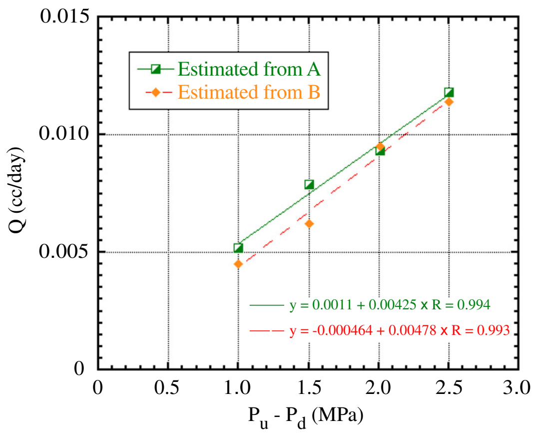

Figure 11

Evolution of the water flow rate with pressure gradient on sample B.

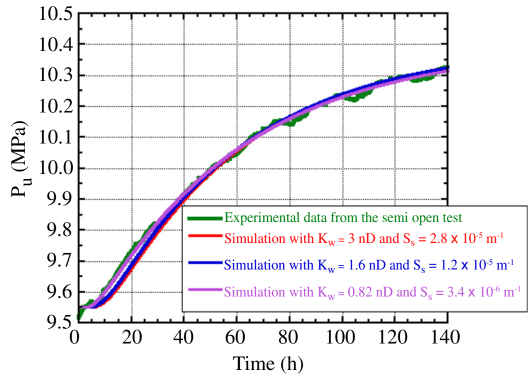

Figure 12

PPTT experiment on sample B. Comparison of simulation curves for different (k_w, S_s) values and experimental ones.

0.01 cc/day. Figure 11 displays the water flow rates against the imposed pressure gradients. The linear regressions of Q *versus* ΔP for upstream and downstream displacements gave 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) was chosen for further calculations. Figure 11 illustrates Darcy's law suitability to describe water flow in very low permeability porous media.

The PPTT lasted six days. It was not the necessary time for the downstream pressure to reach equilibrium *(Fig. 12)*. The simulation pressure curves, for different set of (k_w, S_s) values, can all describe the experimental data reasonably well 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 downstream pressure curve, but larger volume would also involve longer experiment which is unrealistic. This fact is also available for samples A and C but is only illustrated on sample B experiments. The permeabilities obtained by the steady state method were used as reference to fit the curves and to obtain S_s. The permeability of 0.82 nD (8.2×10^{-22} m^2) previously determined, provided a S_s value of 3.4×10^{-6} m^{-1}. Those two values are consistent with *in situ* permeability test performed in Bertrand *et al.* (2002) where permeability is estimated at 0.62 nD (6×10^{-21} m^2) and the specific storage is 1.24×10^{-6} m^{-1}.

The pulse decay test lasted three days. The experimental data were compared with simulations made with the set of (k_w, S_s) values estimated respectively from steady state experiment and the PPTT. As shown in Figure 13, simulation (green curves) does not match the experimental data (red 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 14 shows simulations performed to fit independently P_u or P_d. k_w lay between 0.2 nD (2×10^{-22} m^2) and 2 nD (2×10^{-21} m^2).

Figure 15 shows how the natural log of the pulse decay, ($P_u - P_d$), changes with time for the simulated curves obtained for different (k_w, S_s) values. Pulse decay interpreation can be based on Brace *et al.*'s (1968) relationship as Chenevert and Sharma (1993) did:

$$P_u - P_d = Ae^{-Bt} \tag{14}$$

$$B = \frac{k_w A}{\mu_w L}\left(\rho_w g\left(\frac{1}{S_d} + \frac{1}{S_u}\right)\right) \tag{15}$$

The permeability estimated for Figure 15 and Equation (15) are reported in Table 2. Those permeability values can

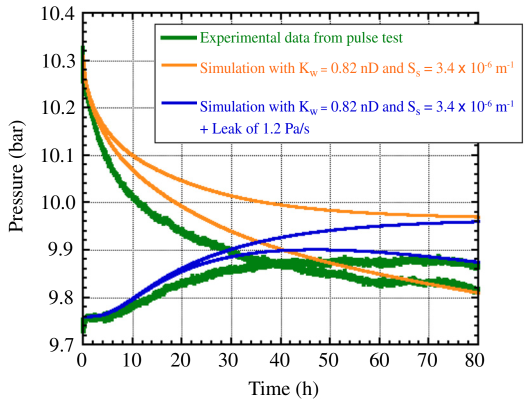

Figure 13

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

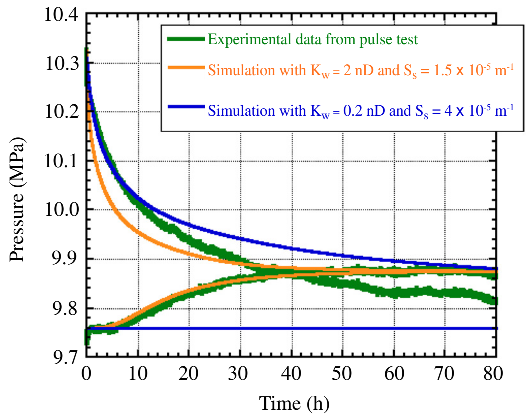

Figure 14

Experimental data compared to simulations fitting each pressure evolution through the pulse decay test.

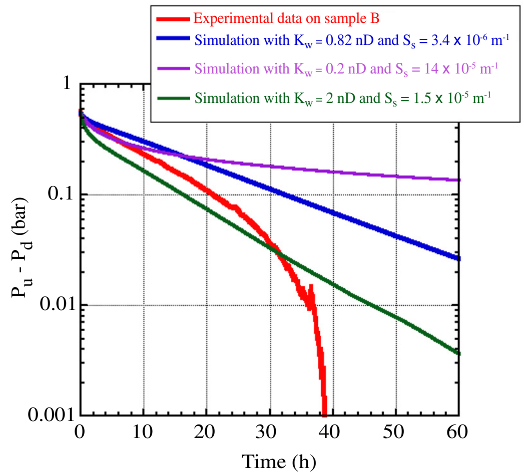

Figure 15

$(P_u - P_d)$ *vs* time for experimental data on sample B and simulations.

be in some case 50% of the original permeability value. In fact, the hypothesis of intant pressure equilibrium stated by Brace *et al.* (1968) is not true and affects permeability measurement in very low permeability porous media. Nevertheless, Chenevert and Sharma (1993) did find consistant data between pulse decay experiment interpreted by Equation (15) and steady state measurement, their findings can be affected by uncertainties that were not reported in their work.

TABLE 2

Permeability estimated from Equation (15) and pulse decay curves

Pulse decay curve	B (h^{-1})	k_w (nD)
Simulation of a 2 nD sample	0.0725	1.39
Simulation of a 0.82 nD sample	0.0485	0.93
Experiment on a 0.82 nD sample (B)	0.0708	1.36
Simulation of a 0.2 nD sample	0.006	0.12

2.4 Sample C - Experiments on a 3 nD Sample

The steady state experiment lasted one day. Three pressure gradients were applied for seven hours. The water flow rate estimated from Figure 16 lay in the range of 0.01 and 0.05 cc/day. The permeability estimated from Darcy's law *(Fig. 17)* was 2.2 nD (2.2×10^{-21} m^2) from the upstream water flow rate values and 2.95 nD (2.95×10^{-21} m^2) from the downstream data. An average value of 2.6 nD (2.6×10^{-21} m^2) was chosen for further calculations. S_s value was obtained from the PPTT. This experiment lasted one day, the pressure equilibrium was not reached at the end of the experiment but the experimental data can be analyzed to investigate S_s values. A value of 1.42×10^{-5} m^{-1} permitted to fit the experimental data *(Fig. 18)*. Those values can be compared with Escoffier's (2001) results on Callovo-oxfordian argilittes cored from a depth close to 500 m. Thus, there is a 100 m difference with our sample. The permeability lies from 5 nD to 50 nD (5 to 50 $\times 10^{-21}$ m^2) and S_s from 0.5 to 2×10^{-5} m^{-1}. The order of magnitude of S_s is correct and permeability is lower than expected. The permeability measured by the steady state experiment and the specific storage estimated by the PPTT were used to simulate pressure curves in the pulse decay experiment (*Fig. 19*). The pulse decay experiment lasted one day, initial pressure gradient was 2 MPa and initial pore pressure was 5.9 MPa, at the end of the experiment pressure reached

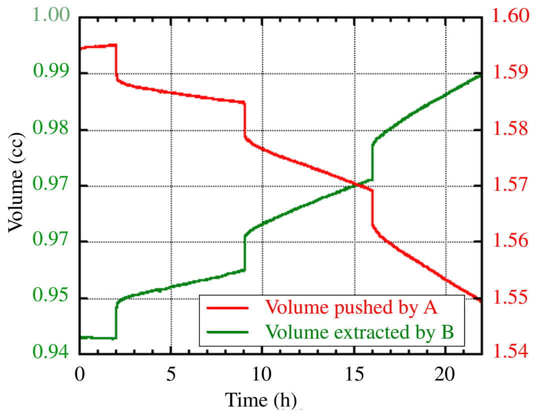

Figure 16

Piston displacements throughout the steady state experiment on sample C.

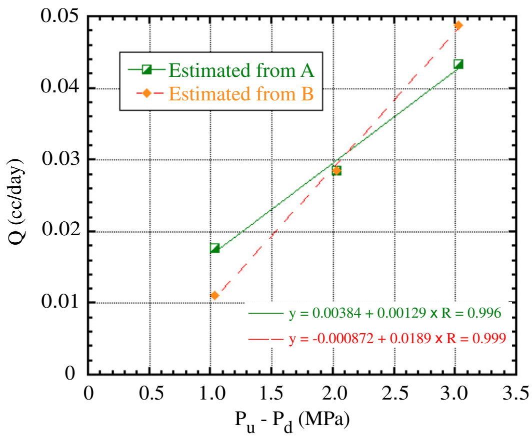

Figure 17

Evolution of the water flow rate with pressure gradient on sample C.

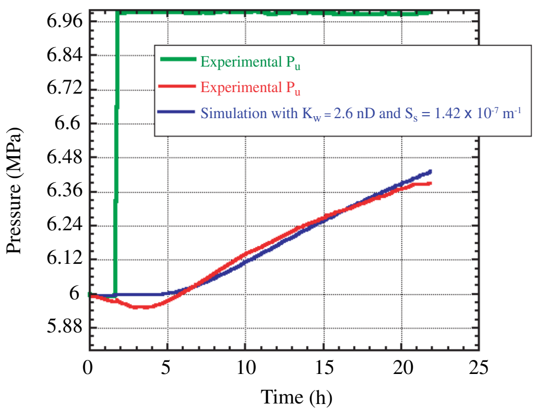

Figure 18

PPTT experiment on sample B. Experimental data compared to simulations.

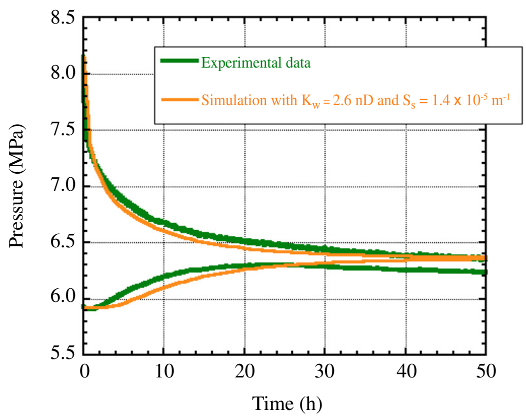

Figure 19

Downstream and upstream pressure evolution through pulse decay test. Experimental data compared to simulations.

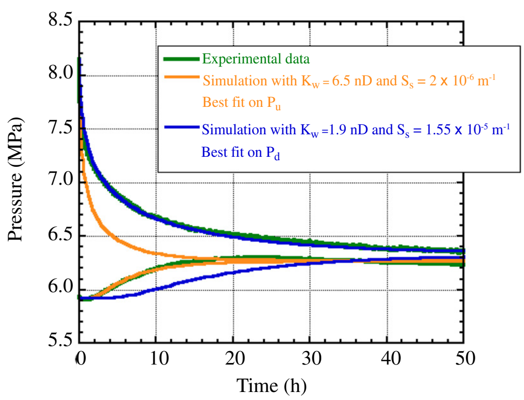

Figure 20

Experimental data compared to simulations fitting each pressure evolution through the pulse decay test.

its equilibrium around 6.35 MPa. P_d started to decrease after one day experiment which might be related to possible leaks localized at the downstream reservoir. The simulated curves fit well the experimental data. The simulation curve of the downstream reservoir seems a bit late compared to the experiment. Simulations were performed to find the best fit for both P_d curve and P_u curves *(Fig. 20)*. The best fit was obtained for permeabilities from 1.9 nD (1.9×10^{-21} m^2) to 6.5 nD (6.5×10^{-21} m^2).

3 DISCUSSION

This study was undertaken with the objective of demonstrating that steady state experiments can be preferred to pulse decay experiments regarding uncertainties control and experiment duration.

3.1 Main Uncertainties

Permeability error estimation *(Tab. 3)* was based on the uncertainties listed in Table 4. In very low permeability rocks, the errors depend mainly on how accurate is the pump to measure water volume. The pump manufacturer provides a pump resolution of 10 nL but this value cannot be used for a proper error estimation. The pump has been calibrated to estimate uncertainties on the pumped water volume. Scales, with a precision of $\pm 10^{-4}$ mg was used to measure water going out of the pump. Ten points of calibration were used for a total of 20 cc pumped. The volume uncertainty is estimated at 0.01 cc for this calibration and 0.07 cc taking into account the uncertainty on the last drop of water. Those values lead to permeability uncertainties higher than 1 000% *(Tab. 3)*. The steady state methods can indeed provide a good estimation of the permeability if it is beleived that the pump resolution is in the order of 100 nL. Nonetheless, with classical calibration technics, it cannot be properly proved.

Malkovsky *et al.* (2009) claimed that the transient experiments are more accurate experiments than the steady state method since pressure is more precisely measured than volume. The second statement is true but the transient experiments are also sensitive to water volume measurement. Pressure variation are linked to water volume through the reservoir specific storages. In fact, the main uncertainties rely on S_u and S_d. A compressibility of 10^{-9} Pa^{-1} corresponds in a 5 cc reservoir to a water volume of 0.005 cc/MPa. When pressure increases by 2 MPa, 0.01 cc is injected by the pump to the reservoir. It is within the pumped volume uncertainty. Error on the compressibility measurement is closed to 100%. The uncertainties on the two reservoir specific storage factors, S_u and S_d, have a direct impact on (k_w, S_s) estimation.

TABLE 3

Uncertainties on permeability for different values of pump volume precision

Uncertainties on pump volume	276 nD sample (%)	0.78 nD sample (%)	2.9 nD sample (%)
10 nL	9	6	5
100 nL	9	13	6
1 000 nL	9	113	36
0.01 cc	10	1 131	360
0.07 cc	37	7 916	2 521

TABLE 4

Uncertainties on the main measurements

Cte	Name	Uncertainties
μ	Viscosity	± 0.01 cP
L	Length	± 0.025 cm
P	Pressure	± 0.03 MPa
T	Duration	± 0.01 h

Additional uncertainties make difficult a proper estimation of k_w in transient techniques such as pulse decay:

- pore pressure is assumed initially constant. It is thus important to wait for pressure stabilization within the sample prior do any transient tests. The duration of the pore pressure equilibrium phase can only be determined once k_w and S_s are known. However, it is impossible to know for sure when pressure stabilization is done within the sample unless a PPTT is performed;
- 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. In pulse decay, S_s can be estimated independently of k_w. From the ending pressure P_{eq} (Hsieh *et al.*, 1981):

$$S_s = \frac{1}{AL}\left(S_u \frac{P_{init,u} - P_{eq}}{P_{eq} - P_{init,d}} - S_d\right) \tag{16}$$

  When the initial pore pressure and P_{eq} are well determined, and S_u and S_d in the order of magnitude of S_sAL, S_s can be estimated accuratly. In contrast, when pulse decay is not conducted until the end or in the case of leakage (sample B) this technique fails to provide suitable S_s value;
- assuming that water transfer in the pulse decay is a 1D problem.

Figures 14 and 20 show the difficulty to conciliate both upstream and downstream pressure curves with simulations in very low permeability rocks. In most cases, experimentators have access to both downstream and upstream pressure curves, permeability is thus well estimated compared to the order of magnitude observed between the two permeabilities in Figure 14. Attention should be focused on pressure curves partially obtained where major uncertainties remain on k_w. Figure 20 shows that, even with minor leakage effect, two different permeabilities fit roughly the experimental data for a pulse decay. This discrepancies observed in Figure 19 can be related to the specific storage change with pressure when pore pressure is lower than 8 MPa.

In Figure 9, the simulated curve gave a perfect fit. In fact for permeability higher than 50 nD (5×10^{-20} m^2) the pulse decay only lasts few hours, even less. It is unlikely to be affected by leaks or temperature fluctuation. The steady state method can also be performed easily but it would be pointless to avoid a transient test that can gave access to the specific storage of the sample.

3.2 Duration of the Experiments

Few steady state experiments have been reported in the literature on low permeable media (El-Dieb and Hooton, 1995; Morrow and Lockner, 1997). In general, the pulse decay method is preferred since the duration of the experiment seems shorter (Jones and Meredith, 1993). However, this

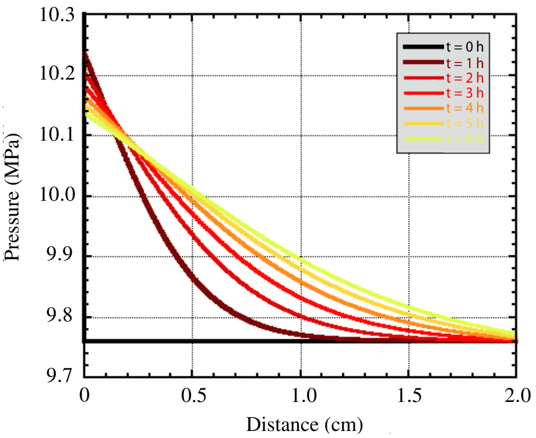

Figure 21

Pressure stabilization over 6 h within sample B throughout the steady state experiment.

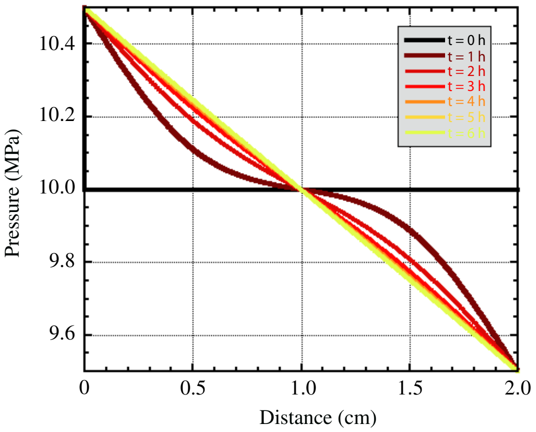

Figure 22

Pressure stabilization over 6 h within sample B throughout the pulse decay test.

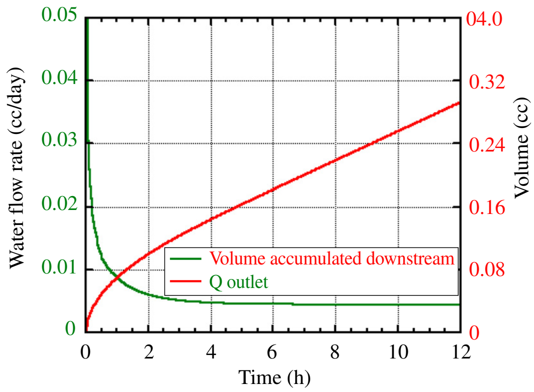

Figure 23

Water flow rate stabilization within the clay sample at 0.82 nD through the steady state test, pressure gradient 1 MPa. Associated water accumulation.

study shows that the two methods can be comparable in time. Steady state flow rate measurement is a two step process: waiting for flux stabilization, then measuring the flow rate by accumulation of water out of the sample.

First of all, 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 $k_w L^2/S_s$ (in Brace *et al.* (1968), it is supposed to be instantaneous). Based on simulations, pore pressure equilibriums within the sample are presented in Figures 21 and 22 for steady state and pulse decay tests carried out on sample B. Over a transient test, the downstream pressure can only start to increase when the pressure wave has propagated throughout the sample. In Figures 12 and 13, the downstream pore pressure increase happened after three to five hours of test, which is consistent with the time required for the flow stabilization observed in Figure 22. Thus, pulse decay cannot be preferred to steady state experiments on the grounds that establishing steady state conditions is a very long process.

In addition, in the steady state method, due to the modification of both upstream and downstream pressure, the pressure propagates in only half of the sample. Compared to a traditional approach in which only the upstream pressure is increased by steps, water flux stabilization is four times faster in this configuration.

Pore pressure stabilization is fast *(Fig. 21)*. Steady state test duration depends mainly on the time required to be able to measure accurately the water flow rate. Water flow rate 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 that are measurable by the pump, the shorter the time required to evaluate water flow rates. Simulations were carried out to estimate water flow rate stabilization for the steady state method on sample B. Figure 23 shows both water flow rate and water accumulation out of the sample. The flow rate is within 5% of its final value at equilibrium after four hours. After that, water accumulation increases linearly with time, the water flow rate can be measured.

Other techniques might be applied here to measure nanodarcy permeabilities. On sample B, an estimation of oscillation test results was made based on Kanz *et al.*'s (1990) equations *(Tab. 5)*. The amplitude ratio of upstream and downstream signals can be noticed (R > 0.01) only if one oscillation lasts 9 hours. Amplitude ratio is estimated after five to ten oscillations (Suri *et al.*, 1997), 3 day experiments can be possible. Oscillation methods can be as long as pulse decay if high precision on pressure variation can be achieved. However, uncertainties are high in this case

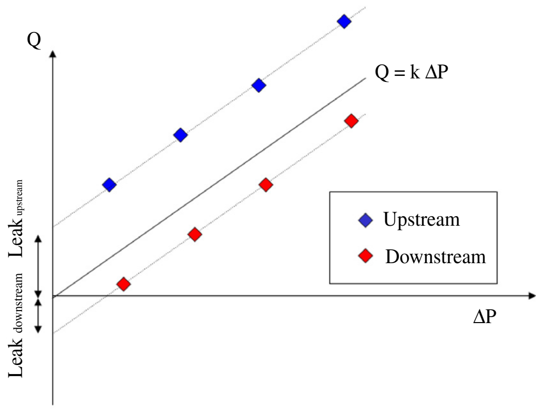

Figure 24

Illustration of leaks (assumed constant with pressure) localized in the upstream and downstream reservoirs.

(Barnabé *et al.*, 2006), actually, permeability and specific storage are highly interdependent parameters. Furthermore, the second steady state method with constant injection flow rate can be considered to measure nanodarcy permeabilities. For sample B, a constant injection rate of 0.005 cc/day would lead to a pressure gradient of 1 MPa. If 0.005 cc/day is injected upstream it would take at least 48 hours to raise pressure by 1 MPa in an upstream volume of 10 cc with a compressibility of 10^{-9} Pa^{-1}. Due to the compressibility of the reservoirs, this steady state method is not appropriate to measure nanodracy permeabilities.

TABLE 5

Oscillation method. Results of amplitude and phase difference between upstream and downstream signals for sample B. Different frequencies are considered

Oscillation frenquencies	Amplitude ratio R	Phase difference (rad)
5 hours	0.0019	0.29
9 hours	0.0095	-1.45
12 hours	0.018	-0.89
24 hours	0.064	0.18

3.3 General Recommendations

The permeability measurement of 0.8 nD (8×10^{-22} m^2) lasted three days with the steady state technique and one day for a 2.6 nD sample (2.6×10^{-21} m^2) . It would have been possible to shorten the experiment if only one pressure gradient had been applied or if a sample of larger diameter was used. The largest diameter leading to the largest flow rates, it easier to measure them in a shorter period of time (Zhang *et al.*, 2002). However, it is recommended to perform at least three different pressure gradients in order to verify Darcy's law (it is a subject of controversy in nanoDarcy porous medium) and to avoid any problem due to possible leakages. Meanwhile, even with a leak, the linear regressions in Figures 7, 11 and 17 remain proportional to k_w as illustrated in Figure 24. Leaks observed in Figure 13 (5×10^{-4} cc/h) are not noticeable on Darcy plot *(Fig. 11)*, still it affects the pulse decay interpretation.

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

- S_s can be estimated;
- possibility to highlight the effects of heterogeneities, fractures (Ning *et al.*, 1993);
- applied effective stress is more homogeneously distributed within the sample (Chenevert and Sharma, 1993). In the steady state, effective stress is, indeed, different from the top to the bottom of the sample and is high ($\Delta P_{max,SS}$ = 3 MPa > $\Delta P_{max,PD}$ = 1 MPa).

The pulse decay test can be done easily when permeability is larger than 50 nD (5×10^{-20} m^2), without being impacted by uncertainties linked to leakage and temperature variations (example on sample A, pulse decay within the hour). The pulse decay test should be done in a range of pore pressure reservoir storage factors are known to be constant (> 8 MPa in our set-up).

The same conclusion cannot be applied to gas experiments. Firstly, the measurement of gas flux is direct and does not involve gas accumulation downstream. Specific gas flow meter exists (Boulin *et al.*, 2008) to measure gas flow through very low permeable media (even lower than nanoDarcy). Secondly, when upstream and downstream volumes are in the order of magnitude of the sample pore volume, pulse tests require twice to three times the duration needed for flux stabilization in a steady state method. In a small sample close to saturation, the gas pore volume is so low that transient method is mainly controlled by upstream and downstream volume size. Only in this specific case, the steady state methods become more efficient in terms of duration. Thirdly, porosity (equivalent of S_s with water) can be estimated directly from the transient part of the gas flow stabilization (Boulin *et al.*, 2011). The estimation of permeability and porosity remains indirect in a pulse decay test with gas. Fourthly, the application of non darcean model is straightforward in steady state method analysis (Wu *et al.*, 1998).

3.4 Mechanical Tests

Moreover, 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. Morrow and Lockner (1997) use this techniques to determine the variation of

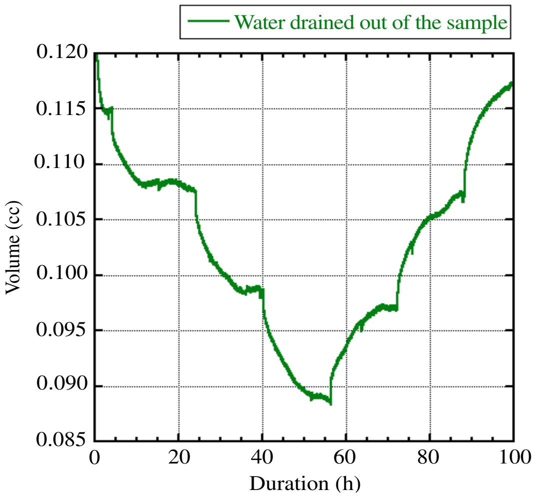

Figure 25

Water drained from the sample for different confining pressure steps on sample B.

porosity with increasing confining pressure. 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 led to erroneous measurements. Before performing the permeability measurement, for sample B, a delay of one day was required after each confining pressure increase *(Fig. 25)*. This stabilization delay should be assessed if the evolution of permeability with confining pressure is investigated. On sample C, a delay of fifteen hours was required. The delay is similar to the sample B delay whereas sample C is three times more permeable. In fact, its specific storage is high. It means longer equilibrium and a rock more sensitive to stress. There is thus more water to expel out of the sample when confinement is increased. Actually, we measured volumes two to three time bigger for sample C than for sample B. The similar delay observed for sample B and C is related to higher permeability compensate by higher specific storage.

CONCLUSIONS

This study shows that, for very low permeability porous 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. The duration of steady state experiments depends mainly on the time needed to estimate the water flow and not on the time required for flow stabilization. This measurement duration becomes shorter as soon as high volumetric resolution pumps are used. Pulse decay should be mainly used to investigate pressure wave propagation within the sample. With an appropriate experimental set up and control of uncertainties (leaks, storage factor), a first approximation of the specific storage S_s can be obtained by the pulse test. The next step would be the extraction of geomechanical data from pulse tests such as Giot *et al.*'s (2011) investigations.

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