# A FAST AND ACCURATE METHOD TO MEASURE THRESHOLD CAPILLARY PRESSURE OF CAPROCKS UNDER REPRESENTATIVE CONDITIONS

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## ABSTRACT

Assessing representative threshold capillary properties of fine grained rocks is very important in the context of the reservoir production, the basin modeling and the geological storage (natural gas or CO2)

This paper presents an innovative approach to measuring representative values of threshold capillary pressure under in-situ conditions based on the analysis of a dynamic displacement of the non-wetting fluid in the sample. When the injection is conducted at a constant pressure drop (higher than the threshold capillary pressure), the pressure profile within the core can be divided into three parts : the upstream invaded region, the front zone with a pressure jump associated with the threshold capillary pressure and the virgin region still saturated with the brine. Just before the non-wetting phase contacts the inlet face of the core, the brine production rate corresponds to Darcy's law using the overall pressure drop and the absolute permeability value. When the non-wetting fluid starts flowing within the core, a reduction of the production rate is observed due to the threshold pressure jump at the displacement front. Because the upstream invaded region extension is limited, its pressure drop can be neglected. The pressure drop in the virgin region can then be calculated from Darcy's law using the reduced production rate and the absolute permeability value. Finally, the threshold capillary pressure is deduced by subtracting this pressure drop value from the overall pressure drop value.

The proposed approach has been compared to the other available techniques to measure threshold capillary pressure : 1) step-by-step increase of the non-wetting phase pressure (standard approach) and 2) transient evolution of inlet and outlet pressure (residual capillary pressure approach). The results demonstrate that the new method is as accurate as the standard approach and is as fast as the residual capillary pressure approach. When local pressure taps are available, the method can also detect threshold Pc variations along the sample.

## INTRODUCTION

### Background on the Threshold Pc

The threshold capillary pressure (also referred as capillary entry pressure in the literature) characterizes the ability of a porous medium saturated with a wetting phase to block the

flow of a non-wetting phase. Its value corresponds to the size of the largest pore throat in the porous medium (Figure 1a) and can be calculated using Laplace's equation :

$$P_c^{th} = \frac{2\sigma\cos\theta}{R_{throat}^{\max}} \quad (1)$$

where $\sigma$ is the interfacial tension for the fluid system considered,
$\theta$ is the contact angle,
$R_{throat}^{\max}$ the largest throat radius.

Therefore, the pressure difference between the non-wetting and the wetting phase (Pc) must exceed $P_c^{th}$ before the non-wetting phase can start to drain the porous medium and flow. Threshold Pc rules the behaviour of caprocks but can also impact the reservoir production. Cunningham and Chaliha (2002) explained a poor waterflood conformance by the presence of a capillary pressure jump in the reservoir (negative threshold Pc in a low permeability rock-type) higher than the capillary pressure induced by gravity. Most of the applications related to the threshold Pc are restricted to fine grained rock materials such as caprocks either to parameterize basin modelling or to evaluate the integrity of a geological formation in the context of the storage of natural gas or $CO_2$. Reliable threshold Pc values are needed in basin modelling because they control the fluxes of fluids through the caprocks over the geological times. Therefore, threshold Pc impacts directly the volume and the localization of the hydrocarbons trapped (best locations for exploration) and also the over-pressured formation, which are important to localize in order to design adapted drilling programs (Thomas et al., 1968; Katz and Ibrahim, 1971).

Threshold Pc is also a key parameter to design geological storage and avoid an upward migration of the injected fluid into the overlaying formations (Horseman et al., 1999; Marschall et al., 2005). Once in place, the injected fluid (natural gas or $CO_2$) forms a column in the reservoir leading to a pressure difference with the water phase due to the density contrast. Because the pressures are equal at the aquifer level, the maximum pressure difference is reached just below the caprock. Figure 1b shows the pressure difference between the two phases at two different steps during the filling of the geological storage ($CO_2$ case). The injection itself can also induce local pressure increases in the injected phase that must be added to the static capillary pressure. One of the objectives of the reservoir storage simulations is to check that the capillary pressure at the contact between the reservoir and the caprock never exceeds the threshold pressure, in order to insure the long-term containment of the injected fluid.

**Outline**

This paper presents an innovative methodology to measure the threshold Pc value of rocks in a fast and accurate manner under reservoir conditions. The proposed approach relies on the interpretation of a dynamic injection test. By definition, the threshold Pc corresponds to a pressure difference between the injected and water phase which is needed to overcome capillary forces and does not contribute to flow of the injected fluid. Therefore, the threshold Pc can be obtained by using the recorded production rate to

calculate the actual driving pressure gradient and subtracting this from the total pressure difference.

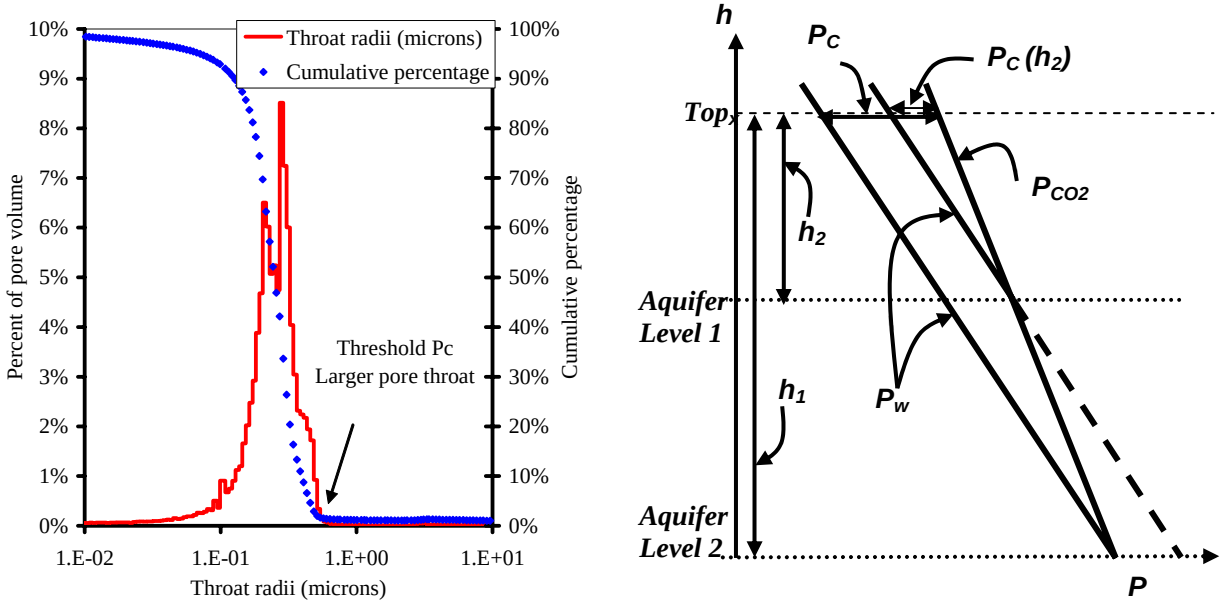


*Figure 1:(a) Threshold Pc and mercury porosimetry – (b) Static pressure profiles in the water and the $CO_2$ phases in the geological formation*

The first part of the paper is devoted to a literature review of the existing approaches to measure threshold Pc value with a listing of their respective advantages and drawbacks. The second part is dedicated to the principle of the proposed method and to a comparative study with two other existing methods on the same set of samples. Then, the method is discussed in terms of detection of heterogeneity of threshold Pc along the core through a synthetic case and also in terms of its applicability under reservoir conditions. Finally, conclusions are drawn on the future works to be conducted.

## EXISTING METHODS TO MEASURE THRESHOLD $P_C$ VALUES

This section reviews the different existing techniques to measure caprock threshold Pc. The principle of each method is recalled and discussed in terms of advantages and drawbacks.

### Mercury Porosimetry

The simplest approach consists of deriving the in-situ threshold Pc value from a mercury porosimetry curve (Monicard, 1981), knowing the values of the interfacial tension and the contact angle for the fluid system and the rock considered. The main added value of this approach is its rapidity and simplicity. Results can be obtained a few days after the sampling.

The first drawback of this approach can be the lack of data to convert the mercury based threshold Pc. For example, if we consider the context of the storage of $CO_2$, some

publications give a reasonable range of $\sigma$ for $CO_2$-pure water system (Chun and Wilkinson, 1995; Da Rocha, 2005) but $CO_2$-brine systems have been less studied (Chalbaud et al., 2006). In addition, the contact angle is often unknown and zero is frequently assumed (water wettability) as a first approximation although recent work has demonstrated that caprocks could be mixed-wet towards $CO_2$ (Chiquet et al., 2005).

Another issue is the preparation procedure followed to dry the caprock samples without alteration of the pore structure. Dewhurst et al. (1999) have compared several preparation procedures :

- air drying at laboratory temperature to remove the pore fluid but not all the interlayer water,
- freeze drying in liquid nitrogen and transfer to vacuum freeze drier to sublimate the solid ice without rock fabric alteration,
- vacuum drying of wet shale in vacuum dessicator.

The results showed that threshold capillary pressure is not significantly affected by the drying methodology when well-indurated caprocks are studied. In the other cases, especially for smectite-rich caprocks, accuracy and data consistency are improved significantly by the freeze-drying method because it preserves the rock structure.

Another shortcoming of the mercury porosimetry approach is the lack of confining pressure. It is well established that the petrophysical properties of low-permeability rock samples are very sensitive to the confining stress either on tight reservoir rock (Shanley, 2004) or on shale samples (Vasseur et al., 1995). Permeability reduction on the order of one decade is commonly observed on low-permeability samples. If we consider that Leverett's function can be applied, it means that an increase of a factor of 3 is expected for threshold capillary pressure. All these limitations make this approach very inaccurate and only adequate to collect early "trends" when no other data under in-situ conditions are available.

**The Step-by-Step Approach (or Standard Approach)**

The standard approach consists of a step-by-step increase of the non-wetting phase pressure at the inlet face of a sample fully saturated with brine while recording of the brine production at the outlet (Al-Bazali et al., 2005; Li et al., 2005). This approach can be conducted under in-situ conditions and is also very simple to interpret since it relies on the definition of the threshold capillary pressure. Identification of the threshold capillary pressure from experimental data is illustrated in Figure 2a. The step-by-step increase of the inlet pressure is conducted with variable pressure steps and the establishment of the flow is controlled using a pump in regulated pressure mode and connected at the outlet.

Nevertheless, the onset of brine production is generally hard to detect accurately because the brine production rates just above the threshold capillary pressure are very small. Therefore these experimental artefacts can contribute to an overestimation of the threshold Pc value if the pressure step duration is not long enough. Moreover, experiments can be long using the standard approach since a trial-and-error process is

followed to reach an unknown value of the inlet pressure. The duration can be shortened by increasing the pressure steps but it makes the accuracy decrease accordingly.

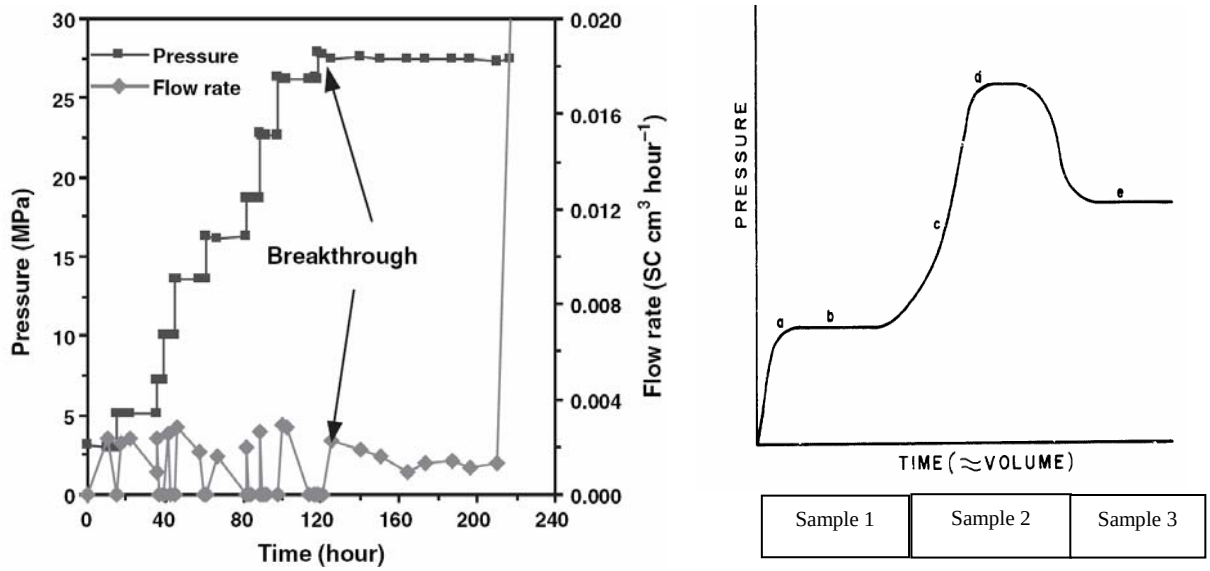


*Figure 2 : (a) Threshold pressure identification with the standard approach (Li et al., 2005) – (b) pressure evolution as a function of time with the continuous injection approach for a composite laminated core (Rudd et al., 1973)*

**The Continuous Injection Approach**

This approach relies on a continuous injection of the non-wetting fluid at a very small rate (Rudd et al., 1973). Initially, the inlet pressure in the non-wetting fluid continuously increases until it exceeds the threshold pressure of the rock located near the inlet face and the flow is initiated in the sample. When the caprock sample is heterogeneous, as is often the case with such material, the pressure evolution as a function of time exhibits pressure jumps (increases and decreases) that corresponds to longitudinal variations of the threshold Pc values (Figure 2b). The main limitation of this method is related to the assumption that the viscous gradient in the water phase is negligible when a very small rate is used. The authors mentioned a minimum injection rate equal to 0.0273 $cm^3$/hr, which can generate for a $10^{-4}$ md, 6 cm length, 5 cm diameter sample, a counter pressure in the order of 23 bar with brine (1 cP). It demonstrates that this approach can contribute to significant overestimation of the threshold Pc.

**The Residual Capillary Pressure Approach**

The residual capillary pressure approach was proposed more recently (Hildenbrand et al., 2002) to cope with the limitations of the standard approach mainly in terms of experiment duration. It is based on a transient flow of non-wetting fluid into the brine saturated sample using pressure vessels at the inlet (P1) and the outlet (P2). At the beginning, P1-P2 is set above the expected value of the threshold capillary pressure. As the non-wetting fluid flows through the sample, P1 decreases whereas P2 increases. P1

can also be kept constant with a large volume reservoir as the P2 increase is recorded. In both cases, a residual capillary pressure is measured at the end of the test, which is interpreted as a threshold capillary pressure (Figure 3a).

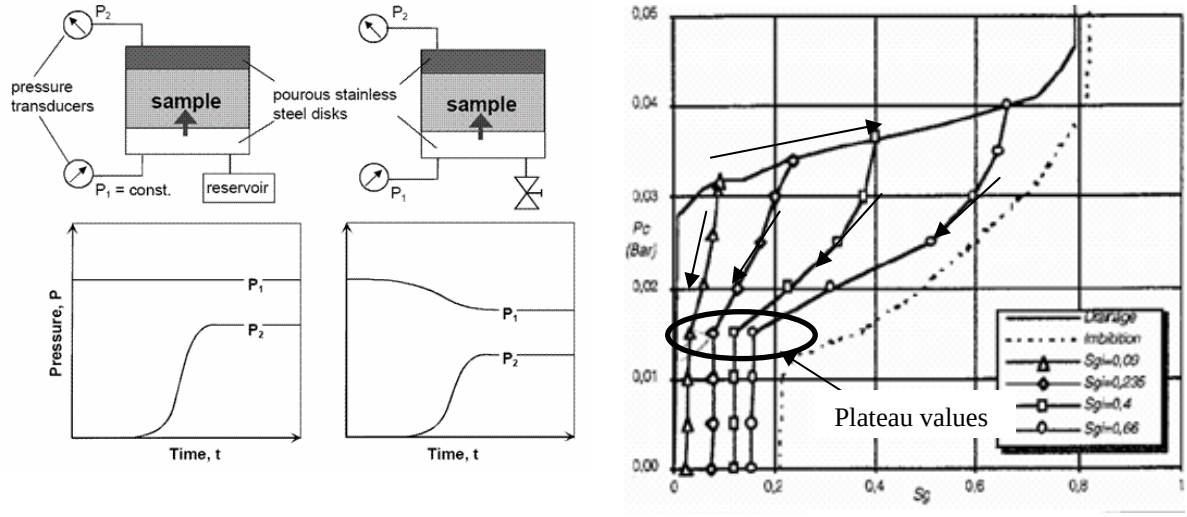


*Figure 3 : (a) principle of the residual Pc method (Hildenbrand, 2002) – (b) scanning curves of imbibition Pc curves (Kleppe et al., 1997)*

Recently, Zweigel et al. (2004) demonstrated that the interpretation of such tests is more complex since both drainage and imbibition processes have to be considered. The recorded residual Pc observed at the end of the test corresponds to the end-point of one scanning imbibition curve rather than the starting point of the first drainage curve, leading to a significant underestimation of the effective threshold Pc (Figure 3b from Kleppe et al., 1997). Therefore, this approach tends to provide very conservative threshold Pc values although it is very fast compared to the standard approach.

**Advantages and Drawbacks of The existing Approaches**

Table 1 demonstrates that none of the existing methods is efficient for all of the listed criteria, which motivated the present work to propose an alternative approach.

*Table 1 : Comparison of the different methods*

| | Duration | Rock structure | In-situ | Accuracy |
|---|---|---|---|---|
| mercury | Good | Medium | Bad | Medium |
| step-by-step | Bad | Good | Good | Good / |
| continuous | Good | Good | Good | Medium / Bad |
| residual Pc | Good | Good | Good | Bad |
| dynamic Pc | Good | Good | Good | Good |

# THE DYNAMIC THRESHOLD PC APPROACH

## Principle of the Proposed Approach

Apart the continuous injection method, most of the existing methods to determine threshold Pc are based on "static" (standard) or "semi-static" (residual) approaches based on the primary definition of the threshold Pc. From a "dynamic" point of view, the threshold Pc can also be considered as a pressure difference between the non-wetting and the wetting phases that does not contribute to the flow. Let's consider a rock sample initially saturated with brine and put under flowing conditions with a non-wetting fluid by applying a constant overall pressure drop ($\Delta P_t$), above the threshold Pc, across the sample. The pressure profile can then be divided into several parts as developed below :

$$\Delta P_t = P_{nw}^{inlet} - P_w^{outlet} = P_{nw}^{inlet} - P_{nw}^{fr} + P_{nw}^{fr} - P_w^{fr} + P_w^{fr} - P_w^{outlet} \quad (2)$$

$$\Delta P_t = \Delta P_{nw} + P_c^{fr} + \Delta P_w = \Delta P_{nw} + P_c^{threshold} + \Delta P_w \quad (3)$$

where $\Delta P_{nw}$ : the pressure drop in the non-wetting fluid invaded region

$\Delta P_w$ : the pressure drop in the virgin region

$P_c^{fr}$ : capillary pressure jump at the front that is also equal to $P_c^{threshold}$

If the start of the non-wetting fluid injection is considered only, two assumptions can be made:

- The pressure drop in the non-wetting invaded region can be neglected ($\Delta P_{nw}$ equal to zero). This results from the limited extent of this region at the start of the injection,
- The pressure drop in the virgin region, that generates a counter pressure in the brine phase, can be deduced using the effective brine production rate ($Q_w^{effective}$) that is recorded at the outlet and the Darcy's law :

$$\Delta P_w = \frac{\mu_w L}{K A} Q_w^{effective} \quad (4)$$

It is then possible to determine directly the threshold capillary pressure value using the expression hereafter :

$$P_c^{threshold} = \Delta P_t - \Delta P_w \quad (5)$$

## Experimental Set-up and Procedure

The experimental apparatus used to conduct the test is very conventional and is mainly composed of a core holder, a regulated pump to injected the non-wetting fluid under controlled pressure condition, a differential pressure sensor and a separator at the outlet to record the brine production. The core holder can be placed in an oven and the confining pressure adjust in order to mimic the reservoir conditions. Figure 4 describes the experimental apparatus.

The test can be divided into several steps :

- Preparation : the non-wetting fluid is injected into the brine saturated sample at a constant $\Delta P_t$ chosen a priori higher than the expected threshold Pc (one correlation function of the absolute permeability is available in Monicard, 1981). In contrast to the standard approach, it is recommended to start this step with brine in the inlet part of the system,
- First period : While the non-wetting fluid is still located in the inlet part of the system, only the wetting fluid flows in the sample with the overall pressure drop ($\Delta P_t$) as pressure gradient. The associated brine flow rate (slope of the brine production curve) that is recorded at the outlet then directly corresponds to Darcy's law using the overall absolute permeability value. This first period, that results from the presence of brine dead volume at the inlet, is very important because it provides the baseline of the brine flow rate at the conditions of the test.
- Second period : as soon as the non-wetting fluid reaches the inlet face of the core, the threshold Pc causes a decrease in the effective pressure gradient displacing the brine, which can be easily detected at the outlet by a bending of the production curve (lower brine flow rate).

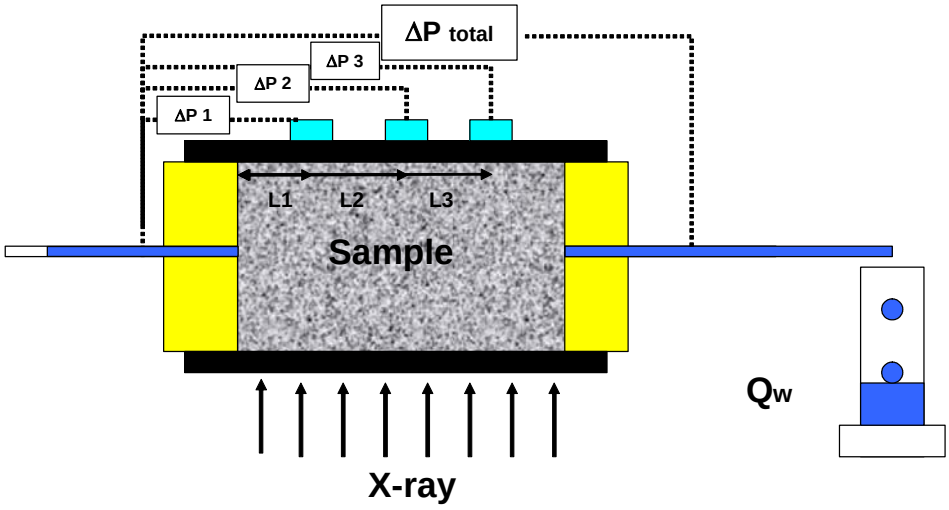


*Figure 4 : Experimental set-up*

Figure 5a shows the evolution of the brine production curve for a test where a brine dead volume was kept in the inlet part of the core holder before starting the injection. The initial slope corresponds to the single phase flow of brine through the sample. As soon as the non-wetting fluid arrives at the inlet face of the core, a significant decrease of the slope (brine flow rate) is recorded due to the capillary pressure jump at the non-wetting phase front. Figure 5b shows that the brine production stops when the injection pressure is set too low. In this example, the test was continued by increasing the injection pressure by a factor of three.

It is worth mentioning that the principle of the proposed approach (equation 3) can be applied to each intermediate pressure tap when available (Figure 4). For local pressure taps, the main difference is that the total pressure is not fixed but depends on the multiphase phase flow in the upstream part of the core. Nevertheless, $P_c^{fr}$ can be detected directly when standard pressure taps are used since they measure the higher pressure between the wetting and the non-wetting phases. Therefore, local pressure taps will measure first the wetting phase pressure, then the non-wetting pressure. The pressure jump, observed as the front passes, directly corresponds to the local threshold Pc value. X-ray measurements can also provide valuable information to detect the arrival time of the front at a given pressure tap (Figure 4). This approach can be very interesting to obtain several measurements of the threshold Pc capillary pressure along the core from one experiment, which is explored numerically in the discussion section.

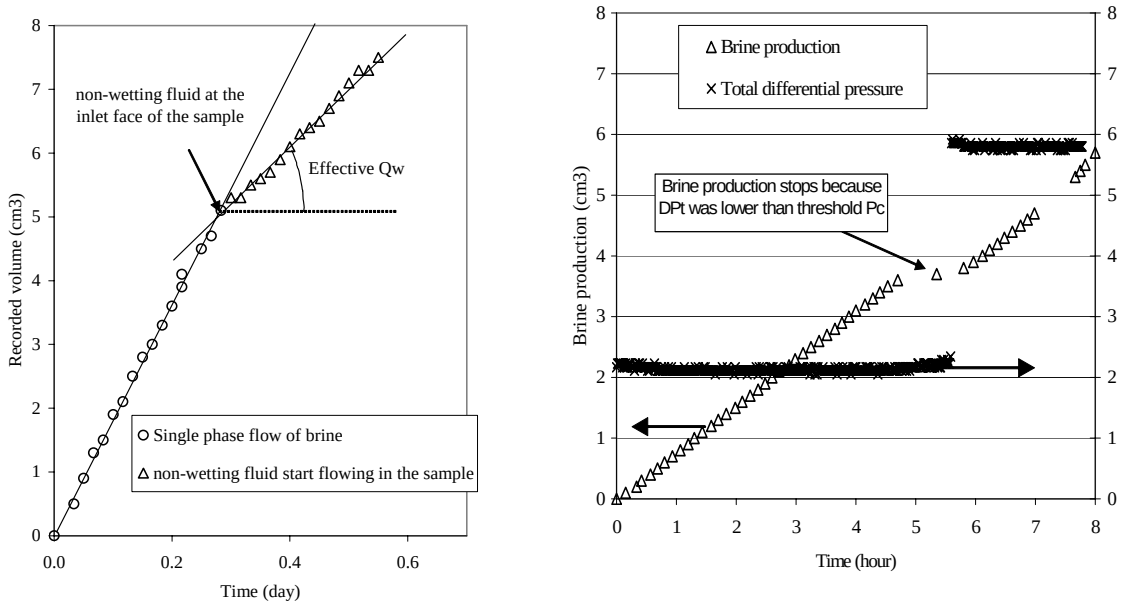


*Figure 5 : (a) typical production curve recorded at the outlet with a break in the slope– (b) typical production when $DP_t$ is lower than threshold Pc with a stop of the brine production*

## Comparison Study Between the Methods

The proposed method has been compared with both the standard and the residual Pc approaches using brine – nitrogen fluid system and five different rock samples having various threshold Pc values and petrophysical properties. For a given rock-type, measurements have been done on the same sample to simplify the comparison of the methods. All the results are gathered in Table 2.

Very good consistency is found between the dynamic and the standard approach (Figure 6), whereas it is confirmed that the residual Pc approach leads to a systematic underestimation of the true threshold capillary pressure value. This latest behavior of the

residual Pc method has been observed whatever the initial inlet pressure P1. Figure 7a shows that the same residual Pc value (2.9 bar) was obtained for R3 (Table 2) with P1 equal to 14 bar and 9.5 bar. This behavior is in line with Figure 3b, which exhibits a plateau for the threshold imbibition Pc values whatever scanning curve is considered. It has also to be pointed out that the residual Pc method may not be applicable if P1 is set to high. In this case, and as shown in Figure 7b for R2, the non-wetting phase can breakthrough leading to a zero residual Pc value at the end of the test.

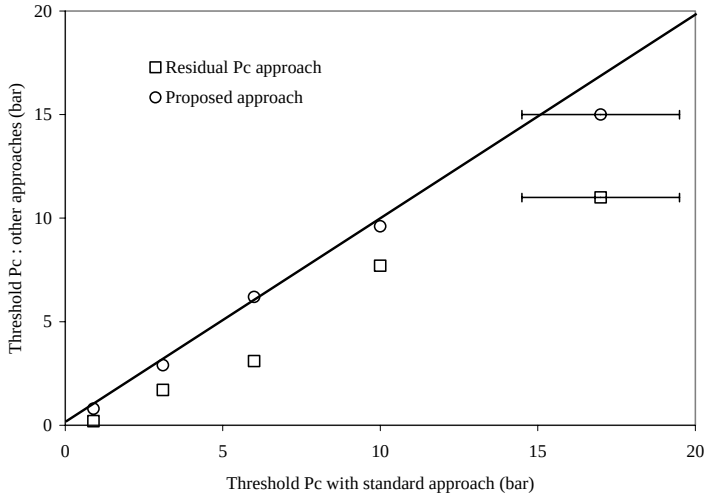


*Figure 6 : Comparison of the results obtained with different techniques*

*Table 2 : Comparison of the results with different techniques (air/brine system, ambient conditions)*

| Sample | Rock type | K (md) | Porosity % | Standard bar | Residual bar | Dynamic bar |
|---|---|---|---|---|---|---|
| R1 | Chalk | 1.7 | 40 | 0.9 | 0.2 | 0.8 |
| R2 | Carbonate | 0.044 | 17.0 | 3.1 | 1.7 | 2.9 |
| R3 | Carbonate | 0.016 | 14.5 | 6 | 2.9 | 6.2 |
| R4 | Fine grained sandstone | 0.0014 | 13 | 10 | 7.7 | 9.6 |
| R5 | Caprock | 0.0006 | 11.4 | 17 | 11.0 | 14.6 |

The dynamic method provides very promising results when compared with the standard method over a large range of threshold Pc values. As the proposed method relies on a simple injection, it has led to a time saving in the order of a factor of 5 compared to the standard method for the set of samples used.

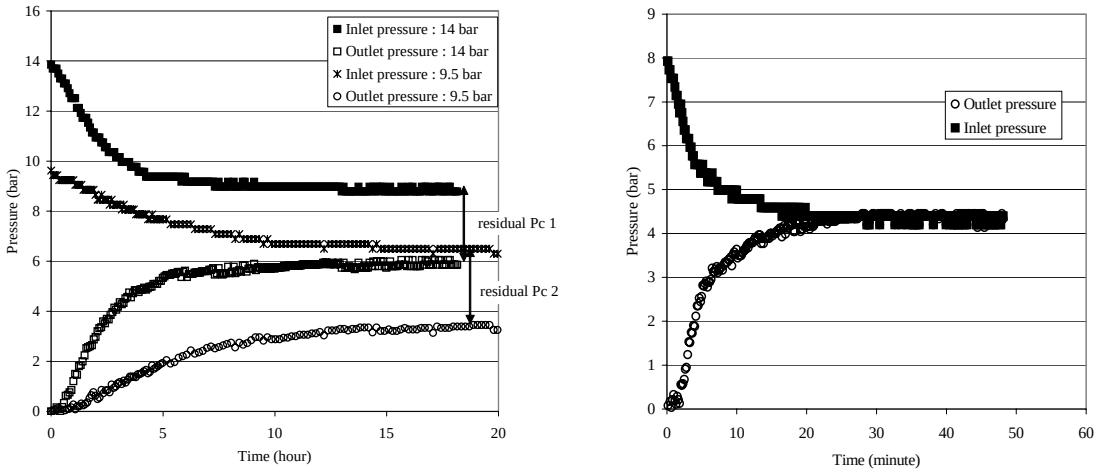


*Figure 7 : (a) Influence of the initial pressure difference on the residual Pc value (R3) – (b) Failing test when initial pressure difference was set too high (R2)*

# DISCUSSION

Heterogeneity Level in the Sample

In the previous section, it was pointed out that local pressure taps could provide additional data about the threshold Pc variation along the sample since the principle of the method can be applied at each location where the pressure is recorded. So far, as no tests have been conducted to check this potentiality of the proposed method, an investigation was conducted from a synthetic case using the composite core described below.

| | | | |
|---|---|---|---|
| $Pc^{th}$=20 b<br>k=5E-4 md | $Pc^{th}$=40 b<br>k=3.5E-4 md | $Pc^{th}$=10 b<br>k=7.1E-4 md | $Pc^{th}$=20 b<br>k=5E-4 md |

A set of Pc (Brook-Corey type) and relative permeability (Corey) curves was built and attributed to the $5.10^{-4}$ md rock-type. The Pc curves attributed to the two other rock-types were obtained by applying the Leverett's J-function in order to be as representative as possible of a real case. Figure 8a plots the numerical brine production curves for two different injection pressures (60 and 30 bar). A slope break is obtained in both cases as recorded experimentally (Figure 5a). The break is more pronounced when the injection pressure is close to the threshold Pc leading to more accurate results. Nevertheless, even when the injection pressure is triple the threshold Pc, it is clear from the example that the test is still interpretable.

Figure 8b shows the evolution of the maximum pressure between the non-wetting and the wetting phases at three points located in each of the three rock-types. All the curves follow the same evolution : 1) constant pressure during the single-phase flow period; 2)

decrease of the pressure when the non-wetting fluid starts to flow in the upstream part of the core; 3) increase of the pressure at the arrival of the non-wetting front. It is observed from these curves, that the pressure jumps detected at the non-wetting front arrival time correspond exactly to the values of the three threshold Pc values introduced in the model (10, 20 and 40 bar). Therefore, the use of local pressure taps could be very promising to capture the threshold Pc heterogeneity within the sample.

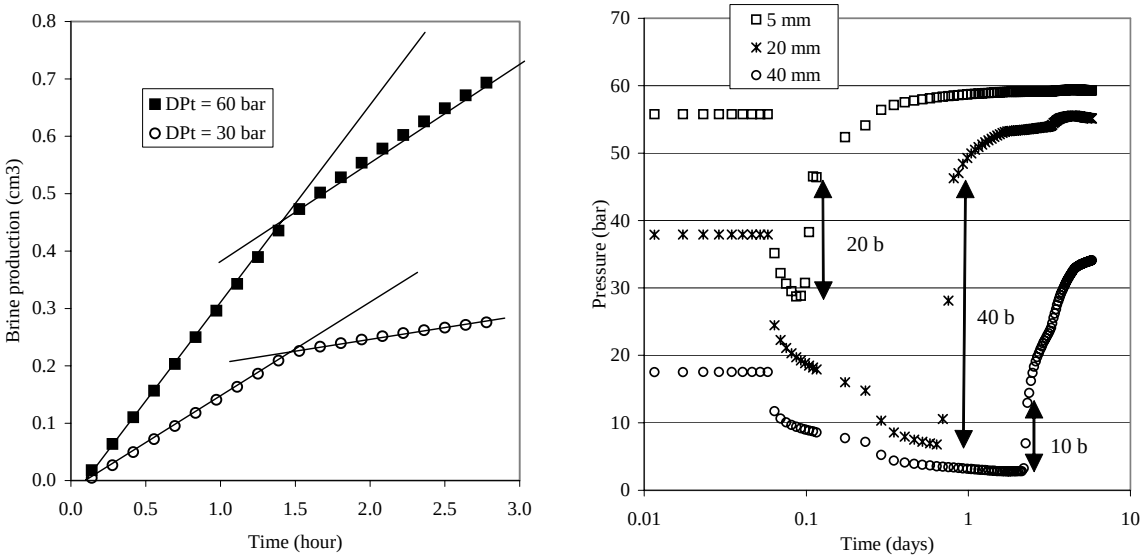


*Figure 8 : (a) Numerical simulation of the slope break of the brine production curve – (b) Pressure evolution as a function of time with local pressure taps*

Solubility of the Injected Fluid in the In-situ Brine

In most applications, threshold Pc is evaluated for a fluid system involving brine and a gas-like phase (natural gas, $CO_2$). One characteristic of the gas phase is its solubility in the in-situ brine especially under reservoir conditions. This mechanism can then impact the slope of the brine production after the non-wetting fluid starts to flow in the sample and cause a misleading interpretation of the threshold Pc with the dynamic method. One way to limit this artifact is to use a brine saturated with solution gas at the experimental conditions. This can be easily handled with the brine-methane system but is more difficult to perform with brine-$CO_2$ system since the $CO_2$ makes the brine acidic with potential rock-fluid interaction phenomena (Egermann et al., 2005). Therefore, it is recommended in this case to simulate the injection with a compositional simulator to history match the threshold Pc in order to take into account both the two-phase flow and the phase exchanges between the $CO_2$ and the fresh brine. Recent work consisting of $CO_2$ injections in saturated reservoir samples (without threshold) under various conditions demonstrated that dedicated numerical simulator provided consistent results to predict the change of slope due to $CO_2$ solubility only (Egermann et al, 2006).

## CONCLUSIONS AND PERSPECTIVES

An innovative methodology was developed to measure threshold Pc under in-situ conditions. From the experimental and the numerical results obtained so far, the proposed method of threshold Pc pressure measurement is as accurate as the standard approach and as fast as the residual capillary pressure approach. In addition, the principle of the method permits multiple measurements of the threshold Pc value in one experiment when local pressure taps are available.

This method will be tested under reservoir conditions with $CO_2$ to investigate the possible impact of wettability for different caprock-types in further works.

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